

LABORATORY AND FIELD STUDY OF A LIQUID IONIC SOIL STABILIZER

by

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Abstract

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Chemical stabilization of expansive soil has been practiced for quite a time now. The use of lime/cement stabilization as a traditional method has been well acknowledged, understood and standard guidelines for practice have been developed. However, owing to certain demerits like high production and application cost, environmental impact, durability issues and most importantly incompatibility with high-sulfate soil leading to excessive heaving and swell, there is need to develop alternatives to these stabilizers. Non-traditional stabilizers like ionic soil stabilizer have been used by manufacturers in the past and claimed to effectively reduce the swell-shrink behavior of expansive soil. Undocumented results, the absence of laboratory tests, proprietary issues, however, have forced engineers to be reluctant about its use and accept it as suitable options to existing stabilizers.

This study has focused on meeting existing shortcomings of the ionic stabilizers by studying their effect on expansive soil both in the laboratory and field. For this purpose, an ionic soil stabilizer was selected from the available commercial products in the market. The stabilizer was used to treat the expansive soil from Carrollton, Texas. The stabilizer was also used to treat a site in Burleson, Texas. The laboratory study focused on observing the effect of treatment on swell potential and strength of the soil at different application

rates (1:150 and 1:300, volume of chemical to volume of water ratio) and curing days and validating the results through micro analysis of the soil. The laboratory tests include basic soil physical property and mechanical property tests such as Atterberg Limits test, bar linear shrinkage test, hydrometer test, standard compaction test, 1-D swell, and unconfined compressive strength test. Soil mineralogy tests including cation exchange capacity and specific surface area were performed to determine soil mineralogy behaviors. In addition micro tests such as Scanning Electron Microscope (SEM) imaging, energy dispersive spectrometer tests were performed to determine micro structure and elemental behavior of both treated and untreated soils. Further studies were conducted on the samples treated in the field as well.

With Carrollton soil, it is found the standard compaction curve is altered for the treated soil. The optimum moisture content increases while the maximum dry density decreases. For treated soil compacted at the optimum moisture content and maximum dry density of the treated soil, more than 50% of swell reduction is observed and the UCS of the treated soil slightly decreases. For Burleson soil, the standard compaction curve of the treated soil is similar to the one of untreated soil. The ionic soil stabilizer is found to successfully reduce the swelling potential and PVR of the active zone of expansive soil generally extending up to 10 feet in field treatment. In the field, the soil is usually wetted to near saturation with the liquid stabilizer. The application mass ratio which is the amount of stabilizer available for soil solids is relatively higher because of the treatment method used in the field. The tests in the laboratory were done at the moisture content near to optimum. In the light that improvements were observed with the Burleson soil, the stabilizer shows a potential in expansive soil stabilization. There are some discrepancies in findings from the lab and the field which can be ruled out in the future with the development of techniques to closely simulate the field condition. Ineffectiveness of stabilizer on Carrollton soil at given

test conditions might be an indication that stabilizers work under certain specified conditions only and concludes that pre-study of the suitability of stabilizer is essential. Nevertheless, incorporating all the shortcomings in the current study, ionic stabilizers can have a good potential in the future for stabilization of expansive soil.

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Chapter 1

Introduction

1.1 Introduction

Expansive soil poses great challenges to geotechnical engineers. The property of expansive soil to expand and shrink with moisture variation makes it a prime concern in the field of transportation and foundation work. The shrinkage after the expansion is not a reversible process (Holtz & Kovacs, 1981). The volume change thus causes permanent damage to the structure. The damage might be severe in many cases. The problematic nature of the expansive soil is attributed to the presence of some minerals which cause the soil to expand with moisture content variation. The expansive mineral called montmorillonite is highly sensitive to moisture change and swell and shrink tremendously under such situations. Expansive soil is predominant in the United States. Every year the government must spend billions of dollars to damaged homes and pavements built on expansive soils (Jones and Jones, 1987). Unfortunately, the treatment of expansive soil is not as easy as it seems.

Texas is one of the states in the United States plagued with problems of expansive soil. The frequency of expansive soil in Texas can be seen in figure 1-1. Severe weather condition aid to the swell-shrink phenomenon of the soil. TxDOT has reported a number of pavement failures constructed on expansive soil. Not only pavement but foundations of buildings are also affected by the swell-shrink movement of expansive soil. The difference in movement at edges and center of the soil under foundation because of variation in moisture content results in differential movement of the foundation leading to the failure.

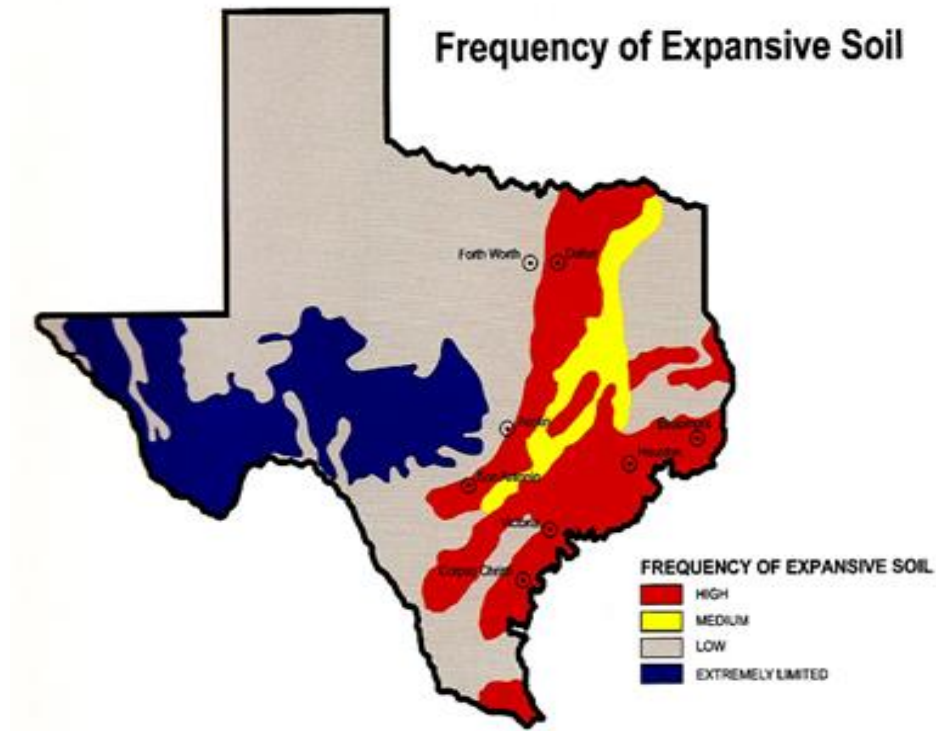


Figure 1-1 Frequency of expansive soil in Texas

(Source: Stratum Foundation Repair)

Stabilization of expansive soil has been a topic of discussion for many years now. Many researchers in the past have introduced several methods of soil stabilization. Mechanical stabilization by surcharge loading, replacement of weak soil with firm compacted soil, prewetting, lime/cement stabilization, is some of the methods used for stabilization of expansive soil. When it comes to chemical stabilization, lime/cement stabilization are often pronounced. However, many Department of Transportations (DOTs) in the US have reported the failure of pavement propagating from subgrade even after stabilization. There are some limitations of traditional stabilization of expansive soil. Lime stabilization is not always preferred especially when the soil encountered are high sulfate soil.

