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16. Abstract

The Texas Department of Transportation has reported difficulty stabilizing soils bearing high and low concentrations of organic matter with lime. Problems include: the stabilizer disappearing over time, difficulty measuring organic matter (ignition oven), and rough pavement due to poor subgrade support. The researchers wanted to identify a good test to measure organic matter in soils and identify the problematic fraction of organic matter that causes problems with lime stabilization. Secondly, we wanted to determine what mechanism or mechanisms were responsible for organic matter interfering with lime stabilization. We developed a simple technique to measure the humic acid content of organic matter using a UV-Vis spectrophotometer. We also constructed hundreds of manufactured soils consisting of a humic acid standard mixed with different clay mineral standards, lime, and quartz sand. These soils were used to measure changes in engineering properties as well as monitor chemical changes to elucidate factors controlling reaction mechanisms. From the manufactured soils, we determined that humic acid does interfere with lime stabilization by inhibiting the formation of Calcium Silicate Hydrate reaction products between the lime and clay minerals. This results in reduced unconfined compressive strength values for stabilized cores. The concentration of humic acid that inhibits the formation of pozzolanic reaction products is around 1 percent. Calcareous soils containing organic matter appear to respond better to lime stabilization than acid soils. This study was the first to provide direct evidence of the effects of organic matter on soil stabilization.

17. Key Words
Humic Acid, Lime Stabilization, Organic Soils, UV-Vis Method, Organic Matter Measurement

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MITIGATING THE EFFECTS OF ORGANICS IN STABILIZED SOILS: TECHNICAL REPORT

by

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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 past few years, the Texas Department of Transportation (TxDOT) has experienced difficulties in stabilizing expansive subgrade soils bearing organic matter (OM) with calcium-based additives. In particular, several districts have treated subgrade soils bearing high concentrations of organic matter with calcium-based additives, and the desired improvement in the soil is never achieved or the improvement disappears over time. In other instances, subgrade soils containing concentrations of organic matter in excess of 20 percent have been successfully stabilized with calcium-based additives (Arman and Munfakh, 1970).

In the paragraphs that follow, factors affecting soil development and the evolution of organic matter will be discussed so that we may better appreciate the complexity of organic soils. This discussion will be followed by a review of what previous investigations have found with calcium-based additives in organic soils. Some of the proposed mechanisms for calcium-based additives not working in organic soils will be reviewed. Ways to identify and quantify organic matter in soils will be discussed in addition to a comparison of the ignition oven to the Elementar technique. And finally, a review of successful stabilization projects in organic soils will be presented.

SOIL DEVELOPMENT IN TEXAS

The development of soils in Texas is extremely diverse and is directly related to the composition of the rocks on which they are produced. There are four key factors that control the fragmentation and decay of rocks (i.e., soil generation); they are properties of the parent rocks, climate, presence or absence of soil and vegetation, and length of exposure (Press and Siever, 2001).

The composition and texture (i.e., grain size and orientation of crystals) of the parent rocks are important in soil formation because different minerals weather at different rates, and the texture affects their susceptibility to cracking and fragmentation. An illustration is comparing tombstones made from granite and limestone. A tombstone composed of limestone will alter more quickly than the granite one. In a few years, the lettering may become illegible on the limestone tombstone, but the lettering on the granite tombstone will still be fresh.

The climate (rainfall and temperature) has a profound impact on soil generation. Using the tombstone analogy again, a limestone tombstone in southeast Texas will decompose more rapidly than a limestone tombstone in west Texas of the same age. Climate and living organisms strongly affect chemical breakdown of rocks. A hot and humid climate encourages growth of organisms, but cold, dry climates impede organism growth and chemical alteration of the rocks.

The presence of preexisting soil will increase the rate of alteration of the rock. Once soil starts to form, it works to weather the rock more rapidly. It retains rainwater, and it hosts a variety of vegetation, bacteria, and other organisms, which all combine to create an environment that promotes mineral alteration and/or dissolution.

Time is the last element in soil formation. A tombstone composed of limestone that is 100 years old in a graveyard in southeast Texas will be badly weathered, but a limestone tombstone that is 10 years old in that same graveyard will still be relatively unblemished. Therefore, the longer a rock is exposed to a certain climate, the more weathered it will become.

Because climate strongly influences weathering, it has a profound impact on the characteristics of the soil formed on any given parent rock. Figure 1.1 shows how soil scientists have characterized soils in general. There are generally three horizons designated by A, B, and C, with the O horizon only developed in temperate climates. The thickness and/or presence of each horizon will vary in different climates. For example, the A horizon will lie directly on the C horizon in wet, tropical climates. There are no O or B horizons because the organic matter is quickly oxidized and recycled into the vegetation. In temperate climates, there is a better developed B horizon, and there can be a pronounced O horizon. In arid climates, there is a small A horizon and a large B horizon with no O horizon.

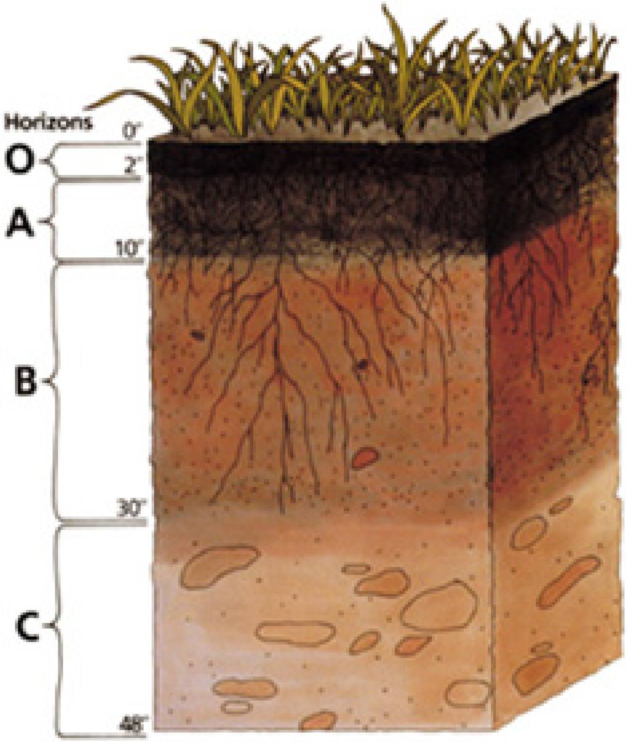


Figure 1.1. A Typical Soil Profile for a Temperate Climate.

The O horizon is dominated by undecomposed or partially decomposed organic material (less than half of the weight is mineral matter). An O horizon may be at the surface or any depth beneath the surface if it has been buried.

The A horizon is composed mostly of clay and insoluble minerals like quartz in a soil that has formed over a long period of time. Soluble minerals have been leached from this layer. Beneath the A horizon is the B horizon, where there is little organic matter. Soluble minerals and iron oxides have accumulated in small pods, lenses, and coatings in this layer. The C horizon is the lowest layer and is slightly altered bedrock (parent material) fragments that have been broken and weathered, mixed with clay from chemical alteration.

ORGANIC MATTER IN TEXAS SOILS

Vegetation is also controlled to a large extent by the geology. The map on the left side of Figure 1.2 was assembled by Benjamin Tharp in 1939; it breaks the state up into different regions based on differences in the vegetation. Geologists often identify different rock units based upon the dominant vegetation growing in the area. For example, Spanish oak characterizes the Walnut Springs limestone; an open stand of cedar, the Glenrose; liveoak and cedar, the Edwards (Tharp, 1939). The rock types illustrated in the geologic map on the right side of Figure 2 shows a very nice correlation with the vegetation zones of Tharp (1939).

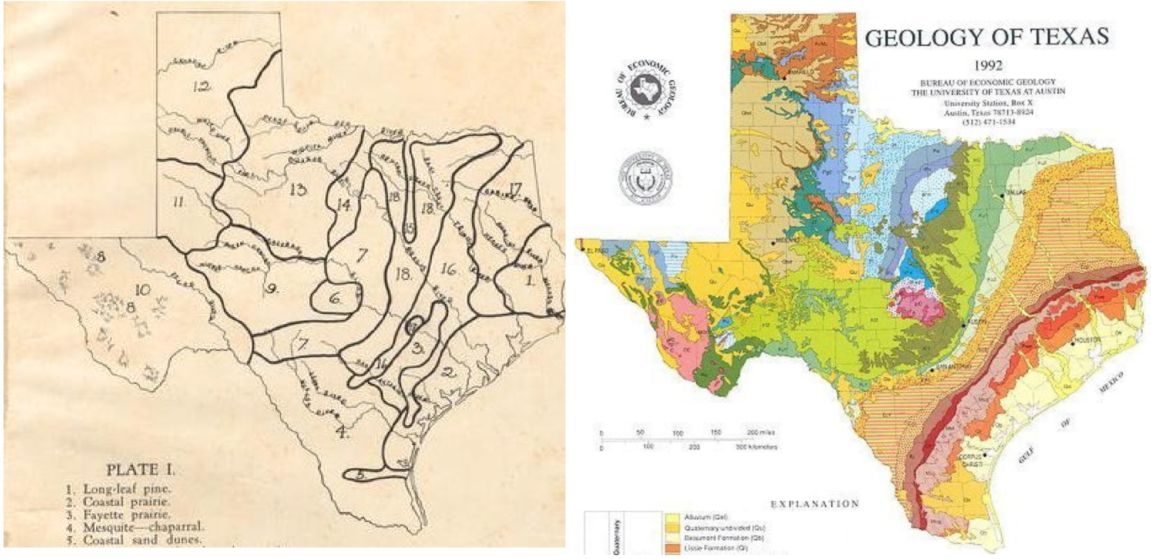


Figure 1.2. Vegetational Regions of Texas Showing How Vegetation [left] Varies with Geology [right]. (vegetation map from Tharp, 1939; geologic map from the Bureau of Economic Geology 1992).

The vegetation will impact the type of organic matter in a soil. Wardwell et al. (1983) explain that an important constituent in OM decomposition is lignin which varies widely from one species to another. Several factors influence the amount of organic matter in a soil. Jenny (1941) arranged the importance of the soil forming factors as follows:

Climate > vegetation > topography = parent material (rock formations) > age

The concentration of organic matter in undisturbed soils will vary, and some generalizations were made based on Jenny's work:

- Grassland soils have higher organic matter than forest soils.
- High plasticity index (PI) soils have higher organic matter than low PI soils.
- Poorly drained soils have higher organic matter than well-drained soils.
- Lowland soils have more organic matter than upland soils.
- Organic matter increases as precipitation increases.
- Organic matter decreases as temperature increases.

Soil organic matter is composed of many parts, such as (1) intact plant and animal tissues and microorganisms, (2) dead roots and other recognizable plant residues, and (3) a mixture of complex amorphous and colloidal organic substances no longer identifiable as plant tissues (Plank, 2001).

Soil humus or humic material makes up 60 to 80 percent of the organic matter in soil; humus is a complex system of substances remaining in the soil after extensive chemical and biological breakdown of fresh plant and animal residues (Plank, 2001). The other 20 to 40 percent OM is less stable and partially decomposed. Humus is stable and relatively resistant to microbial attack; it is responsible for the cation exchange capacity (CEC) of OM and can be divided into three groups (Brady and Weil, 1999):

- **Fulvic a cid-** low molecular weight, light color, soluble in both acid and alkali, and most susceptible to microbial attack. Depending on conditions, the half-life (time it takes to destroy half of the material) is approximately 10 to 15 years.
- **Humic acid-** moderate molecular weight and color, soluble in alkali but insoluble in acid, and intermediate in degradation potential with a half-life >100 years.
- **Humin-** high molecular weight, dark color, insoluble in acid and alkali, and most resistant to microbial attack.

Soil contents of humic and fulvic acids vary by depth, climate, and geography (Thurman, 1985). Forest soils such as alfisols, spodosols, and ultisols generally are high in fulvic acids, whereas grassland soils such as mollisols are high in humic acids (Tindall and Kunkel, 1999). Tindall and Kunkel (1999) report that forest soils like spodosols, significant amounts of aluminum, iron, and organic matter have been mobilized and transported deeper into the profile in the B horizon. Podzols evaluated by Clare and Sherwood (1954) also showed variations in organic matter content. They concluded that "active" organic matter is formed in the vegetable top-soil and subsequently leached by rainwater into the B horizon where it is deposited at depths as great as 5 feet.

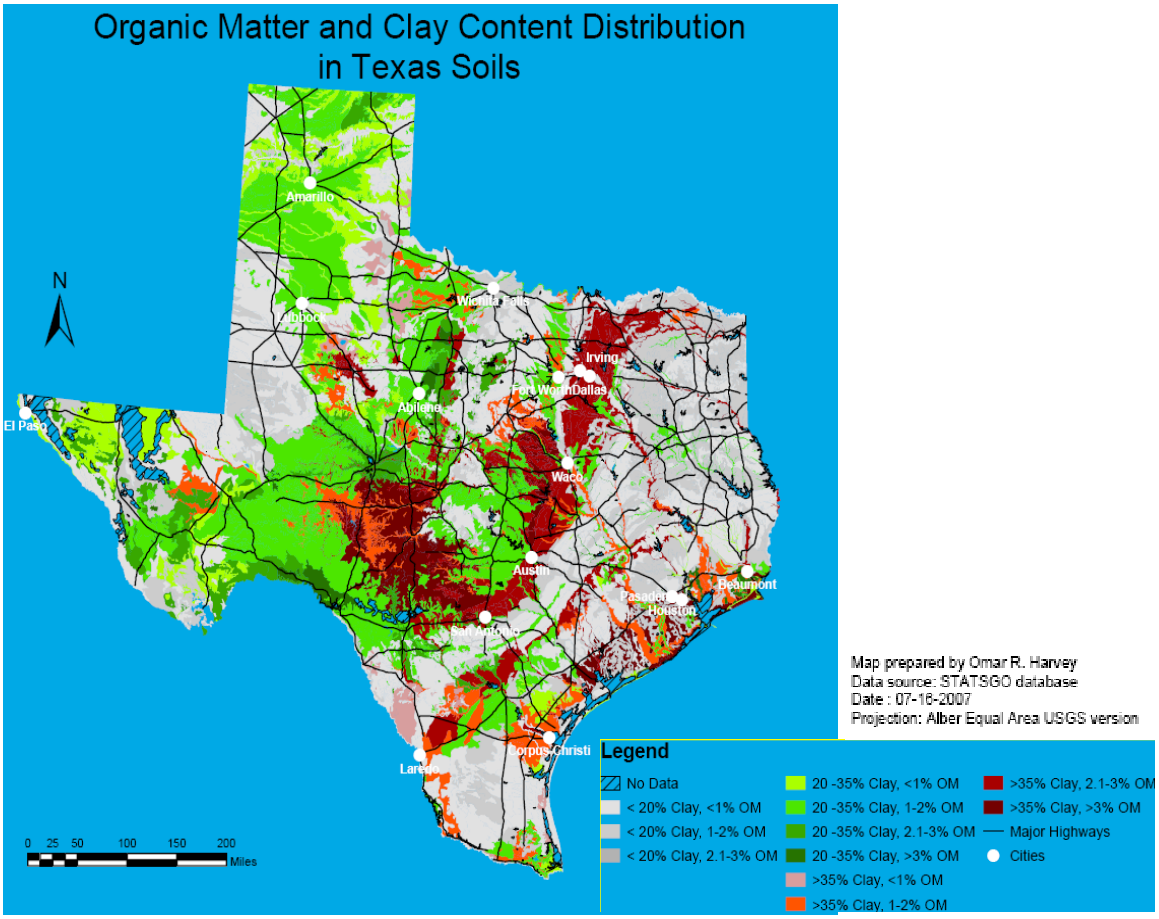
Brady and Weil (1999) state that soil humus has a much higher CEC than clay minerals common in soils of the southern United States (Table 1.1). The CEC contributed by the organic fraction may range from 30 to 90 percent of the soil's CEC (Tindall and Kunkel, 1999). This high CEC will affect the absorption of Ca, Mg, and K.

Table 1.1. Cation Exchange Capacity of Soil Colloids at pH 7 (from Brady and Weil, 1999).

Colloid CE	C (cmol$^{(+)}$/kg) or meq/100g
Humus	200
Vermiculite	100
Smectite	150
Fine-grained micas	30
Kaolinite	8
Hydrous oxides	4

IDENTIFYING ORGANIC MATTER IN SOILS

Soil scientists have long been measuring organic matter in soils because organic matter is important for growing crops. Due to the importance of OM for soil fertility, soil scientists have developed methods to analyze different fractions of organic matter; these data can be accessed in the State Soil Geographic (STATSGO) database and plotted on maps like the one depicted in Figure 1.3.



Map prepared by Omar R. Harvey
Data source: STATSGO database
Date : 07-16-2007
Projection: Alber Equal Area USGS version

Figure 1.3. Surface Distribution of Organic Matter and Clay in Texas Soils. Map prepared using Data from STATSGO database

Figure 1.3 shows that the soils with high organic matter and high clay contents (typical of soils stabilized with lime) are concentrated along the I-35 corridor, along river drainages, and along the coast.

The techniques developed for measuring organic matter are too numerous to count; they were all developed for measuring specific organic fractions and have different applications. All of the methods developed for measuring organic matter have their drawbacks. The following paragraphs will describe a few of the methods relevant to soil stabilization.

One procedure commonly used is to place a small amount of soil on filter paper and moisten the soil lightly to bring out the color. The color is then compared to a color chart to estimate the organic content of the soil. This technique is often not very accurate because there is a lot of interpolation from one concentration to the next. There are also soils with high concentrations of organic matter that do not have the dark color typical of organic soils.

Another procedure that can be applied in the field is to add ethylenediamenetetraacetic acid (EDTA) to air-dried soil samples and mix for a specified time. The color of the extract is directly proportional to the concentration of the organic matter.

The most common procedures for measuring organic matter in soils have traditionally been categorized as either wet or dry combustion techniques. Both techniques involve oxidation of the organic carbon (C) and quantifying the CO_2 evolved.

Using the dry combustion technique, the soil is heated to high temperatures (>400ºC) where the organic matter is oxidized to CO_2 and H_2O with the majority of the soil minerals left intact. Most of the organic matter goes off as a gas, and the difference in weight can be used to estimate OM content. The loss on ignition method (LOI) is easy to perform and inexpensive. However, problems arise when minerals such as gypsum, smectite, vermiculite, and gibbsite are present because these minerals can lose 20-30 percent of their total weight at these temperatures.

The wet combustion technique known as the Walkley-Black method has been considered the standard method to estimate organic matter in soils for a number of years. This procedure measures the organic carbon that can be easily oxidized by using dichromate (a powerful oxidizing agent when mixed with sulfuric acid). The Walkley-Black method has been shown to oxidize approximately 77 percent of the organic matter in a soil.

More recent work has been done with near infrared diffuse reflectance spectrometry for measuring moisture, organic carbon, and total nitrogen (Dalal and Henry, 1986). They concluded that organic C can be measured in soils containing moderate amounts of organic matter (0.3-2.5 percent).

Sherwood (1962) correlated the pH of soil-cement mixtures during early stages of hydration to unconfined compressive strength (UCS); he concluded that the measurement of the pH of the soil-cement mixture one hour after being mixed could be used to find the presence of deleterious organic matter.

EVALUATION OF TXDOT'S AND TAMU'S SOIL AND CROP SCIENCES ORGANIC MATTER PROCEDURES

The Bryan District of TxDOT has been using the LOI method to measure organic matter in soils. They have been heating the soil in an ignition oven at 450ºC until no change in mass occurs. The difference in the weights is interpreted to be the organic matter that has been removed. The Department of Soil and Crop Sciences at Texas A&M University uses an Elementar VarioMax carbon-hydrogen-nitrogen (C-H-N) analyzer (Figure 1.4).

Figure 1.4. Elementar VarioMax C-H-N Analyzer Used for Organic C Analysis.

The procedure used in the Department of Soil and Crop Sciences calls for a wet chemical treatment to remove carbonates, which can give false high organic C results. The samples are heated to 650ºC (for organic C) in purified O_2 stream and heated to 900ºC (for total C). The CO_2 evolved is measured, and percents of organic carbon and total carbon are calculated.

The LOI technique yields higher percentages of organic matter because it does not discriminate between organic carbon and inorganic carbon. Hydrated (water-bearing) minerals can contribute to the loss in weight as well, which can create some large errors in organic carbon measurement.

American Society for Testing and Materials (ASTM) 2974-00 calls for drying the soil in an oven at 105ºC (to remove moisture) prior to placing in the ignition oven to burn off the organic matter. Research at Texas Transportation Institute (TTI) also shows that there may be a problem with this practice. Samples dried at 25ºC prior to analysis with the Elementar contained up to 50 percent more organic carbon than samples from the same location dried at 60ºC.

PREVIOUS INVESTIGATIONS

A review of the engineering literature over the last 50 plus years yields mixed results with regard to calcium-based stabilizer performance in soils bearing organic matter. Firstly, all of the literature found in this cursory review pertains to cement and/or cement + other stabilizers (lime, ground granulated blastfurnace slag [GGBFS], fly ash). The researchers have yet to find a study documenting the use of only lime in organic soil stabilization. Clare and Sherwood (1954) performed the first systematic study of the effects of organic matter on cement stabilization. They evaluated the UCS (7 day) of different organic fractions mixed in sand and stabilized with 10 percent cement and determined that the organic fractions could be classified into three groups: 1) inactive or slightly active- cellulose, alginic acid, wood, and gelatin; 2) active- carboxymethylcellulose, pectin, and casein; and 3) very active- glucose, and nucleic acid.

In a subsequent investigation, Clare and Sherwood (1956) measured the UCS of cement stabilized natural soils bearing organic matter and concluded that the ability of cement to stabilize soils will vary from soil to soil depending on the chemical activity of the organics. In the latter investigation, they analyzed a soil developed on limestone ($CaCO_3$) and determined that the organic matter did not retard the setting of the cement due to saturation of the active constituent with calcium (Ca^{2+}) ions. The limestone in the soil supplied the Ca^{2+} to the organic matter so that there was no competition for the Ca^{2+} ions in the cement.

There have been a number of studies that report initially low strengths for calcium-based additives (Clare and Sherwood, 1954; Pousette et al., 1999; Cortellazzo and Cola, 1999). Åhnberg and Holm (1999) reported that higher curing temperatures have a detrimental effect on cement and lime mixtures.

EFFECTS OF ORGANIC MATTER ON CALCIUM-BASED STABILIZER CHEMICAL REACTIONS

For soils with low (<1%) organic matter, Eades et al. (1962) determined that interlayer cations in smectite (expansive clay) are first replaced with calcium from lime (CaO). The lime forms $Ca(OH)_2$, which raises the pH to over 12. Once the interlayer positions have been saturated with calcium, the clay mineral structure deteriorates (at $pH > 10.5$), and new minerals (CSH gel) are formed creating the strength gain and textural improvements.

Clare and Sherwood (1954) report that the retardation of hardening of organic soil-cement mixtures is due to the retention by the organic matter of calcium ions liberated during the hydrolysis of the calcium silicates and aluminates in the cement grains. These ions are not available to form the cementitious products, making the cement ineffective.

Tindall and Kunkel (1999) report that organic soils usually require higher rates of pesticide application than mineral soils due to two factors: (1) the chemical is strongly retained onto humic substances; and (2) organic soils contain more water on a volume basis, requiring more solute to obtain a given concentration. The researchers believe that lime will behave in a similar fashion to the pesticides.

There are concerns regarding leaching of calcium-based stabilizers from organic soils. Huat et al. (2005) reported that high concentrations of organic matter resulted in a loss of stabilizer (cement and lime). Hydrogeologists report that water soluble organic carbon (WSOC) commonly transports contaminants through the soil profile and into the groundwater (Tindall and Kunkel, 1999). WSOC may leach calcium from the stabilizers over time and render the stabilizers ineffective.

Based on the above literature review and the researchers' personal experiences with stabilizing organic-rich soils, we believe that the organics may interfere with lime stabilization in the following manner:

- use of Ca^{2+} ions to satisfy the exchange capacity of the organic matter,
- lowering pH to the point where dissolution of clay minerals no longer takes place,
- formation of complexes that interfere with the soil-lime reactions, and
- the dilution factor of high water contents of organic soils requiring higher stabilizer contents.

SUCCESSFUL STABILIZATION OF ORGANIC SOILS

Success has been variable, and most of the literature documents peat stabilization with regard to deep soil mixing. den Haan (1998) showed that high strengths could be achieved in organic soils when using a mixture of slag cement and anhydrite. Hebib and Farrell (1999) evaluated two Irish peats and concluded that highly organic soils can effectively be stabilized if enough stabilizer is added to the soil. They determined that cement increased UCS for both peats, but a slag and gypsum combination only improved UCS on one of the peats. Hampton and Edil (1998) summed it up when they concluded that research is needed on the fundamental chemical reactions contributing to changes in the geotechnical properties of stabilized organic ground.

CHAPTER 2

TxDOT SURVEY ON ORGANIC SOILS IN TEXAS

INTRODUCTION

To evaluate the impact of organic matter on stabilized soils in Texas, we circulated a survey among 25 TxDOT district offices. The survey questionnaire is presented in the Appendix. The responses received from this survey have been analyzed and are presented in the following sections. Sixteen of the 25 districts responded to the survey, which constitute 64 percent of the total number of districts in Texas (Figure 2.1).

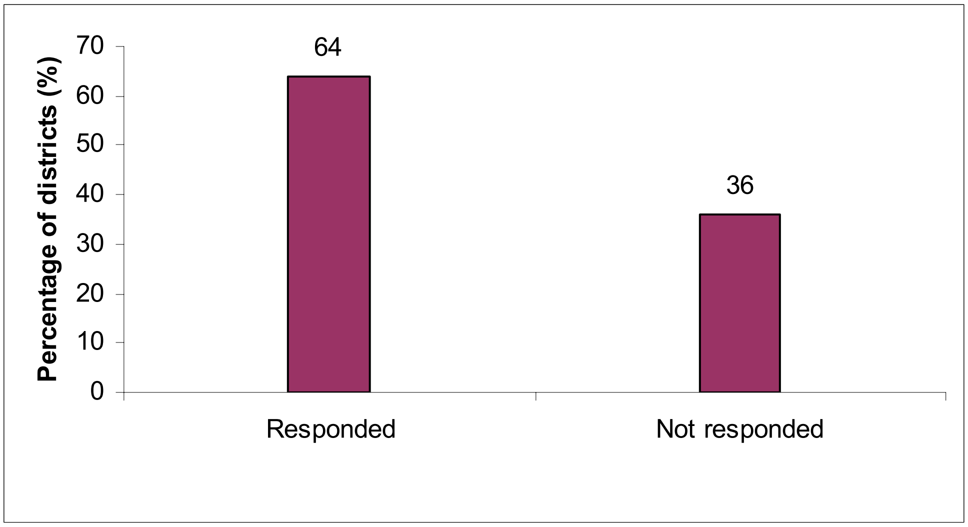


Figure 2.1. Responses to the Questionnaire.

SURVEY ANALYSIS

Q1. Have you encountered any organics in subgrade soils from your district?

Organic soils are encountered occasionally by 44 percent of the total number of districts that responded to the survey and none on a regular basis. Of the total number of districts responding, 56 percent had not encountered any organics in their subgrade soils. Figure 2.2 graphically shows these results.

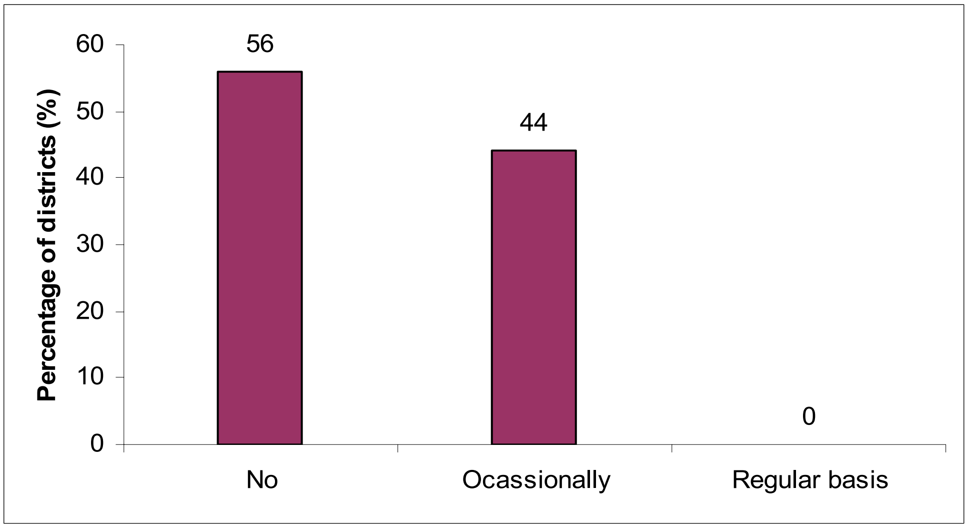


Figure 2.2. The Number of Districts Encountering Organic Soils.

Q2. In your opinion, how do you assess problems due to organic-rich subgrades in your district?

Organic soils have been encountered in 44 percent of the total number of districts that responded to this survey (i.e., 7 districts out of 16). In other words, 56 percent of total respondents have not encountered any organic soils in their districts. Among the districts that have encountered organic soils, 72 percent of districts reported problems due to the organic-rich soil subgrades. The Amarillo District reports no problems due to organics, although they report having organic-rich subgrade soils (Figure 2.3). The Austin District is the only district to report considerable problems due to organic-rich subgrade soil.

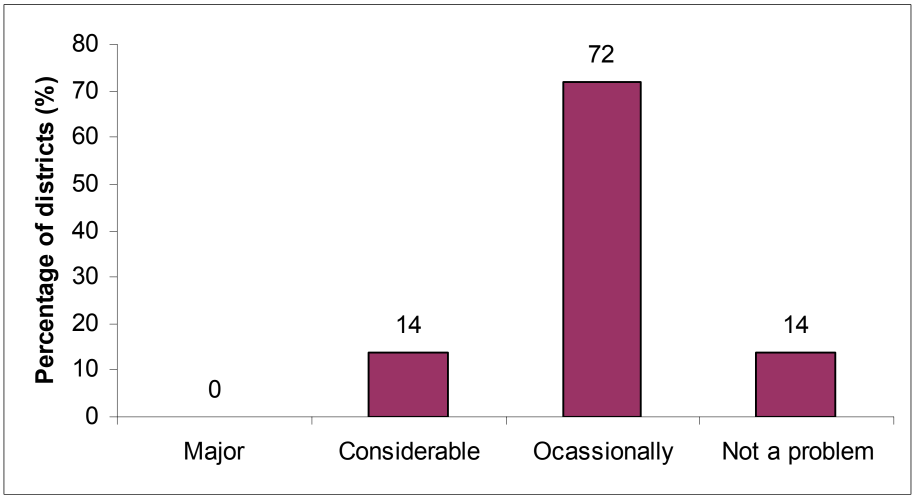


Figure 2.3. Problems Due to Organic Soils.

Q3. *What laboratory method have you followed for the measurement of organics?*

TxDOT districts have been using different procedures to determine organic content in the soil. Seventy-two percent or five of the districts that had encountered organic soils use the loss on ignition test (TxDOT procedure) to determine organic content. Amarillo is the only district that uses an ASTM procedure, whereas, the Austin District mentioned that it had not tested any soil to determine organic content, but it would measure organic content in the future. Figure 2.4 shows these results.

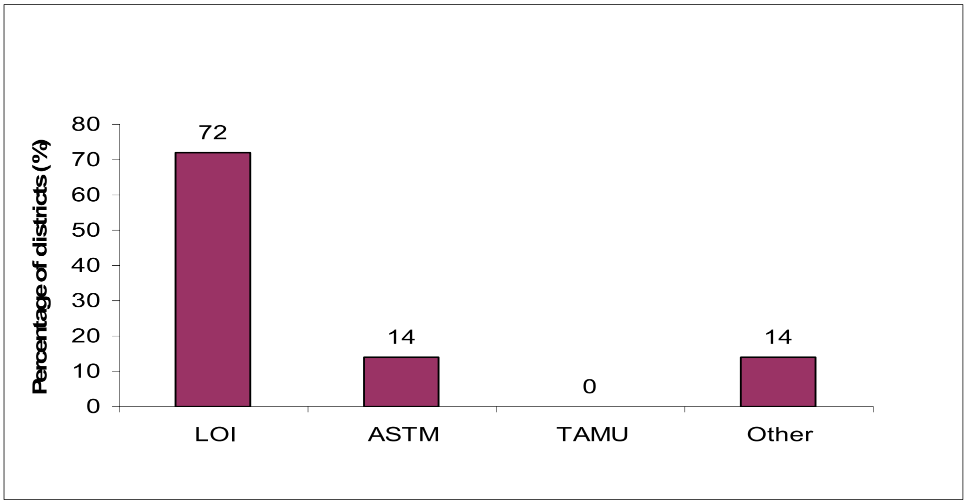


Figure 2.4. Methods Used to Find Out Organics by Different Districts.

Q4. *At what depths have you encountered the organics?*

Organics are at an intermediate depth of 1-3 feet, for 58 percent of the districts that have encountered organic soils. Austin and Amarillo experienced organics at an intermediate depth of 3-5 feet and less than 1 foot, respectively. The Waco District has identified organics anywhere from 0-5 feet (Figure 2.5).

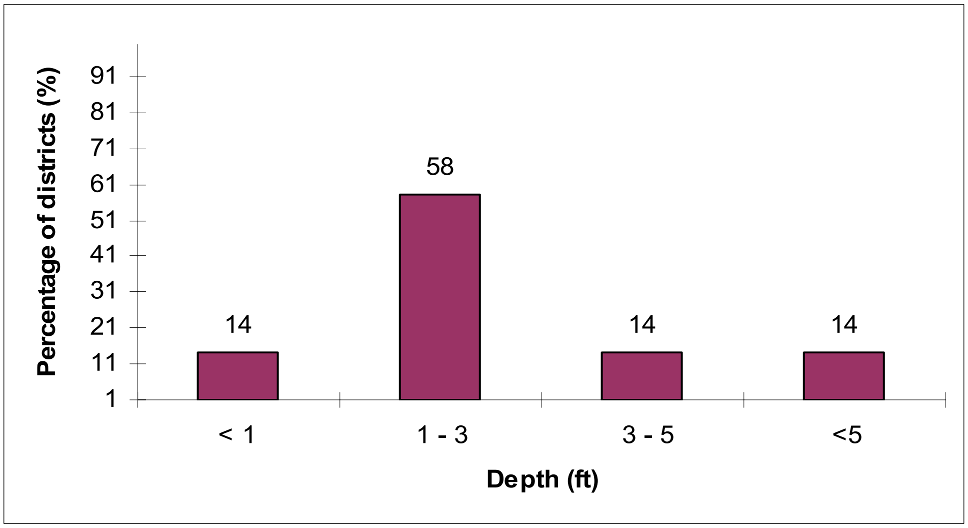


Figure 2.5. The Depth at Which Organic Soils Were Encountered by TxDOT Districts.

Q5. Have you implemented any stabilization on the organic-rich subgrades?

Figure 2.6 shows that 72 percent of the districts implemented a stabilization plan when they encountered organics in the subgrade soils. However, 28 percent of the districts did not use any stabilization technique when they encountered organics in the subgrade.