With the existing shortcomings of the traditional stabilizers, there is a need to develop new methods of stabilization for those soil which do not comply with the traditional stabilizers. Non-traditional stabilizers include ionic stabilizers, enzyme stabilizers, polymer stabilizers. They have been applied in the field in the past and have been claimed to be successful by the companies that produce them. However, there is no clear understanding of how these chemicals work, and although manufacturers claim that the chemicals are effective in stabilizing soil, lack of documentation and scientific procedures of application and evaluation of effectiveness makes it unreliable and not-a-potential option when it comes to making the decision for engineers.

As of now, there is a need to evaluate the efficiency of these stabilizers and document the findings in a scientific and logical way so that the effects of these stabilizers can be compared with other stabilizers on a one-to-one basis, listing all the pros and cons. If these stabilizers can work as claimed by the manufacturers, then there is a possibility of replacement of traditional stabilizers with the new stabilizers in the future which will be more economical and environment-friendly. For this to happen, there is a need for continuous research, experiment and modification of the stabilizers as required by the field needs.

1.2 Problem Statement

Liquid ionic stabilizers are non-traditional stabilizers which have been used in the stabilization of expansive soil in different parts of Texas. The stabilizer consists of sulfuric acid, phosphoric acid, and citric acid and has been effectively used in reducing the swell potential of expansive foundation soil in residential building. However, there is a dearth of supporting documents which can validate its efficacies in soil stabilization. The results are limited to field observations which are often not documented and laboratory tests that can validate the results obtained in the field have not been performed. Hence, the current study

focuses on the evaluation of the stabilization effect of ionic stabilizer liquid on expansive soil from Carrollton, Texas and study the field treatment in Burleson, Texas integrating with laboratory studies.

1.3 Research Objective

The main objective of this research is to evaluate the stabilizing effect of liquid ionic stabilizer available as a commercial product in the market in reducing swell and strengthening the soil and validate the results through the study of microscopic aspects of stabilization. The specific objective can be listed as follows:

- Conduct laboratory tests on control and stabilized soil to monitor the performance of stabilized soil compared to control soil.
- Select a suitable dose of stabilizer based on the performance of the stabilized soil.
- Validate the results from soil test in the laboratory through microanalysis of soil.

1.4 Organization of Chapters

This thesis consists of five chapters. Chapter 1 is the introduction which introduces the topic, explains about research objectives and outlines the chapters of the thesis.

Chapter 2 is the literature review. This chapter reviews some of the literature on expansive soil. A brief introduction of clay minerals and structure is presented along with stabilization of expansive soils using ionic stabilizers. It also presents a review of literature for sample preparation for electronic imaging and microanalysis of soil.

Chapter 3 is the methodology section which presents the methods of performing laboratory tests. It explains in detail how the test is performed, the conformity of test with the standards, deviation from standards and the test methodology from past literature.

Chapter 4 is the result and analysis section. This chapter presents all the results obtained from the laboratory, compares the result and presents the finding through illustrations and explanations.

Chapter 5 presents the case study of one of the sites. It presents the result obtained from the treatment of soil in the field and compares the results obtained with the laboratory treatment method.

Finally, chapter 6 is the summary and conclusion section which summarizes all the results and provides conclusions based on the observations made in the laboratory. It also provides recommendations for future studies.

Chapter 2

Literature Review

2.1 Introduction to Expansive Soil

Expansive soils are one of the problematic soils especially when it comes to geotechnical engineering. The soils exhibit moderate to high plasticity, low to moderate strength and high swell and shrinkage behavior with the fluctuation of the moisture content (Holtz & Gibbs, 1956). As the soil water system changes, the internal stress equilibrium is disturbed. The disturbance results in redistribution of the forces so that the system comes to a new equilibrium position which results in the expansion of soil (Nelson & Miller, 1992). The main reason for these type of behavior in the soil is the presence of a mineral called montmorillonite which has an expanding lattice. Expansive soils are found in all the continents except perhaps Antarctica (Nelson et al., 2015). Soils rich in these types of minerals are especially found in arid and semi-arid regions (Elarabi, 2002). Arid and semi-arid regions are the places often characterized by the precipitation lower than the evapotranspiration. Expansive soil cover one-third of the Earth's surface (Chen, 1988) especially in the arid and semi-arid regions where precipitation is lower than the evapotranspiration (Jones & Holtz, 1973). One-fourth of the United States is covered with expansive soil (Buhler & Cerato, 2007). Mostly the western and Midwestern part of United States is plagued with major concern of expansive foundation soil problems (Nelson & Miller, 1992).

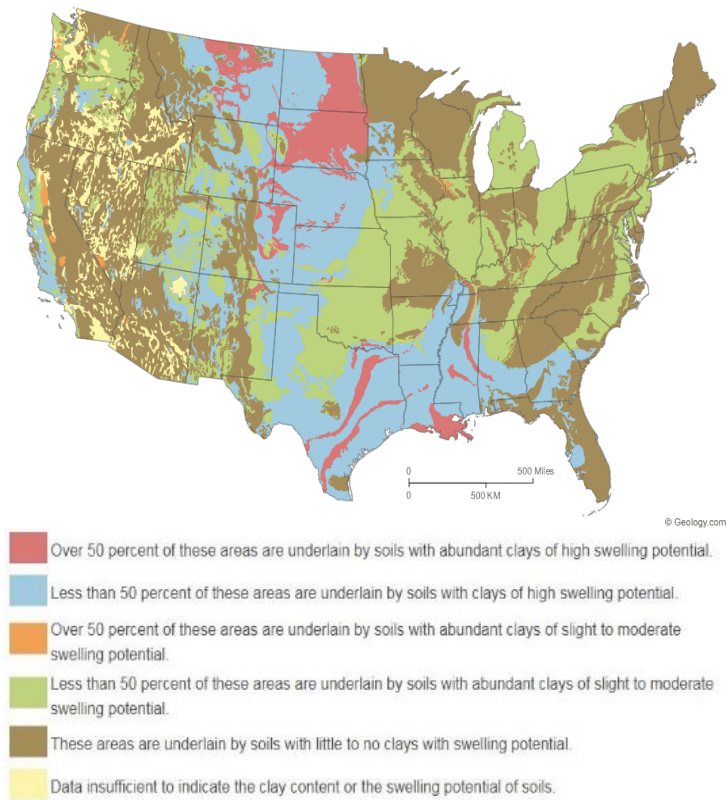


Figure 2-1 Expansive Soil Distribution over the United States (USGS Publication)

2.2 Problems Caused by Expansive Soils

With increased urbanization and population growth, construction over expansive soil increases (Williams, 2003). The expansive soils, however, are considered very poorly suitable for urban uses. The swelling and shrinkage of expansive soil cause damage to the infrastructure built on them. Swelling of the expansive clays can generate a pressure up to 273 metric tons/m² resulting damage to the foundations, pipelines, and pavements (Chen, 1988). The damage caused by expansive soil to the structures, particularly light buildings and pavements is more than the damage caused by any other natural hazards, including earthquakes and floods (Jones & Holtz, 1973). In arid and semi-arid regions, soils with even moderated expansiveness can cause significant damage (Jones & Jefferson, 2012).