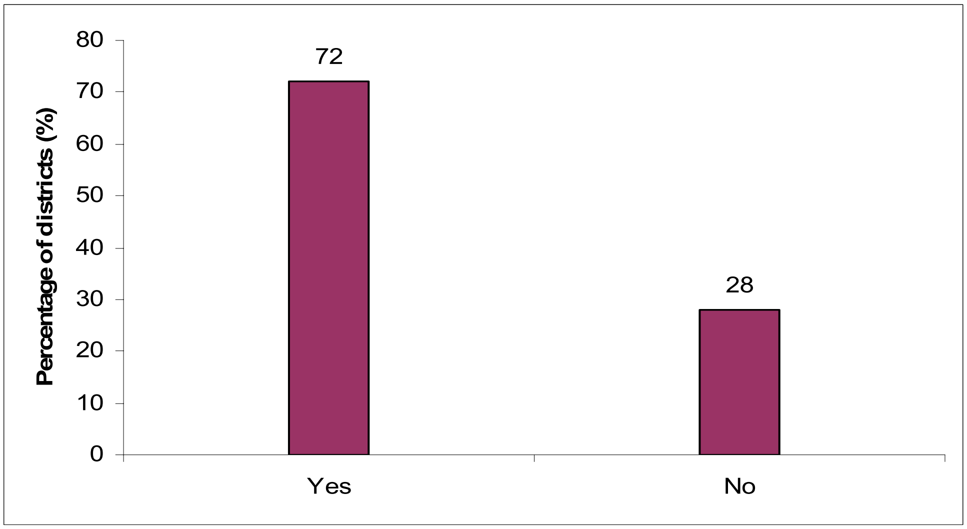


Figure 2.6. Implementation of Stabilization on Organic-Rich Subgrades.

Q6. If chemical modification is the choice, please check the stabilizer that has been used.

Chemical modification is the major stabilization technique used for the organic soils in Texas. Eighty percent of the districts that stabilize the subgrade of organic soils use lime as the stabilizer (Figure 2.7). However, 20 percent use fly ash as a stabilizer to reduce the effects of organic-rich soils.

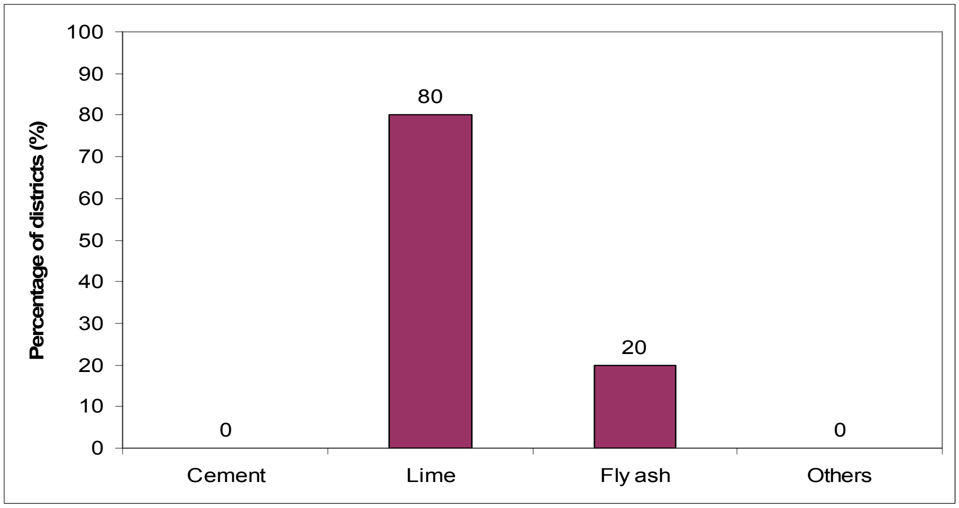


Figure 2.7. Types of Stabilizers Used.

Q7. Which soil property was the major focus of your subgrade design?

TxDOT districts have been using different soil properties for the subgrade design. The majority of the districts (72 percent) use swell-shrink changes as the major design property for stabilizing organic soils. However, strength and stiffness/resilient modulus properties are the focus of 14 percent of the districts that have encountered organic soils in the subgrade (Figure 2.8).

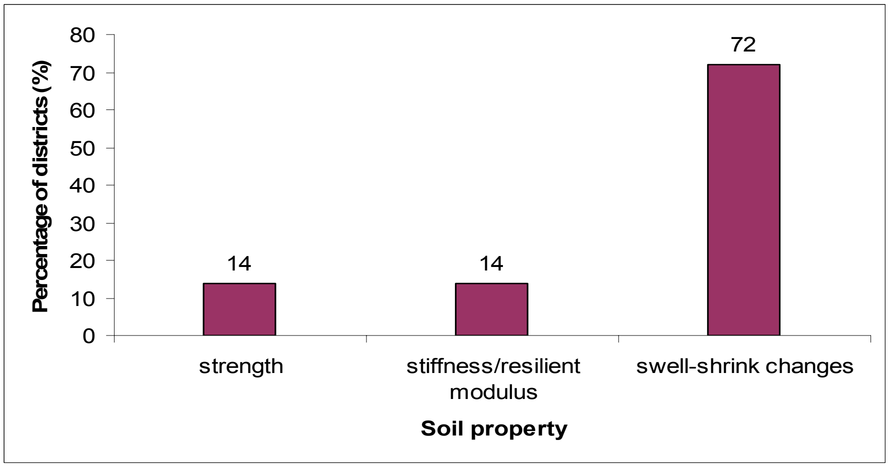


Figure 2.8. Soil Properties Used as the Major Focus of Subgrade Design.

Q8. If any other modification or replacement of organics is implemented, please list the method followed.

Amarillo and Fort Worth Districts did not answer the question, whereas Austin doesn't use any modification or replacement techniques. However, Beaumont, Waco, and Wichita Falls Districts use a method to remove and replace the problematic material with a better local fill material. San Angelo mixes limestone, which is locally available, with the organic-rich material.

Q9. Have you experienced any major distress on stabilized organic-rich subgrades?

San Angelo and Wichita Falls did not answer the question, whereas Amarillo and Waco do not report any problems after stabilization of the subgrades. However, Beaumont, and Fort Worth experienced rutting and poor strength gain on the subgrade after stabilization of the subgrades. The Austin District experienced problems such as shrink-swell due to the ineffectiveness of the calcium-based additive. These results indicate that 60 percent of the districts that have implemented stabilization of organic-rich subgrades have problems after stabilization.

SUMMARY

The researchers prepared a short survey to measure the extent of problems experienced by TxDOT districts when organic-rich soils are encountered. Sixteen districts responded to this survey, which acccounts for 64 percent of the total number of districts present in Texas. From the responses received, organics in the subgrade are encountered by seven districts, which accounts for 43 percent of the total number of districts that responded to the survey. Five of the districts that have encountered organic soils have used stabilization. Lime is used as a stabilizer by four of the districts that have implemented stabilization in organic-rich subgrade soils. However, the survey indicates that three of the districts that have stabilized organic-rich subgrades report problems after stabilization. So, the effects of organics on stabilized soils are a major part of this research.

CHAPTER 3

INVESTIGATION OF METHODS TO MEASURE ORGANIC MATTER IN SOILS-LABORATORY TESTS

INTRODUCTION

To evaluate the effectiveness of a stabilizer in organic soils one needs to accurately measure the organic content of a soil. The researchers evaluated three different techniques to measure organic matter in soils. Two of the techniques are currently used by TxDOT laboratories to measure organic matter in soils. They are loss on ignition and a colorimetric technique where soil is mixed with a sodium hydroxide solution and a color change indicates OM content. There is concern that LOI will yield too high OM values when a soil contains carbonates or water-bearing minerals like smectite or gypsum. The researchers also question the ability of the colorimetric technique to measure small changes in OM content.

There is a need to be able to rapidly identify small variations of organic matter content in soils. Two of the three techniques evaluated are relatively simple, so sources of error can be minimized. The researchers wanted to see if the techniques are sensitive to changes in the level of organics present. Secondly, the researchers wanted to see if soil mineralogy impacts the test results. To accomplish this objective, the researchers constructed artificial samples with known concentrations of four types of OM and different mineral compositions to see if mineralogical differences affect the results.

METHODS OF IDENTIFYING ORGANIC MATTER IN SOILS

There are many methods to identify organic matter in soils. However, a satisfactory method to measure soil organic matter has yet to be formulated (Tan, 1996). This situation is attributed to the high variability in content and composition of soil organic matter.

For quantitative analysis of organic matter, the C_{org} is generally measured, not the total C content of soils. The total C is composed of C_{org} plus C_{inorg} in the soil. The soil organic matter is obtained by multiplying the C_{org} by a factor ranging from 1.724 to 2.0 (Jackson, 1979).

Several TxDOT districts have been using the LOI method, a variant of ASTM D 2974-00 and AASHTO T267, to measure organic matter in soils. Initially, the soil is heated at 105°C to remove moisture. Then they heat the soil in an ignition oven at 440ºC until no change in mass occurs. The difference in the weights is interpreted to be the organic matter that has been removed. This technique is called dry combustion, where the organic matter is oxidized at high temperatures (>400°C) to $CO_{2(g)}$ and $H_2O_{(g)}$ with the majority of the soil minerals left intact. However, problems arise when minerals such as gypsum, smectite, vermiculite, and gibbsite are present because these minerals can lose 20-30 percent of their total weight at these temperatures.

A second technique (Tex-408-A) that TxDOT uses to measure organic matter in fine aggregates used for cement mortar involves placing a 3 percent sodium hydroxide solution in a glass jar with 300 g of air-dried soil. After 24 hours, the color of the supernatant is compared with a standard color chart to determine the amount of organic material in the sample. The British used this test in BS 812 but quit using this test because certain naturally occurring organic impurities that are deleterious give negative results in the color test (Lea, 1971).

The Department of Soil and Crop Sciences at Texas A&M University uses an Elementar VarioMax C-H-N analyzer (Figure 1.4). This procedure calls for a wet chemical treatment to remove carbonates, which can give false high organic C results. The samples are then heated to 650ºC (for organic C) in a purified O_2 stream and heated to 900ºC (for total C). The CO_2 evolved is measured, and percents of organic carbon and total carbon are calculated.

METHODS

The researchers sent artificially prepared "manufactured" soil samples to the Department of Soil and Crop Sciences at Texas A&M University where an Elementar C-H-N analyzer was used to measure organic C in the soils. This equipment measures CO_2 evolved from combustion of organic matter and calculates the percentage of organic C. The researchers measured organic C using the LOI (ASTM D 2974-00) method. The researchers also used test method Tex-408-A to estimate the concentration of organic matter in the artificially prepared soil samples.

To investigate methods for measuring the organic content of soils, four soils of differing mineralogy were "manufactured" in the lab using known standards and treated with four different types of organic compounds at five different concentrations. Table 3.1 shows the soils, organic types, and treatment levels tested.

Table 3.1. Test Plan for Evaluating Organics Tests.

Mineralogies	Organic Types	Organic Concentrations (%)
100% Quartz Sand	Sugar (standard)	0
80% Quartz / 20% Smectite	Compost	0.5
90% Quartz / 10% Calcite	Leaves	1
90% Quartz / 10% Dolomite	Peat	3
		5

Each combination of mineralogy/organic type/organic concentration was tested in duplicate both with ASTM D2974 and with the Elementar Carbon-Hydrogen-Nitrogen analyzer. TTI labs used the ASTM method, which is based on loss-on-ignition principles, and Texas A&M's Soil and Crop Sciences Department performed the C-H-N testing.

RESULTS OF TEST METHOD TEX-408-A

As stated before, the TEX-408-A test method requires comparing the color of the supernatant to a color comparator (Figure 3.1) after soaking in a sodium hydroxide solution for 24 hours. This method is supposed to give an estimate of the percent organic matter in the soil. As stated previously, the British quit using this test because there were deleterious organic particles that did not show up in this test (Lea, 1971).

After testing six of the manufactured samples, the researchers concluded that this test could not distinguish between 0.5, 1, and 3 percent compost (Figure 3.2). We decided to discontinue further testing at that point and pursue other methods.

Figure 3.1. Equipment Used for Test Method TEX-408-A.

Figure 3.2. Samples with 1, 3, and 0.5 Percent Compost after TEX-408-A Test.

SUMMARY OF ASTM D 2974 (LOI) AND ELEMENTAR C-H-N ANALYZER RESULTS

Using a series of tests employing multiple soil mineralogies, organic types, and organic concentrations, the researchers discovered:

- Both the ignition oven and the C-H-N could be impacted by mineralogy. Both could be impacted by the presence of smectite in the soil mineralogy. The ignition oven method particularly was impacted by smectite, and the presence of smectite significantly increased the value of the test result.
- Although the impact of smectite on results from the C-H-N was statistically significant in certain cases, resulting in a reduced test result, it is not understood why smectite impacted the result in these cases.
- With the ignition oven method, the standard deviation of test results was approximately 0.13 percent loss.
- With the C-H-N analyzer, the standard deviation of test results was approximately 0.17 percent organic matter.
- Both methods can distinguish differing levels of organics. However, the number of replicates should be increased to three for both methods to better enable discerning small changes in the quantity of organics. Using three replicates would enable discernment between levels of percent loss from the ASTM method as low as approximately 0.2. Similarly, using three replicates with the C-H-N analyzer would enable discernment between levels of organic matter as low as approximately 0.25 percent.

RESULTS FROM SUGAR STANDARD TESTS

Table 3.2 presents the results from the sugar standard tests. For purposes of the C-H-N analyzer results, it must be noted that the sugar used contains approximately 44 percent organic carbon. In contrast, the ASTM method cannot distinguish between organic and inorganic C. It burns off all of the sugar, and the mass difference is taken as total organic matter.

DISCUSSION OF RESULTS FROM ASTM D2974 WITH SUGAR STANDARD

Figure 3.3 shows the results from the ASTM method. Examination of the figure indicates the method was sensitive to changes in the level of sugar regardless of mineralogy. Additionally, the data indicate the presence of smectite in the soil led to a higher value of the test result as contrasted with other mineralogies, particularly when the known level of sugar added was low.

To substantiate these observations, researchers used two-way analysis of variance to investigate the effects of the percent sugar added and mineralogy on the test result. Table 3.3 presents the summary test results, and Table 3.4 presents the ANOVA output. Although the output suggests the test results were strongly impacted both by percent sugar added and mineralogy (as indicated by the P-values less than 0.05), the test results must be evaluated by contrasts of the cell means in Table 3.3 due to the presence of interactions.

Table 3.2. Sugar Standard Results.

	ASTM Result		C-H-N Result
Mineralogy	% Sugar Added	Total Loss (%)	Organic C%
100% quartz	0	0.10	0.03
100% quartz	0	0.14	0.02
100% quartz	0.5	0.52	0.35
100% quartz	0.5	0.47	0.24
100% quartz	1	0.99	0.47
100% quartz	1	0.95	0.50
100% quartz	3	2.74	1.59
100% quartz	3	2.85	1.85
100% quartz	5	4.85	2.43
100% quartz	5	4.70	3.36
80% qtz 20% smectite	0	1.39	0.14
80% qtz 20% smectite	0	1.27	0.04
80% qtz 20% smectite	0.5	1.86	0.19
80% qtz 20% smectite	0.5	1.76	0.21
80% qtz 20% smectite	1	2.12	0.35
80% qtz 20% smectite	1	1.73	0.38
80% qtz 20% smectite	3	3.65	1.04
80% qtz 20% smectite	3	3.66	0.99
80% qtz 20% smectite	5	5.31	1.80
80% qtz 20% smectite	5	5.34	1.75
90% qtz 10% calcite	0	0.22	0.44
90% qtz 10% calcite	0	0.26	0.14
90% qtz 10% calcite	0.5	0.76	0.28
90% qtz 10% calcite	0.5	0.72	0.31
90% qtz 10% calcite	1	1.23	0.49
90% qtz 10% calcite	1	1.25	0.46
90% qtz 10% calcite	3	3.22	1.17
90% qtz 10% calcite	3	3.20	1.11
90% qtz 10% calcite	5	5.19	1.96
90% qtz 10% calcite	5	5.15	2.01
90% qtz 10% Dolo.	0	0.30	0.18
90% qtz 10% Dolo.	0	0.50	0.10
90% qtz 10% Dolo.	0.5	0.73	0.31
90% qtz 10% Dolo.	0.5	0.80	0.26
90% qtz 10% Dolo.	1	1.16	0.39
90% qtz 10% Dolo.	1	1.62	0.51
90% qtz 10% Dolo.	3	3.20	1.03
90% qtz 10% Dolo.	3	3.27	1.31
90% qtz 10% Dolo.	5	5.24	2.12
90% qtz 10% Dolo.	5	5.16	2.33

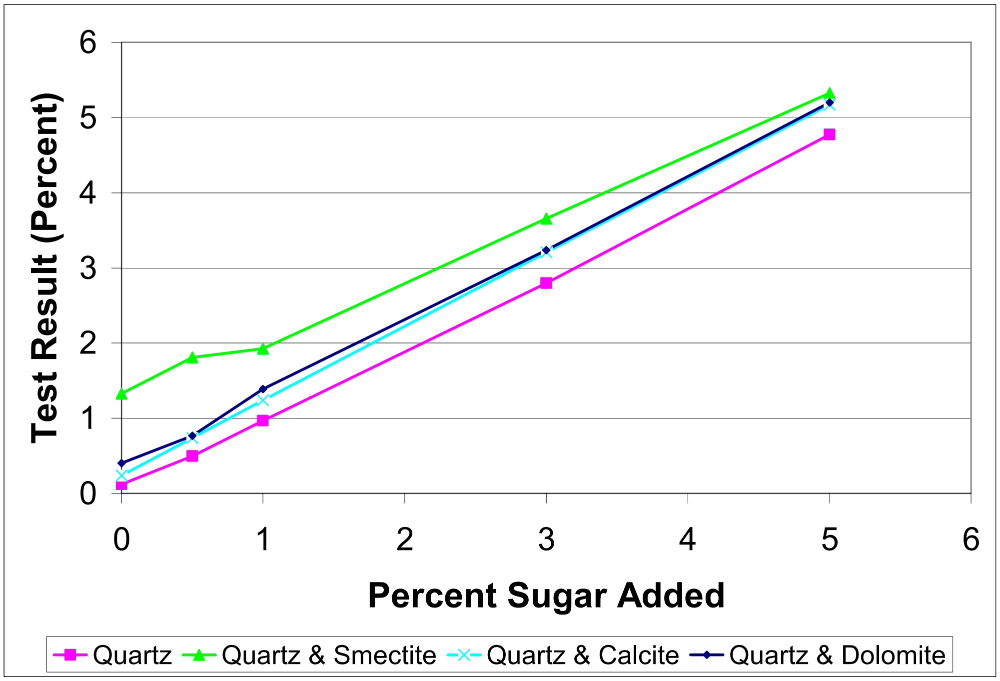


Figure 3.3. Sugar Standard Results from ASTM D2974.

Table 3.3. Summary Results for Sugar Standard Tests with ASTM D2974.

	Mineralogy			
% Sugar Added	Quartz	Quartz & Smectite	Quartz & Calcite	Quartz & Dolomite
0	0.12	1.33	0.24	0.4
0.5	0.495	1.81	0.74	0.765
1	0.97	1.925	1.24	1.39
3	2.795	3.655	3.21	3.235
5	4.775	5.325	5.17	5.2

Table 3.4. Two-Way ANOVA Output for Sugar Standard Results from ASTM D2974.

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
% Sugar Added	116.741	4	29.1854	2376.66	1.8E-26	2.86608
Mineralogy	5.07205	3	1.69068	137.678	1.6E-13	3.09839
Interaction	0.84527	12	0.07044	5.73612	0.00032	2.27758
Within	0.2456	20	0.01228			
Total	122.904	39				

To perform these contrasts of means, researchers used the Scheffe procedure to evaluate: for a given mineralogy, could the test distinguish between levels of percentage sugar added; and for a given percentage sugar added, did a difference in the mean test result exist among differing mineralogy. The analysis performed indicates that, with 95 percent confidence, the following family of statements is true:

- With all mineralogies examined, the test could distinguish differences in level of percent sugar added between 1 and 3 percent, and between 3 and 5 percent.
- The test did not distinguish differences in level of percent sugar added between 0 and 0.5 percent, nor between 0.5 and 1 percent, regardless of mineralogy.
- For all mineralogies except quartz and smectite, the test could distinguish differences in level of percent sugar added between 0 and 1 percent.
- At low levels of sugar added (both 0 and 0.5 percent), the test result for the quartz and smectite mineralogy exceeded the test result for all three other mineralogies.
- At a level of percent sugar added of both 1 and 3 percent, the test result for quartz and smectite mineralogy only exceeded the test result for the quartz mineralogy.
- With 5 percent sugar added, the mean test result among all mineralogies was statistically equivalent.
- No difference in mean test result existed among quartz, quartz and calcite, and quartz and dolomite mineralogy, regardless of the level of percent sugar added.

This analysis therefore confirmed the generalized observations previously noted. Specifically, at low levels of sugar added, the mineralogy of quartz and smectite resulted in an inflated test result as compared to other mineralogy; and regardless of mineralogy, the test was sensitive to changes in the level of percent sugar added. When an array of mineralogies were tested, and with two replicates performed, the test was sensitive to changes in the level of percent sugar added as low as 1 percent.

Realizing that soils do change in the field, yet hoping that a series of construction project tests would not be plagued by as drastic shifts in mineralogy as were used in this experiment, researchers then analyzed the individual data set for each mineralogy to investigate the sensitivity of the test when mineralogy was held constant. Applying Tukey’s multiple pairwise comparison procedure to each data set revealed that, for quartz and quartz and calcite mineralogy, differences were detected between all pairwise comparisons; i.e., the test detected changes as small as 0.5 percent in the level of sugar added. However, with the quartz and smectite mineralogy, the test did not detect a difference in means between the 0.5 and 1 percent treatment level; with quartz and dolomite mineralogy, the test did not detect a statistical difference in means between the 0 and 0.5 percent treatment level. The researchers therefore concluded that, to consistently discriminate between small changes ($\leq$ 0.5 percent) in the amount of sugar present, a larger number of replicates would be needed. Based upon the observed dispersion in the data, increasing the number of replicates to three would allow for consistent discernment between changes as small as 0.3 percent.

DISCUSSION OF RESULTS FROM C-H-N WITH SUGAR STANDARD

Figure 3.4 shows the results from the C-H-N. The C-H-N is considered the standard with respect to analyzing organic C content in soils. In Figure 3.4, the divergence of the results with the quartz mineralogy at levels of 3 and 5 percent (1.3 and 2.1 percent organic C added, respectively) raised the researchers' suspicions. After discussions with an organic geochemist (Paul Comet), the consensus was that these two data points were suspect and likely artificially high due to inadequate cleanout of the analyzer between samples. The researchers therefore discarded the results of the quartz mineralogy from further analysis.

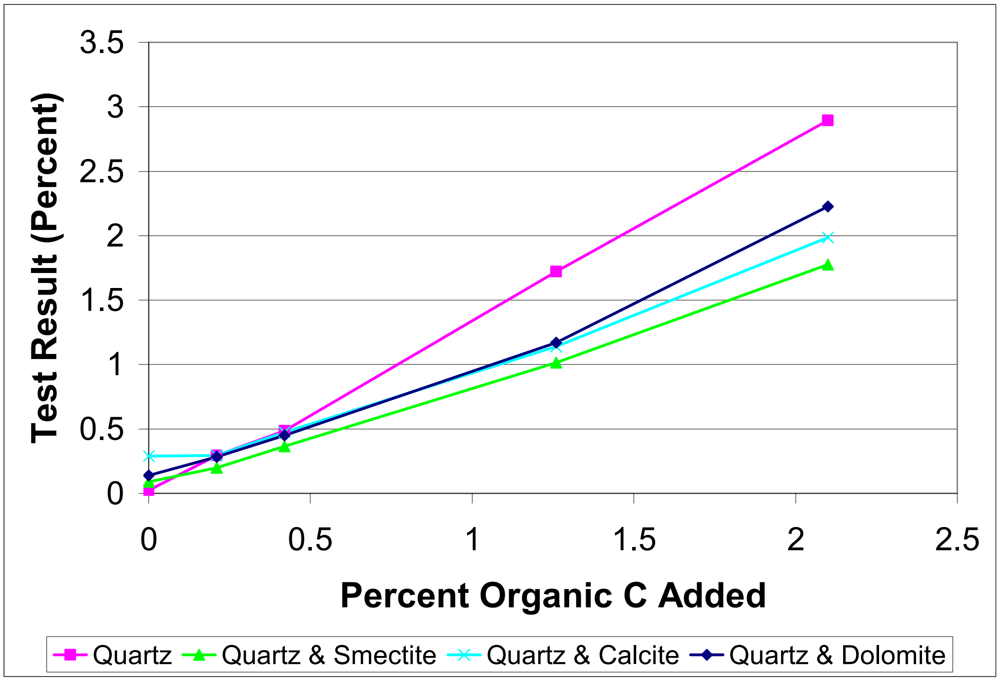


Figure 3.4. Sugar Standard Results from C-H-N Analyzer.

Table 3.5 summarizes the remaining data, and the ANOVA output from these data (shown in Table 3.6) indicate the C-H-N analyzer indeed is responsive to changes in the level of organic C added and to mineralogy. Additionally, no interactions were observed between the level of organic C added and the mineralogy.

Table 3.5. Summary Results for Sugar Standard from C-H-N Analyzer.

% Organic C Added	Mineralogy			
	Quartz & Smectite	Quartz & Calcite	Quartz & Dolomite	Row Average
0	0.09	0.29	0.14	0.13625
0.21	0.2	0.295	0.285	0.26875
0.42	0.365	0.475	0.45	0.44375
1.26	1.015	1.14	1.17	1.26125
2.1	1.775	1.985	2.225	2.22
Column Average	0.689	0.837	0.854	

Table 3.6. Two-Way ANOVA Output for Sugar Standard Results from C-H-N Analyzer.

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
% Organic C added	14.0645	4	3.51613	405.707	4.4E-15	3.05557
Mineralogy	0.16473	2	0.08236	9.50346	0.00216	3.68232
Interaction	0.13264	8	0.01658	1.91308	0.13274	2.6408
Within	0.13	15	0.00867			
Total	14.4919	29				

Due to the lack of interactions between the level of organic C and mineralogy, the impacts of mineralogy and amount of organic C added can be analyzed by contrasting the column and row means, respectively, presented in Table 3.5. Using the Bonferroni procedure for multiple pairwise comparisons, the following family of observations is true with 95 percent confidence:

- Results from the quartz and smectite mineralogy were lower than results for both quartz and calcite and quartz and dolomite mineralogy. The estimated reduction in the organic C result with the quartz and smectite mineralogy was 0.16 percent. The 95 percent confidence interval for this reduction is 0.02 to 0.29 percent.
- No difference in results existed between quartz and calcite and quartz and dolomite mineralogy.
- The test did not distinguish between the blank and the lowest level (0.21 percent) of organic C added.
- The test did distinguish between different levels of organic C added for all other increments tested.

These results indicate that mineralogy can impact results from the C-H-N analyzer. However, although statistically significant, the researchers believe the difference noted among the quartz and smectite mineralogy and the other mineralogies may be of minimal practical significance since the presence of smectite was found to result in an expected decrease in the test result by only 0.16 percent organic C.

The results also show that, with only two replicates tested, the C-H-N can statistically detect a difference when the organic C level increases from 0.21 to 0.42 percent (0.5 to 1 percent sugar, respectively) but not from 0 to 0.21 percent (no sugar to 0.5 percent sugar, respectively). Increasing the number of replicates to three would enable discernment between the blank and 0.5 percent sugar.

RESULTS FROM VARYING ORGANICS SOURCES

Table 3.7 presents the results from the manufactured soils treated with varying levels of organics in the form of compost, leaves, or peat.

Table 3.7. Organic Content Results from Soils with Compost, Leaves, and Peat.

			ASTM 2974		**Elementar**	
			TTI		Soil & Crop Science	
Soil	Organic Material	% Organic Material Added	Moisture Content (%)	Organic Ash (%)	Organic Carbon (%)	% Organic Matter (=%OC x 1.724)
100% Quartz	Blank	0	4.25	0.55	0.005	0.01
100% Quartz	Blank	0	4.15	0.25	0.016	0.03
80% Quartz & 20% Smectite	Blank	0	7.60	0.72	0.045	0.08
80% Quartz & 20% Smectite	Blank	0	8.58	0.70	0.023	0.04
90% Quartz & 10% Calcite	Blank	0	3.32	0.20	0.059	0.10
90% Quartz & 10% Calcite	Blank	0	3.73	0.15	0.066	0.11
90% Quartz & 10% Dolomite	Blank	0	2.96	0.24	0.072	0.12
90% Quartz & 10% Dolomite	Blank	0	2.91	0.16	0.073	0.13
100% Quartz	Compost	0.5	4.18	0.19	0.048	0.08
100% Quartz	Compost	0.5	3.43	0.20	0.051	0.09
80% Quartz & 20% Smectite	Compost	0.5	8.13	0.92	0.074	0.13
80% Quartz & 20% Smectite	Compost	0.5	8.78	0.90	0.061	0.11
90% Quartz & 10% Calcite	Compost	0.5	3.26	0.24	0.106	0.18
90% Quartz & 10% Calcite	Compost	0.5	3.15	0.22	0.099	0.17
90% Quartz & 10% Dolomite	Compost	0.5	3.23	0.22	0.096	0.17
90% Quartz & 10% Dolomite	Compost	0.5	3.01	0.21	0.116	0.20
100% Quartz	Compost	1	4.14	0.32	0.097	0.17
100% Quartz	Compost	1	4.19	0.36	0.077	0.13
80% Quartz & 20% Smectite	Compost	1	5.02	1.21	0.137	0.24
80% Quartz & 20% Smectite	Compost	1	7.65	1.03	0.085	0.15
90% Quartz & 10% Calcite	Compost	1	2.81	0.51	0.157	0.27
90% Quartz & 10% Calcite	Compost	1	3.09	0.41	0.131	0.23
90% Quartz & 10% Dolomite	Compost	1	2.49	0.37	0.149	0.26
90% Quartz & 10% Dolomite	Compost	1	0.57	0.35	0.142	0.25
100% Quartz	Compost	3	5.25	0.73	0.231	0.40
100% Quartz	Compost	3	4.51	0.63	0.188	0.32
80% Quartz & 20% Smectite	Compost	3	6.97	1.27	1.010	1.74
80% Quartz & 20% Smectite	Compost	3	3.66	1.06	0.275	0.47
90% Quartz & 10% Calcite	Compost	3	3.44	0.86	0.288	0.50
90% Quartz & 10% Calcite	Compost	3	3.60	0.88	0.306	0.53
90% Quartz & 10% Dolomite	Compost	3	2.30	0.69	0.312	0.54
90% Quartz & 10% Dolomite	Compost	3	2.13	1.01	0.267	0.46
100% Quartz	Compost	5	3.81	1.17	0.431	0.74
100% Quartz	Compost	5	3.80	1.20	0.391	0.67
80% Quartz & 20% Smectite	Compost	5	6.61	1.90	0.483	0.83
80% Quartz & 20% Smectite	Compost	5	5.80	1.76	0.379	0.65
90% Quartz & 10% Calcite	Compost	5	3.16	1.26	0.477	0.82
90% Quartz & 10% Calcite	Compost	5	2.71	1.38	0.379	0.65
90% Quartz & 10% Dolomite	Compost	5	1.79	1.33	0.541	0.93
90% Quartz & 10% Dolomite	Compost	5	1.60	1.14	0.516	0.89

Table 3.7 Organic Content Results from Soils with Compost, Leaves, and Peat (continued).

			ASTM 2974		Elementar	
			TTI		Soil & Crop Science	
Soil	Organic	% Organic	Moisture	Organic	Organic	% Organic
100% Quartz	Leaves	0.5	3.92	0.39	0.117	0.20
100% Quartz	Leaves	0.5	3.59	0.44	0.117	0.20
80% Quartz & 20% Smectite	Leaves	0.5	7.30	1.29	0.079	0.14
80% Quartz & 20% Smectite	Leaves	0.5	8.55	1.24	0.169	0.29
90% Quartz & 10% Calcite	Leaves	0.5	2.90	0.43	0.172	0.30
90% Quartz & 10% Calcite	Leaves	0.5	2.57	0.43	0.163	0.28
90% Quartz & 10% Dolomite	Leaves	0.5	2.72	0.39	0.193	0.33
90% Quartz & 10% Dolomite	Leaves	0.5	2.82	0.38	0.161	0.28
100% Quartz	Leaves	1	4.86	0.74	0.247	0.43
100% Quartz	Leaves	1	5.14	0.73	0.213	0.37
80% Quartz & 20% Smectite	Leaves	1	7.30	1.13	0.205	0.35
80% Quartz & 20% Smectite	Leaves	1	7.42	0.92	0.215	0.37
90% Quartz & 10% Calcite	Leaves	1	3.44	0.80	0.258	0.45
90% Quartz & 10% Calcite	Leaves	1	2.94	0.74	0.261	0.45
90% Quartz & 10% Dolomite	Leaves	1	3.57	0.83	0.162	0.28
90% Quartz & 10% Dolomite	Leaves	1	2.80	0.71	0.156	0.27
100% Quartz	Leaves	3	4.12	1.94	0.566	0.98
100% Quartz	Leaves	3	4.32	2.00	0.709	1.22
80% Quartz & 20% Smectite	Leaves	3	7.19	2.53	0.413	0.71
80% Quartz & 20% Smectite	Leaves	3	7.78	2.45	0.482	0.83
90% Quartz & 10% Calcite	Leaves	3	3.67	1.78	0.743	1.28
90% Quartz & 10% Calcite	Leaves	3	3.13	1.87	0.697	1.20
90% Quartz & 10% Dolomite	Leaves	3	3.09	2.03	0.271	0.47
90% Quartz & 10% Dolomite	Leaves	3	2.89	2.00	0.323	0.56
100% Quartz	Leaves	5	3.87	3.29	0.932	1.61
100% Quartz	Leaves	5	9.11	3.32	1.010	1.74
80% Quartz & 20% Smectite	Leaves	5	5.64	3.72	0.650	1.12
80% Quartz & 20% Smectite	Leaves	5	4.78	4.81	0.778	1.34
90% Quartz & 10% Calcite	Leaves	5	3.22	3.17	1.050	1.81
90% Quartz & 10% Calcite	Leaves	5	3.82	3.13	1.231	2.12
90% Quartz & 10% Dolomite	Leaves	5	2.81	3.10	0.626	1.08
90% Quartz & 10% Dolomite	Leaves	5	3.06	3.03	0.515	0.89
100% Quartz	Peat	0.5	3.99	0.42	0.203	0.35
100% Quartz	Peat	0.5	3.87	0.46	0.192	0.33
80% Quartz & 20% Smectite	Peat	0.5	8.82	1.11	0.074	0.13
80% Quartz & 20% Smectite	Peat	0.5	7.74	1.10	0.168	0.29
90% Quartz & 10% Calcite	Peat	0.5	3.15	0.47	0.192	0.33
90% Quartz & 10% Calcite	Peat	0.5	3.39	0.50	0.218	0.38
90% Quartz & 10% Dolomite	Peat	0.5	2.21	0.45	0.245	0.42
90% Quartz & 10% Dolomite	Peat	0.5	2.64	0.42	0.243	0.42
100% Quartz	Peat	1	3.52	0.74	0.334	0.58
100% Quartz	Peat	1	3.73	0.79	0.330	0.57
80% Quartz & 20% Smectite	Peat	1	8.23	1.74	0.660	1.14
80% Quartz & 20% Smectite	Peat	1	7.53	1.67	0.322	0.56
90% Quartz & 10% Calcite	Peat	1	3.15	0.11	0.354	0.61
90% Quartz & 10% Calcite	Peat	1	3.08	0.98	0.358	0.62
90% Quartz & 10% Dolomite	Peat	1	2.39	0.77	0.488	0.84
90% Quartz & 10% Dolomite	Peat	1	2.17	0.83	0.502	0.87
100% Quartz	Peat	3	7.00	2.23	0.905	1.56
100% Quartz	Peat	3	6.60	2.30	0.974	1.68
80% Quartz & 20% Smectite	Peat	3	6.87	2.75	0.847	1.46
80% Quartz & 20% Smectite	Peat	3	7.35	2.67	0.313	0.54
90% Quartz & 10% Calcite	Peat	3	3.36	2.28	1.114	1.92
90% Quartz & 10% Calcite	Peat	3	3.64	2.28	1.253	2.16
90% Quartz & 10% Dolomite	Peat	3	3.07	2.26	0.991	1.71
90% Quartz & 10% Dolomite	Peat	3	2.57	2.19	0.952	1.64
100% Quartz	Peat	5	3.99	3.68	1.581	2.73
100% Quartz	Peat	5	4.36	3.81	1.902	3.28
80% Quartz & 20% Smectite	Peat	5	7.98	4.65	1.425	2.46
80% Quartz & 20% Smectite	Peat	5	7.74	4.79	1.655	2.85
90% Quartz & 10% Calcite	Peat	5	3.03	3.88	1.968	3.39
90% Quartz & 10% Calcite	Peat	5	3.77	3.94	1.690	2.91

DISCUSSION OF ASTM D2974 RESULTS FOR COMPOST, LEAVES, AND PEAT

To analyze the data, researchers used a three-way analysis of variance approach to investigate the effects of changing the level added, organic type added, and mineralogy. Table 3.8 presents the output, which shows:

- Level, organic type, and mineralogy all impacted the test result.
- Level and organic type interacted (i.e., the influence of changing the level added differed by organic type).
- Level and mineralogy interacted (i.e., the impact of varying the level added was not constant among the different mineralogy).