According to the American Society of Civil Engineers, one in four homes have some damage caused by expansive soils (Jones & Jefferson, 2012). The damage mostly includes foundation crack and differential settlement. If the foundations are built on soil with montmorillonite which has very high swell-shrink potential, the frequency of damage is over three times compared to the foundations built on sandy loam soils with moderate shrink-swell potential (Hudak, 1998).

Apart from foundations, a number of failures of the pavement built on expansive soil has also been reported in the past. The shrinkage of expansive soil owing to the drying following the dry season causes the cracks to propagate through the pavements in different directions. Heaving due to expansion causes another vital issue in pavement causing discomfort in riding and reducing the serviceability of the pavement. Swelling and shrinkage are not fully reversible (Holtz & Kovacs, 1981). As a result, cracks formed during shrinkage do not close-up perfectly. The cracks are further worsened by the percolation of water through these channels.



(a)

(b)

Figure 2-2 Cracks caused by expansive soil on (a) Pavement (b) Foundation

2.3 Cost of Damage Caused by Expansive Soil

Loss caused by expansive soil on residential buildings in the United States was \$798.1 million/year in 1970 (Wiggens et al., 1978); (Petak et al., 1978). Total cost including other commercial/industrial buildings and transportation facilities was about 2 to 3 times this value. Estimated average annual loss due to shrink-swell phenomenon reached \$9 billion by 1981 (Jones, 1981). The swell-shrink damage is particularly severe in Texas, Wyoming, and Colorado as the soils have much montmorillonite. Around the 1970s, the total cost of pavement maintenance due to expansive soil was from six to ten million dollars a year in Texas (Center for Highway Research, 1971). Another case study from Tulsa, Oklahoma shows that shrinkage damage due to the drought of 1980 was so severe that relief fund was made available through Small Business Administration (Nelson & Miller, 1992). All the studies show that the country's economy is severely affected by the problems due to expansive soil. However, if the initial stabilization is performed before construction, it is more economical than carrying out the remedial treatments in the existing structures at a later stage (Petry & Armstrong, 1989).

2.4 Mechanism of Swell/Shrink in Expansive Soil

Clay particles generally are platelets having negative electrical charges on their surfaces and positively charged edges. Cations in the soil water that are attached to the surfaces of the platelets by electrical forces balance the negative charge. The internal electrical force system which is the function of both negative surface charges and the electrochemistry of the soil water must be in equilibrium with the externally applied force and matric suction. Any change in soil water chemistry or the amount of water or chemical composition results in change in interparticle force field which if not balanced by externally applied stress, the force system achieves new equilibrium by adjusting the particle spacing. Ultimately swelling and shrinkage occur.

Some theories for explanation of volume change in expansive soil are explained in Mitchell and Soga 2005. Two important theories are osmosis theory and water adsorption theory. According to osmosis theory, expansion occurs because of osmotic flow of water into the interspace of soil particles which is triggered by the concentration difference. Cations are strongly adsorbed to the clay surface to neutralize the electronegativity of clay particles. They are not free to diffuse. As the moisture content of the soil increases, concentration difference occurs between the solution surrounding the clay and the region in between the clay particles. The difference in concentration develops a pseudo semipermeable membrane and water diffuses into the region between the clay particle through osmosis across this pseudo semipermeable membrane as shown in the figure 2-3. The clay particles are pushed away increasing the interparticle spacing.

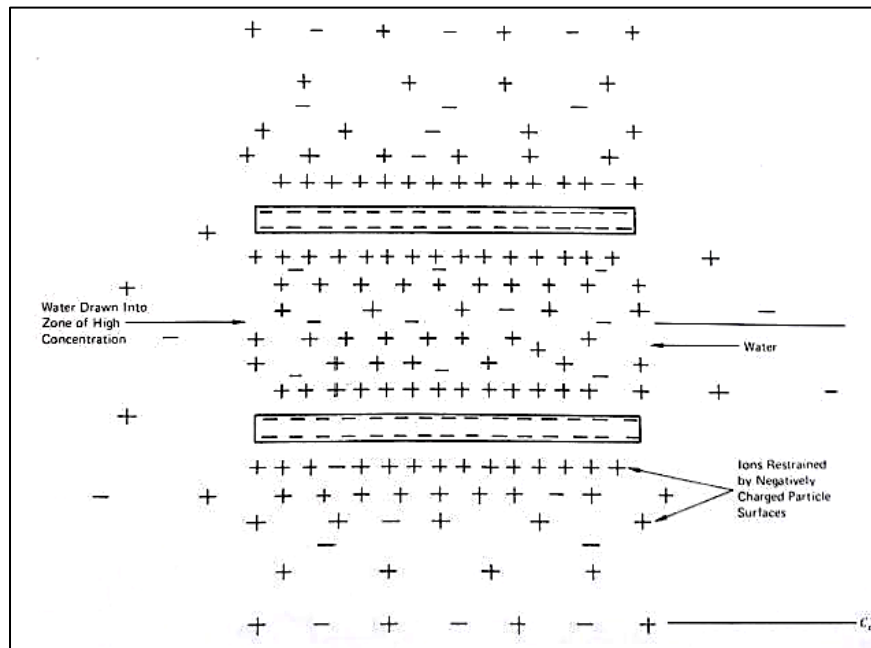


Figure 2-3 Mechanism of Swelling by Osmosis in Expansive Clay

(Source: Mitchel and Soga, 2005)

According to water adsorption theory, swelling occurs due to surface hydration (Low, 1987). As the water interacts with the clay surface, the chemical potential of the water is reduced. As a result, the gradient of chemical potential is created which causes further flow of additional water into the system until equilibrium is achieved.

2.5 Factors affecting Swell/Shrink behavior

Several factors affect the expansion of the soil. Three principal factors affecting the shrink-swell potential of soil are soil characteristics, environmental factors and state of stress (Nelson & Miller, 1992). If the soil swells by more than 6% volumetrically during wetting, it implies that the soil has high shrink-swell potential and if the soil swells by more than 9% volumetrically, it implies that the soil has very high shrink-swell potential (Ford & Pauls, 1980). Expansion up to 10% is usually common for many expansive soils (Chen, 1988) (Nelson & Miller, 1992).

Soil that exhibits plasticity over a wide range of moisture content and has higher liquid limit have great potential for swelling and shrinkage. Swelling potential is also dependent on the concentration of cation and cation valence. Increase in cation concentration and cation valence reduces potential for swelling. Replacement of Na^+ cations by Mg^+ cations result in less swelling (Mitchell, 1976).

Dry density of the soil also affects the shrink-swell potential of the clay — higher dry density results in closer spacing of the particles. As the particles come closer, greater repulsive forces are experienced by the particles which increase their swelling potential.

Stress history is another major factor affecting the swell-shrink potential of expansive soil. If two soils with same void ratio are compared, the expansion potential of the overconsolidated soil is larger than the normally consolidated soil. Swell pressure increases with the aging of the compacted clays (Mitchell, 1976).

2.6 Clay mineralogy

Soil mineralogy is an important topic in soil engineering. Studies by Teresa et al., 2004; Chittori and Puppala, 2011; Pedarla, 2013 have shown that mineralogy is one of the fundamental parameters governing the swell shrink behavior of any expansive soils. Mineralogy of the soil controls shape, size and properties (physical and chemical) of the soil particles. Generally, soils contain various amounts of crystalline non-clay and clay minerals, non-crystalline matter and precipitated salts (Mitchell & Soga, 2005). In engineering practice, crystalline non-clay minerals comprise the greatest constituent of the soil. Crystalline clay minerals are relatively low. However, their influence on the properties of soil is far more than abundance.

Minerals comprise more than half of the soil volume. The presence and absence of certain minerals explain the mechanical and chemical weathering that results in the formation of the soil (Schulze, 2002). As a mineral type, clay has small particle size, net negative electrical charge, plasticity when mixed with water and high weathering resistance (Mitchell & Soga, 2005). Clay minerals primarily consist of hydrous aluminum silicates and are generally platy and in some cases tubular or needle-shaped.

The primary molecular building blocks of clay are silicon tetrahedron and aluminum octahedron (Mitchell & Soga, 2005). Silicon tetrahedron joins only at their corners and sometimes not at all. The silica tetrahedral are connected in a sheet to form silica sheet in which three of the four oxygens are shared to form a hexagonal net the bases of which are all in the same plane. The same structure repeats indefinitely.

Octahedral sheets are formed by the octahedral coordination of oxygens or hydroxyls with magnesium or aluminum. Cations like Fe^{2+} , Fe^{3+} , Mn^{2+} , Ti^{4+} , Ni^{2+} etc. can also be available instead of Al^{2+} and Mg^{2+} . Depending upon the valency of the cations, dioctahedral or trioctahedral structure might be form. Divalent cations when octahedrally

coordinated occupy all possible cationic site and result in trioctahedral structure whereas if the trivalent cations are present, only two-thirds of the cationic sites are occupied and hence the structure is called dioctahedral structure.

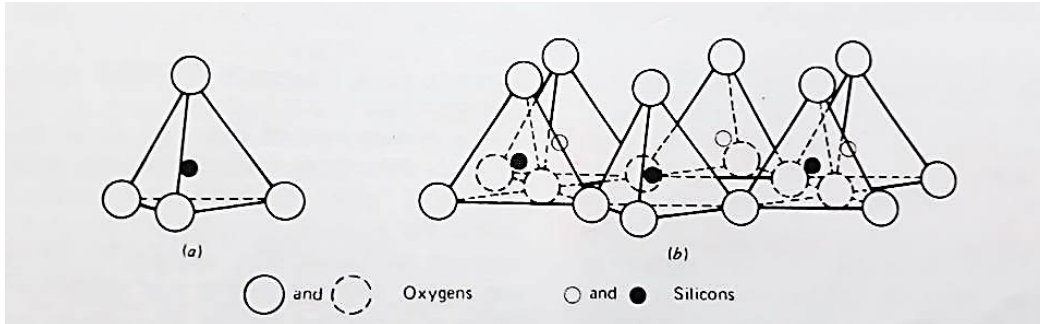


Figure 2-4 (a) Silicon tetrahedron (b) silica tetrahedra arrange in hexagonal network

(Source: Mitchell and Soga, 2005)

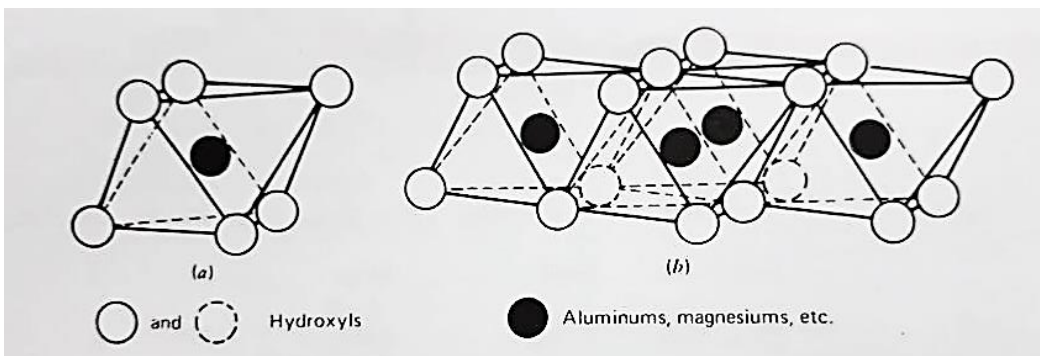


Figure 2-5 Octahedral unit and sheet structure of octahedral unit

(Source: Mitchell and Soga, 2005)

Expansion in soils is mainly caused by the clay minerals montmorillonites, vermiculites and some mixed layer minerals. Illites and kaolinites can also cause volume change when particle sizes are extremely fine (less than few-tenths of a micron) (Grim, 1968); (Mithcell & Raad, 1973); (Snethen et al., 1977).

When the Feldspar weathers, its mineral gets hydrolyzed into mineral Kaolinite and amorphous Silica (Mitchell, 1993). Kaolinite is rich in alumina whereas illite (which is

formed due to transformation of Kaolinite during transportation in streams and deposition) and smectite have increased percentage of silica. Illite is dominated by potassium (K) and smectite is rich in sodium or calcium.

2.6.1 Kaolinite

Kaolinite is a common phyllosilicate mineral. The kaolinite minerals are composed of alternating silica and octahedral sheets. Kaolinite is also called 1:1 mineral. It is found where precipitation is relatively high, and its formation is favored when silica is scarce, and alumina is abundant (Holtz & Kovacs, 1981).

As seen in the figure 2-6, the tip of the silicon tetrahedral point towards the center of the unit layer and are shared by one of the planes of octahedral sheet. In the common plane of tetrahedral and octahedral, two-third of the atoms are oxygen and the remaining are (OH) located directly below the hole of the hexagonal net.

The bonding between successive layers in Kaolinite is both by van der Waals forces and hydrogen bonds. As the bond is sufficiently strong, there is no any interlayer swelling in the presence of water as well has a low cation exchange capacity (White et al., 2002). The CEC usually range from 3 to 15 meq/100g for kaolinite (White et al., 2002). The specific surface area for Kaolinite ranges from 5 m²/gm to 39 m²/gm. Therefore, Kaolinite exhibits less plasticity and swelling potential compared to other minerals.