Table 3.8. ANOVA Output for ASTM Method with Compost, Leaves and Peat.

3-Way Analysis of Variance

Source of Variation	**SS**	**df**	**MS**	**F**	**P-value**	**F-Crit**
Level	112.05	4	28.01	1248.3153	0.000	3.36439
Organic Type	19.82	2	9.91	441.70578	0.000	4.50785
Mineralogy	9.14	3	3.05	135.75429	0.000	3.77747
Level-Type Interaction	21.37	8	2.67	119.02683	0.000	2.6414
Level-Mineralogy Interaction*	0.83	12	0.07	3.067031	0.002	2.35195
Type-Mineralogy Interaction	0.12	6	0.02	0.886157	0.511	2.2586
Level-Type-Mineralogy Interaction	0.80	24	0.03	1.4900206	0.108	1.8681
Error	1.35	60	0.02			
Total	165.47	119				

*A review of the data led researchers to conclude this interaction was non-important

Prior to any further analysis, researchers reviewed the interactions noted. For example, Figure 3.5 shows an example of varying the level and organic type while keeping mineralogy constant. The data indeed strongly indicate that the impact of varying the level is not constant with changing organic types. For all mineralogies evaluated, the test result did not increase near as much with increasing levels of compost added. The researchers therefore concluded the level-organic type interaction needed to be considered in the data evaluation. The data indicate that the compost, as a percent by mass, does not contain as high concentrations of organic matter as the leaves or peat.

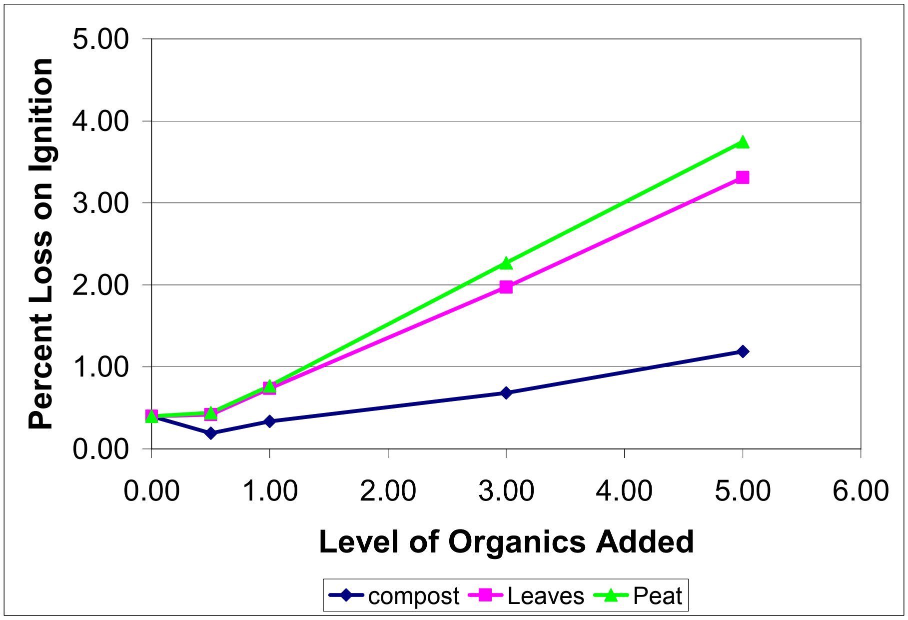


Figure 3.5. ASTM Percent Loss versus Level Added for Quartz Mineralogy.

Researchers next examined the level-mineralogy interaction. Figures 3.6-3.8 illustrate the changes in test result when the level and mineralogy are changed and the organic type added remains constant. If within each plot all lines are parallel, no interaction exists. Although the ANOVA analysis showed that statistically an interaction exists, additional analysis and examination of Figures 3.6-3.8 led the researchers to conclude that the general trends for each organic type are close enough to parallel to ignore the level-mineralogy interaction. The data do indicate a mineralogy effect, where particularly quartz and smectite appears to produce a higher test result.

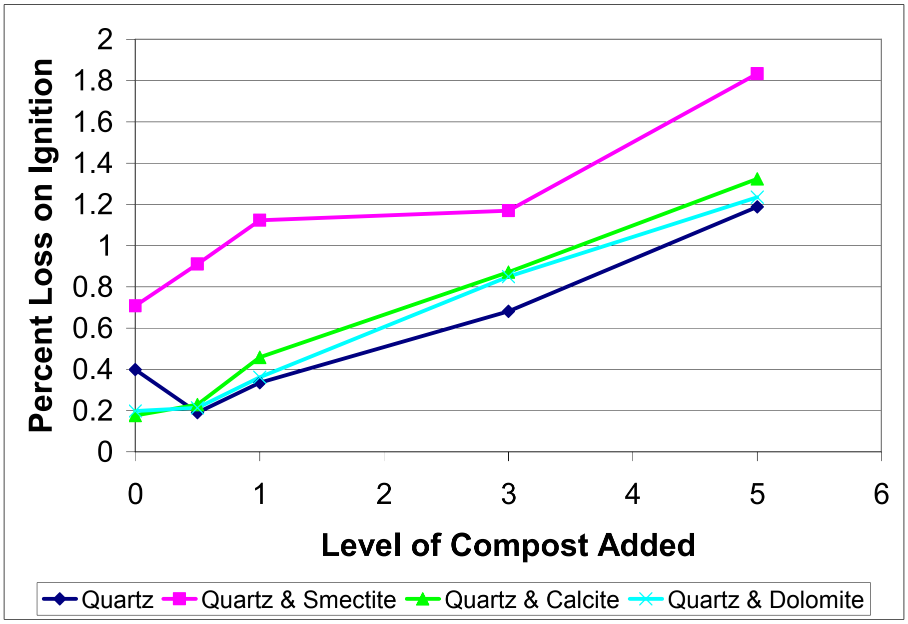


Figure 3.6. ASTM Test Result for Compost.

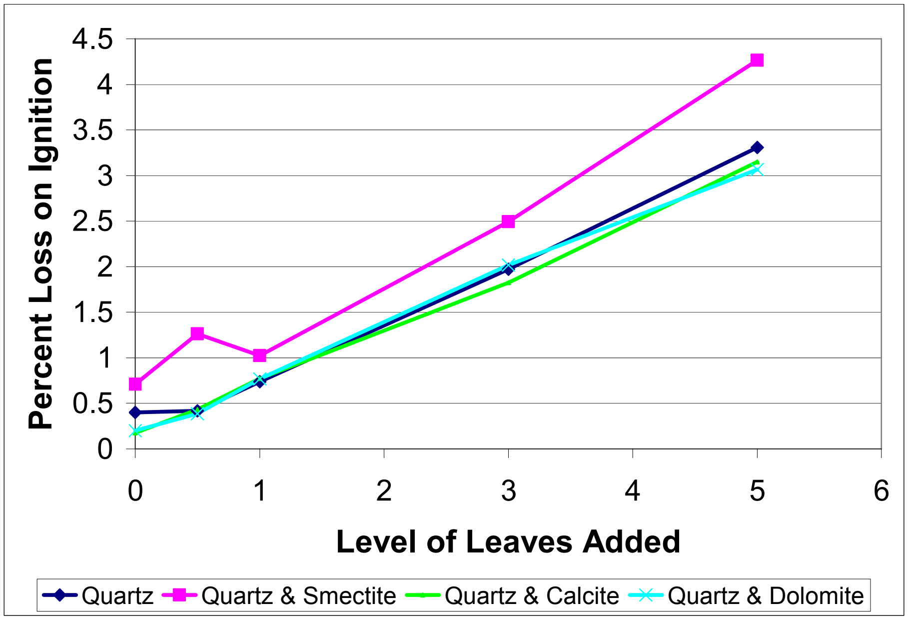


Figure 3.7. ASTM Test Result for Leaves.

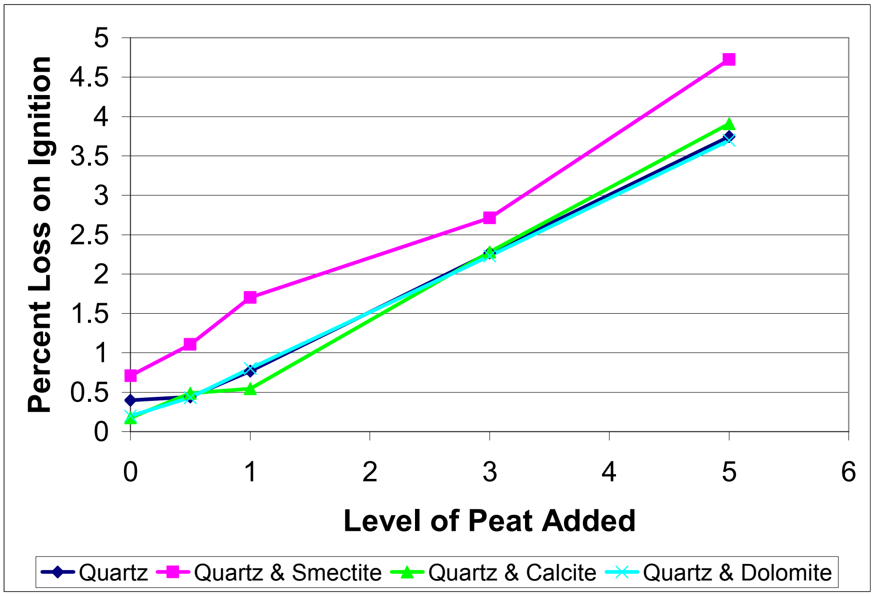
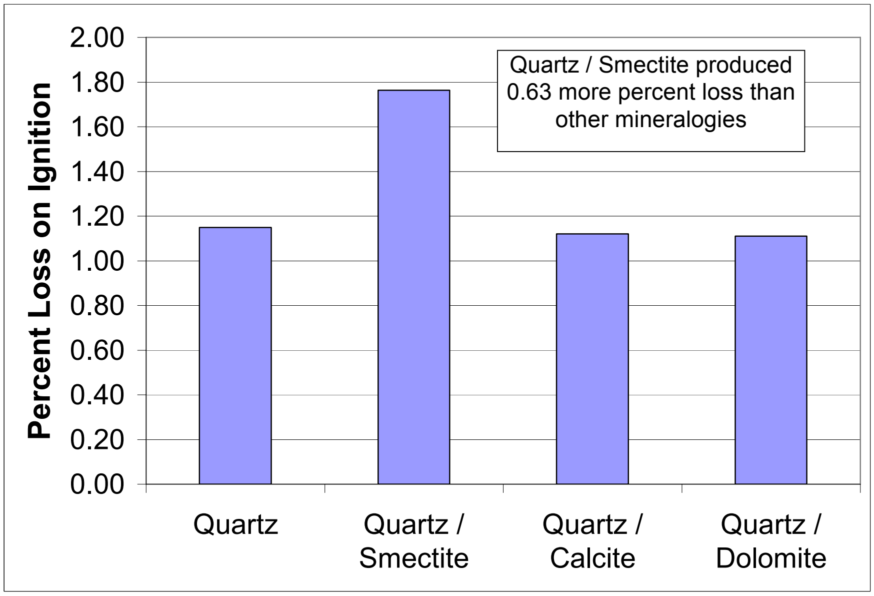


Figure 3.8. ASTM Test Results for Peat.

With the level-mineralogy interaction deemed non-critical, the impact of mineralogy can be analyzed on the basis of its factor level means. Figure 3.9 presents these values. Analysis of the data shows the following family of statements is all true with 95 percent confidence:

- The quartz/smectite mineralogy produced a higher test result than the other three mineralogies tested. The expected value of this increase is 0.63 percent loss.

- No difference in the test result existed among quartz, quartz and calcite, and quartz and dolomite mineralogy.



Quartz / Smectite produced 0.63 more percent loss than other mineralogies

Figure 3.9. Effect of Mineralogy on ASTM Results with Compost, Leaves, and Peat.

Due to the presence of interactions between the level of organics added and the organic type added, the impact of varying organic type and varying level must be analyzed by averaging the test results over all mineralogies for each combination of level and organic type. Table 3.9 presents these data, which are further represented graphically in Figure 3.10.

Table 3.9. ASTM Results for Varying Level and Organic Type.

	Organic Type Added		
Level	**Compost**	**Leaves**	**Peat**
0.00	0.37	0.37	0.37
0.50	0.39	0.62	0.62
1.00	0.57	0.82	0.95
3.00	0.89	2.08	2.37
5.00	1.39	3.45	4.02

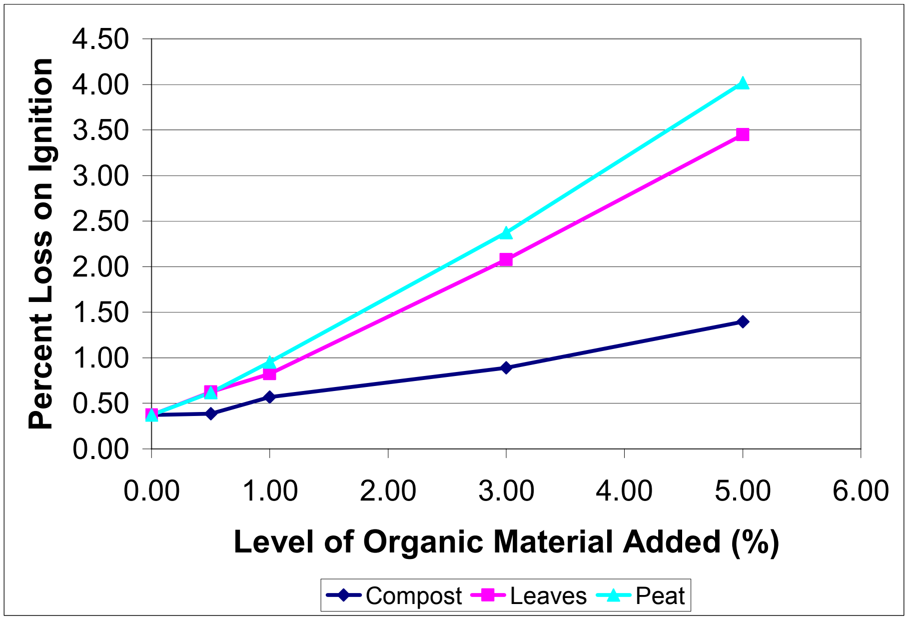


Figure 3.10. ASTM Results for Varying Level and Organic Type with Compost, Leaves, and Peat.

The two pertinent questions from these data include: 1) How does differing organic type impact the test result, and 2) How sensitive is the test result to varying level of organic material added for a given organic type.

To answer the first question, researchers used a linear regression analysis to investigate the plots shown in Figure 3.10. Table 3.10 shows the results, which indicate:

- The compost, leaves, and peat contain approximately 21, 62, and 73 percent organic matter by mass, respectively, which is lost on ignition.
- The ASTM test produced a result of approximately 0.3 percent loss on ignition even when no organic matter was present. This value is apparent by averaging the intercepts, which did not significantly differ from each other regardless of organic type.

Table 3.10. Regression Results for ASTM Method with Compost, Leaves, and Peat.

	Compost	Leaves	Peat
Slope	0.21	0.62	0.73
S.E. (slope)	0.01	0.02	0.02
Lower 95%	0.16	0.55	0.66
Upper 95%	0.25	0.69	0.81
Intercept	0.33	0.29	0.27
S.E. (intercept)	0.04	0.06	0.06
Lower 95%	0.22	0.11	0.07
Upper 95%	0.44	0.47	0.47
S.E. of Estimate	0.06	0.09	0.10
R^2	0.99	1.00	1.00

To investigate the sensitivity of the test to changes in the level of organics added for each organic type, the researchers used Bonferroni's multiple contrast procedure to evaluate contrasts of means between levels for each of the organic types used. Figure 3.11 illustrates these mean values. The results show:

- With the observed spread in the data, using two replicates, researchers were able to discern differences in loss on ignition as low as 0.2 percent.
- With compost, the test could not distinguish between the blank and 0.5 percent level added (0.37 and 0.39 percent loss, respectively), or between the 0.5 and 1 percent level (0.39 and 0.57 percent loss, respectively). The test could distinguish between 1 percent compost added and the blank, and could distinguish between all other incremental levels of compost added (1 to 3 percent, and 3 to 5 percent).
- Increasing the number of tests to three would enable discernment between the 1.0 and 0.5 percent level (0.57 and 0.39 percent loss, respectively) for compost. The results from the blank and 0.5 percent level are so close together that no reasonable number of tests would enable discerning a difference between these two levels.
- With leaves, the test was able to distinguish differences in means between all increments in the level added.
- With peat, the test was able to distinguish differences in means between all increments in the level added.

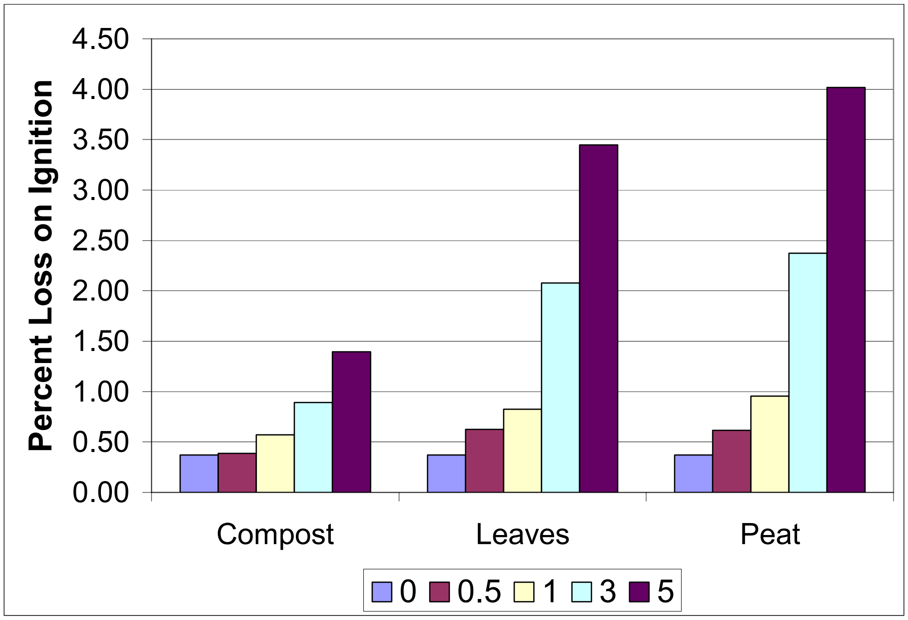


Figure 3.11. ASTM Results for Compost, Leaves, and Peat.

DISCUSSION OF C-H-N RESULTS FOR COMPOST, LEAVES, AND PEAT

Researchers again used a three-way analysis of variance approach to analyze the results from the C-H-N when known amounts of compost, leaves, or peat were added to soils of differing mineralogy. Unfortunately, the dispersion in the data and the number of replicates performed only allowed statistical discernment between levels when the difference exceeded 0.37 percent organic matter. Increasing the number of replicates to three would enable discernment between levels when the treatment means changed by 0.30 percent organic matter or more.

Table 3.11 shows the ANOVA output. Analysis of the data to produce generalized conclusions is complicated by the fact that, not only are several two-way interactions present, but three-way interactions also exist. Due to these interactions, overall factor effects for level, organic type, or mineralogy cannot be statistically determined. Instead, the data must be evaluated on the basis of contrasting the treatment means, shown in Table 3.12.

Table 3.11. ANOVA Output for C-H-N with Compost, Leaves, and Peat.

3-Way Analysis of Variance						
Source of Variation	**SS**	**df**	**MS**	**F**	**P-value**	**F-Crit**
Level	45.97	4	11.49	343.68709	0.000	3.36439
Organic Type	13.02	2	6.51	194.68751	0.000	4.50785
Mineralogy	0.44	3	0.15	4.3926387	0.007	3.77747
Level-Type Interaction	14.03	8	1.75	52.462766	0.000	2.6414
Level-Mineralogy Interaction	0.67	12	0.06	1.6738743	0.096	2.35195
Type-Mineralogy Interaction	1.29	6	0.22	6.4516834	0.000	2.2586
Level-Type-Mineralogy Interaction	2.13	24	0.09	2.6584367	0.001	1.8681
Error	2.01	60	0.03			
Total	79.57	119				

Table 3.12. Summary Results from C-H-N with Compost, Leaves, and Peat.

	Compost				Leaves				Peat			
Level	**Quartz**	**Quartz / Smectite**	**Quartz / Calcite**	**Quartz / Dolomite**	**Quartz**	**Quartz / Smectite**	**Quartz / Calcite**	**Quartz / Dolomite**	**Quartz**	**Quartz / Smectite**	**Quartz / Calcite**	**Quartz / Dolomite**
0.00	0.02	0.06	0.11	0.13	0.02	0.06	0.11	0.13	0.02	0.06	0.11	0.13
0.50	0.09	0.12	0.18	0.18	0.20	0.21	0.29	0.31	0.34	0.21	0.35	0.42
1.00	0.15	0.19	0.25	0.25	0.40	0.36	0.45	0.27	0.57	0.85	0.61	0.85
3.00	0.36	1.11	0.51	0.50	1.10	0.77	1.24	0.51	1.62	1.00	2.04	1.67
5.00	0.71	0.74	0.74	0.91	1.67	1.23	1.97	0.98	3.00	2.65	3.15	3.41

Performing the aforementioned analysis for mineralogy effects shows that at levels added up to 1 percent, the test was not impacted by mineralogy. At levels added of 3 and 5 percent, the test was impacted by mineralogy. Figures 3.12-3.14 show the results from the C-H-N with compost, leaves, and peat, respectively. Statistical differences in means exist as follows:

- With a level of 3 percent compost added, the result from quartz/smectite mineralogy exceeded all other mineralogy's result.
- With 5 percent compost added, mineralogy did not impact the result.
- With 3 percent leaves added, the quartz/smectite result was significantly less than the result from quartz/calcite mineralogy. The quartz/dolomite mineralogy was significantly different from both the quartz and quartz/calcite result.
- With 5 percent leaves added, the quartz/smectite and quartz/dolomite results were significantly less than both the quartz and quartz/calcite mineralogy.
- With 3 percent peat added, the quartz/smectite result was significantly less than the result from all other mineralogies. Additionally, the quartz/dolomite result was less than the quartz/calcite result.
- With 5 percent peat added, the quartz/smectite result was less than both the quartz/calcite and quartz/dolomite mineralogy. Additionally, the quartz/dolomite result exceeded the result from quartz mineralogy.

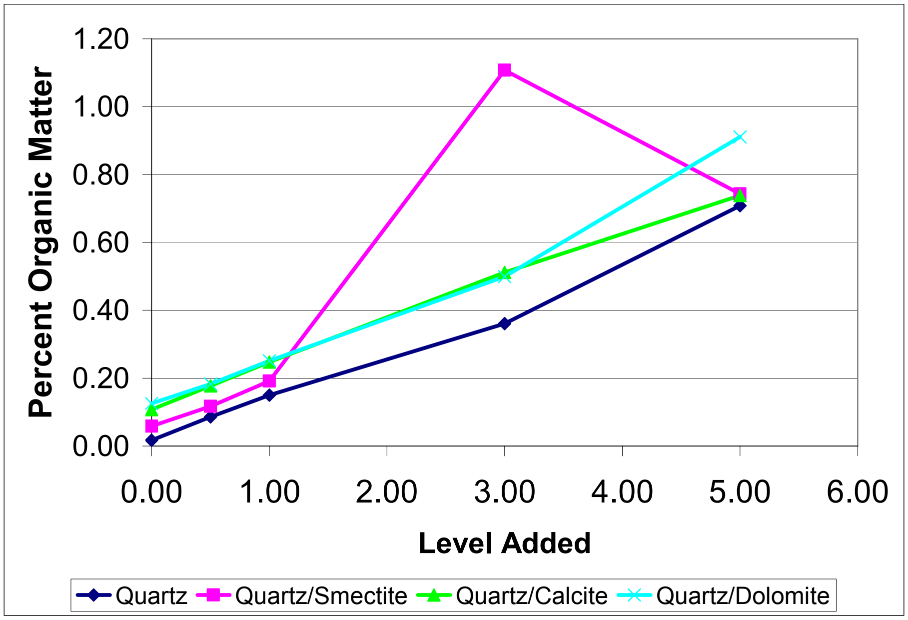


Figure 3.12. C-H-N Result for Compost.

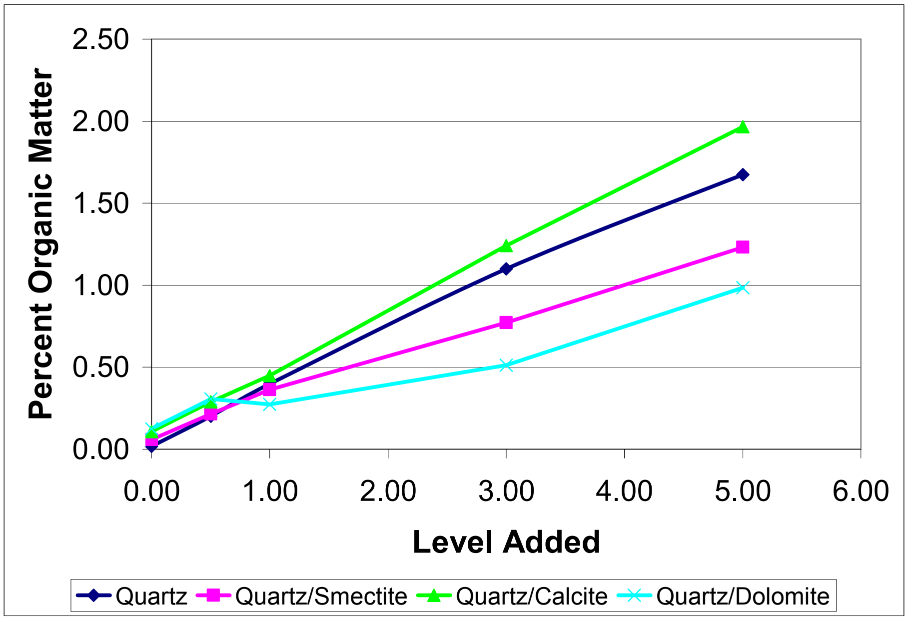


Figure 3.13. C-H-N Result for Leaves.

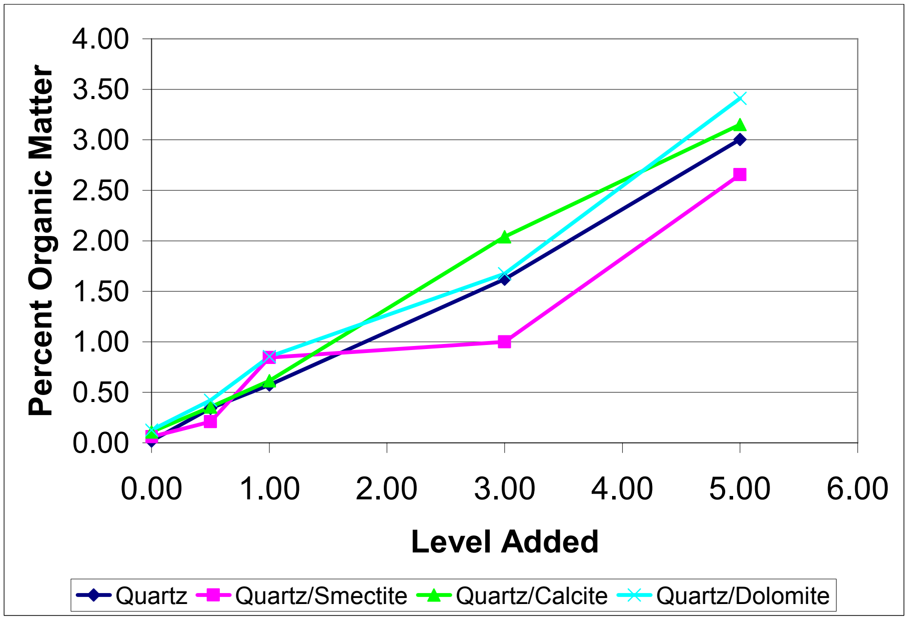


Figure 3.14. C-H-N Result for Peat.

Due to the complicated nature of the data using the three-way ANOVA and the lack of suitability of the data for producing generalized conclusions, researchers decided to instead consider each of the data shown in Figures 3.12-3.14 as individual two-factor problems, with the two factors being level added and mineralogy. Performing these two-factor analyses for compost, leaves, and peat showed:

- With compost, the test was sensitive to level and was not impacted by mineralogy. With two replicates, the test statistically discerned differences of 0.22 in the mean percent organic matter if evaluating a single pairwise comparison. To maintain 95 percent confidence for a family of contrasts, this discernment limit increases to approximately 0.30.
- With leaves, the test was sensitive to both level and mineralogy, and the two factors interacted (i.e., the impact of increasing the level was not the same for all mineralogies). This interaction is apparent in Figure 3.13. For unknown reasons, the test result responded differently to the level of leaves added depending on the mineralogy, particularly at the 3 and 5 percent levels. With two replicates, the test discerned differences of 0.26 in the mean percent organic matter.
- With peat, the test was sensitive to both level and mineralogy. The quartz/smectite mineralogy produced a significantly lower result than both quartz/calcite and quartz/dolomite. The 95 percent confidence interval for this reduction was from 0.02 to 0.58 percent organic matter, and the observed reduction was 0.30. No significant difference existed among quartz, quartz/calcite, and quartz/dolomite mineralogy. If performing a single pairwise comparison, the test discerned differences of 0.24 percent organic matter. To maintain 95 percent confidence for a family of contrasts, this discernment limit increases to approximately 0.32.

CONCLUSIONS

The results obtained from the ASTM ignition oven method and the C-H-N analyzer technique for measuring organics in soils show that both techniques are sensitive to changes in the level of organic matter present, and both techniques can at times be impacted by mineralogy. In particular, the presence of 2:1 clays such as smectite tends to inflate the result from the ignition oven method and, at times, can result in a reduction in the result from the C-H-N. Certainly, the loss of clay interlayer water accounts for the inflated test result in the ignition oven method. The reason for the occasional reduced test result from the C-H-N when clays are present is not known.

To better discern between low-level changes in the test result, the number of replicates should be increased to three for both techniques. Performing three replicates would enable discernment in mean percent loss of 0.2 for the ASTM method and approximately 0.25 percent organic matter for the C-H-N method.

CHAPTER 4

A NEW SPECTROPHOTOMETRIC METHOD FOR ESTIMATING SOIL ORGANIC CARBON CONTENT—A FIELD TEST

INTRODUCTION

Over the past few years, the Texas Department of Transportation has experienced difficulties in stabilizing expansive subgrade soils bearing organic matter with calcium-based additives. In particular, several districts (i.e., Bryan, Houston, Beaumont) have treated subgrade soils bearing high concentrations of organic matter with calcium-based additives, and the desired improvement in the soil is never achieved or the improvement disappears over time. In other districts (Fort Worth, San Antonio), soils bearing much higher concentrations of organic matter have been successfully stabilized with calcium-based additives.

The ignition oven and C-H-N analyzer are useful for measuring organic matter in soils, but they are not designed to be used in the field. The researchers wanted to find a test that can be done in the field that would enable TxDOT to determine if a soil will be problematic with respect to organic matter. Other research has shown that the humic acid (HA) fraction of soil organic matter (SOM) is the fraction of organic matter that is problematic with respect to soil stabilization (Kuno et al., 1989). Therefore, the researchers wanted to identify a method that focused on the detection of the HA fraction in SOM.

Humic acids are the fraction of humic substances that are not soluble in water under acidic conditions ($pH < 2$) but are soluble at higher pH values. They can be extracted from soil by various reagents and are insoluble in dilute acid. Humic acids are the major extractable component of soil humic substances. They are dark brown to black in color (Stevenson, 1994).

The purpose of this investigation was to develop an alternative semi-quantitative method for estimating SOC. In addition to being quick, easily executable, and easily amendable to field use, the method needs to show potential for use across large geographic areas composed of soils from different parent material and/or land use. Procedures for a new spectrophotometric method for indirectly estimating SOC are presented. We evaluated the method to determine: (1) the feasibility of using a single empirical equation for large-scale SOC determination, and (2) the effect of calibration sample size on method performance. Issues such as errors associated with SOC predictions, and method applicability and limitations are also discussed.

BACKGROUND

Methods of Identifying Soil Organic Matter

Both quantitative and semi-quantitative methods are available for determining soil organic carbon. Quantitative methods, such as elemental analysis, despite being more accurate are typically cost prohibitive and time consuming. The ability to analyze a large number of samples in a short period of time and the often lower cost associated with semi-quantitative approaches make them an attractive alternative to quantitative methods, especially in cases where a large number of samples are to be analyzed or where spatial variability is more critical than accuracy. Semi-quantitative methods for SOC determination also have an added advantage in that they are often easily amendable for field use.

A number of semi-quantitative methods are available for laboratory and field estimation of SOC. These include loss on ignition (Konen et al., 2002), remote sensing (Chen et al., 2005), soil color measurements (Konen et al., 2003), and colorimetric techniques (Bowman et al., 1991). Many field-based or field-amendable semi-quantitative methods for SOC, however, often produce variable results, tend to be site specific, limited to field-scale or landscape-scale investigations, or may generate wastes that are environmental hazards. For example, correlation co-efficients between soil color and SOC, reported in studies using quantitative soil color measurements as a proxy for SOC, range from 0.01 to 0.94 (Wills et al., 2007; Konen et al., 2003; Lindbo et al., 1998; Ibarra-F et al., 1995; Schulze et al., 1993; Fernandez et al., 1988). Variability in these studies existed at various spatial scales, leading some researchers to conclude that soil color-SOC relationship was more suitable for a given landscape than for large geographic areas (Schulze et al., 1993; Fernandez et al., 1988). Schulze et al. (1993) found that soil color-SOC correlations were stronger when soils were grouped by textural class and landscape with similar parent material. Wills et al. (2007), however, found that even with soils derived from similar parent material, soil color-SOC relationships can vary significantly. They found that soil color-SOC relationships for prairie soils, derived from similar parent material as an agricultural soil within the same landscape, were weak and unsatisfactory for predictive purposes and suggested also separating soils by land use. Studies evaluating loss on ignition for estimating SOC also indicated strong landscape and land use influences on method performance (Konen et al., 2002; Abella and Zimmer, 2007). Colorimetric methods such as the Walkley-Black method (Walkley, 1947) are easily amendable for field use, less prone to scale and soil property effects but generates wastes that are environmental hazards. Bowman et al. (1991) developed a colorimetric method that was environmentally friendly; however, strong potential for interference from Fe^{3+} (a common constituent of soil) coupled with the need for heating makes the approach impractical for field use.