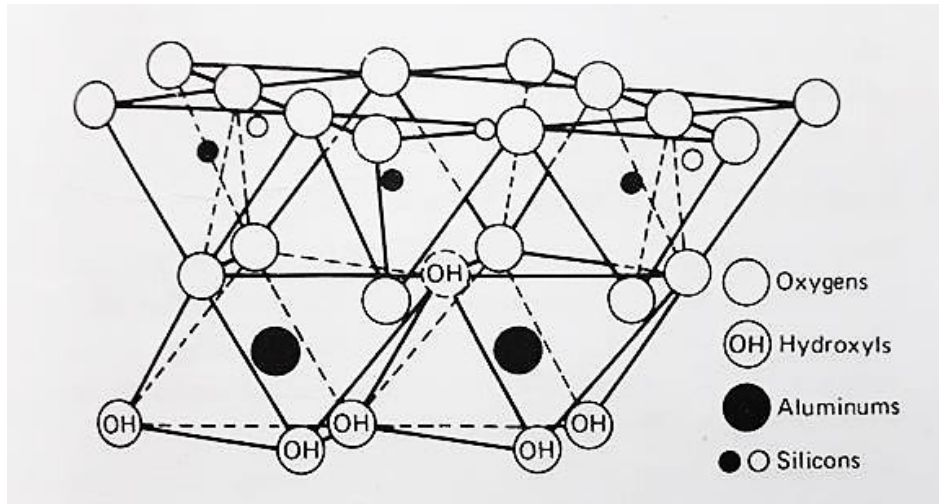


Figure 2-6 Mineral structure of Kaolinite

(Source: Mitchell & Soga, 2005)

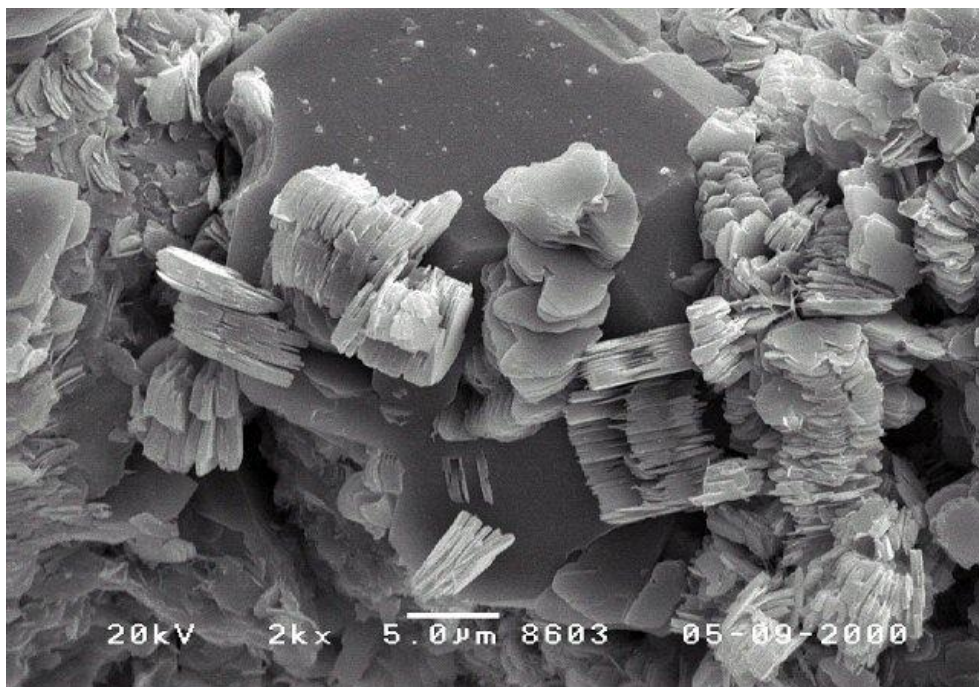


Figure 2-7 Kaolinite observed under Scanning Electron Microscope

(Source: OMNI Laboratories, Inc)

2.6.2 Montmorillonite

Montmorillonite is the most common mineral in the smectite group. It is formed due to weathering of volcanic ash under poor drainage or marine water. It is 2:1 layer silicate mineral. The interlayer of montmorillonite is expansible. During wetting and drying cycle montmorillonite containing soil can undergo up to 30% of volume change (Katz et al., 2001). Structurally, it consists of octahedral sheet sandwiched between silica sheet as shown schematically in the figure 2-8. The layers formed are stacked one above another which are bonded by weak van der Waals force and the cations that balance the charge deficiencies. As the bond is weak, it can be easily separated by adsorption of the water into the interlayer (Grim, 1968).

Various minerals can be formed by isomorphous substitution in smectite minerals. Montmorillonite are formed during dioctahedral substitution of aluminum. As there is large amount of unbalanced substitution, the cation exchange capacity of these minerals is high. The cation exchange capacity of montmorillonite is in the range of 47 m²/gm to 162 m²/gm. Specific surface area value ranges from 600 to 800 m²/gm. Montmorillonites are the most likely minerals to exhibit swelling. As these minerals have large specific surface area, the structure results in unsatisfied water adsorption forces at lower water content (Mitchell & Soga, 2005).

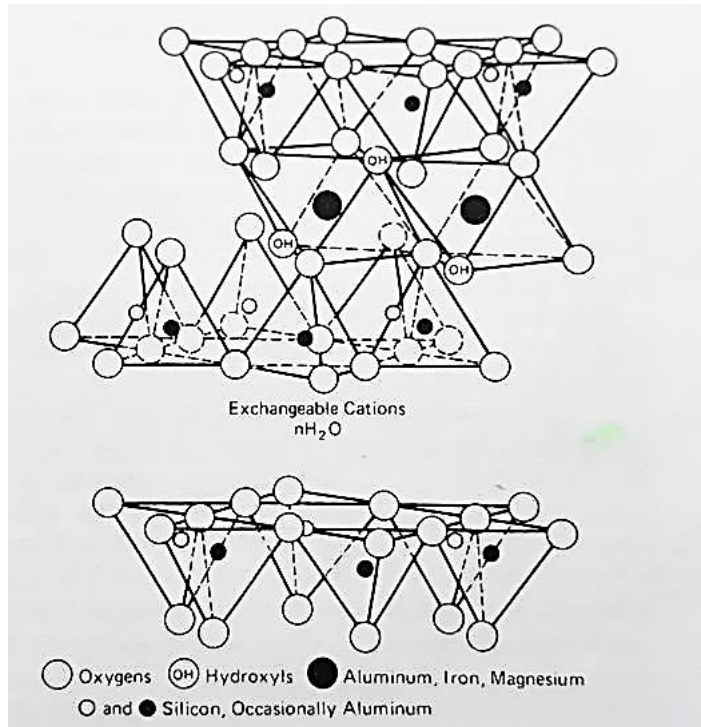


Figure 2-8 Mineral Structure of Montmorillonite

(Source: Mitchell and Soga, 2005)

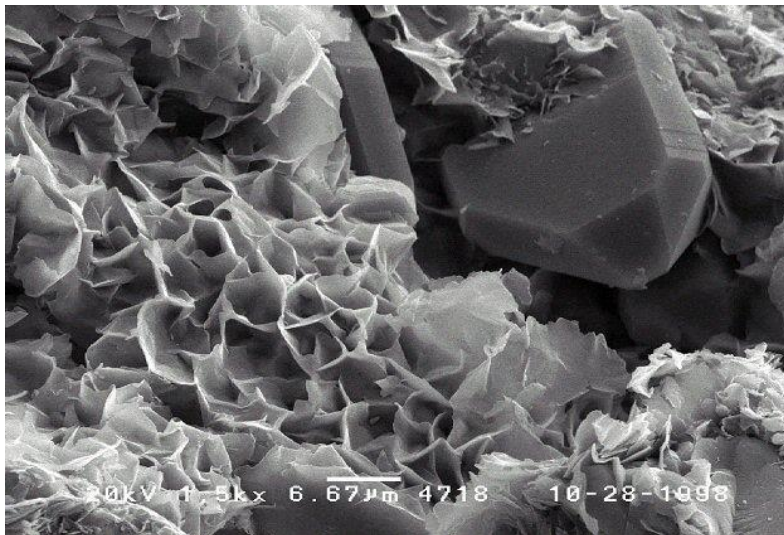


Figure 2-9 Montmorillonite observed under Scanning Electron Microscope

(Source: OMNI Laboratories, Inc)

2.6.3 Illite

Illite is the most commonly found mineral in soils in the engineering practices (Mitchell & Soga, 2005). It is a non-expanding clay mineral. Potassium is usually found as an interlayer cation in the illite which neutralizes the negative charge and makes the structure stable with bonding. Its formation is generally favored by alkaline environment and high aluminum and potassium.

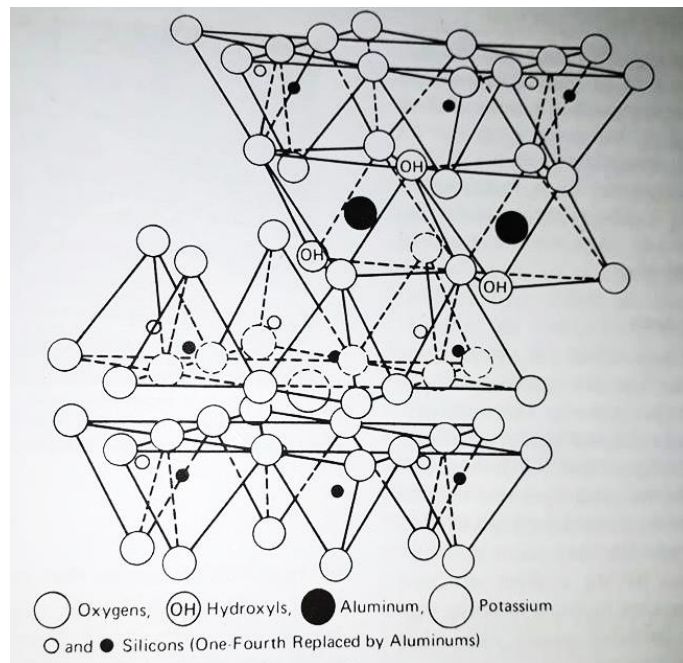


Figure 2-10 Mineral Structure of Illite

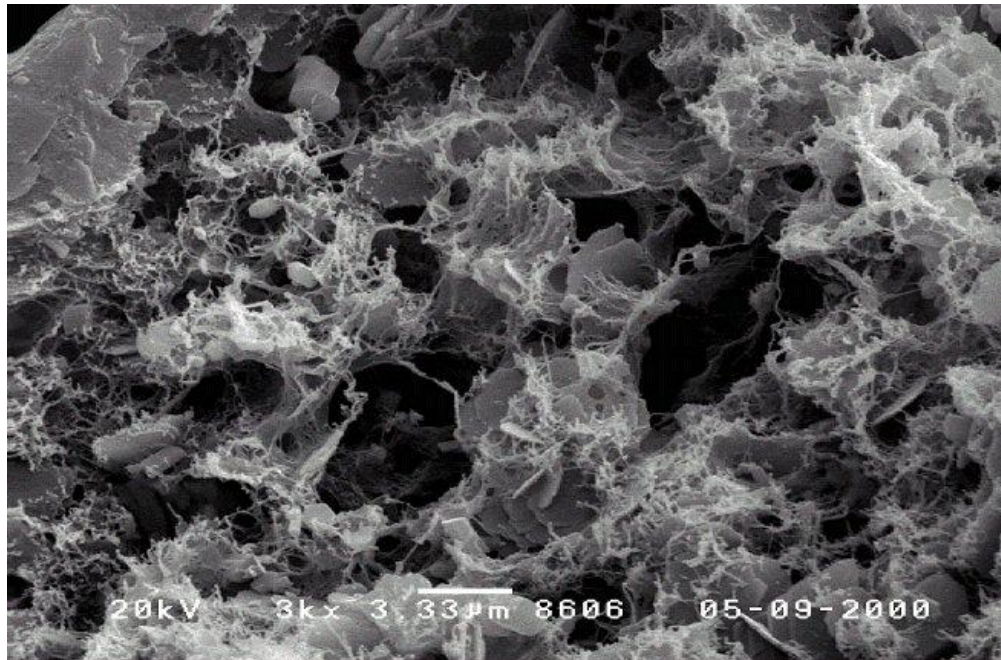


Figure 2-11 Illite observed under Scanning Electron Microscope

(Source: OMNI Laboratories, Inc)

Dominating clay minerals, montmorillonite, illite and kaolinite can be calculated for their percentage of presence in any expansive soil using relationships of soil properties like cation exchange capacity, specific surface area and total potassium.

2.7 Cation Exchange Capacity

Cation exchange capacity is the quantity of exchangeable cations required to balance the negative charge on the surface of the clay particles. It is the quantity of negative charges in soil prevailing on surfaces of clay and organic matter (Camberato, 2001). Especially in soil, the negative charges are imparted during isomorphous substitution within the structure of silicate mineral layers. Cation exchange capacity is expressed in milliequivalents per 100 grams of dry clay. It is a good indicator of soil reactivity. Swelling of expansive clay minerals in presence of water depends on the cationic environment of the soil (Mitchell & Soga, 2005). Examples include sodium and lithium

montmorillonite which undergo excessive swelling in comparison to divalent and trivalent forms of montmorillonite as the confining pressure is too small in the former case. Swell potential of the soil is also associated with increased cation exchange capacity of the soil. Presence of cations like Na^+ , K^+ , Mg^{++} , Ca^{++} creates increased water affinity causing swell.

2.8 Specific Surface Area

Specific surface area refers to the total surface area in a unit mass of soil. This is another important parameter of soil since various phenomenon in the soil are governed by its specific surface area. Higher specific area may imply high water holding capacity and greater swell potential. Moreover, the particle size distribution can give idea of the specific area of any soil. This was explained by Campbell, 2005 with a simple experiment where the cube when divided into smaller cubes resulted in higher specific area however the mass remained same, thus verifying the statement.

From among various approaches to determine specific surface area, the adsorption of ethylene glycol monomethyl ether (EGME) is the most commonly used method (Carter et al., 1986). In this two-day long test procedure, the soil sample is saturated and equilibrated in a vacuum over a CaCl_2 -EGME solvate. The weight of the sample at the point of equilibrium is noted. The specific surface area is obtained from the mass of retained EGME compared to the amount retained by pure Montmorillonite clay which is assumed to have a surface area of $810 \text{ m}^2/\text{g}$ (Carter et al., 1986). This method was identified applicable to a wide range of mineralogies with the capability of determining surface area ranging from 15 to $800 \text{ m}^2/\text{gm}$ by (Cerato & Lutenegeger, 2002) after fully evaluating it for geotechnical usage.