The development of field-based or field-amendable semi-quantitative methods for SOC estimation that are environmentally friendly and independent of soil properties, soil origin, or land use are important for several reasons. In addition to providing a quick method for SOC estimation, such methods would allow for: (1) better standardization of semi-quantitative SOC methods, (2) better estimation and modeling of the spatio-temporal variability in SOC across small and large geographic areas, (3) cross study comparisons, and (4) the development of larger and more consistent databases of SOC pools under different land use and management practices (Lal et al., 2001, Kern, 1994).

MATERIALS AND METHODS

Study Area and Soils

A total of 146 (82 calcareous, 62 non-calcareous) soil samples from 11 major land resource areas (MLRA) across the State of Texas (Figure 4.1) were used in this study. Soils as well as data on their classification and physico-chemical properties were obtained from the Texas A&M University Soil Characterization Laboratory. Samples were already air-dried and crushed to pass through a 2 mm sieve when received and were used with no further processing. Researchers determined soil organic carbon content of 25 randomly selected samples by difference between the total organic content (determined by elemental analysis using an Elementar CNS analyzer) and the total inorganic carbon content (calculated from calcite and dolomite content determined according to Dreimanis [1962]). Soil organic carbon determined for these samples was similar to that determined by the soil characterization lab using dry combustion; hence, SOC contents from the database were used in all analyses. Sampling and analytical techniques used by the Texas A&M Soil Characterization Laboratory are outlined on their website (http://soildata.tamu.edu/methods.pdf).

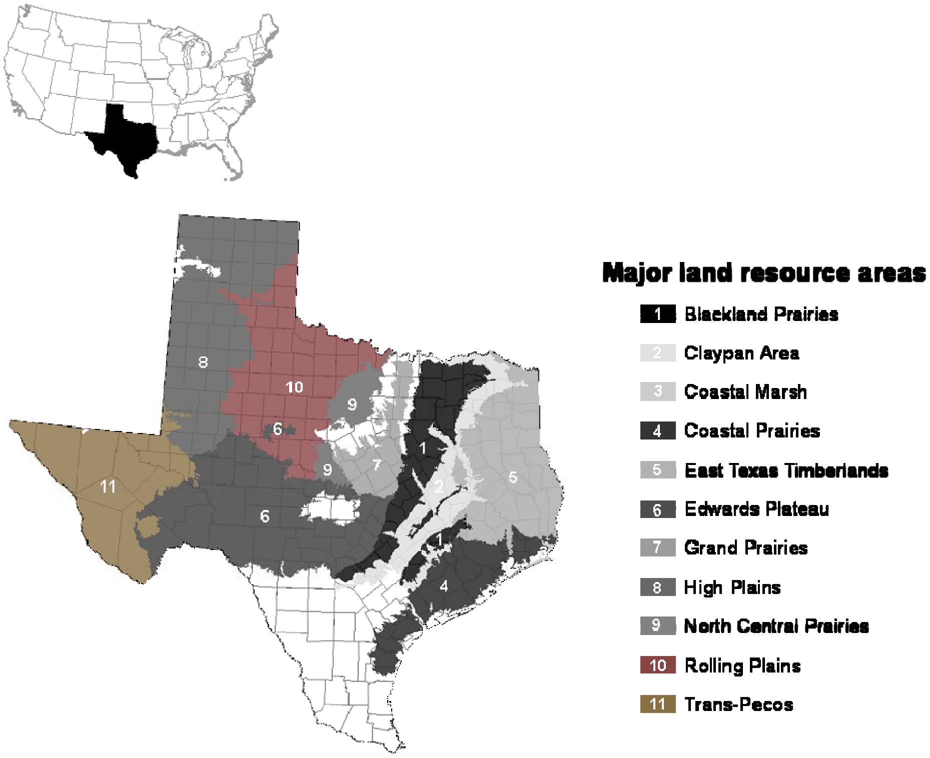


Figure 4.1. Map Showing the Areas Where Samples Were Collected to Evaluate the UV-Vis Method (Soil Survey Staff, 2007).

Method Calibration and Evaluation

For method calibration and evaluation, 73 (50 percent) of the 146 soil samples were randomly selected to develop an empirical relationship between extract absorbance at 300 nm and dry combustion determined SOC. The extract absorbance of the remaining 73 samples was used to predict the SOC of the soils measured by dry combustion. Method performance was evaluated by comparing regression parameters (r^2, slope and intercept) for the dry combustion determined SOC versus spectrophotometric estimated SOC line, for the validation samples, to those of the ideal 1:1 line with an intercept and slope of 0 and 1, respectively. Standard error (SE_p) associated with predicted SOC, and the relative prediction deviation (RPD) was also calculated as follows:

$$\mathrm{SE}_p = (\sum(\mathrm{SOC}_{\mathrm{DC}} - \mathrm{SOC}_{\mathrm{S}})^2 / n)^{0.5} \qquad [1]$$

$$RPD = \mathrm{SD}_{\mathrm{DC}} / \mathrm{SE}_{\mathrm{p}} \qquad [2]$$

where, SOC_{DC} is SOC determined by dry combustion, SOC_S is the SOC estimated for validation samples (using the calibrated spectrophotometric method), and n is the number of validation samples.

A very important consideration, though often not addressed in many studies, when evaluating indirect methods is the optimal number of samples required to develop a robust calibration. In this study, the effect of calibration sample size on method performance was evaluated by considering calibration: validation splits of 1: 9, 1: 3, 1: 2, 1: 1, 2: 1, 3: 1, and 9: 1. Here, a more stochastic approach to evaluating method performance was adopted. Rather than a single randomization, multiple randomizations were calculated, and distributions of regression parameters SE_p and RPD were compared. A total of 63 randomizations were made for each calibration: validation split.

RESULTS AND DISCUSSION

Soil Properties and Organic Carbon Extraction

Soils used in the study covered a wide range of moisture and temperature regimes as well as taxonomic families (Table 4.1), textural classes (Figure 4.2), and chemical properties (Table 4.2). Such diversity is critical to method development since both the physical and chemical properties of soils are likely to influence organic carbon extractability and subsequently UV absorbance-concentration relationships (Stevenson, 1994). Results from initial experiments showed that while NaOH provided quantitative extractions for SOC determination in non-calcareous soils, for calcareous soils, NaOH alone was not an efficient extractant (Figure 4.3).

Table 4.1. Taxonomic Classification of Soils Used from Each Major Land Resource Area.

MLRA[†]	Soil Family[‡]	Soil Series
1	Udorthentic Haplustolls; Udic Haplusterts	Austin; Heiden, Dimebox, Branyon, Houston Black
2	Aquic Arenic Paleustalfs; Chromic Vertic Albaqualfs	Robco; Bonneville
	Oxyaquic Vertic Paleustalfs; Typic Albaqualfs	Tabor, Lufkin, Mabank; Zulch
	Vertic Paleudalfs; Udic Haplusterts	Annona; Burleson
	Aquic Paleudalfs; Udic Paleustalfs	Falba; Shiro
3	Typic Psammaqunts	Mustang
4	Aquic Glossudalfs; Oxyaquic Glossudalfs	Snake Creek; Orcadia
	Plinthic Paleudalfs	Hockley
5	Albaquultic Hapludalfs; Chromic Drystruderts	Spurger; Raylake
	Fluvaquentic Dystrudepts; Glossic Paleudalfs	Mooreville; Gallime
	Natric Vermaqualfs; Typic Glossaqualfs	Nona; Evadale
	Vermic Natraqualfs; Aquic Paleudalfs	Vidor; Fetzer
6	Lithic Haplustolls; Lithic Petrocalcic Calciustolls	Eckrant; Noelke
	Petrocalcic Calciustolls; Typic Argiustolls	Cho, Ozona; Rumple
	Typic Paleustalfs; Udertic Haplustolls	Pedernales; Krum
	Udic Calciustolls	Venus
7	Udic Calciustolls; Typic Haplustepts	Denton, Lewisville, Topsey; Brackett
8	Aeric Endoaqalfs; Aeric Halaquepts	Lamesa; Lenorah
	Torrifluventic Haplustepts; Typic Halaqeupts	Hindman; Cedarlake
	Typic Haplustalfs	Seagraves
9	Pachic Argiustolls; Udic Paleustalfs	Abilene; Bastrop
10	Typic Haplustepts; Calcic Haplustepts	Vernon; Shep
11	Fluventic Haplocambids; Sodic Ustic Haplocambids	Tornillo; Martillo, Straddlebug
	Ustic Haplocalcids; Ustic Torrifluvents	Chilicotal, Reagan, Potrillo, Pandale; Nillo

[†] Numbers correspond to legend shown in Fig. 4.1

[‡] Subgroup level classification (Soil Survey Staff, 1993).

Both clay and $CaCO_3$ can physically protect the organic matter from extraction (Stevenson, 1994). While physical protection of SOC from extraction was likely in our study, we have reason to believe that the dominant exchangeable cation(s) may also influence organic carbon extractability. In comparing the solubility of a reference humic acid (Sigma-Aldrich, Switzerland) in NaOH (0.05N) and saturated $Ca(OH)_2$ (~0.025M or 0.05N OH), we found that the absorbance of the NaOH extract was at least an order of magnitude higher than that of the $Ca(OH)_2$ extract. Since equivalent OH^- concentration and subsequently the pH of both solutions were similar (pH > 12), we believe the primary reason for the difference in absorbance observed was due to differences in interaction of Ca^{2+} and Na^+ with the humic acid. We suspect that calcium forms an organo-calcium complex with the humic acid that is more flocculated, and hence less prone to dissolution, than an organo-sodium complex, which is likely to be more dispersed.

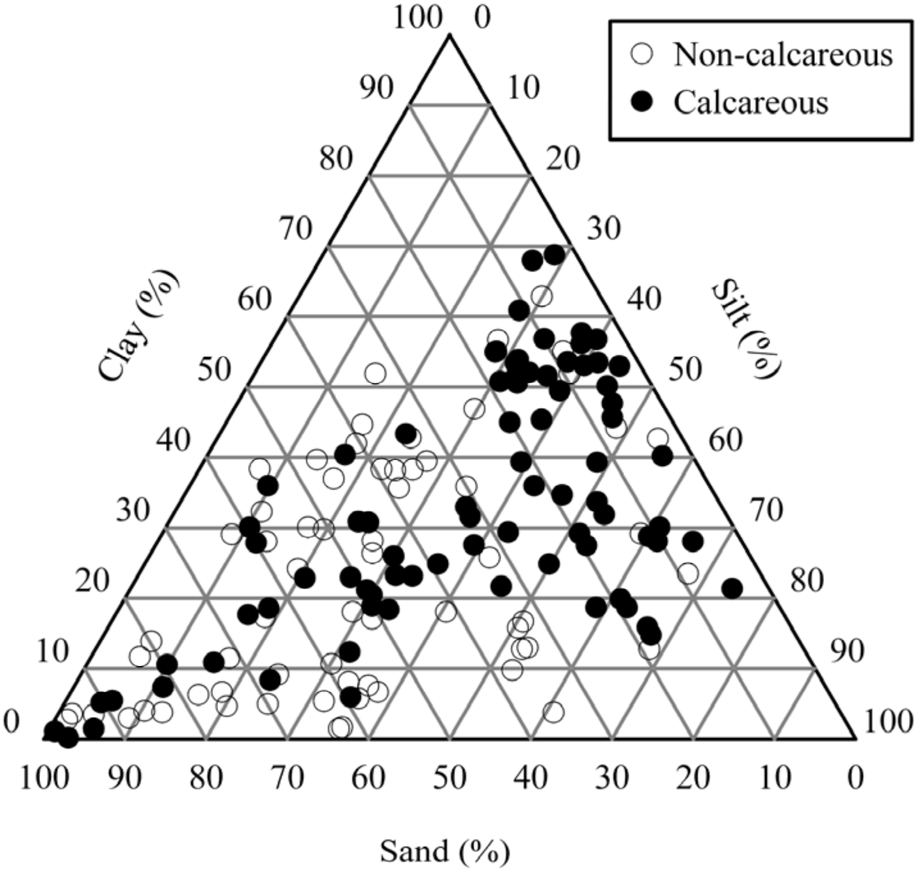


Figure 4.2 Particle Size Distribution of Soils Used Shows a Broad Range in Texture.

Table 4.2 Summary of Selected Soil Chemical Properties for Soil Samples Used (n=146).

	pH[†]	CEC[‡]	SOC[§]	$CaCO_3$[¶]	$CaMg(CO_3)_2$[¶]	Base saturation
		$cmol.kg^{-1}$	----------------------------	%	-------------------------	
Min	3.6	1.4	0.0	0.1	0.1	4
Max	9.0	61.6	4.6	79.7	15.2	100
Mean (sd)	6.8(1.5)	21.0(15.6)	0.8(0.9)	21.7(22.8)	1.5(1.9)	81(29)

[†] determined in 1: 1 soil: water mixtures
[‡] cation exchange capacity
[§] dry combustion soil organic carbon
[¶] for calcareous samples only, n= 82.

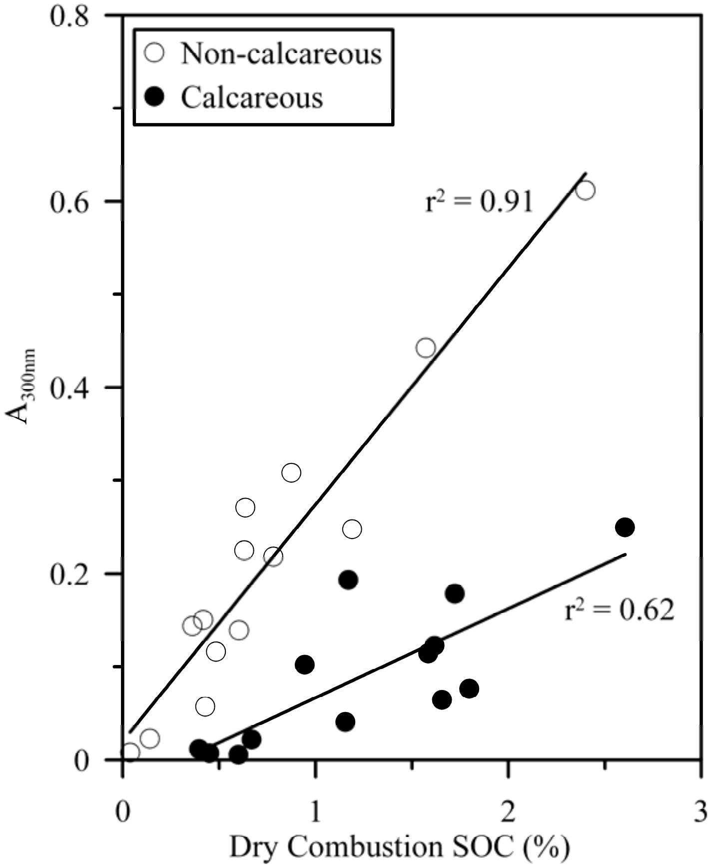


Figure 4.3. Correlation between Extract Absorbance at 300 nm (A_{300nm}) and Dry Combustion Determined SOC for Selected Soils Using 1 N NaOH as an Extractant.

Bowman et al. (1991) found that using a mixture of potassium hydroxide (KOH) and EDTA provided more efficient extraction of SOC from calcareous soils, in comparison to $NaHCO_3$ or KOH only. In our evaluations using a 0.25 N NaOH-0.05M EDTA extractant mixture and 13 calcareous soils with SOC ranging from 0 - 2.4 percent, both extract adsorbance and its correlation with SOC increased (r^2= 0.82) compared to NaOH extracts. We, however, observed that the absorbance-SOC relationship was only linear up to 1 percent SOC with an intercept that was 2.5 times that of the absorbance-SOC relationship of $Na_4P_2O_7.10H_20$ extracts. These observations suggested that the NaOH-EDTA extractant was inefficient at high SOC contents and was likely to overestimate SOC, particularly for lower SOC-containing soils. These findings are consistent with those of Bowman et al. (1991) who found that for high organic matter, high clay content soils heating at 85°C for 2 hours was needed for efficient extraction of SOC. They also noted that the formation of EDTA-Fe^{3+} complexes were likely to be a major interferent in estimating SOC using the KOH-EDTA extractant. Both the temperature and interferent effects are likely to have affected our results since our experiments were conducted at room temperature, and Fe^{3+} is usually abundant in most soils.

The best extraction results were obtained using the HCl + NaOH-pyroP extractant at a soil:extractant ratio of 1:300. In addition to having intercepts close to zero, A_{300nm}-SOC relationships were linear over the full SOC evaluation range (0-2.5 percent SOC) with statistically similar slopes and intercepts (α = 0.05) for both non-calcareous (r^2 = 0.89) and calcareous soils (r^2 = 0.91). Therefore, a common equation could be used to describe the A_{300nm}-SOC relationship for both soil types suggesting that the HCl + NaOH-pyroP extractant was equally efficient at extracting ultraviolet (UV)-active SOC from both classes of soils. This was consistent with analysis of covariance results using all 146 soil samples, which showed no significant influence on A_{300nm} whether soils were calcareous or not (Table 4.3). Table 4.3 also shows no influence of MLRA or soil textural class on A_{300nm}, thereby suggesting that neither SOC source nor clay content had any significant impact on extraction efficiency of UV-active SOC when HCl + NaOH-pyroP was used as the extractant. That source of SOC did not significantly influence the A300 nm-SOC relationship and is consistent with recent work by Sierra et al. (2005) which showed that irrespective of source, all organic matter exhibited common fluorescence behavior in the UV region and were attributable to humic components.

Table 4.3. Analysis of Covariance of Effect of SOC, MLRA, Soil Texture, and Whether Soils Were Calcareous or Non-Calcareous on Soil Extract Absorbance.

Source of variation	*df*	*SS*	*MS*	*F-value*	*P > F*
SOC	1	0.527	0.527	25.326	<0.001
MLRA	10	0.115	0.012	0.553	0.848
Calcareous	1	0.016	0.016	0.787	0.377
Texture	13	0.143	0.011	0.529	0.901
MLRA * SOC	10	0.171	0.017	0.822	0.608
Calcareous * SOC	1	0.000	0.000	0.007	0.935
Texture * SOC	13	0.302	0.023	1.116	0.356
Error	96	1.997	0.021		
Total	145	30.032			

The ability of the pyrophosphate anion to form insoluble or colorless soluble complexes with metals (Alexandrova, 1960) was important in enhancing SOC extractability and removing interference effects. We believe the initial addition of excess HCl, while important for dissolving $CaCO_3$, was also critical in disrupting soil aggregation and clay-organic interactions resulting in increasing soil dispersion. Although the pH of the NaOH-pyrophosphate extracts were on average lower (11.6 ±0.3) compared to NaOH (12.7 ±0.02) and NaOH-EDTA (12.6 ±0.01) extracts, UV-Vis adsorbance spectra of soil extracts were similar irrespective of extractant used. In addition to suggesting no apparent effect of extractant on the UV-Vis active SOC fraction, such finding was indicative of the extractants targeting similar SOC fractions. Comparison of the UV-Vis spectra of selected soil extracts to that of a reference humic acid indicated that humic substances were the primary UV-Vis active SOC fraction present in the soil extracts (Figure 4.4). By normalizing the soil extract's spectra to that of the reference humic acid, we found that spectra were most similar around 290-320 nm (Figure 4.4). Although we chose 300 nm for our analyses, other wavelengths within the 290-320 nm range are likely to produce similar results.

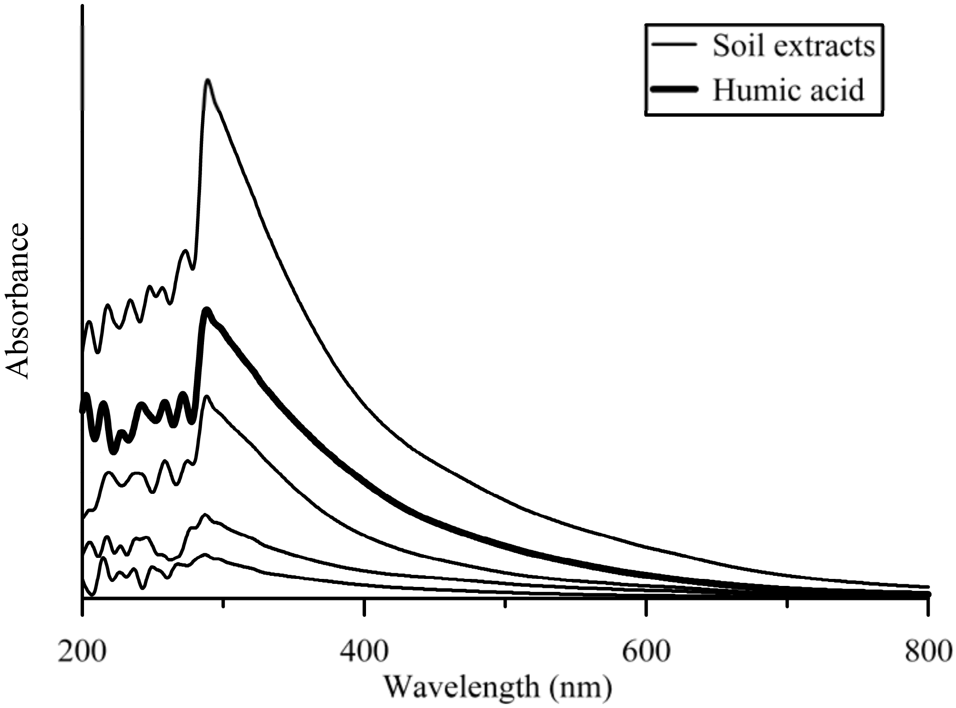


Figure 4.4. UV-Vis Spectra of Soil Extracts and Reference Humic Acid.

Soil Organic Carbon Extraction and Analysis

Initial experiments geared toward identifying a suitable extractant, soil: solution extraction ratio, and wavelength for analysis were made using 28 soil samples (13 calcareous, 15 non-calcareous) with SOC contents ranging from 0 to 2.5 percent. In these experiments, we evaluated solid: solution ratios ranging from 1: 20 to 1: 300, three basic extractants, and UV-Vis spectra of selected soil extracts. The three basic extractants evaluated were NaOH, NaOH-EDTA (Bowman et al., 1991), and NaOH- $Na_4P_2O_7.10H_20$ (Schnitzer, 1982).

Based on initial experimental results, which will be discussed later, a modified approach to that described by Schnitzer (1982) was adopted for organic carbon extraction, soil: solution extraction ratio of 1: 250, and a wavelength of 300 nm for organic carbon estimation. Modifications were made to the Schnitzer (1982) procedure to make it less time consuming, simpler, and more field amendable. Modifications include: (1) the use of disposable syringe filters rather than centrifugation for separation; (2) hand shaking for 10 minutes instead of mechanical shaking for 24 hours; and (3) the elimination of the washing and air-drying step following the addition of HCl.

The newly proposed extraction and analysis procedure for determining SOC follows:
(1) Weigh 0.1g of soil into a 50 ml polypropylene centrifuge tube and add 5 ml of 1 N HCl.
(2) Shake by hand at 1-minute intervals for 5 minutes.
(3) Add 20 ml of extractant solution (0.25 N NaOH- 0.1 M $Na_4P_2O_7.10H_20$), and shake by hand at 1-minute intervals for an additional 5 minutes.
(4) Filter approximately 10 ml of extract through a 0.45 μm polycarbonate syringe filter.
(5) Measure absorbance of the filtrate relative to a reagent blank at a wavelength of 300 nm in the UV-Vis spectrophotometer (Figure 4.5).
(6) Use the calibration curve to estimate SOC content based on measured absorbance.

Figure 4.5. Portable UV-Vis Spectrophotometer Used to Estimate SOC in Soil.

Method Performance, Applicability, and Limitations

Figure 4.6 shows the correlation between absorbance of soil extracts (obtained using the proposed method) and SOC for the calibration samples (n = 73). When the resulting calibration equation in Figure 4.6 was used to predict SOC in the remaining 73 validation samples, predicted SOC were in close agreement with dry combustion determined SOC (Figure 4.7). Standard error and relative prediction deviation associated with predicted SOC (compared to dry combustion SOC) was 0.29 percent SOC and 3.2, respectively, indicating that in addition to being fairly accurate, the proposed method was also stable.

Based on Figure 4.7 we propose the use of the equation:

$$\text{SOC } (\%) = 2\ (\text{Absorbance}_{\text{soil extract}}) \qquad [3]$$

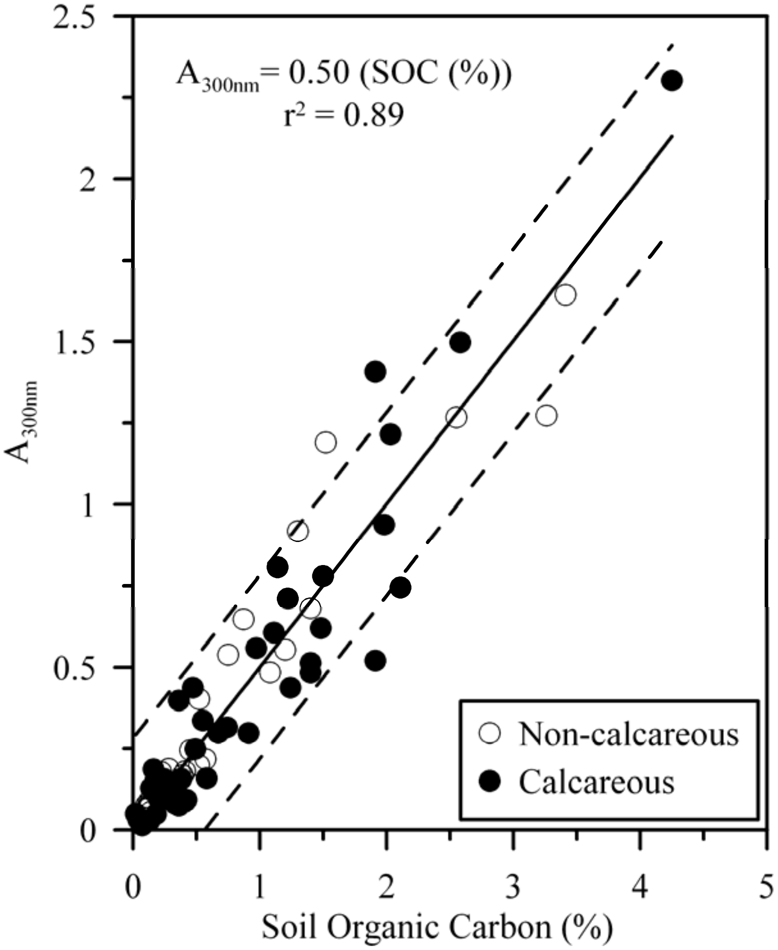


Figure 4.6. Relationship between Extract Absorbance and SOC for Calibration Samples. ([n = 73] using proposed extraction approach, envelope [----] shows 95 percent confidence interval).

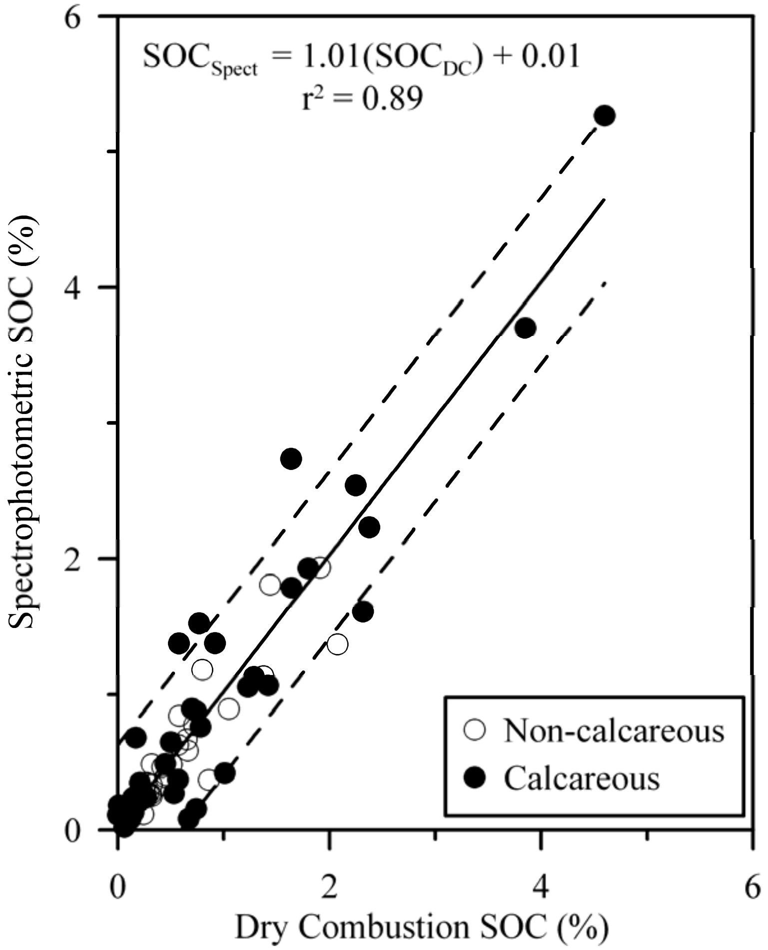


Figure 4.7. Comparison of SOC Measured by Dry Combustion and That Estimated from Extract Absorbance. (Using the equation in [Figure 4.6] for 73 samples, not used in calibration, envelope [----] shows 95 percent confidence interval).

Further evidence of method stability was seen from comparing the effect of calibration sample size on calibration and validation regression parameters. For a calibration sample size ranging from 10 percent (1: 9 calibration: validation split) to 90 percent (9: 1 calibration: validation split) of the 146 samples used in this study, ε and r2 for calibration equations were similar (Table 4.4). Table 4.4 also showed that irrespective of calibration sample size, accuracy of the proposed method in determining SOC was similar, indicating that by using a single calibration equation developed using quantitatively determined SOC and soil extract absorbance from as few as 15 samples, SOC in a large number of soils could be determined. As with any other analytical techniques, researchers recommend that SOC in the calibration and prediction datasets cover a similar range. Results shown in Table 4.4 indicate that the use of equation 3, with soil extracts obtained using the proposed method, was suitable for estimating SOC in the soils used in this study. Since the method was not evaluated with samples containing >5 percent SOC contents, we would recommend developing a separate calibration for soils with higher SOC.

Table 4.4. Mean and Standard Deviation for Method Calibration and Validation Regression Parameters When Data Set (*n = 146*) Was Split Randomly into Calibration and Validation Sets of Different Sizes. Values were based on 63 independent randomizations for each calibration: validation split.

	Calibration			Validation				
C: V	εl	R^2		Slope	Intercept	R^2	SE_p	RPD
1:9	0.52(0.05)	0.87(0.09)		0.97(0.10)	0.02(0.02)	0.89(0.01)	0.29(0.03)	3.0(0.3)
1:3	0.51(0.03)	0.88(0.06)		0.99(0.07)	0.02(0.02)	0.89(0.02)	0.29(0.02)	2.9(0.3)
1:2	0.52(0.02)	0.89(0.04)		0.96(0.07)	0.02(0.01)	0.89(0.02)	0.29(0.03)	3.0(0.3)
1:1	0.51(0.02)	0.89(0.03)		0.98(0.07)	0.02(0.03)	0.89(0.03)	0.30(0.03)	2.9(0.4)
2:1	0.51(0.01)	0.89(0.02)		0.97(0.09)	0.03(0.03)	0.89(0.04)	0.29(0.04)	3.0(0.6)
3:1	0.51(0.01)	0.89(0.02)		0.97(0.09)	0.03(0.04)	0.87(0.06)	0.30(0.04)	2.9(0.6)
9:1	0.51(0.01)	0.89(0.01)		0.96(0.16)	0.03(0.09)	0.87(0.12)	0.26(0.08)	3.4(1.4)

CONCLUDING REMARKS

The ease of execution, rapid analysis, and good accuracy of the proposed method makes it an attractive alternative for both field and laboratory use. Although not as accurate as quantitative methods, the proposed method would be suitable for quick analysis of a large number of samples where accuracy to within ±10 percent was not required. With good quality field spectrophotometers now readily available, the proposed method could be easily adopted for field use. Requirements for field use include: substituting weighing of soil samples with using a scoop, and making corrections for soil moisture and temperature effects, if necessary. Given the small required sample size, except in clay soils, soil moisture is not anticipated to significantly affect estimates of SOC, but it is worth mentioning since we used only air-dried samples in this study. Additionally, since our analyses were made at room temperature, it is not known how temperature may affect predictions in the field.

The ratio of humic to non-humic substances present in the organic fraction of the soil is likely to be the major limitation to applicability of the proposed method. The method assumes that humic substances are the dominant organic fraction present in soils and therefore can be used as a proxy for total soil organic carbon. This may be true for most soils since non-humics are usually rapidly biodegraded (Schnitzer, 1982). However, for soils containing freshly deposited organic matter that contains a significant non-humic fraction, the method will likely underestimate soil organic carbon content.

CHAPTER 5

MEASURING THE IMPACT OF ORGANICS ON ENGINEERING AND CHEMICAL PROPERTIES—LAB TESTING

INTRODUCTION

To develop a proper stabilization scheme in organic-rich soils, we need to understand the fundamental properties affecting lime stabilization, be it chemical reactions and/or physical factors. Due to the complex nature of natural soils, the researchers designed a series of experiments with manufactured soils where the components were tightly controlled to aid in monitoring physical and chemical changes. A series of paste and core experiments were carried out using the manufactured soils and a commercial humic acid.

The manufactured soils were composed of 60 percent quartz sand and 40 percent clay. We used monocrystalline quartz sand from the St. Peter formation in Ottawa, Illinois. We looked at two smectitic clays and a kaolinite because these are the clay minerals commonly found in Texas soils. One smectite was a Ca-dominated bentonite from Gonzales, Texas, and the second smectite was a Na-dominated clay from Panther Creek, Colorado. The kaolinite we used was a clay mineral standard from Georgia. We used a humic acid standard purchased from Fisher Scientific to represent organic matter because earlier research reported that the humic acid fraction was the most important in controlling lime/soil reactions (Kuno et al., 1989).

The primary objectives in this task were to; (1) evaluate chemical reactions of lime with manufactured soils bearing different concentrations of organic matter using differential scanning calorimetry; and (2) quantify the effect (on engineering properties) of organic matter content on lime stabilization in soils with different mineralogies.

MATERIALS AND METHODS

To evaluate the chemical reactions between lime and the manufactured soils, we prepared 1:1, solid to solution pastes in 40 ml Nalgene centrifuge tubes. The solutions were cured under conditions of 100 percent humidity and 23^{o}C for 10 days. The compositions of the pastes are shown in Table 5.1. Researchers prepared samples by mixing 60 percent by weight of quartz sand and 40 percent of either kaolinite, Ca-smectite, or Na-smectite. Zero percent hydrated lime was added to control samples, and 6 weight percent lime was added to others; humic acid was added to all combinations in concentrations ranging from zero to 5 weight percent. An equivalent mass of de-ionized water was added to all samples. Each combination was produced in triplicate for a total of 90 samples. Following curing, the pastes (Figure 5.1) were flash frozen (using liquid nitrogen), freeze dried, light crushed, and sieved through a 63 micron (#230) sieve. Figure 5.1 also shows how the pastes responded to the lime: the compartment on the far left contained only smectite sand and water; it remained liquid due to no stabilizer being present. The other four compartments contained 6 percent lime, but the organic content ranged (left to right) from 0 percent to 0.5, 2.0, and 5.0 percent humic acid. Note, with increasing humic acid, the samples become less viscous, indicating that lime is not reacting with the soil.

Mineralogical composition of the < 63 micron fraction was assessed using X-ray diffraction (XRD) on a Rigaku Diffractometer and differential scanning calorimetry (DSC) using a SDT Q600 calorimeter. For XRD analysis, samples were mounted as dry powders and scanned over the 2.1 to 60° two-theta range at a speed of 1°/min. For DSC analysis, approximately 20 mg of <63 micron fraction was placed in a ceramic sample crucible and heated from 25 to 1050°C at a ramping rate of 10°C min^{-1}.

Table 5.1. Manufactured Sample Matrix.

Sand (wt.%)	Clay (wt.%)	Lime (wt.%)	Humic Acid (wt.%)
60 Quartz	40 Kaolinite	0	0.0
	40 Ca-Smectite	6	0.5
	40 Na-Smectite		1.0
			2.0
			5.0

Figure 5.1. Smectite Paste Consistency Prior to Flash Freezing. Note, paste with no lime is liquid and with increasing humic acid content, the paste again reaches liquid consistency.

RESULTS AND DISCUSSION

Differential scanning calorimetry data for the Ca-bentonite and hydrated lime starting materials (before they are combined and reacted) are shown in Figure 5.2. Labels show peaks attributable to clay dehydration (Cd), lime dehydration (Ld), lime and clay degradation (Ldeg and Cdeg, respectively), and clay recrystallization (Cr). The clay and lime dehydration peaks are endotherms (absorb heat), and the clay recrystallization peak is an exotherm (gives off heat). Karathanasis (2008) states that phase transitions, dehydration, dehydroxylation, and some decomposition reactions produce endothermic peaks while oxidation, recrystallization, and certain decomposition reactions generate exothermic peaks.

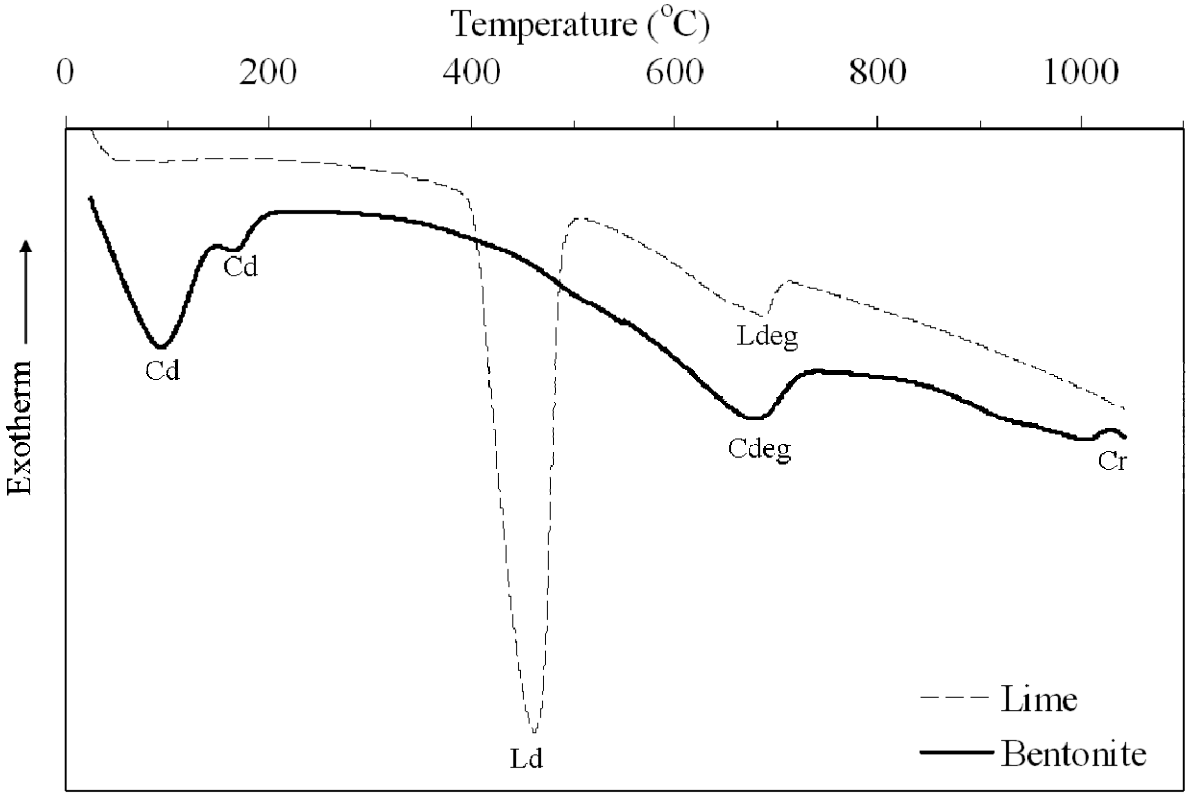


Figure 5.2. Differential Scanning Calorimetry Thermograms for Lime and Bentonite Starting Materials Used in Paste and Core Lime Stabilization Experiments.

Figure 5.3 shows DSC thermograms for manufactured soils cured for 10 days. Several common, as well as distinct, features are apparent from these scans. The endothermic peaks at approximately 100 and 170°C and the exothermic peak at around 1025°C are common to both the Ca-bentonite and the reacted pastes: these peaks are consistent with the dehydration and the recrystallization of the clay, respectively. The endotherm with the minimum occurring around 670°C is common to all DSC scans and is attributable to: (1) the degradation of quick lime (CaO) formed as a result of dehydration of the hydrated lime (peak around 450°C); or (2) the degradation of the Ca-Bentonite. The absence of the hydrated lime peak (450°C) from DSC scans for the pastes suggests consumption of the added hydrated lime during curing, which is consistent with expectations of stabilization.

The most significant differences between DSC scans for starting materials (Figure 5.2) and the reacted pastes were observed in the 250-500°C and the 800-900°C temperature ranges (Figure 5.3). Exothermic peaks at around 350 and 425°C were observed only in samples containing humic acid. As stated by Karathanasis (2008), oxidation reactions produce exothermic peaks. Increase in intensity of these peaks with increasing HA content suggests that the peaks are attributable to HA oxidation. The exothermic peak that occurred around 850°C was observed in the pastes containing 0, 0.5 or 1 percent HA but not in the starting materials or in the

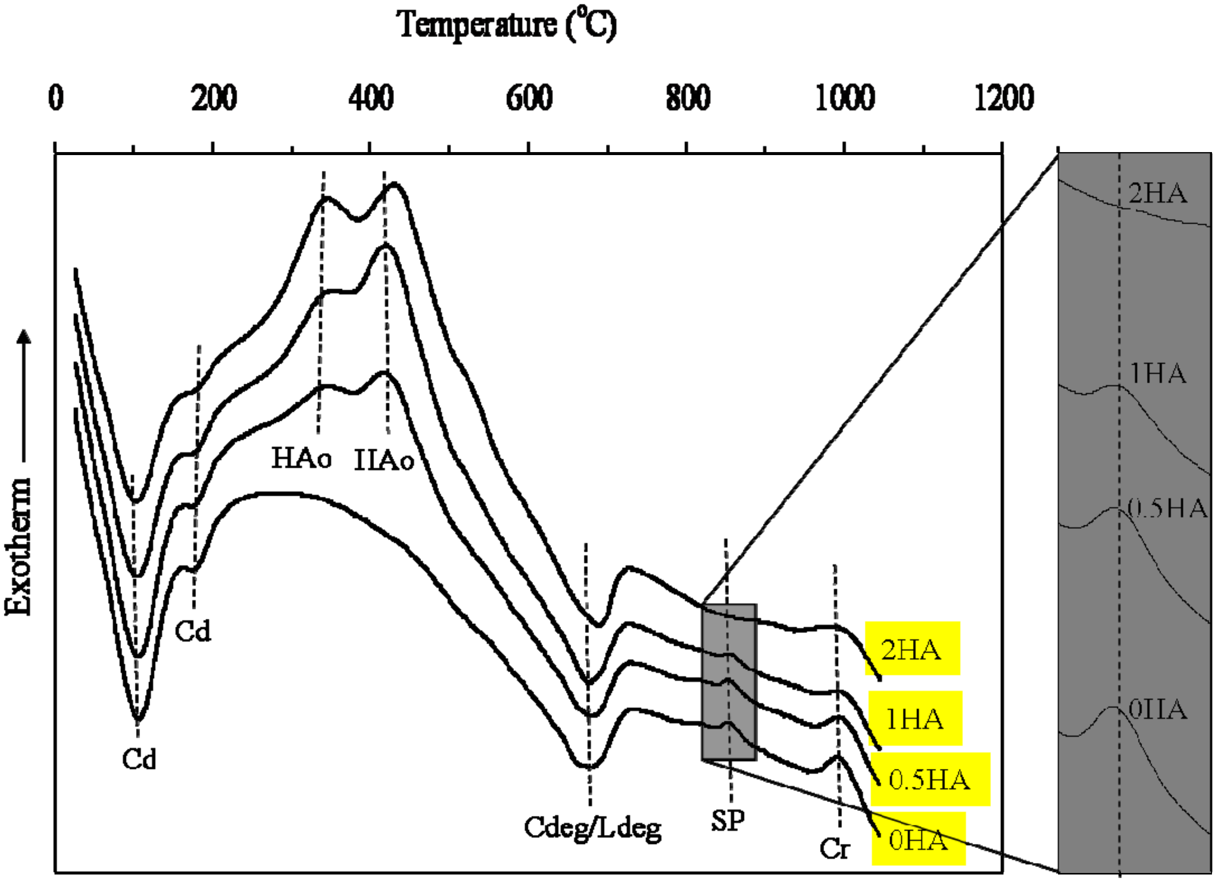


Figure 5.3. Differential Scanning Calorimetry Thermograms of 10 Day Cured 1: 1 Solid: Solution Lime Stabilized Pastes Containing 0, 0.5, 1, and 2 Percent Humic Acid.

2 percent HA paste, which was attributable to the presence of stabilization product. The concomitant decrease in the intensity of the peak (Figure 5.3 inset) with increasing HA content indicates that the presence of HA in the system inhibits the formation of stabilization products. X-ray diffraction patterns (Figure 5.4) show no evidence of a crystalline phase, suggesting that the stabilization product formed is likely amorphous in nature. A comparison of the unstabilized clay (0 HA 0 Lime) to the lime stabilized clay with various concentrations of HA in Figure 5.4 reveal almost identical XRD patterns. The X-ray technique only detects crystalline material; therefore, the XRD patterns shown in Figure 5.4 suggest that the reaction products detected by the DSC (Figure 5.3) are noncrystalline.

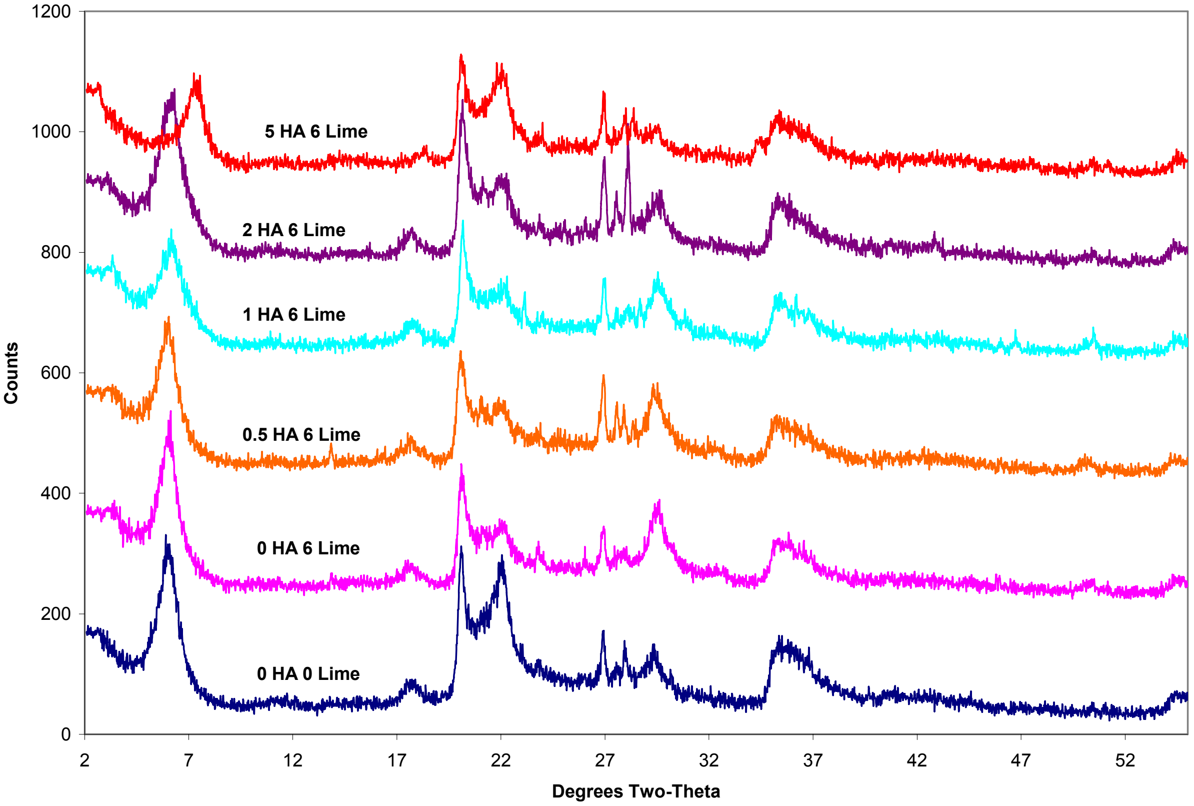


Figure 5.4. X-Ray Diffraction Patterns of Manufactured Soils Treated with Different Concentrations of Humic Acid.

Scanning electron microscopy (SEM) analyses confirm what was observed with the XRD data. The SEM image shown in Figure 5.5A is from the raw smectite with no lime and no humic acid added. It shows crystals with a cornflake morphology, which is typical of smectite. There is no evidence of any reaction products in this sample. The accompanying energy dispersive spectrometer (EDS) pattern (Figure 5.5B) shows the relative elemental abundances of the Ca-smectite. Note the very low calcium (Ca) concentration with respect to silicon (Si) and aluminum (Al) in this smectite that has no lime or humic acid.

Figure 5.6A shows evidence of reaction products formed when lime is added to the smectite. The long acicular crystals are typical of reaction products formed from the reaction of lime with the smectite. This is direct evidence of reaction products being formed in the lime-stabilized sample.

The humic acid treated plus lime-stabilized sample shown in Figure 5.7A shows no evidence of reaction product formed from lime reacting with the smectite. This sample looks very similar to the raw soil; the light-colored areas are humic acid aggregates. Figure 5.7B is a representative EDS pattern for this sample. Note how the calcium (Ca) is higher than the raw soil, but it is much lower than the lime-treated soil with no HA added.

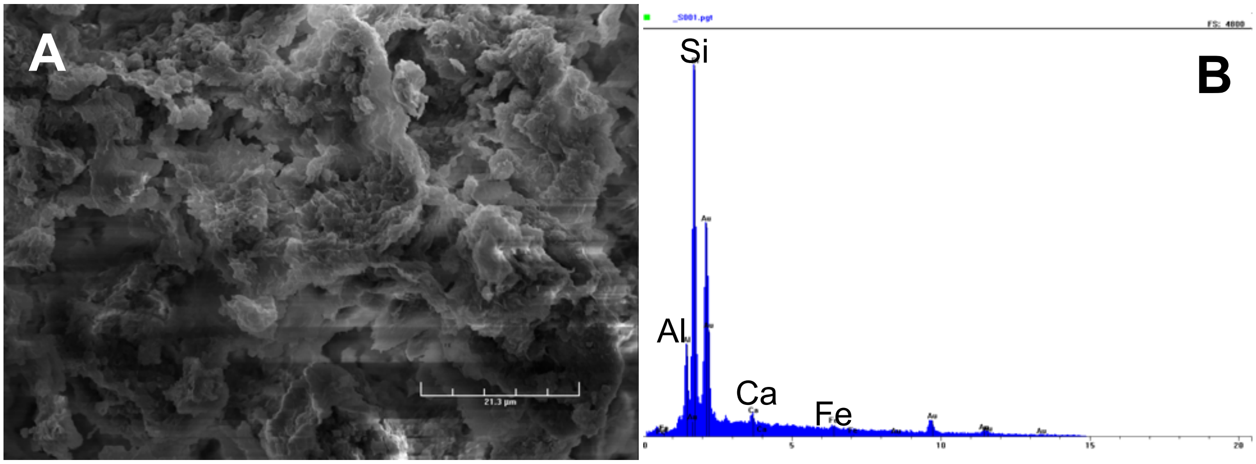


Figure 5.5. A) SEM Image of Untreated Ca-Smectite, B) EDS Pattern of Same Ca-Smectite.

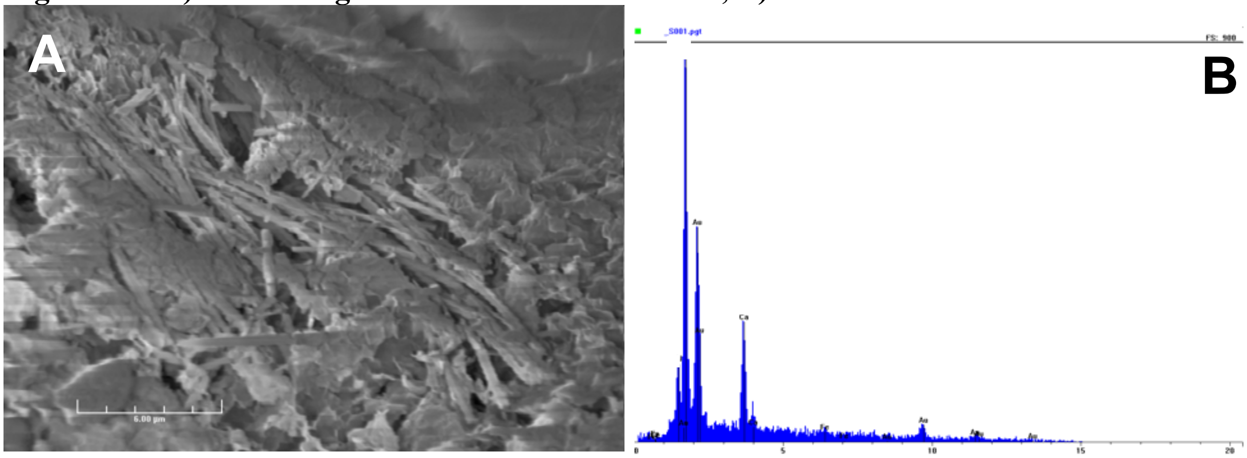


Figure 5.6 A) SEM Image of Lime-Treated Ca-Smectite, B) EDS Pattern of Same Sample.

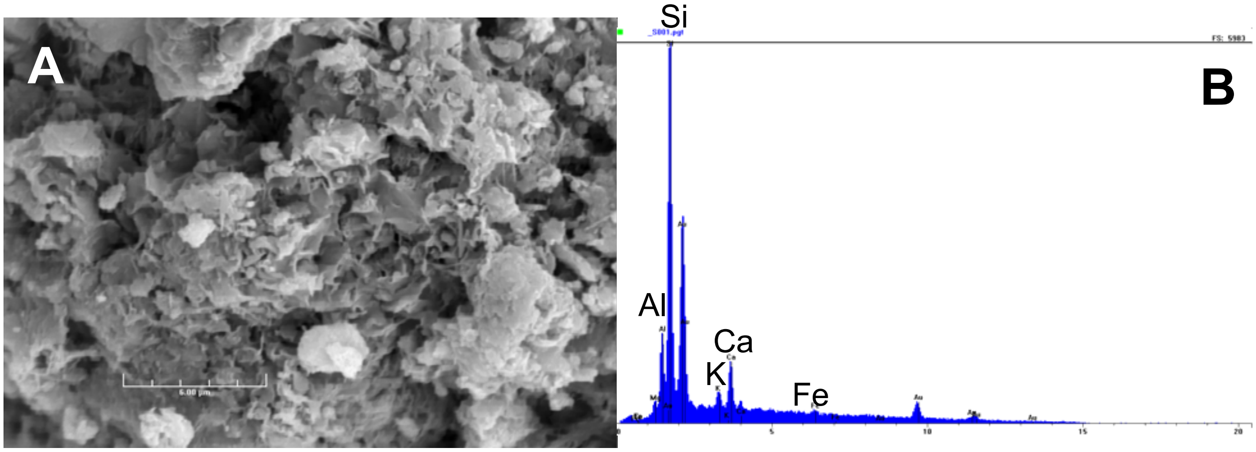


Figure 5.7. A) SEM Image of HA + Lime-Treated Ca-Smectite, B) EDS Pattern of Same Sample.

THE EFFECTS OF ORGANIC MATTER CONTENT ON THE ENGINEERING PROPERTIES OF LIME-STABILIZED SOILS

The first part of this chapter discussed the use of DSC to evaluate the reaction of lime with manufactured soils containing different concentrations of a reference humic acid. The utility of DSC was shown by its ability to detect amorphous phases, which are difficult to detect using other techniques. In this section we will use the DSC technique to evaluate the engineering properties (UCS, optimum lime content) of Harvard Miniature samples (Figure 5.8) using the same sample matrix (Table 5.1) discussed previously.

Figure 5.8. Harvard Miniature Samples of Sand, Smectite, Lime, and Different Contents of HA Ranging from (left to right) 0, 0.5, 2.0, and 5.0 Percent.

Materials and Methods

A series of 2 inch (height) by 1 inch (diameter) cylindrical cores were made using the Harvard miniature apparatus. Each core contained 60 percent sand, 40 percent clay, 6 percent lime, and 0, 0.5, 2, or 5 percent HA and was compacted to a dry bulk density of 1.63 g/cm^3 at a moisture content of 22.5 percent by weight. To simulate different smectitic soil types, cores were made from; a Ca-dominated bentonite (representing a di-valent cation dominated smectite); and a Na-dominated montmorillonite (representing a mono-valent cation dominated smectite). Cores were cured for 3, 7, 10, 14, and 28 days at 100 percent humidity and 23^{o}C. For each curing period, triplicates of cores were made for each of the four HA contents resulting in a total of 36 (4 HA content × 3 clay types × 3 replicates) cores per curing period or 180 total core samples.

Following curing, the unconfined compressive strengths of the cores were measured using an Instron model 5583 load frame. Samples were then dried in an oven overnight at 70^{o}C, lightly crushed, and sieved through a 63 μm sieve. About 40 mg of the less than 63 μm fraction were then analyzed by DSC at a ramping rate of 10°C min^{-1} from 25 to 1050^{o}C.

To evaluate how pH varies with organic content, we manufactured soils with monocrystalline quartz, Ca-dominated bentonite, various concentrations of humic acid, and different concentrations of lime to determine the optimum lime content for soils of uniform mineralogy with varying amounts of organic matter. We then measured the pH following the Eades and Grim procedure.

Results and Discussion

Tables 5.2 and 5.3 report the unconfined compressive strength data for all Harvard Miniature cores. There are two trends evident in these data. First, the calcium smectite samples all attained higher strengths when compared to the sodium smectite samples with equal concentrations of lime and humic acid. Secondly, as the humic acid content increases, there is a precipitous drop in the strengths of the lime-stabilized samples.

Table 5.2. UCS Data for the Ca-Smectite Samples.

	UCS (lb in^{-2})				
	3d	7d	10d	14d	28d
0HA	198 (26)[†]	291 (29)	434 (19)	454 (90)	471 (3)
0.5HA	156 (4)	262 (37)	309 (78)	296 (30)	381 (52)
2HA	107(9)	123 (8)	126 (7)	116 (4)	177 (17)
5HA	98 (2)	95 (2)	88 (14)	91 (6)	71 (6)

[†] numbers in parentheses are standard deviations based on triplicate samples.

Table 5.3. UCS Data for the Na-Smectite Samples.

	UCS (lb in^{-2})				
	3d	7d	10d	14d	28d
0HA	82 (7)[†]	108(8)	115(2)	118(1)	105(9)
0.5HA	79 (7)	80(10)	83(7)	94(19)	78(15)
2HA	67(1)	70(10)	62(4)	63(10)	56(3)
5HA	67 (2)	58(3)	56(5)	62(2)	52(2)

[†] numbers in parentheses are standard deviations based on triplicate samples.

A graphical representation of UCS as a function of the amount of HA added, is shown in Figures 5.9 and 5.10. Figure 5.9 shows data from both the Ca-dominated (filled symbols) and Na-dominated (open symbols) smectite. Note that for all curing times up to 28 days moist cure, UCS drops as more humic acid is added to the manufactured soil. The portion of the graph shaded in gray shows the limit of stabilization product formation as observed in the DSC thermograms. Figure 5.10 is an exploded view of the Na-dominated smectite samples that are shown in the lower portion of Figure 5.9.

The data indicated that: (1) For a given curing time, UCS decreased with increasing amounts of HA added; and (2) UCS were considerably higher for the Ca-bentonite cores than for Na-bentonite cores.

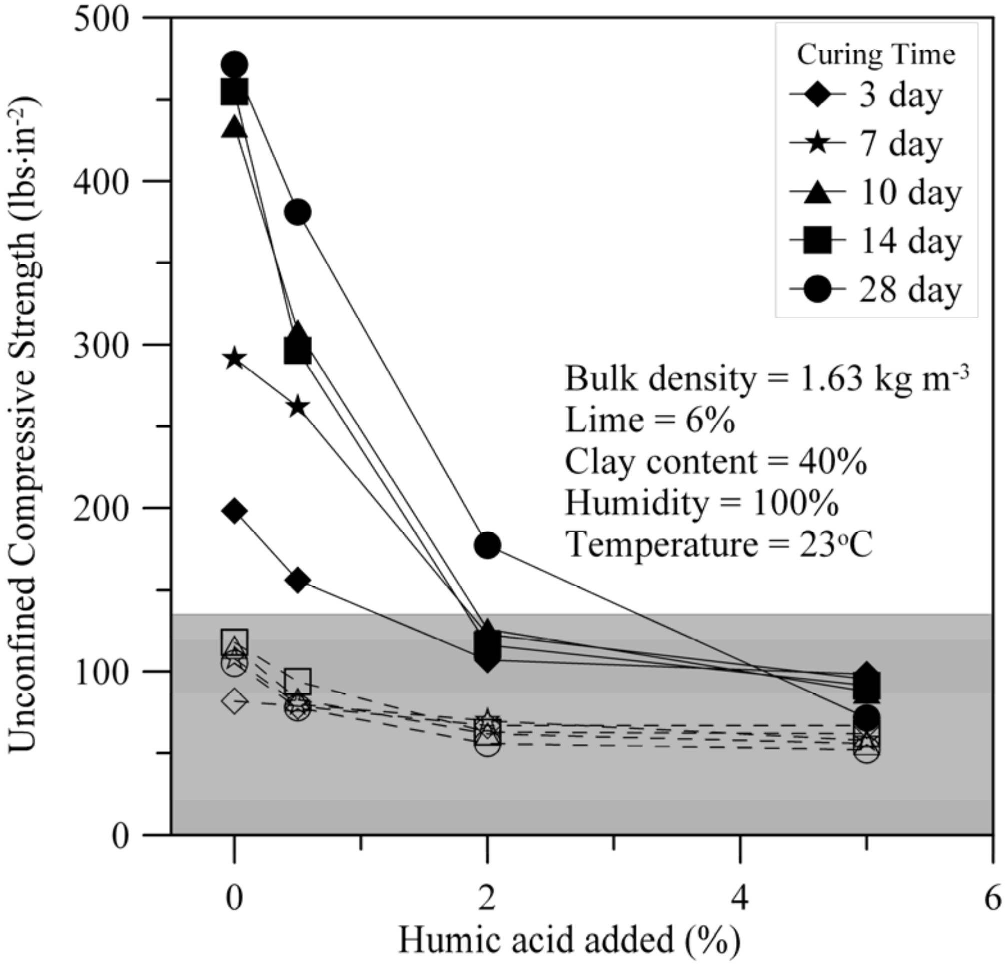


Figure 5.9. Post-Curing Unconfined Compressive Strengths for Lime-Stabilized Harvard Miniature Cores, Containing Varying Amounts of Humic Acid.

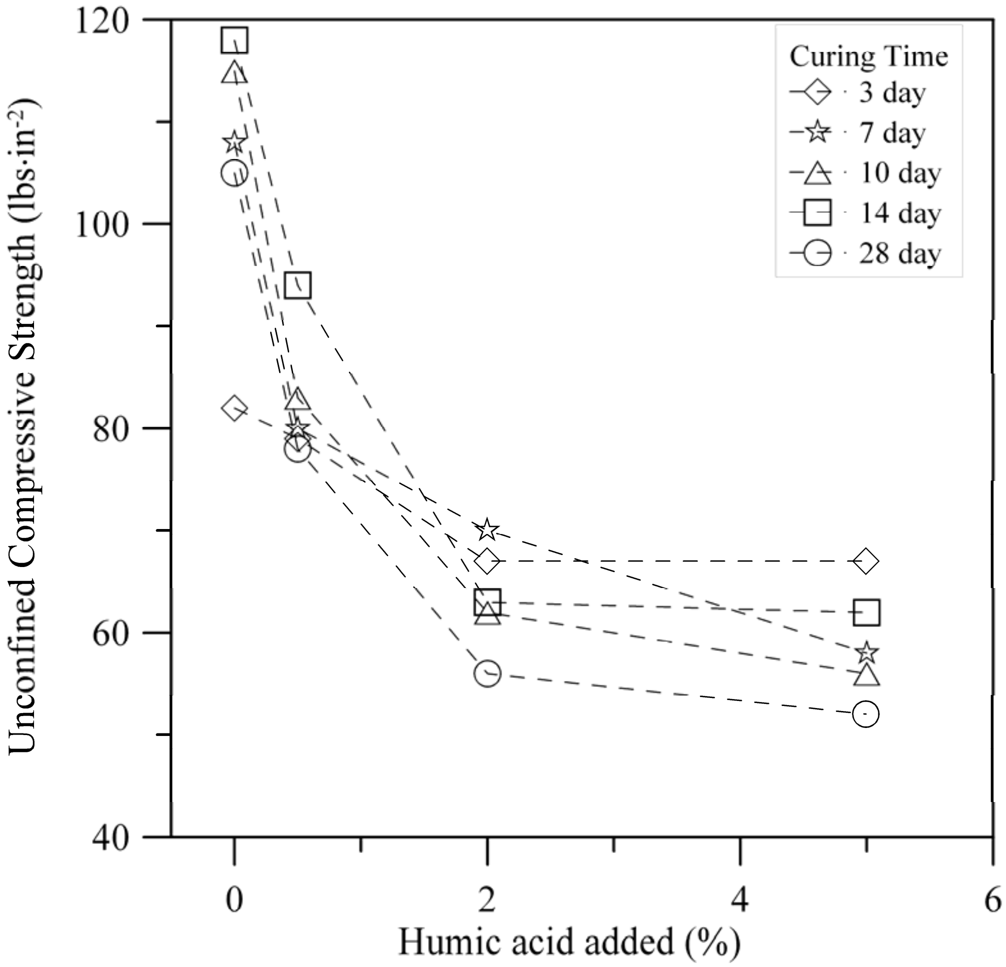


Figure 5.10. UCS Data of the Na-Dominated Smectite Samples Show Dramatically Lower Strengths.

The decrease in UCS with increasing HA content that we observed is consistent with DSC results from paste experiments, which show a concomitant decrease in the intensity of the exothermic peak at 850^{o}C with increasing HA content. These results suggest a direct relationship between the exothermic peak at 850^{o}C and the degree of stabilization in the cores. Further evidence of this relationship is seen in Figure 5.11, which shows a strong correlation between the UCS and DSC peak area for samples in which the exotherm was observed. The regression equation in Figure 5.11 suggests that for experimental conditions used in the study, the peak would only be present in cores with strengths in excess of 133 lbs in^{-2}. This hypothesis was supported by the data. In addition to being of significantly lower strengths than samples that showed the exotherm at 850^{o}C, samples in which the exotherm did not occur had UCS values that were all lower than 133 lbs in^{-2}.

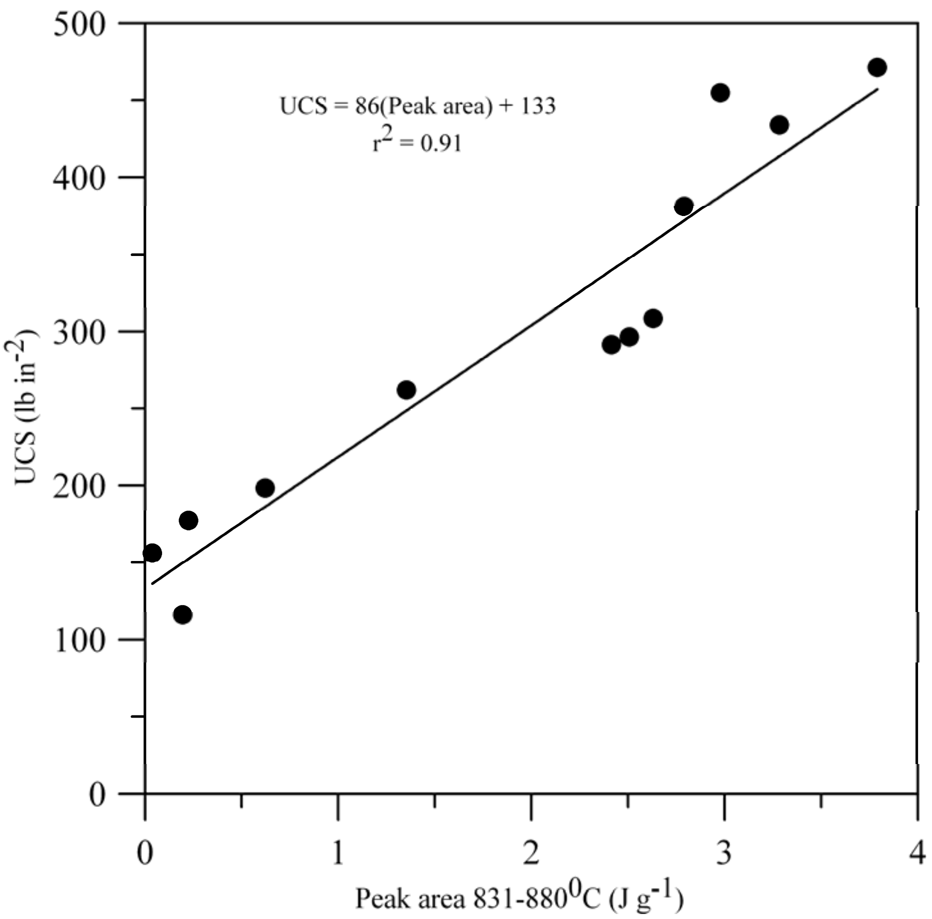


Figure 5.11. Relationship between DSC Peak Area and UCS for Lime-Stabilized Cores Showing Evidence of Stabilization Product Formation, Based on DSC Thermograms.