2.9 Total Potassium

The presence of mineral illite can be detected by measuring the amount of potassium ion in the soil. This is because illite is the only clay mineral to have potassium

in the structure (Mitchell & Soga, 2005). The method proposed by Knudsen et al. (1982) is one of the most common method for determining the potassium in soils. In this method, two acids (Hydrofluoric acid and Perchloric acid) is used to break the mineral structure and extract the potassium ions from the structure, the concentration of which is determined with the help of spectrophotometer or any other suitable device. Percentage of total potassium can also be found out through EDS result. EDS calculates the percentage of potassium in the given amount of soil relative to the presence of all other detected elements based on the intensity of the characteristic x-rays generated during transition of inner electron of an element.

2.10 Clay water interaction

Water molecules are strongly attracted to and are adsorbed to the clay surface. Low (1961) proposed several mechanisms for water adsorption to the clay surface. Some of the popular mechanisms of water adsorption are shown in figure 2-12. Water can interact with the clays through several possible mechanisms like hydrogen bonding, ion hydration, attraction by osmosis, dipole attraction etc.

Cations are tightly adsorbed to the clay surface usually to a distance of about 1 nm (equivalent to three molecular layers) from silicate layer surface (Sposito, 1989). They neutralize the electronegativity of the clay particles. Associated anions and excess cations are available as salt precipitates which go into solution in presence of water. The adsorbed cations try to diffuse away to equalize the concentrations throughout the pore fluid which is restricted by the negative electric field of the clay surface. A balance is achieved during this phenomenon which lead to the ion distribution adjacent to single clay particle in suspension forming diffused double layer in the system as shown in the figure 2-13. The interaction of water and clay results in the increase in the b dimension of the clay particle lattice resulting in the rearrangement of the clay and water structure.

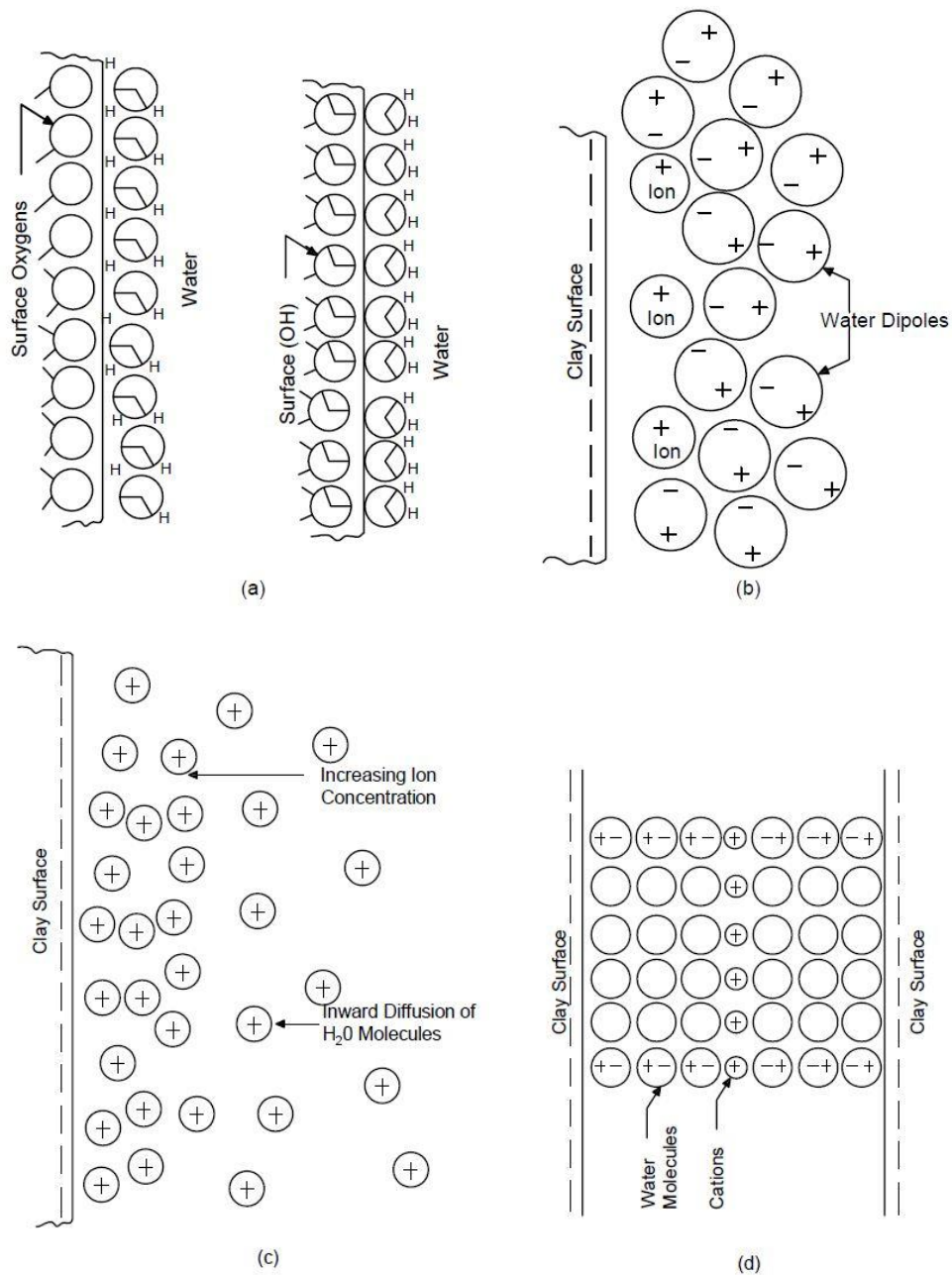


Figure 2-12 Water adsorption to clay by (a) Hydrogen Bonding (b) Ion Hydration (c) Attraction by Osmosis (d) Dipole Attraction

(Source: Mitchell and Soga, 2005)