Despite being of similar compositions and exhibiting similar general trends in response to increasing HA content, the Ca-dominated and Na-dominated smectite respond differently to lime stabilization. Firstly, irrespective of HA content or curing time, cores made of Na-dominated smectite have significantly lower strengths than those made of the Ca-dominated smectite (Tables 5.2 and 5.3; Figure 5.9).

Secondly, compared to the Ca-dominated smectite, improvement in strength over the curing period is much lower and appears to be short-lived for the Na-dominated smectite (Figure 5.12). Figure 5.12 was constructed by normalizing the UCS of all curing times with respect to the UCS of the 3-day cured specimens for both the calcium and sodium dominated smectites. The short-term nature of the strength gain in the Na-dominated smectite is evidenced by the initial increase in strengths over the first 14 days of curing, followed by a decrease in strength for the 28-day samples (0 and 0.5 percent HA cores). The data in Figure 5.12 also suggest that HA has a greater negative impact on stabilization in the Na-smectite than in the Ca-smectite. For example, only the 5 percent HA Ca-smectite cores show strengths that are progressively lower over time than their respective 3-day cores, compared to the 2 and 5 percent HA cores for the Na-smectite.

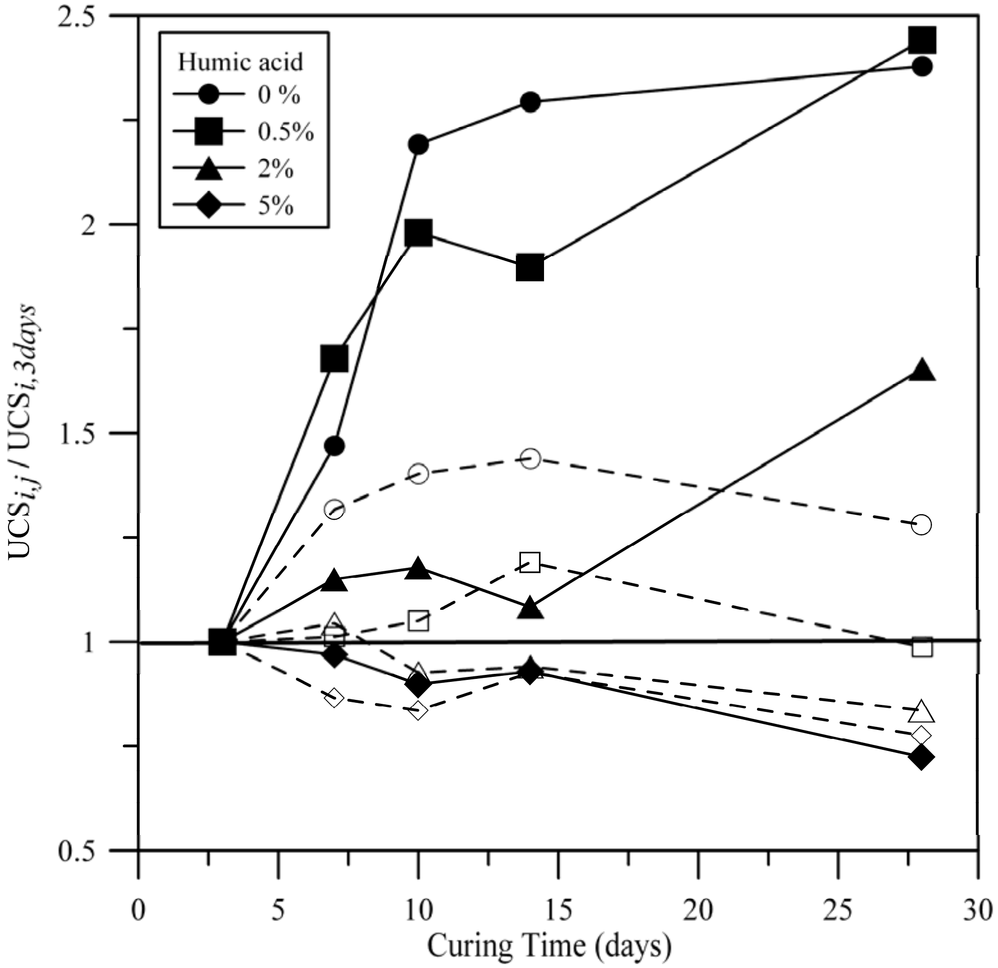


Figure 5.12. Temporal Changes in Unconfined Compressive Strengths for Lime-Stabilized Ca-Dominated (filled symbols) and Na-Dominated Smectite (open symbols) Cores.

We followed the Eades and Grim (1966) procedure to measure the optimum lime content of our manufactured calcium smectite. Table 5.4 shows that the amount of humic acid in the manufactured soil does not appear to have an effect on the lime requirement of the soil. Note how the pH for 5 percent lime is the same for 0 percent HA all the way to 5 percent HA. These measurements were taken after 1 hour of shaking. We also measured the pH after 24 hours and obtained essentially the same results. At present, we do not have an explanation for these results.

Table 5.4. Optimum Lime Content with Varying Amounts of Humic Acid.

	pH of 1 Hour Eades & Grim Samples				
	0% HA	0.5% HA	1% HA	2% HA	5% HA
2% Lime	12.56				
3% Lime	12.6		12.61		
4% Lime	12.58	12.62	12.66		
5% Lime	12.66	12.69	12.7	12.7	12.69
6% Lime	12.68	12.65	12.71	12.72	12.73
7% Lime		12.7	12.74	12.63	12.74
8% Lime		12.6		12.75	12.76
9% Lime				12.71	12.79

CONCLUSIONS

Analysis of the data from this task indicates that:

- DSC provides a useful tool for evaluating the degree of lime stabilization in soils, as well as the potential impact of soil organic matter content on the stabilization process.
- Organic matter content over 1 percent (in the form of humic acid) influences lime stabilization of soils by inhibiting the formation of stabilization products. Negative impacts on lime stabilization increase with organic matter content.
- The type of clay present (Na-smectite, Ca-smectite, kaolinite) in the soil will influence the degree of stabilization and the impact of organic matter content on the stabilization process. Calcareous soils (pH≥8) will respond better to lime stabilization than acid soils with equal amounts of OM.
- Humic acid contents higher than 1 percent will prevent or retard formation of the pozzolanic reaction products responsible for long-term strength gain.
- The pH as measured in the Eades and Grim procedure apparently is not affected with increasing humic acid concentrations.

CHAPTER 6

FIELD EVALUATIONS OF TXDOT PROJECTS

INTRODUCTION

Task 5 is dedicated to evaluation of field sites with high organic contents to see the effects of high percentages of SOM on engineering properties like plasticity, optimum moisture/density curves, optimum lime and cement contents, and unconfined compressive strengths.

Initially, we will give the results for measurement of organics using different techniques. These results will be followed by the engineering properties that were measured at the University of Texas at Arlington (UTA) (Anand Puppala's group).

ORGANICS MEASUREMENT

The researchers measured the organic matter present in the field sites using various techniques (Table 6.1). The C-H-N technique is generally accepted as the standard for all other techniques. Note how the ignition oven results are all much higher than the results obtained with the C-H-N. The UV-Vis results are all lower than those obtained with the C-H-N. That is because the UV-Vis measurements were made up to one year after the researchers initially collected the samples. SOM can be reduced by microbial action if samples are not stored under completely dry conditions.

Table 6.1. Organic Contents for the Field Sites Measured with Different Techniques.

District	City	Location	CHN % OM	*UV-Vis % OM	Ign. Ovn. TTI % OM	Ign. Ovn. UTA % OM
Abilene	Abilene	Abilene		0.5	3.9	4.4
Austin	Austin	Parmer lane	1.4	1.0	3.3	4.8
Beaumont	Beaumont	Beaumont	1.5	0.7	3.6	4.6
Corpus Christi	Corpus Christi	FM 665		0.8	3.5	3.7
		IH 37		0.1	1.7	1.5
Bryan	Huntsvillie	(I 45) Trench 1 (Top)	0.7	0.3		6.1
		(I 45) Trench 3 (Top)	1.6	0.5	3.8	3.7
		(I 45) Trench 3 (Bottom)	0.7		2.0	4.4
		(I 45) Trench 4 (Top)	1.4	0.3	3.6	4.4
	College Station	SH 6, East Side			1.5	2.1
		SH 6, Northbound			2.2	2.2
		SH 6, Southbound		0.2	1.5	2.3

** UV-Vis measurements were made on many samples after prolonged storage.*

MATERIALS (UTA)

We obtained soil samples from 12 sites in six different districts (Table 6.2). The districts include: Abilene, Austin (Parmer Lane), Beaumont, Bryan (Huntsville and College Station), and Corpus Christi. These soil samples are given sample notations as mentioned in Table 6.2. Here after, soil samples are denoted by these sample notations.

Table 6.2. Site Description and Sample Notation.

District, Name of the Site	**Sample Notation**
Abilene	ABL
Austin (Parmer Lane)	AUSPL
Beaumont	BMT
Corpus Christi, (IH 37)	CCI37
Corpus Christi, (FM 665)	CCF665
Bryan, Huntsville, Trench 1 Top (I 45)	BI45T1T
Bryan, Huntsville, Trench 3 Top (I 45)	BI45T3T
Bryan, Huntsville, Trench 3 Bottom (I 45)	BI45T3B
Bryan, Huntsville, Trench 4 Top (I 45)	BI45T4T
Bryan, College Station, Eastside (I 45)	BS6E
Bryan, College Station, Northbound (SH 6)	BS6N
Bryan, College Station, Southbound (SH 6)	BS6S

BASIC PROPERTIES TESTS (UTA)

Tests to measure basic properties of all the soil samples collected include: specific gravity, sieve analysis, hydrometer analysis, Atterberg limits, organic content, and standard proctor tests. These tests were done to characterize the soils prior to the stabilization tests with lime and cement. Atterberg limits, organic content, and standard proctor tests are also made on both lime- and cement-stabilized soils. The total quantity of natural soil obtained from the above-mentioned sites was limited; therefore, compaction and strength measurements were made on Harvard miniature samples.

Researchers performed the particle size analysis according to the Tex–101-E (Method B). The distribution of the grain sizes in test materials finer than 0.0075 mm was determined using Tex-110-E. Finer particle size analysis was performed using hydrometer analyses.

Atterberg limits were determined based on Tex-104-E to determine the liquid limit and Tex-105-E to determine the plastic limit. Specific gravity, which is defined as the ratio of unit weight of soil to unit weight of water, of present test materials was determined as per TxDOT procedure Tex-108-E. The pH of present soils was also determined by following the Tex-128-E procedure.

Organic contents of soils were determined by following the ASTM D-2974-87 procedure. This is an ignition oven procedure.

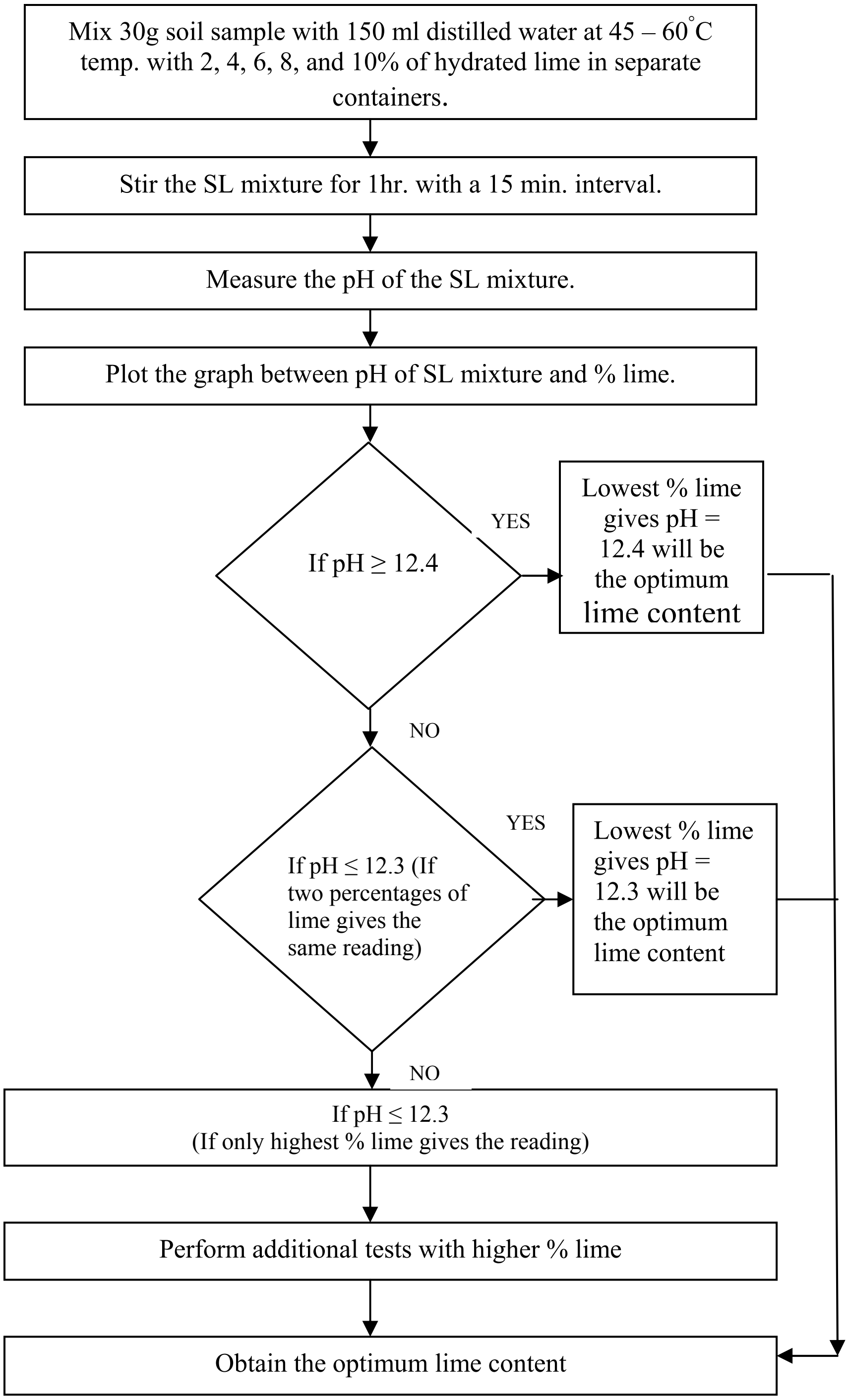
To determine the optimum moisture content and maximum dry unit weight of the soils, researchers prepared Harvard miniature proctor compaction samples. Compaction tests were made on both natural soil and stabilized soil samples to determine moisture content and dry unit weight relationships. We followed the ASTM D 4609 procedure to determine moisture content vs. dry density relationships.

We measured unconfined compressive strength in accordance with the TxDOT procedures. To confirm the reproducibility for each test, duplicate specimens were tested and analyzed.

We determined the optimum lime content for soils following the procedure given by Eades and Grim (1966). The procedure is explained in the form of a flowchart shown in Figure 6.1.

The researchers measured the optimum cement content for soils following the procedure outlined in Tex-120-E. Initially, we determined the optimum moisture content and maximum density for a soil-cement mixture containing 6 percent cement in accordance with Tex-113-E. We recombined the sizes prepared in accordance with Tex-101-E, Part II to make three individual samples and added the optimum moisture content determined from Tex-113-E to each sample.

Subsequently, we measured the unconfined compressive strength and plotted the results between the unconfined compressive strength and percentage of cement added. Optimum cement content is chosen as the percentage that gives an unconfined compressive strength of 150psi or 1035kPa. A typical graph used for the calculation of optimum cement content is shown in Figure 6.2.



Note: SL – Soil Lime

Figure 6.1. Optimum Lime Content for Stabilization of Soil (Eades and Grim Procedure).

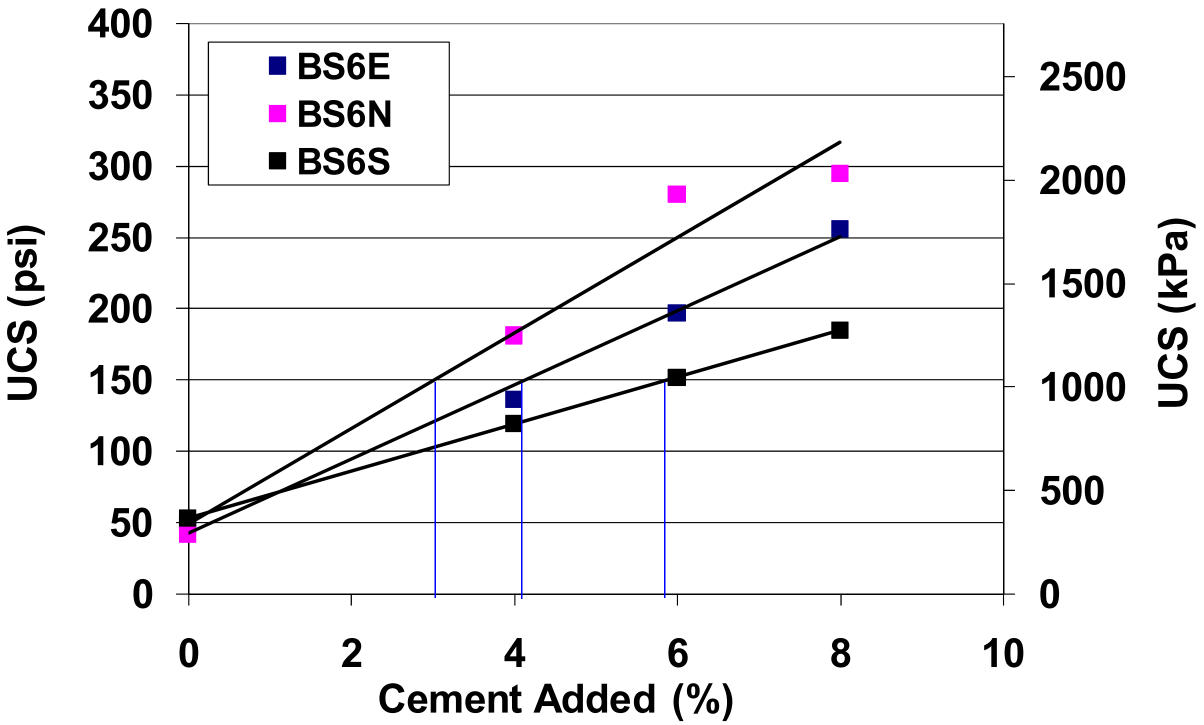


Figure 6.2. UCS versus Percent Cement Added.

RESULTS

Here we present a comprehensive analysis of both basic and engineering tests of soils evaluated in this research. The analysis evaluates the potentials of lime and cement stabilizers to provide enhancements to organic soil properties. We'll also explain the effectiveness of each stabilizer and its influence on strength at 7, 28, and 56 days curing periods and the plasticity index on organic rich soils as well as on the control soil. In this chapter, results are presented in two different sections; the first section describes the basic properties of control soils while the second section deals with the analysis of engineering properties of control and stabilized soils. They are explained in the flowchart shown in Figure 6.3.

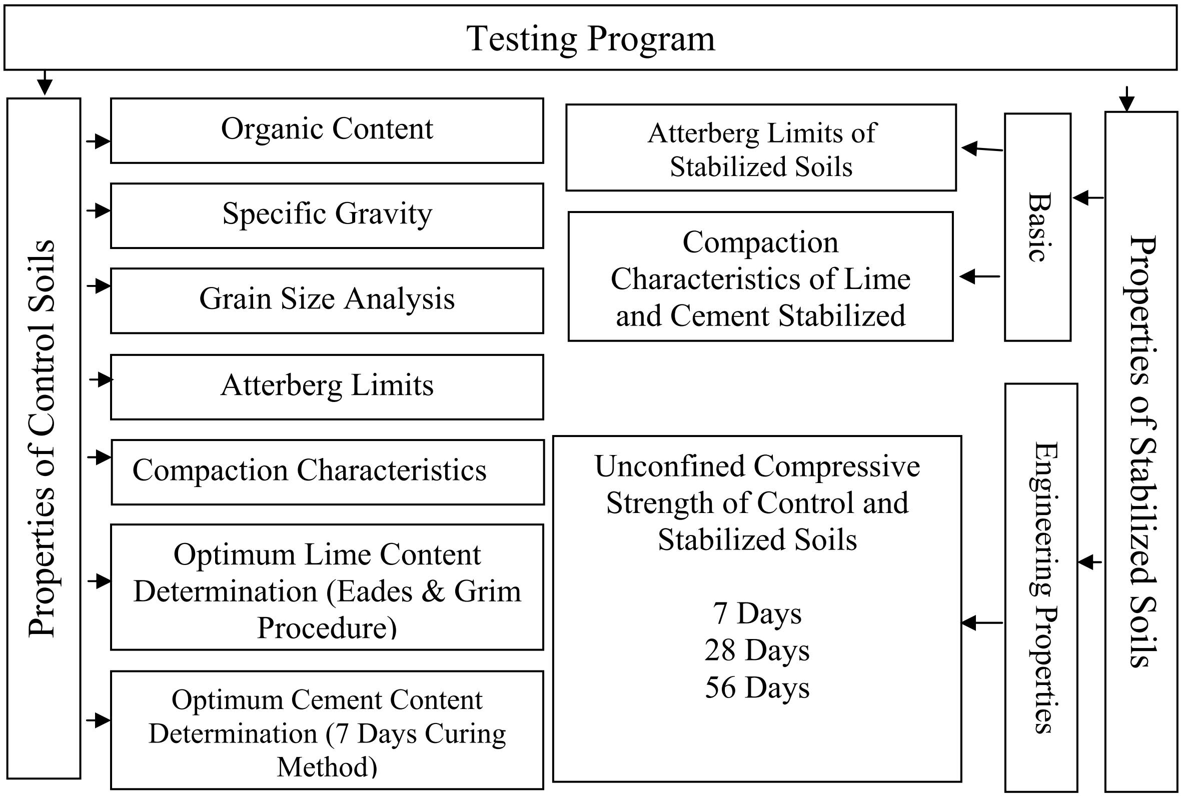


Figure 6.3. Flow Chart of the Engineering Testing Program.

Properties of Control Soils

This section includes the basic properties of control soils such as organic content, specific gravity, Atterberg limits, compaction characteristics, and optimum lime and cement content determination.

We measured the soil organic matter present in natural soil specimens. Table 6.3 depicts the amount of organic matter present in the natural soil samples. The amount of organic matter present in the soil ranges from 1.5 to 6.1 percent.

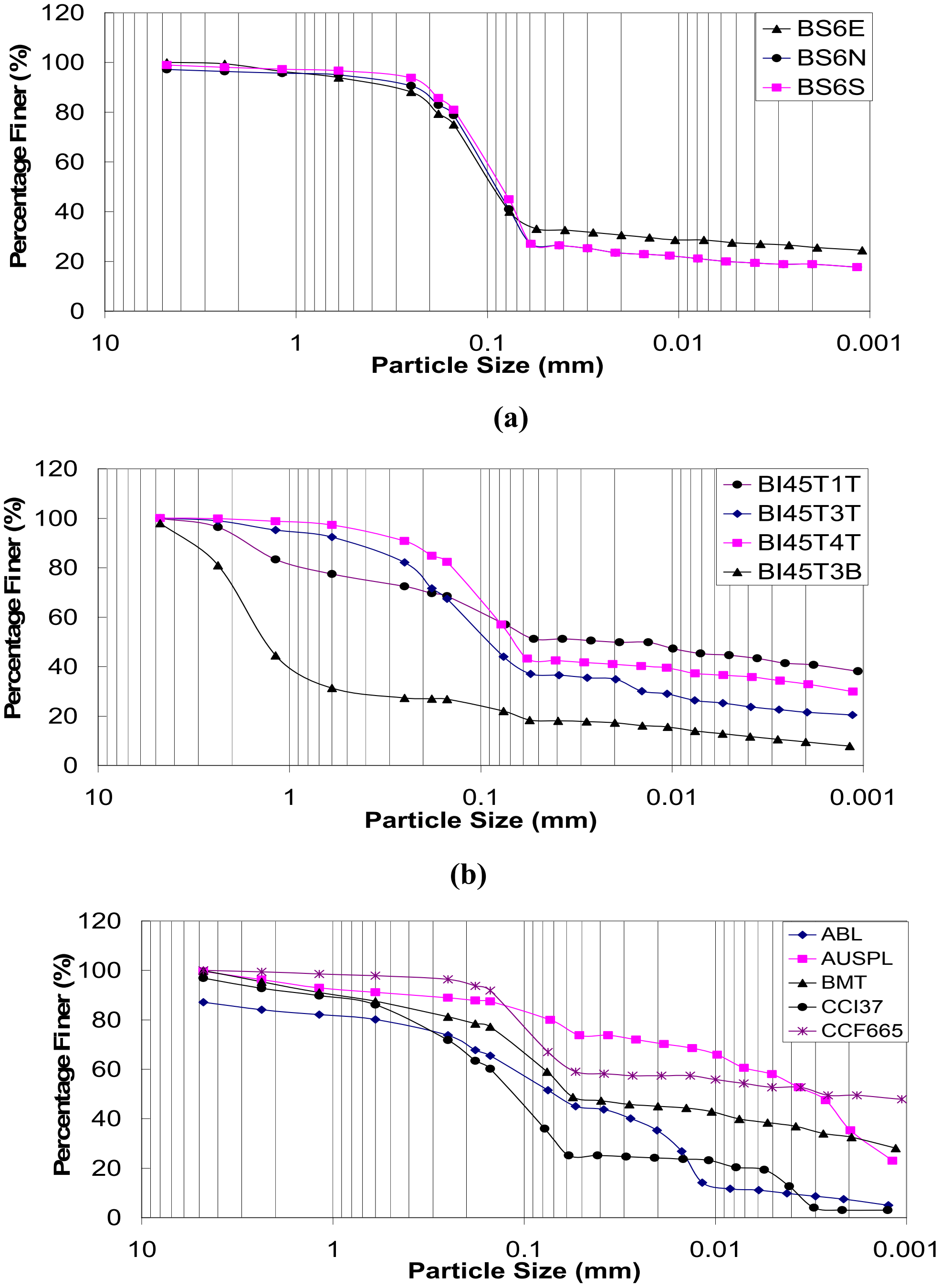
We measured specific gravity of the natural soils. The specific gravity test provides an indirect explanation of the possible constituents of a soil. Table 6.3 presents the specific gravity test results of all control soils. It can be seen that the control soils exhibit specific gravity values in the range of 2.37 to 2.62. These specific gravity values are low when compared to the inorganic soils, which are usually in the range between 2.5 to 2.7. These low values are attributed to the amount of lightweight organic content present in these control soils. Several researchers have reported similar results in the literature (Mitchell and Soga, 2005).

Atterberg limits tests explain the plastic nature of soils. Atterberg tests were conducted to measure the consistency of the natural as well as stabilized soils. The plasticity indices were calculated by first measuring the liquid limit (LL) and plastic limit (PL) values, and then calculating the difference between them (Table 6.3).

Table 6.3. Engineering Properties in Natural Soils.

Sample	Ign. Oven % OM	Specific Gravity	LL	PL	PI
ABL	4.4	2.53	36	17	19
AUSPL	4.8	2.57	46	23	23
BMT	4.6	2.58	42	19	23
CCI37	1.5	2.52	32	22	10
CCF665	3.7	2.51	51	21	30
BI45T1T	6.1	2.37	59	21	38
BI45T3T	3.7	2.45	27	22	5
BI45T3B	4.4	2.62	49	27	22
BI45T4T	4.4	2.48	35	23	12
BS6E	2.1	2.59	24	17	7
BS6N	2.2	2.58	28	16	12
BS6S	2.3	2.57	27	14	13

Sieve and hydrometer analyses were made on the natural soils to determine the grain size distribution (gradation) of all the test materials, and these results are presented in. Figure 6.4 (a), (b), and (c) present the grain size distribution curves of the control soils and Table 6.4.



(a)

(b)

(c)

Figure 6.4. Grain-Size Distribution Curves of Natural Soils.

Table 6.4. Particle Size Details of Natural Soils.

Soil Notation	D_{10} (mm)	D_{30} (mm)	D_{50} (mm)	D_{60} (mm)
ABL	0.004000	0.01500	0.0740	0.0800
AUSPL	0.000950	0.00150	0.0020	0.0065
BMT	0.000930	0.00150	0.0720	0.0750
CCI37	0.003800	0.00380	0.0800	0.1500
CCF665	0.000090	0.00095	0.0025	0.0750
BI45T1T	0.000090	0.00095	0..0150	0.0750
BI45T3T	0.000088	0.00150	0.0700	0.0750
BI45T3B	0.003000	0.50000	1.5000	0.0750
BI45T4T	0.000085	0.00150	0.0800	0.0750
BS6E	0.000900	0.07800	0.0730	0.1000
BS6N	0.000930	0.08500	0.0690	0.0800
BS6S	0.000950	0.08500	0.0710	0.0750

Note: D_{10} *- Diameter of particle at which 10 percent is finer than that size*
D_{30} *- Diameter of particle at which 30 percent is finer than that size*
D_{50} *- Diameter of particle at which 50 percent is finer than that size*
D_{60} *- Diameter of particle at which 60 percent is finer than that size*

From Figure 6.4 and Table 6.4, it can be observed that all the natural soils except BI45T3B and CCI37 have fine fraction (percent passing No. 200 sieve) greater than 50 percent; hence, these soils are classified as silts and clays. CCI37 and BI45T3B are classified as sands, since their fine fraction is less than 50 percent.

Additionally, Table 6.5 illustrates that the soil samples contain organic content greater than 1.5 percent and less than 6.2 percent. According to Karlsson and Hansbo (1981), soils having organics in the range of 2 percent to 6 percent are termed as low organic soils, and soils having organics in the range of 6 percent to 20 percent are termed as medium organic soils. However, their soil classification is based on the tests performed on Scandinavian soils. Therefore, the present soil samples are classified as 'Organic silt, Organic clay, and Organic sand' of medium organic content levels.

Table 6.5 also shows the plasticity indices of the control soils, which varied from 5 to 38. The BI45T3T soil exhibited low PI value and BI45T1T soil exhibited high PI value. Based on the amount of organic content and plasticity indices of natural soils, they are divided into four different groups, as shown in Figure 6.5 and presented as follows:

Group I – Organic Content < 3 percent and PI < 15 percent
Group II – Organic Content > 3 percent and PI < 15 percent
Group III – Organic Content > 3 percent and 15 percent ≤ PI ≤ 25 percent
Group IV – Organic Content > 3 percent and PI > 25 percent

From Figure 6.5, it can be illustrated that plasticity index of the soil has increased with an increase in the organic content of the soil. According to Jenny (1941), soils with a high plasticity index should have a higher organic content than soils with a low plasticity index. This observation confirms that the present soils follow the same trend of results (i.e., soil with a high organic content has a higher plasticity index). Table 6.5 summarizes the organic content and plasticity indices for all grouped control soils.

Table 6.5. Classification of Soils Based on Organic Content and Plasticity Index.

Group I, OC < 3%, PI < 15	Sample Notation	PI	OC (%)
	BS6E	7	2.18
	BS6N	12	2.24
	BS6S	13	2.31
	CCI37	10	1.54
Group II, OC > 3%, PI < 15	Sample Notation	PI	OC (%)
	BI45T3T	5	3.72
	BI45T4T	12	4.47
Group III, OC > 3%, 15≤ PI ≤25	Sample Notation	PI	OC (%)
	ABL	19	4.40
	BI45T3B	22	4.42
	BMT	23	4.65
	AUSPL	23	4.80
Group IV, OC > 3%, PI > 25	Sample Notation	PI	OC (%)
	CCF665	30	3.74
	BI45T1T	38	6.12

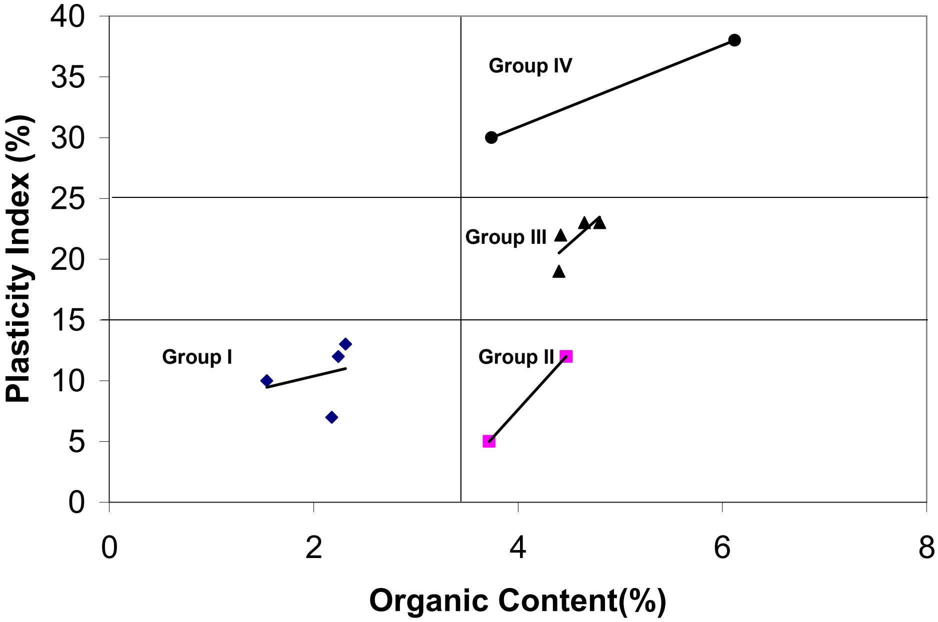


Figure 6.5. Classification of Soils Based on Organic Content (%) and Plasticity Index (%).

Compaction Test Results

Compaction tests were conducted on the control soils to determine the optimum moisture content and dry unit weight relationships. Figure 6.6 presents the typical plot for moisture content - dry unit weight curves of the control soils for BS6 samples. Table 6.6 presents optimum moisture content and dry unit weights of all the control soils; these values were used for soil specimen preparation for engineering tests.

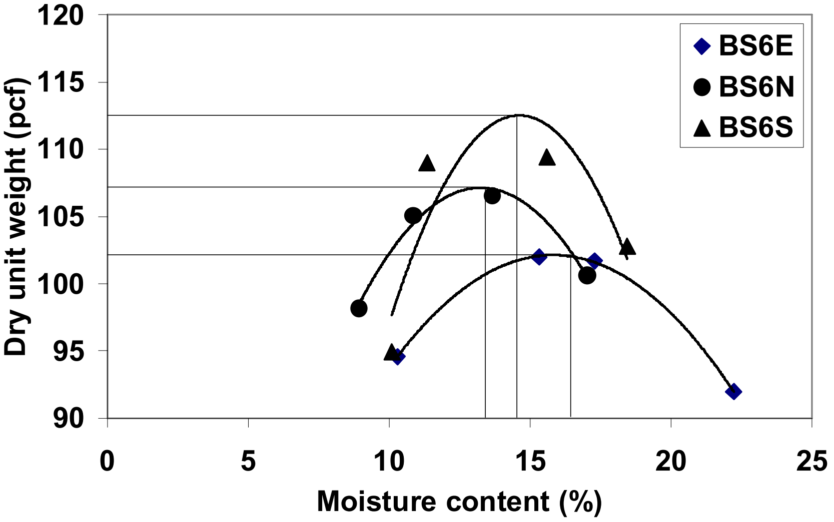


Figure 6.6. Moisture Content – Dry Unit Weight Curves of Control Soils Sampled from State Highway 6 at College Station in Bryan District.

Table 6.6. Optimum Moisture Content and Dry Unit Weight of Control Soils.

Sample Notation	Optimum Moisture Content (%)	Dry Unit Weight (pcf)
ABL	17.7	99.8
AUSPL	20.0	93.2
BMT	17.7	99.1
CCI37	16.3	103.8
CCF665	24.5	90.5
BI45T1T	31.0	79.2
BI45T3T	16.5	97.8
BI45T3B	34.2	75.8
BI45T4T	24.0	91.5
BS6E	16.5	102.0
BS6N	13.5	106.8
BS6S	14.5	112.0

Figure 6.7 presents the influences of organic content (percent) on optimum moisture content of compacted soils. It can be observed from this figure that an increase in the organic content resulted in an increase in optimum moisture content of the organic soils. This result can be explained from the fact that the organic matter is highly hydrophilic in nature. Therefore, with an increase of organic content, there is an increase in optimum moisture content of the compacted soil.

The influence of organic content on the compaction dry unit weight is shown in Figure 6.8. From these data, it can be concluded that with an increase in the organic content, dry unit weight of the soil is decreased.

From Figures 6.7 and 6.8, note that the behavior of the soil samples arrow marked (CCI37 and BI45T3B) come under soil groups I and III, respectively, and are different from their respective groups, which may be due to their clay mineralogy. Further mineralogical and chemical studies are required to understand the constituents of these soils to interpret their behavior.

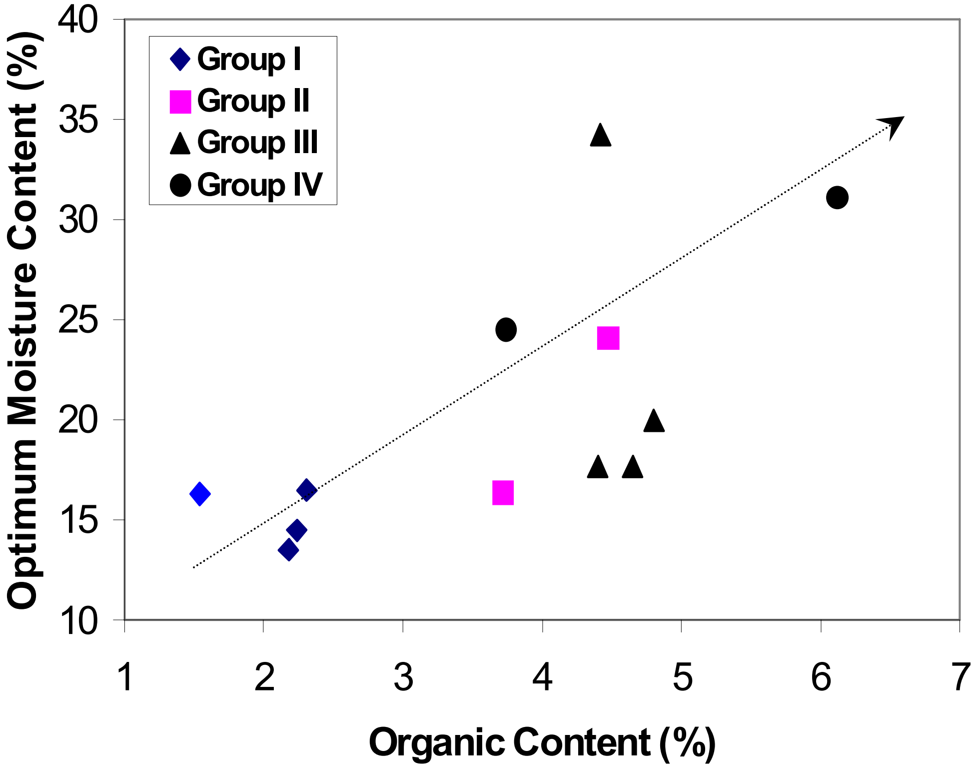


Figure 6.7. Influence of Organic Content (%) on Compaction Optimum Moisture Content.

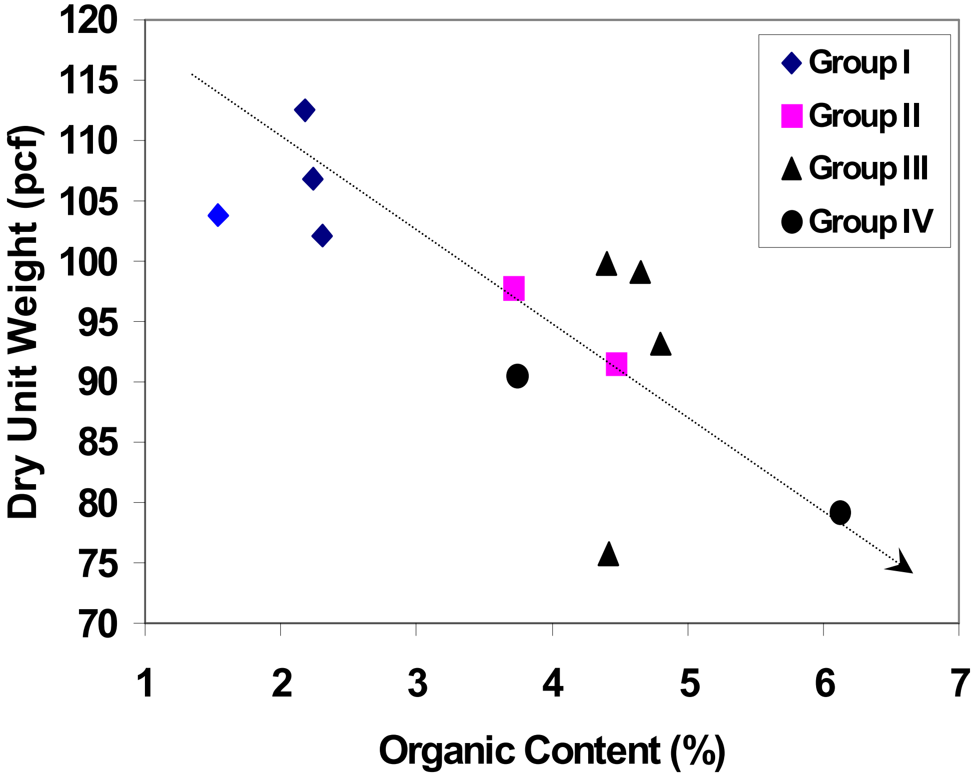


Figure 6.8. Influence of Organic Content (%) on Compaction Optimum Dry Unit Weight.

Optimum Lime Content Determination

The optimum lime content is determined by the Eades and Grim procedure, as explained previously. This test determines the best lime percentage for a particular soil sample because the percent dosage for effective lime stabilization varies with soil type. The lime content corresponding to pH value equal to 12.3 is taken as optimum lime content. A typical graph of optimum lime content determination is shown in Figure 6.9. Optimum lime contents for the field sites are shown in Table 6.7.

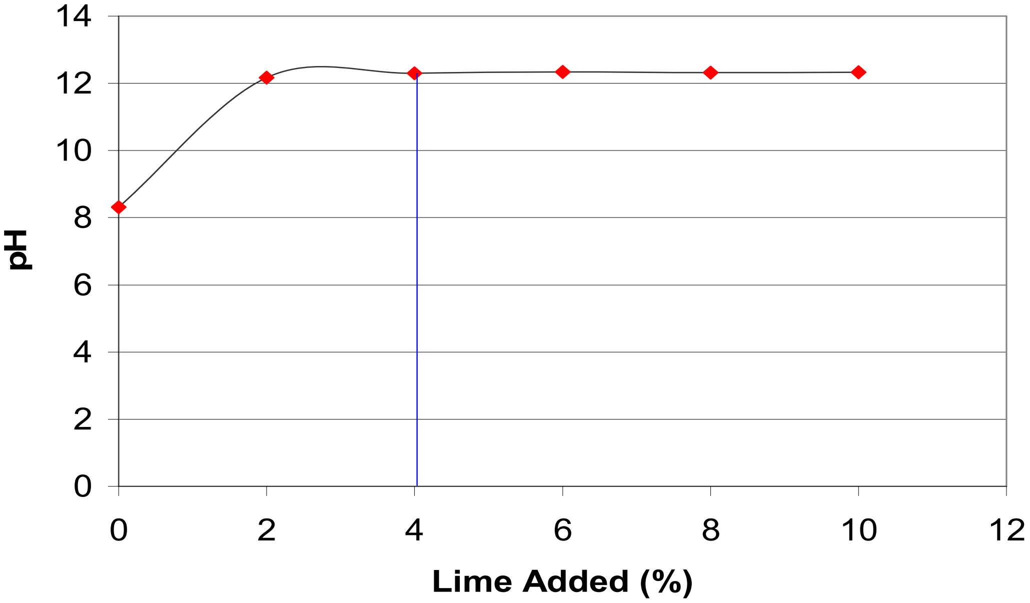


Figure 6.9. Typical Lime Content Determination Graph for Abilene Soil.

Table 6.7. Optimum Lime Content of the Soil Samples.

Sample Notation	Optimum Lime Content (%)
ABL	4.0
AUSPL	8.0
BMT	6.0
CCI37	4.0
CCF665	8.0
BI45T1T	8.0
BI45T3T	6.0
BI45T3B	8.0
BI45T4T	6.0
BS6E	8.0
BS6N	6.0
BS6S	8.0

Optimum Cement Content Determination

Optimum cement content of the field sites is determined by Tex-120-E standard method. Various cement-stabilized soils cured for seven days were used, and UCS tests were conducted on the cured samples. Prior to conducting UCS tests, all soils were stabilized with a cement content of 6 percent, and these stabilized soils were subjected to standard proctor compaction tests to determine the optimum moisture content of each soil for 6 percent cement content. The optimum moisture content and maximum dry unit weight results for the soils stabilized with a cement content of 6 percent are shown in Table 6.8. The molding water content necessary for other cement contents (other than 6 percent) was calculated using the following formula.

% molding water = % optimum moisture from M/D curve + 0.25 (% cement change from 6%) → Equation 6.1

Table 6.8. Compaction Characteristics of 6% Cement-Stabilized Soils.

Name of the Site (Sample Notation)	Optimum Moisture Content (%)	Dry Unit Weight (pcf)
ABL	19.0	93.9
AUSPL	20.7	93.7
BMT	17.9	93.1
CCI37	16.1	98.5
CCF665	22.0	101.0
BI45T1T	29.1	69.5
BI45T3T	16.8	97.1
BI45T3B	31.0	76.0
BI45T4T	21.7	89.1
BS6E	14.2	102.7
BS6N	14.7	104.0
BS6S	16.2	95.7

After determining the optimum moisture content (percent) and dry unit weight (pcf) of soil stabilized with 6 percent cement content, the molding moisture contents for other percentages of cement contents (2, 4, 8, and 10 percent) are determined. A typical calculation is shown below, and the results are summarized in Table 6.9 for Abilene soil.

Let us consider a cement content of 4 percent for calculation purposes. The molding water content for this percent cement is calculated as per equation 6.1:

% molding water content = 19.0 + 0.25*(4% - 6%) = 18.5%

Table 6.9. Moisture-Density Characteristics.

Soil Type	Cement Content, %	Optimum Moisture Content, %	Max. Dry Unit Weight (pcf)
Abilene	0	17.7	99.8
	2	18.0	N/A
	4	18.5	N/A
	6	19.0	93.9
	8	19.5	N/A
	10	20.0	N/A

It is to be noted here that the optimum moisture content for 2, 4, 6, 8 and 10 percent shown in Table 6.9 are not true optimum moisture contents of stabilized soils. It is a molding water content used to mix the soil with cement to ease and reduce the experimental errors.

Tex-120-E does not recommend a target unconfined compressive strength. However, the Tex-121-E (soil lime testing) method mentions that a target strength of 150 psi (1035kPa) should be achieved for a stabilized soil. Higher strengths are not recommended because they can lead to cracking. The optimum cement contents obtained from this procedure are summarized in Table 6.10.

Table 6.10. Optimum Cement Content of the Soil Samples.

Sample Notation	Optimum Cement Content (%)
ABL	2.5
AUSPL	3.5
BMT	5.0
CCI37	5.5
CCF665	6.5
BI45T1T	6.5
BI45T3T	5.0
BI45T3B	8.0
BI45T4T	5.5
BS6E	4.0
BS6N	3.0
BS6S	6.0

Properties of Stabilized Soils

This section includes the basic properties of stabilized soils such as Atterberg limits, compaction characteristics, and engineering properties. UCS tests are performed at different curing periods to evaluate the engineering properties of both natural and stabilized soils. The following sections discuss these results in detail.

Atterberg limits of the soils were determined after treating the soils with optimum cement and lime content. Optimum cement and lime content values are shown in Table 6.10 and Table 6.7. The Atterberg limits of soils stabilized with lime and cement are presented in Tables 6.11 and 6.12, respectively.

Table 6.11. Atterberg Limits of Lime-Stabilized Soils.

Sample Notation	Liquid Limit (%)	Plastic Limit (%)	Plasticity Index(%)
ABL	35	23	12
AUSPL	43	31	21
BMT	42	31	11
CCI37	32	24	8
CCF665	46	30	16
BI45T1T	58	28	30
BI45T3T	31	28	3
BI45T3B	33	27	6
BI45T4T	42	34	8
BS6E	24	21	3
BS6N	30	23	7
BS6S	30	20	10

Table 6.12. Atterberg Limits of Cement Stabilized Soils.

Sample Notation	Liquid Limit (%)	Plastic Limit (%)	Plasticity Index(%)
ABL	38	23	15
AUSPL	49	30	19
BMT	42	22	20
CCI37	35	28	7
CCF665	51	25	26
BI45T1T	68	34	34
BI45T3T	32	28	4
BI45T3B	49	30	19
BI45T4T	41	31	10
BS6E	27	22	5
BS6N	31	22	9
BS6S	28	17	11

Figure 6.10 compares the plasticity indices of both lime- and cement-stabilized soil specimens with control soils. From the graph, it can be illustrated that stabilized soils have low plasticity index when compared to the unstabilized soils. It is to be noted that lime-stabilized soils have exhibited lower PI values than cement-stabilized soils.

To quantify the reduction in PI values, a non-dimensional factor called reduction factor of plasticity index (R_f) is introduced and defined as the ratio of the difference between the stabilized and unstabilized plasticity indices of lime / cement soil to the plasticity index of the unstabilized soil.

$$R_f = \frac{PI_s - PI_u}{PI_u}$$

Where, PI_s = PI of stabilized soil; PI_u = PI of unstabilized soil

The reduction factors for both cement- and lime-stabilized soils were calculated, and these results are summarized in Table 6.13 by separating them according to their grouping. From these results, it can be seen that the reduction factors are more predominant in soil group I. A moderate increase in reduction factors are observed for soil groups II, III, and IV. The influence of chemical treatment is impacted by the amount of organic matter in the control soil, which was subjected to chemical treatment.

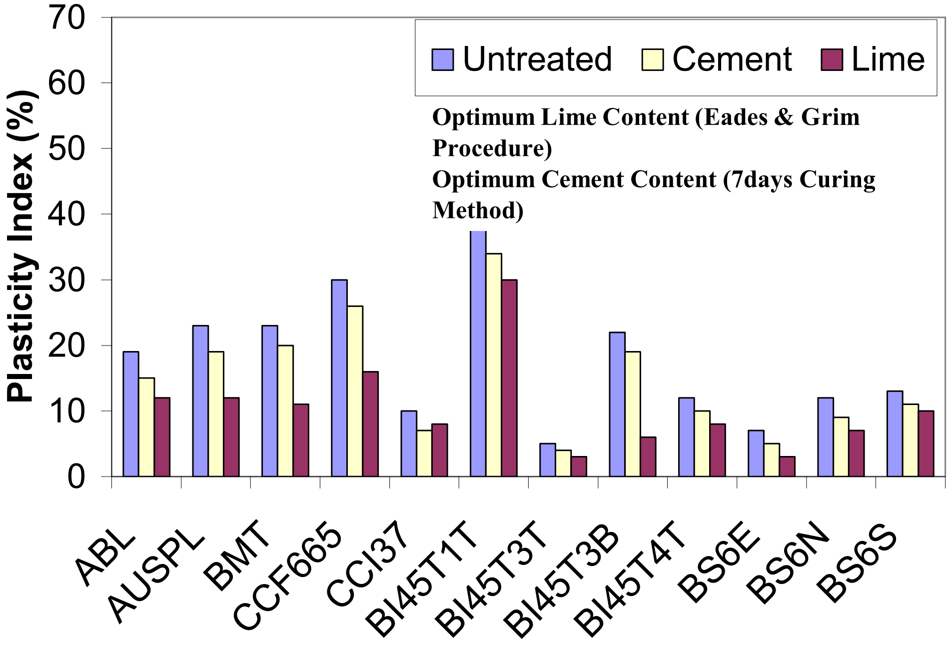


Figure 6.10. Comparisons of Plasticity Indices of Stabilized and Unstabilized Soils.

Table 6.13. Reduction Factors of Soils Stabilized with Cement and Lime.

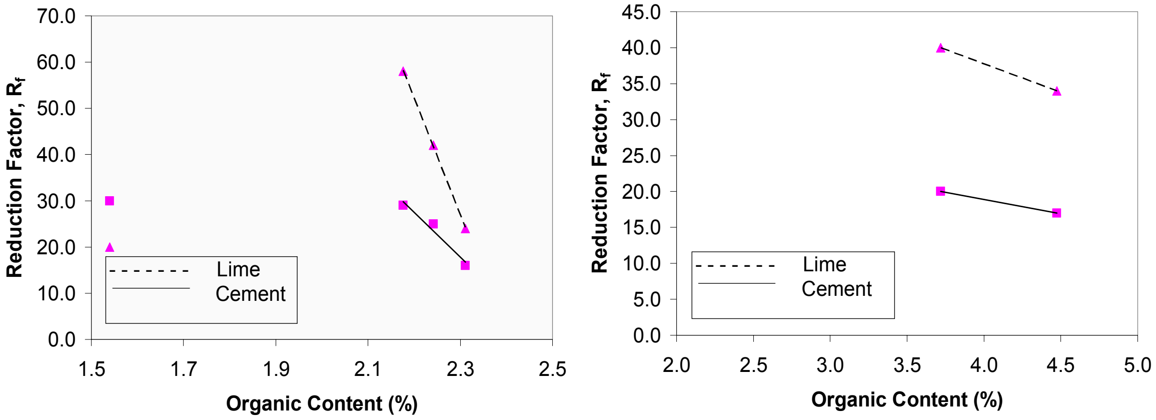
Group I, OC < 3%, PI < 15	Sample Notation	$R_{f(Lime)}$ (%)	$R_{f(Cement)}$	OC (%)
	BS6E	38	29	2.18
	BS6N	42	25	2.24
	BS6S	24	16	2.31
	CCI37	20	30	1.54

Group II, OC > 3%, PI < 15	Sample Notation	$R_{f(Lime)}$ (%)	$R_{f(Cement)}$ (%)	OC (%)
	BI45T3T	40	20	3.72
	BI45T4T	34	17	4.47

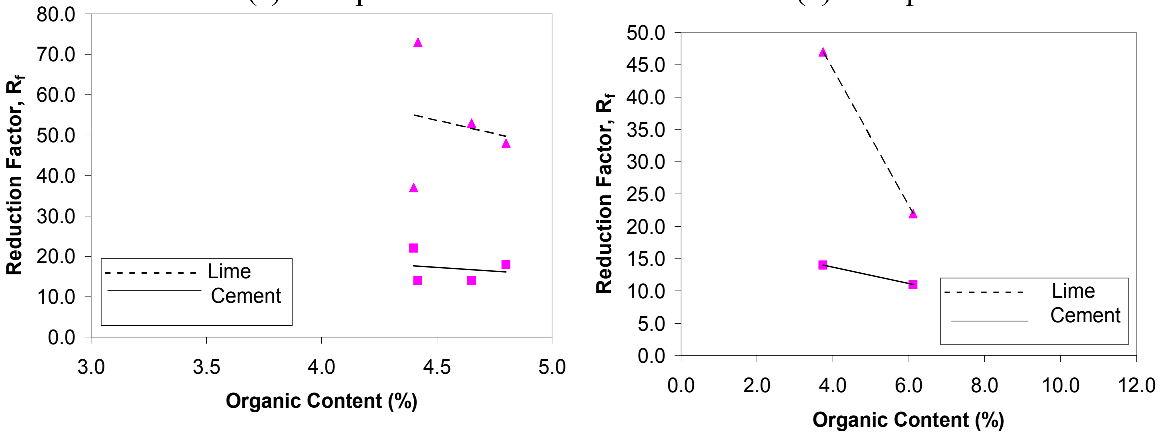
Group III, OC > 3%, 15 <PI < 25	Sample Notation	$R_{f(Lime)}$ (%)	$R_{f(Cement)}$ (%)	OC (%)
	ABL	37	22	4.40
	BI45T3B	73	14	4.42
	BNT	53	14	4.65
	AUSPL	48	18	4.80

Group IV, OC > 3%, PI > 25	Name of the Site	$R_{f(Lime)}$ (%)	$R_{f(Cement)}$ (%)	OC (%)
	CCF665	47	14	3.74
	BI45T1T	22	11	6.12

From Figure 6.11, researchers noticed that the reduction factor (R_f) of the soil decreases with an increase in the organic content (percent). The graph also illustrates that the reduction factor (R_f) of the soil stabilized with lime is high compared to the soil stabilized with cement. The plasticity index of the organic soil stabilized with lime is more efficient than cement. As the organic content increases, it becomes less effective in reducing the plasticity index of the soil for both lime- and cement-stabilized soils. This explains the importance of organics on the chemical treatment.



(a) Group I (b) Group II



(c) Group III (d) Group IV

Figure 6.11. Comparison of Reduction Factors R_f of Lime and Cement with Organic Content (%) (a), (b), (c), (d).

Compaction characteristics of stabilized soil were obtained to perform engineering properties. The optimum lime and cement contents determined earlier (Table 6.7 and Table 6.10) were used to prepare the compaction specimens. The optimum moisture content (percent) and dry unit weights (pcf) of optimum lime- and cement-stabilized soils are presented in Tables 6.14 and 6.15, respectively.

Table 6.14. Optimum Moisture Content and Dry Unit Weight of Lime-Stabilized Soils.

Sample Notation	Optimum Moisture Content (%)		Dry Unit Weight (pcf)	
	Control	Lime treated	Control	Lime treated
ABL	17.7	18.4	99.8	93.6
AUSPL	20	24.7	93.2	87.7
BMT	17.7	20.6	99.1	93.1
CCI37	16.3	16.5	103.8	99.5
CCF665	24.5	25.9	90.5	84.4
BI45T1T	31	33.6	79.2	75.4
BI45T3T	16.5	19.9	97.8	92.1
BI45T3B	34.2	36.0	75.8	78.2
BI45T4T	24	25.9	91.5	85.0
BS6E	16.5	18.2	102	95.0
BS6N	13.5	15.1	106.8	101.0
BS6S	14.5	18.7	112	89.5

Engineering Properties

This section analyzes the results of unconfined compressive strength of both natural (unstabilized) and stabilized soils with lime and cement. Note that these tests were performed on miniature samples prepared and cured at 7, 28, and 56 days.

UCS tests performed on all natural soils at zero days curing period are reported in Figure 6.12. From this figure, it can be seen that there is no specific trend observed to understand the results. Hence, these soils are divided into the four different groups mentioned previously. Franklin et al. (1973) have reported the UCS test data for similar soils having similar organic content. Figure 6.13 compares the present test data (UCS results) with Franklin et al. (1973) test data. A good agreement between test results can be seen. From Figure 6.13, it is observed that samples used by Franklin et al. (1973) having low organic content had a strength ranging from 2 to 3.75 tons/sq-ft, whereas present research samples having low organic content have a strength ranging from 0.5 to 3.65 tons/sq-ft.

Table 6.15. Optimum Moisture Content and Dry Unit Weight of Cement-Stabilized Soils.

Sample Notation	Optimum Moisture Content (%)		Dry Unit Weight (pcf)	
	Control	Cement treated	Control	Cement treated
ABL	17.7	18.6	99.8	95.7
AUSPL	20	20.4	93.2	94.3
BMT	17.7	17.7	99.1	98.9
CCI37	16.3	15.9	103.8	101.8
CCF665	24.5	22.5	90.5	90.6
BI45T1T	31	29.0	79.2	69.7
BI45T3T	16.5	17.2	97.8	96.8
BI45T3B	34.2	29.4	75.8	76.6
BI45T4T	24	21.1	91.5	90.0
BS6E	16.5	14.5	102	103.3
BS6N	13.5	14.6	106.8	104.2
BS6S	14.5	16.2	112	95.7

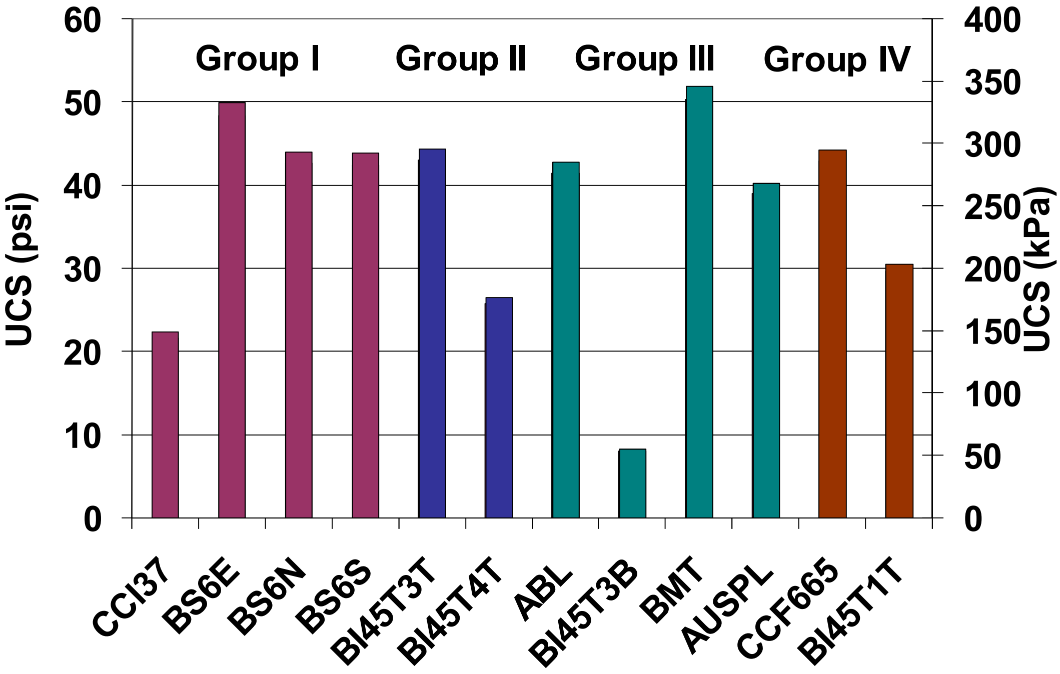


Figure 6.12. Unconfined Compressive Strength of Control Soils at Zero Days.

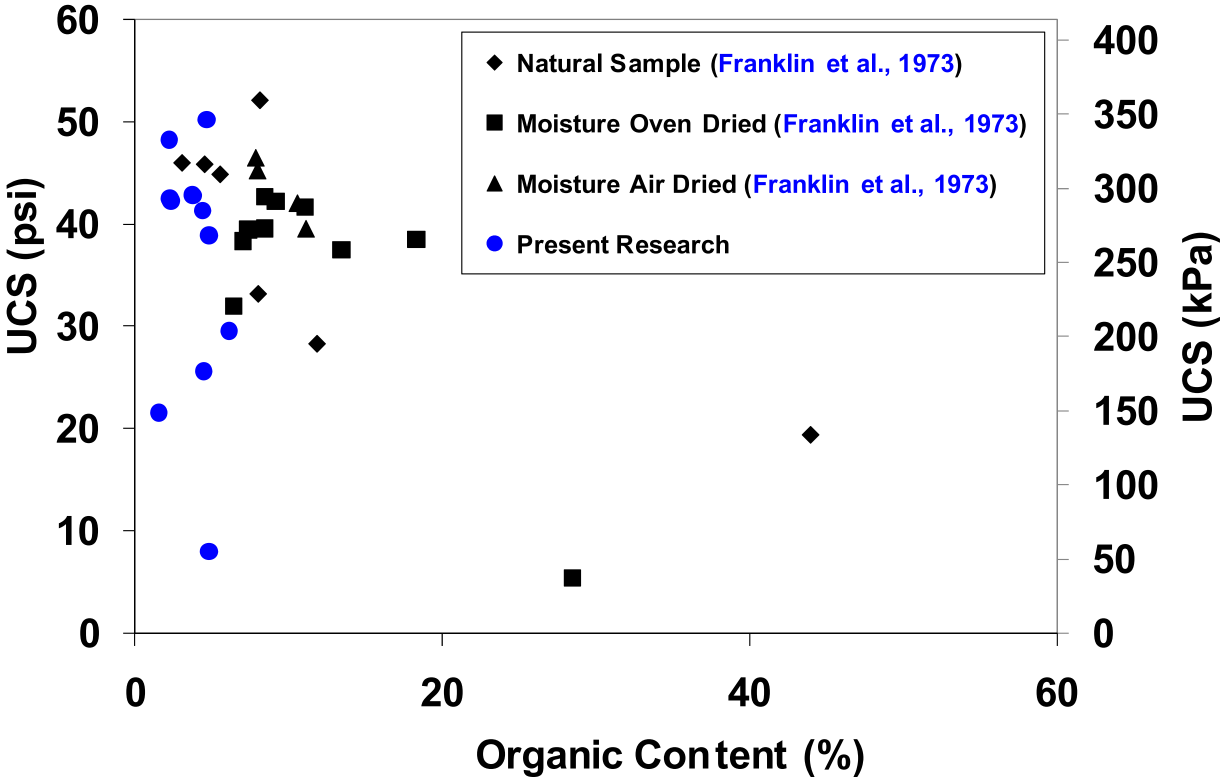


Figure 6.13. Variation of Unconfined Compressive Strength with Organic Content Based on Grouping.

The UCS results presented in Figure 6.13 are re-plotted based on their grouping. From this figure, it can be mentioned that the organic content has moderate influence on the UCS of the present soils. This result is expected as the strength of the soil is dependent of soil type and clay mineralogy.

Table 6.16. Classification of Organic Soils Based on Constant Plasticity Index and Different Organic Content.

Group I	Sample Notation	UCS (psi)	Organic Content (%)
	CCI37	21.6	1.54
	BS6E	48.3	2.18
	BS6N	42.6	2.24
	BS6S	42.3	2.31

Group II	Sample Notation	UCS (psi)	Organic Content (%)
	BI45T3T	42.9	3.72
	BI45T4T	25.7	4.47

Group III	Sample Notation	UCS (psi)	Organic Content (%)
	ABL	41.5	4.40
	BI45T3B	8.1	4.80
	BMT	50.3	4.65
	AUSPL	39.0	4.80

Group IV	Sample Notation	UCS (psi)	Organic Content (%)
	CCF665	42.9	3.74
	BI45T1T	29.6	6.10

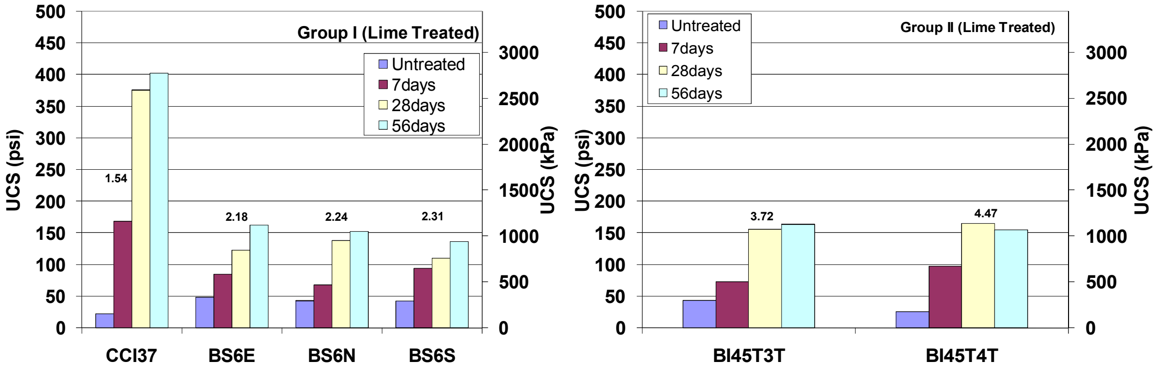
Unconfined Compressive Strength Test for Stabilized Soils

As mentioned earlier, UCS tests on all soil samples stabilized with optimum lime and cement content were carried out at different curing periods. In addition, the pH concentration and the organic content of all the above UCS tested soil specimens were also determined at the end of each curing period. The strength of the soil samples stabilized with lime and cement at different curing periods is presented in Figures 6.14 and 6.15, respectively.

Figure 6.14 compares the UCS test results of a typical soil sample selected from each group. From Figure 6.14, the strength of the lime-stabilized soil is reduced, with an increase in the organic content in each individual group.

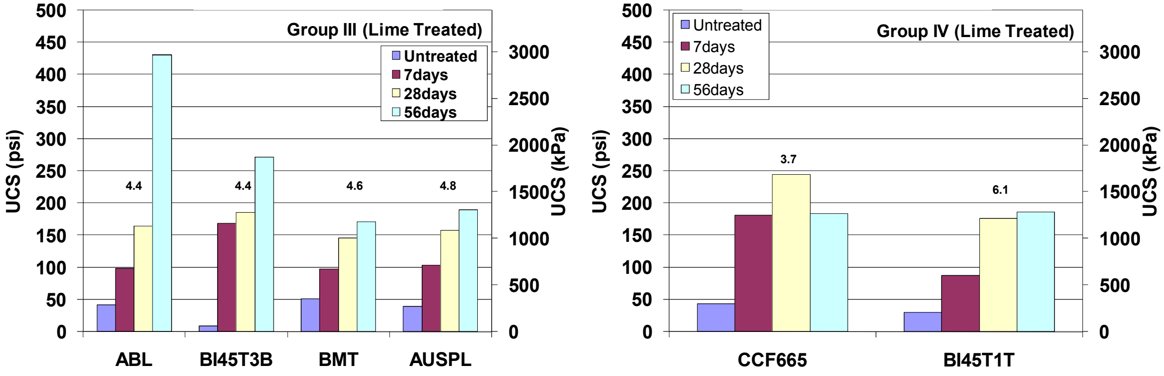
From Figure 6.14, there is also a slight reduction in strength after 56 days for BI45T3T and BI45T4T soils. This reduction in strength may be due to the formation of insoluble calcium humic acid and the moderate reduction of pH concentrations.

Figure 6.15 shows a similar typical plot for cement-treated specimens. From Figure 6.15, the strength of the cement-stabilized soil is reduced with an increase in the organic content. Also notice a slight reduction in strength of cement-stabilized soils after 56 days. This reduction in strength could also be attributed to the formation of insoluble calcium humic acid and the moderate reduction of pH concentrations of the cement-soil mixture after different curing periods. Further analysis is very necessary to quantify these reductions in strength at prolonged curing periods.



(a) Group I

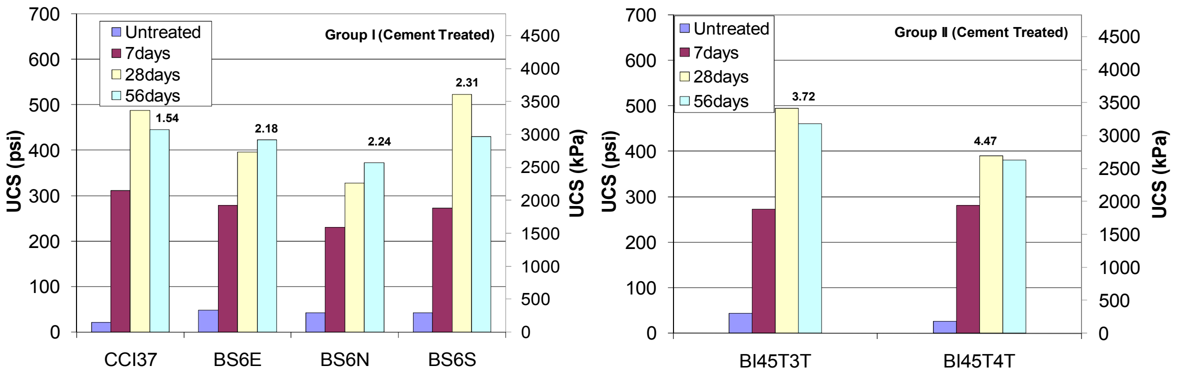
(b) Group II



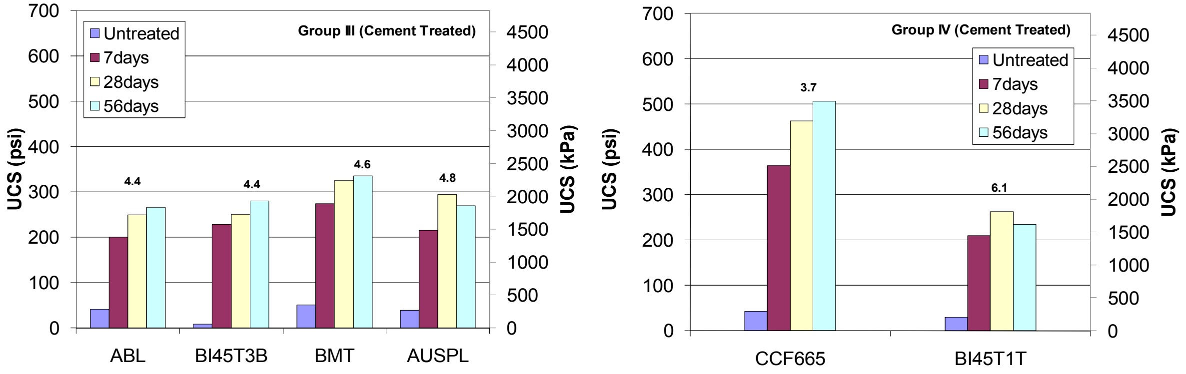
(c) Group III

(d) Group IV

Figure 6.14. UCS of Lime-Stabilized Samples at Intervals of 7, 28, and 56 Days (a), (b), (c), (d), (e).



(a) Group I (b) Group II

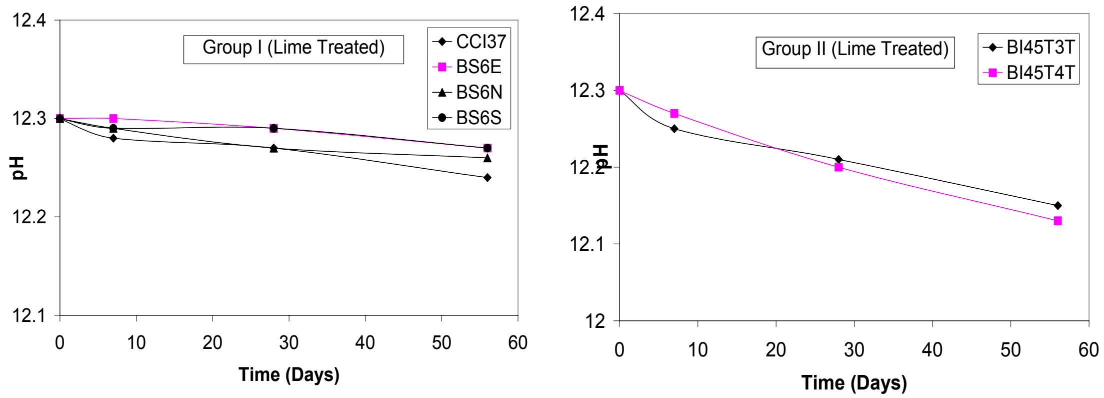


(c) Group III (d) Group IV

Figure 6.15. UCS of Cement-Stabilized Samples at Intervals of 7, 28, and 56 Days (a), (b), (c), (d).

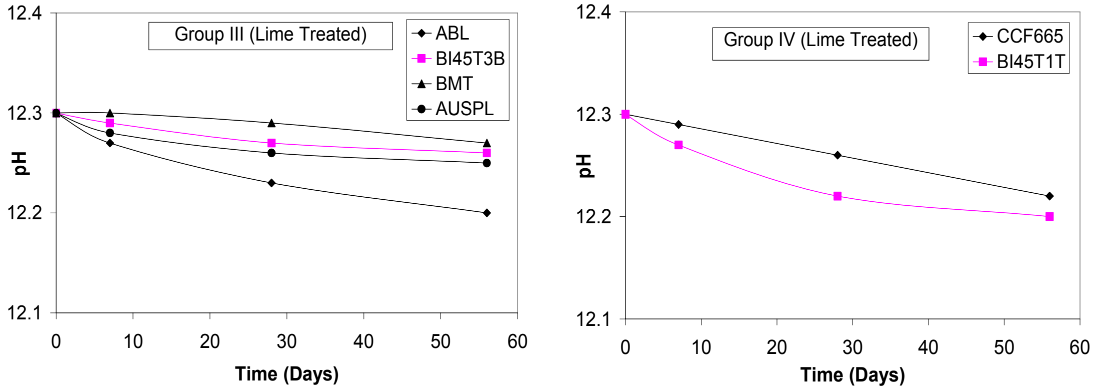
Variations of pH of the lime- and cement-stabilized soils with a curing period (days) is shown in Figures 6.16 and 6.17. It can be seen that the pH of the soil decreases with curing periods. In general, when calcium-based additives are used to stabilize soils, the pH of the stabilized soil mixture increases and leads to the formation of CSH gel by dissolving the silica present in the clay (Eades and Grim, 1966). The CSH gel is known to be the basic hydration product that contributes to the increase in strength of the soil. In contrary, the pH of the lime/cement-stabilized organic soil is decreased with an increase in the curing period. This drop in pH concentration retards the dissolution of silica in the clay, which inhibits the formation of CSH gel and thus the strength of the stabilized soil.

Another factor contributing to the reduction in strength is attributed to the formation of insoluble calcium humic acid. To understand this phenomenon, a brief history of organic soil formation in Texas is needed**.** The main soil formations in Texas are either grassland soils or forest soils (Soil Taxonomy, 2006). These soils are rich in both humic and fulvic acids. We obtained the soils in this study from locations dominated by the following soil orders: Alfisols, Ultisols, Vertisols, and Mollisols (Figure 6.18).



(a) Group I

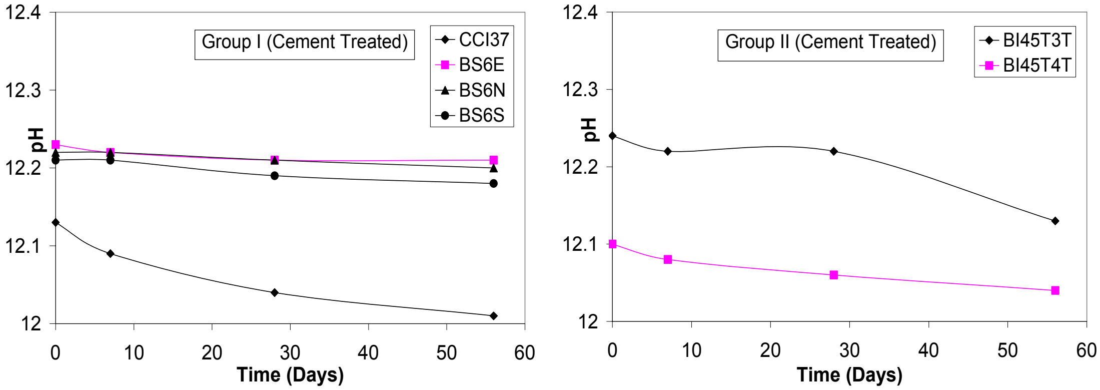
(b) Group II



(a) Group III

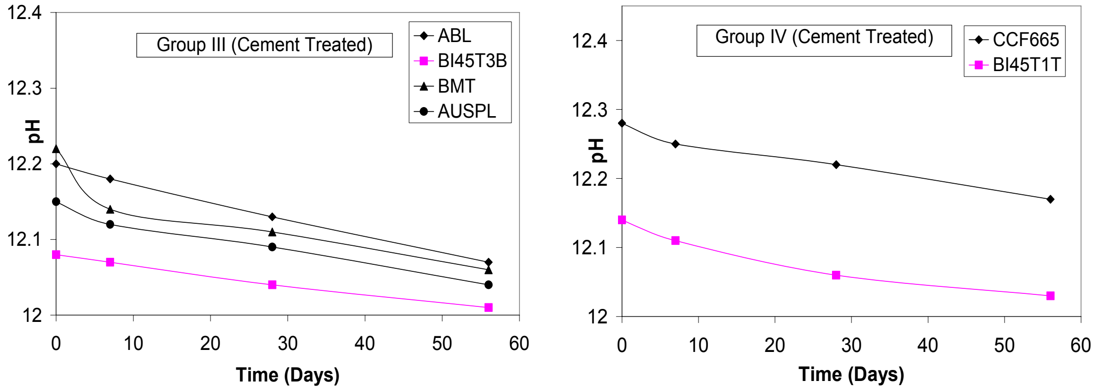
(d) Group IV

Figure 6.16. Variation of pH of Lime-Stabilized Samples at Intervals of 7, 28, and 56 Days (a), (b), (c), (d).



(a) Group I

(b) Group II



(c) Group III

(d) Group IV

Figure 6.17. Variation of pH of Cement-Stabilized Samples at Intervals of 7, 28, and 56 Days (a), (b), (c), (d).

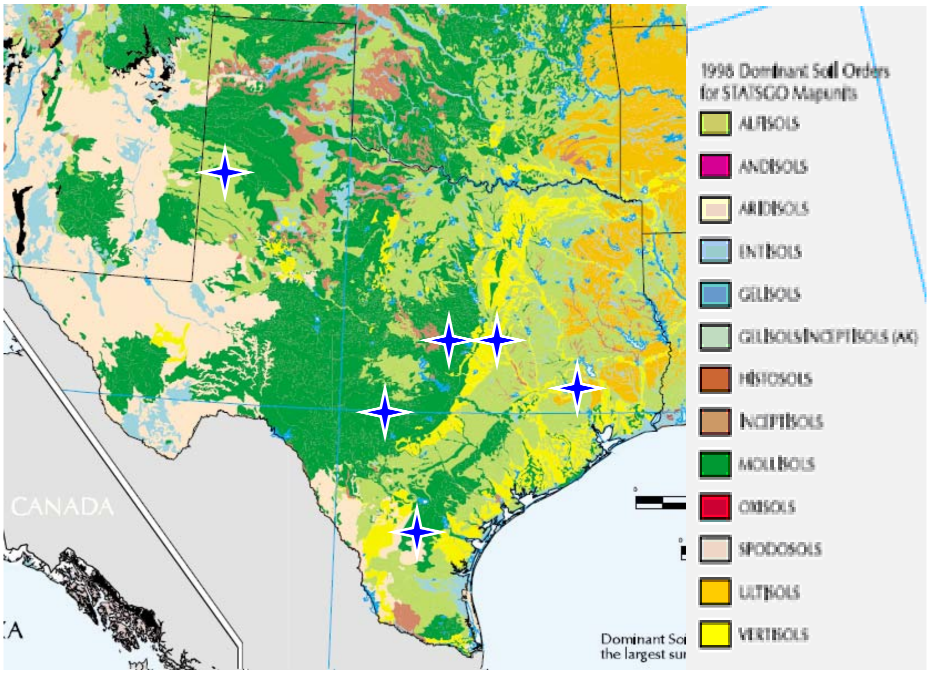
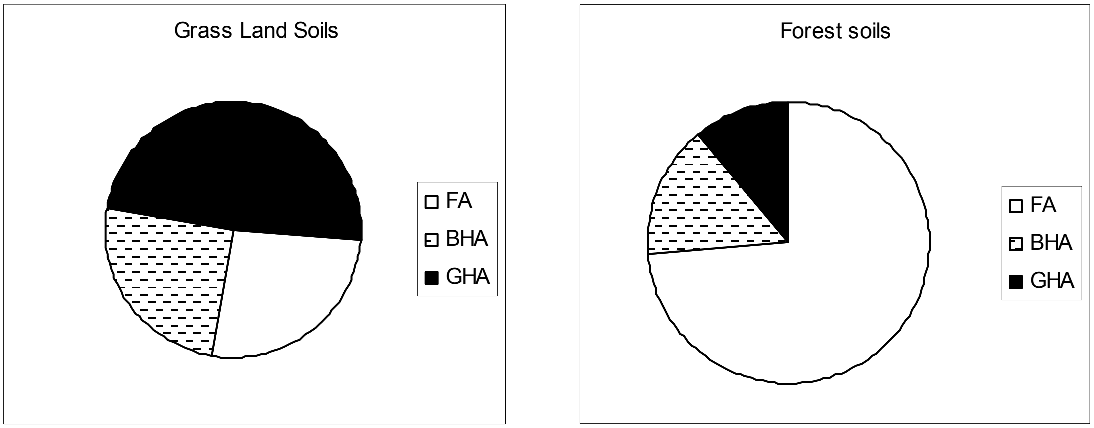


Figure 6.18. Texas Map Showing the Dominant Soil Orders for STATSGO Map units. (Soil Taxonomy, 2006)

According to Tindall and Kunkel (1999), alfisols, spodosols, and ultisols are generally high in fulvic acids classified as forest soils; mollisols are high in humic acids classified as grassland soils. However, vertisol is neither a grassland soil nor forest soil. Moreover, Stevenson (1994) reported that forest soils consist of fulvic acid, whereas grassland soils consist of humic acid in which the major portion is gray humic acid, as shown in the pie chart in Figure 6.19. Among these acids, humic acid has a higher affinity toward calcium if present in the soil. In addition, the half life of humic acid present in any soil formation is reported as more than 100 years (Brady and Weil, 1999). Hence, when calcium-based additives, such as cement, are used to stabilize humic acid rich organic soils, the humic acid will react with the calcium present in the stabilizer and form insoluble calcium humic acid. This compound formation utilizes most of the calcium present in the stabilizer and hence no calcium is available for the formation of CSH gel.



FA = Fulvic acid; BHA = Brown humic acid; GHA = Gray humic acid

Figure 6.19. Pie Chart Showing the Relationship of Humic Acid and Fulvic Acid (Stevenson, 1994).

Figure 6.20 and Figure 6.21 show that the organic content of the soil was decreased when it was treated with lime or cement. From these observations, it can be concluded that reactions are taking place between organic content and lime/cement. If the pH of the soil is decreased, then there is a chance of the formation of acids. The acidic reactions may be in between humic acid and calcium and form insoluble calcium to humic acid. This reaction might have contributed to low strength at high curing periods.

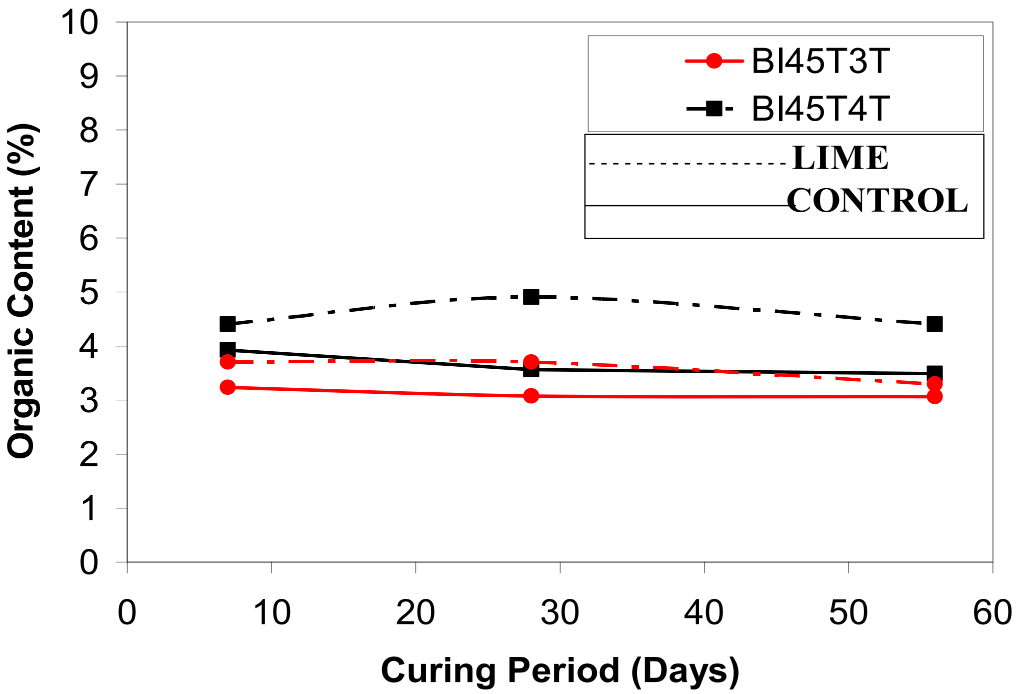


Figure 6.20. Comparison of Lime-Stabilized Sample's Organic Content with Control Soil's Organic Content at Intervals of 7, 28, and 56 Days.

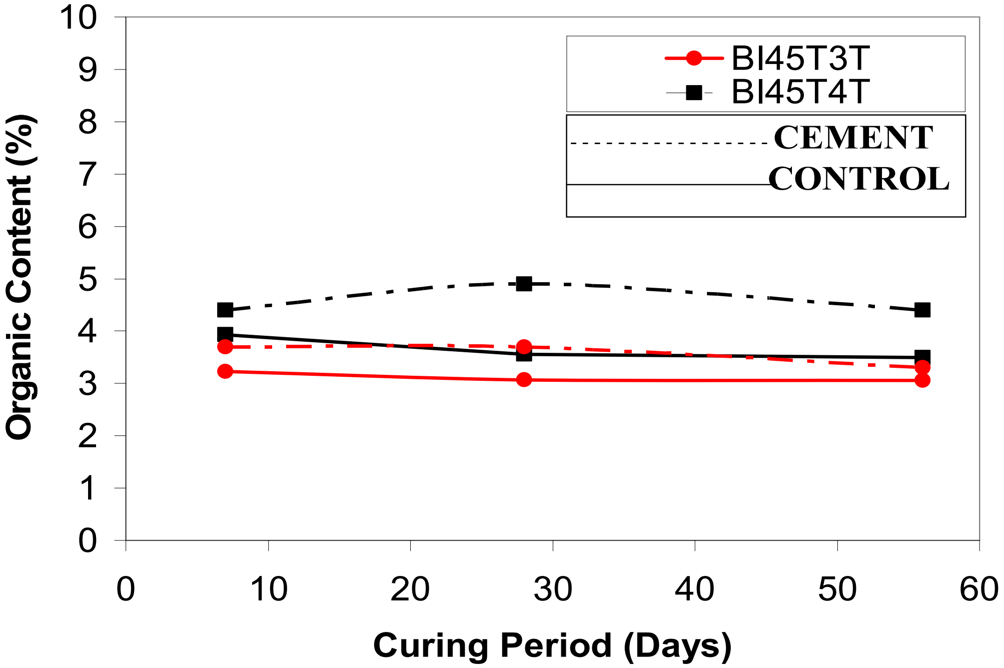


Figure 6.21. Comparison of Cement-Stabilized Sample's Organic Content with Control Soil's Organic Content at Intervals of 7, 28, and 56 Days.

Strength Factors

To explain the strength increment for stabilized soils, two strength factors are introduced and explained below:

1) LISF – Lime-Induced Strength Factor

$$\text{LISF}_n = \frac{\text{Strength of lime-stabilized specimen at n days}}{\text{Strength of unstabilized specimen at n days}}$$

2) CISF – Cement-Induced Strength Factor

$$\text{CISF}_n = \frac{\text{Strength of cement-stabilized specimen at n days}}{\text{Strength of unstabilized specimen at n days}}$$

Where n is the number of curing days.

The LISF and CISF values are determined for 7, 28, and 56 days; graphs are plotted between the strength factors and organic content. The graphs are shown in Figure 6.22 with the three different groups as classified earlier.

From the graphs shown in Figure 6.22, the following conclusions are drawn:

1) Both the lime-induced strength factor and the cement-induced strength factor decreased with an increase in the organic content.
2) Lime-induced strength factors are low when compared to the cement-induced strength factors.

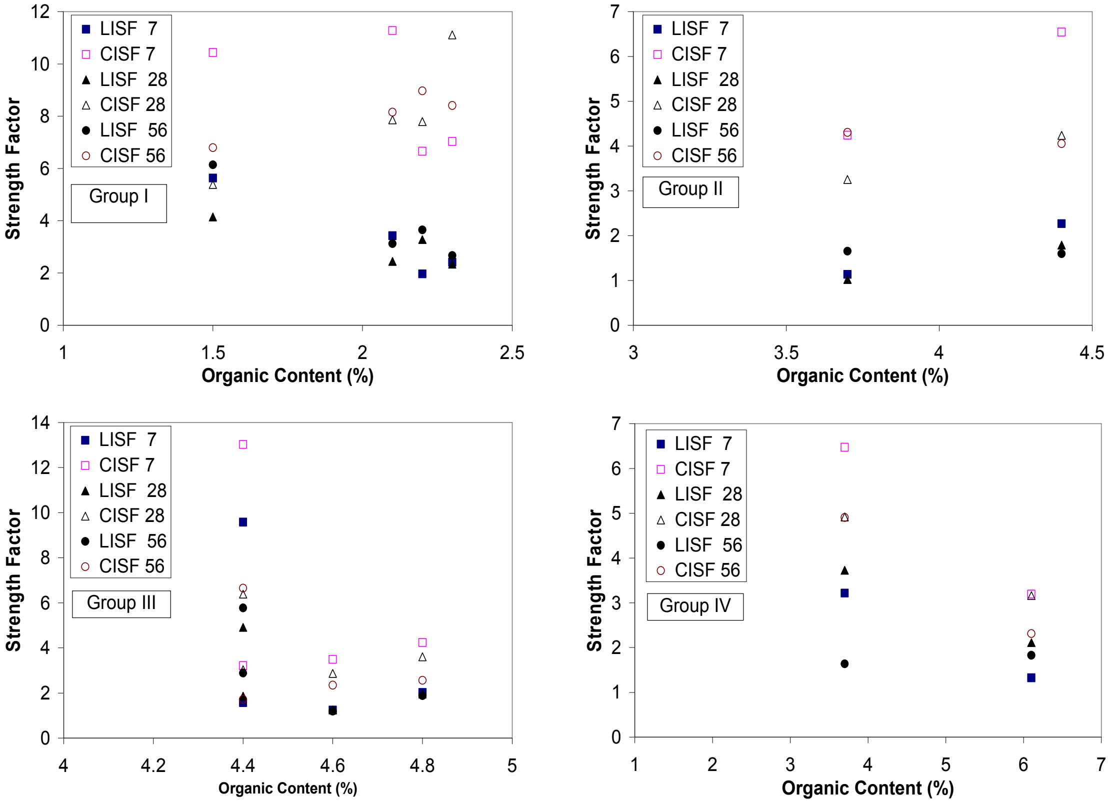


Figure 6.22. Variation of Strength Factors of Stabilized Samples at Intervals of 7, 28, and 56 Days with Organic Content (a), (b), (c), (d).

SUMMARY AND CONCLUSIONS

We collected soil samples from 12 locations at six sites that were thoroughly characterized as the field soils. An experimental program was designed to perform various basic and engineering tests for both natural and stabilized soils. The natural soil is stabilized with optimum lime and cement content. The optimum lime and cement contents were obtained by using the Eades and Grim procedure and a 7-day curing method, respectively. Experimental test results were analyzed to assess the effects of organic content on basic and engineering properties of natural (unstabilized) and stabilized soils.

The following major conclusions can be made from the analysis of laboratory test results:

1. The organic content of all the control soils varied from 1.5 percent to 6.1 percent. The maximum and minimum percentage of 6.1 percent and 1.5 percent was measured in BI45T1T and CCI37 soil samples, respectively. Therefore, soils are classified as organic soils.

2. The specific gravity of soils varied from 2.37 to 2.62. These specific gravity values are low when compared to the inorganic soils. These low values are attributed to the amount of lightweight organic content present in the soils.

3. The plasticity index of the control soils varied from 5 to 38. The plasticity index of the soil is increased with an increase in the organic content. Reduction of plasticity index is low for high organic soils and high for low organic soils when stabilized with lime or cement.

4. The optimum moisture content of soils increases with an increase in the organic content. This increase can be explained from the fact that the organic matter has high water retention capacity. The dry unit weight of the soil decreases with an increase in organic content. This decrease is due to the fact that the increase in organic material decreases the soil unit weight.

5. Organic content has moderate influence on the UCS of the present control soils. This influence is expected as the strength of the soil is dependent of soil type and clay mineralogy. The unconfined compressive strength of the soil is reduced after 56 days of curing, when it is stabilized with lime and cement. This reduction could be due to the following reasons:

    - moderate reduction of pH in the stabilized soils as the curing period progresses, or
    - formation of inorganic calcium humic acid when calcium present in the stabilizer is reacted with humic acid present in the soil.

6. The strength improvement factor of the soil is decreasing with an increase in the organic content, when stabilized with lime or cement.

7. One-dimensional free swell strains of all the soils varied from 2.5 percent to 31.2 percent. The vertical free swell strain increased with an increase in the organic content due to more water retention capacity of the organics.

8. Linear shrinkage strain values of all control and stabilized soils varied between 0.3 percent and 16.5 percent. Linear shrinkage strain values increased with an increase in the organic content. In stabilized soils, cement-stabilized soils exhibited low linear shrinkage strains, when compared to lime-stabilized soils.

CHAPTER 7

CONCLUSIONS AND RECOMMENDATIONS

SURVEYS

The researchers prepared a short survey to measure the extent of problems experienced by TxDOT districts when organic-rich soils are encountered. Sixteen districts responded to this survey, which acccount for 64 percent of the total number of districts present in Texas. From the responses received:

- Organics in the subgrade are encountered by seven districts, which accounts for 43 percent of the total number of districts that responded to the survey.
- Five of the districts that have encountered organic soils have used stabilization.
- Lime is used as a stabilizer by four of the districts that have implemented stabilization in organic-rich subgrade soils.
- Three of the districts that have stabilized organic-rich subgrades report problems after stabilization. So, the effects of organics on stabilized soils are the focus of this research.

LABORATORY MEASUREMENT OF ORGANIC MATTER

The results obtained from the ASTM ignition oven method and the C-H-N analyzer technique for measuring organics in soils show that both techniques are sensitive to changes in the level of organic matter present, and both techniques can at times be impacted by mineralogy. In particular:

- The presence of 2:1 clays such as smectite tends to inflate the result from the ignition oven method and, at times, can result in a reduction in the result from the C-H-N.
- To better discern between low-level changes in the test result, the number of replicates should be increased to three for both techniques. Performing three replicates would enable discernment in mean percent loss of 0.2 for the ASTM method and approximately 0.25 percent organic matter for the C-H-N method.

FIELD MEASUREMENT OF ORGANIC MATTER

The researchers developed a method to measure organic matter in soil which uses a UV-Vis spectrophotometer to measure the humic acid extracted from the soil. Ease of execution, rapid analysis, and good accuracy of the proposed method makes it an attractive alternative for both field and laboratory use. Although not as accurate as quantitative methods, the proposed method would be suitable for quick analysis of a large number of samples where accuracy to within ±10 percent was not required. With good quality field spectrophotometers now readily available, the proposed method could be easily adopted for field use.

Requirements for field use include:

- Substituting weighing of soil samples with using a scoop,

- Making corrections for soil moisture effects, if necessary. We used only air-dried samples in this study.
- Our analyses were made at room temperature, it is not known how temperature may affect predictions in the field.

The ratio of humic to non-humic substances present in the organic fraction of the soil is likely to be the major limitation to applicability of the proposed method. The method assumes that humic substances are the dominant organic fraction present in soils and therefore can be used as a proxy for total soil organic carbon. This may be true for most soils since non-humics are usually rapidly biodegraded (Schnitzer, 1982). However, for soils containing freshly deposited organic matter that contains a significant non-humic fraction, the method will likely underestimate soil organic carbon content.

LAB TESTING OF ORGANIC MATTER IMPACT ON STABILIZATION

The researchers created manufactured soils to assess the impact of known organic constituents on the ability of calcium-based stabilizers (lime) to effectively alter clay soils and improve the engineering properties of the soil.

Analysis of the data from this task indicate that:

- DSC provides a useful tool for evaluating the degree of lime stabilization in soils, as well as the potential impact of soil organic matter content on the stabilization process.

- Organic matter content over 1 percent (in the form of humic acid) influences lime stabilization of soils by inhibiting the formation of stabilization products. Negative impacts on lime stabilization increase with organic matter content.

- The type of clay present (Na-smectite, Ca-smectite, kaolinite) in the soil will influence the degree of stabilization and the impact of organic matter content on the stabilization process. Calcareous soils ($pH \geq 8$) will respond better to lime stabilization than acid soils with equal amounts of OM.

- Humic acid contents higher than 1 percent will prevent or retard formation of the pozzolanic reaction products responsible for long-term strength gain.

- The pH as measured in the Eades and Grim procedure apparently is not affected with increasing humic acid concentrations.

FIELD EVALUATIONS OF TXDOT PROJECTS

We collected soil samples from 12 locations at six sites that were thoroughly characterized as the field soils. An experimental program was designed to perform various basic and engineering tests for both natural and stabilized soils. The natural soil is stabilized with optimum lime and cement content. The optimum lime and cement contents were obtained by using the Eades and Grim procedure and a 7-day curing method, respectively. Experimental test results were analyzed to assess the effects of organic content on basic and engineering properties of natural (unstabilized) and stabilized soils.

The following major conclusions can be made from the laboratory analysis of these field sites:

1. The organic content of all the control soils varied from 1.5 percent to 6.1 percent. The maximum and minimum percentage of 6.1 percent and 1.5 percent was measured in BI45T1T and CCI37 soil samples, respectively. Therefore, soils are classified as organic soils.

2. The specific gravity of soils varied from 2.37 to 2.62. These specific gravity values are low when compared to the inorganic soils. These low values are attributed to the amount of lightweight organic content present in the soils.

3. The plasticity index of the control soils varied from 5 to 38. The plasticity index of the soil is increased with an increase in the organic content. Reduction of plasticity index is low for high organic soils and high for low organic soils when stabilized with lime or cement.

4. The optimum moisture content of soils increases with an increase in the organic content. This increase can be explained from the fact that the organic matter has high water retention capacity. The dry unit weight of the soil decreases with an increase in organic content. This decrease is due to the fact that the increase in organic material decreases the soil unit weight.

5 Organic content has moderate influence on the UCS of the present control soils. This influence is expected as the strength of the soil is dependent of soil type and clay mineralogy. The unconfined compressive strength of the soil is reduced after 56 days of curing, when it is stabilized with lime and cement. This reduction could be due to the following reasons:

    - moderate reduction of pH in the stabilized soils as the curing period progresses, or
    - formation of inorganic calcium humic acid when calcium present in the stabilizer is reacted with humic acid present in the soil.

6. The strength improvement factor of the soil is decreasing with an increase in the organic content, when stabilized with lime or cement.

7. One-dimensional free swell strains of all the soils varied from 2.5 percent to 31.2 percent. The vertical free swell strain increased with an increase in the organic content due to more water retention capacity of the organics.

8. Linear shrinkage strain values of all control and stabilized soils varied between 0.3 percent and 16.5 percent. Linear shrinkage strain values increased with an increase in the

organic content. In stabilized soils, cement-stabilized soils exhibited low linear shrinkage strains, when compared to lime-stabilized soils.

IMPLEMENTATION RECOMMENDATIONS

The researchers would like to purchase three portable UV-Vis systems and deliver them to TxDOT so they can do testing for organics. We would also provide training to the district and construction division personnel on the use of the UV-Vis instruments for measuring organic matter.

We would also supply 20 soil samples, with concentrations of organic matter ranging from 0 to 5 percent, to each laboratory receiving the UV-Vis instruments. Each lab should analyze each sample in triplicate for a total of 60 analyses and send the results back to TTI for determination of repeatability and reproducibility.

We would also continue to assist in the evaluation of field sites determined by TxDOT to have high organic contents. We would run the DSC on lime-treated samples to determine if enough stabilizer has been added to effectively treat the soil.

Additionally, we would develop a series of lab experiments to evaluate the failure mechanisms of stabilized organic rich soils. Strength tests will be conducted and permanency of stabilization measured with the DSC. Once this task is complete, TTI will a) recommend methods to effectively treat the organic rich soils and b) set target (risk) levels where calcium stabilization is not recommended.

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APPENDIX

SURVEY QUESTIONNAIRE

As part of Project 0-5540, “Mitigating the Effects of Organics in Stabilized Soils,” the researchers have prepared a short survey to measure the extent of problems experienced by TxDOT when organic-rich soils are encountered. We would very much appreciate you taking a few minutes to complete this survey.

Organic-rich soils typically have a strong odor similar to the smell of sewage and are often dark in color. These soils typically do not respond well to calcium-based stabilizers (lime and cement) and may have a very low pH. Problems that typically occur are loss of strength over time, loss of stabilizer over time, or large amounts of stabilizer may be required to achieve acceptable strength/swell reduction. The image below shows a soil with a dark, organic-rich horizon in the center of the image.

Figure A1 . Organic-Rich Zone in Subgrade Soil.

"Districts Survey on Organics for TxDOT Project 0-5540"

NAME: District:

Please click or check (with X) the following questions. We thank you in advance for your input.

1. Have you encountered any organics in subgrade soils from your district?

   ☐ Yes, on a regular basis ☐ Yes, but occasionally ☐ No

   If the answer to the above question is NO, please move forward to Question No. 10.

2. In your opinion, how do you assess problems due to organic-rich subgrades in your district?

   ☐ Major ☐ Considerable, but not a major one ☐ Occasional ☐ Not a problem

3. What laboratory method have you followed for the measurement of organics?

   ☐ Loss of Ignition (TxDOT Procedure) ☐ ASTM Method
   ☐ TAMU Soil & Crop Sciences Elementar Analyzer Method
   ☐ Other, specify_____________

4. At what depths you have encountered the organics? (If necessary, please check more than 1.)

   ☐ Less than 1 ft ☐ Between 1 and 3 ft ☐ Between 3 and 5 ft
   ☐ Between 3 and 5 ft

5. Have you implemented any stabilization on the organic-rich subgrades?

   ☐ YES ☐ NO

6. If chemical modification is the choice, please check the stabilizer that has been used:

   ☐ Cement ☐ Lime ☐ Fly Ash ☐ Others

7. Which soil property was the major focus of your subgrade design?

   ☐ Strength ☐ Stiffness or Resilient Modulus ☐ Swell and Shrink Changes

8. If any other modification or replacement of organics is implemented, please list the method followed:

   ____

9. Have you experienced any major distress on stabilized organic-rich subgrades?

☐ Poor Strength Gain ☐ High Leaching of Chemical Stabilizer
☐ Rutting ☐ Others

10. We would like to contact you if we have any follow-up questions. Please list your email or phone number where we can reach you.

Email: Tel:

We thank you very much for your input. Please send your survey response to the email address of anand@uta.edu, or fax it to 817-272-2630, or mail it to: Anand J. Puppala, PhD, PE, Professor, Box 19308, Department of Civil and Environmental Engineering, The University of Texas at Arlington, Arlington, TX 76019, USA.