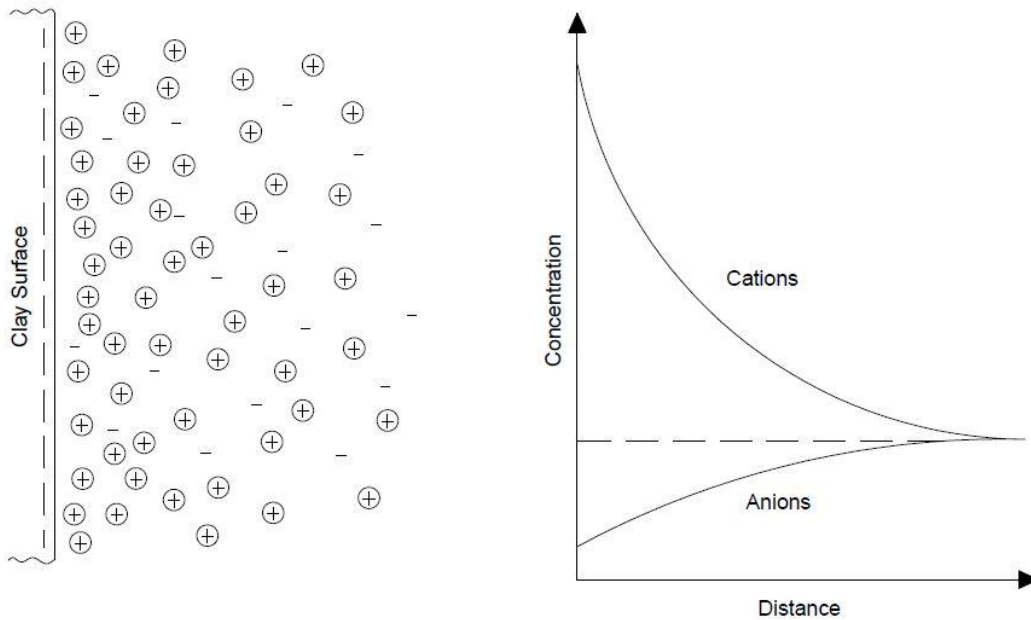


Figure 2-13 Formation of Double Diffusion Layer

2.11 Soil structure and fabric

The fundamentals of soil behavior, particularly the fine-grained soil is governed by the characteristics of the particles. The study of clay particles arrangement and their relationship to their mechanical behavior started after the microanalysis techniques such as SEM, XRD aided to the exploration around mid-1950s. The tendency of expansion is more in flocculated clay than in dispersed clay. Flocculation implies edge to edge (EE) or edge to face (EF) arrangement which have more voids and open channels. Compaction at higher water content and remolding alters fabric and structure. Kneading compaction results dispersed structures with low swell potential. than static compaction at lower moisture content (Johnson & Snethen, 1978); (Seed et al., 1962).

van Olphen (1977) illustrated various particle associations in clay suspension. The association can be face-to-face (aggregated), no face-to-face (dispersed), edge-to-edge or edge-to-face (flocculated) or no association (deflocculated). Face-to-face aggregated

fabric structure results in thicker and larger particles whereas flocculated association produces card house structures which are voluminous.

2.12 Micro Characterization of soil

Micro characterization of soil involves study of the internal arrangement, soil fabric, measurement of basal spacing, orientation of fabric, study of soil morphology etc. The importance of microstructures was recognized from early days and concepts of microstructures of soil were proposed by Terzaghi (1925), Casagrande (1932) as well as Lambe (1953 and 1958) & (Barden & Sides, 1971). Particle arrangements in soil were unknown until the techniques like x-ray diffraction and scanning electron microscopes made it possible around mid-1950s (Mitchell & Soga, 2005). The shape and size of particles, their arrangements and interparticle forces determine control the properties of the soil like compressibility, permeability, strength etc. These factors are even more important in deciding the properties of fine-grained soils. Micromechanics theories relate the microstructure to macroscopic behavior which were developed in the 1970s and 1980s. Even though sufficient theories to correlate all the microscopic behavior to macroscale properties are not available at the present scenario, the studies are ongoing and hopefully in the future it might be possible to predict the mechanical behavior of soil masses in terms of characteristics of the particles they are composed of.

2.12.1 X-ray Diffraction

X-ray diffraction is one of the most widely used techniques for identification of fine-grained soil minerals. The modern story of soil material and clay mineralogy discipline was commenced after crystalline nature of the soil minerals were confirmed by X-ray diffraction in two independent studies by Hendricks & Fry (1930) and Kelley et al. (1931) (Grim, 1988). Clay minerals are crystalline in nature. This property makes them possible to be identified under X-ray through diffraction by the planes separated by certain distance. The spacing

of the interatomic planes is unique to each mineral which acts as characteristic fingerprint for mineral identification. The 001 spacing is characteristic for each clay mineral group. X-ray diffraction provides a measure of the characteristic basal spacing, which describes the thickness and spacing of the individual bonded layers. The intensity varies according to the density of the atomic packing and other factors. Non-crystalline or amorphous minerals are identified using vibration spectroscopy or X-ray absorption (Whittig & Allardice, 1986). The basal spacing of crystalline clay minerals can be calculated following Bragg's law. The diffraction angle θ can be used to find the interplanar d spacing using the following relation.

$$n\lambda = 2d_{hkl}\sin\theta$$

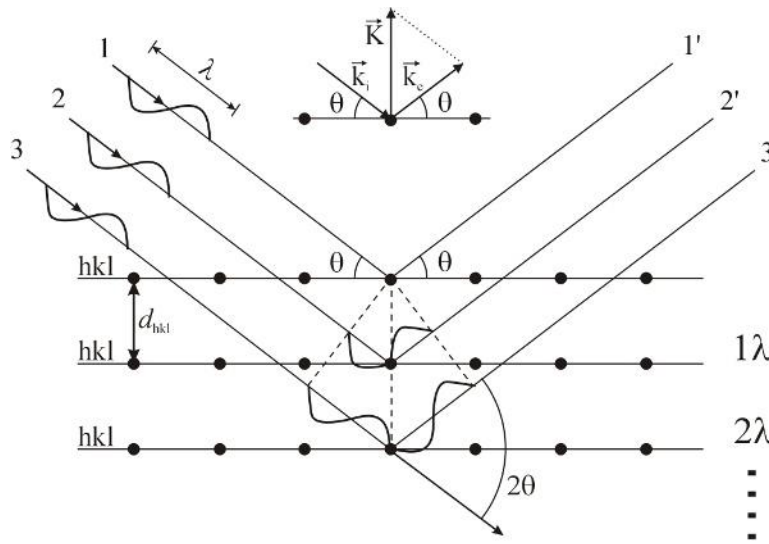


Figure 2-14 X-ray Scattering after reflection through crystallographic planes

(Source: KU Leuven)

X-rays are produced at the point of impact where the electrons are accelerated towards the anode to hit the target. One percent of the energy of the impact is converted into X-rays. The incident electron excites the electron in the inner shell of the atom to higher energy state. The vacancy due to this transition is filled by electron from outer shell

releasing characteristic X-rays. X-rays falling on equally spaced planes of atoms are either transmitted, absorbed or scattered (Cullity & Stock, 2001). The coherently scattered light will form the interference pattern. The diffracted X-rays are captured by the detector and the intensity is shown as a plot by converting analog signal into digital. The intensity pattern can be compared to standard diffraction pattern of different minerals from the database and the presence of minerals can be confirmed through matching of these patterns. Generally, powder diffraction method is used for the soil which can contain all possible orientations. As large number of particles are arranged in a different orientation; some particles might be in the right orientation to produce desired reflection (Whittig & Allardice, 1986).

2.12.2 Scanning Electron Microscope

The scanning electron microscope is a powerful tool that can be used for examination and study of the microstructural characteristics of solid objects. It has a very high resolution of about 1nm. It uses the reflection of electron beams from the surface of the objects to generate an image. SEM was first used in the study of the soil by Roscoe and his co-workers in Cambridge University (Barden & Sides, 1971). It mainly consists of a vacuum system, microscope column, sample chamber, and image system. SEM produces an image by scanning system which generates a raster on both specimen and screen by scanning specimen point by point in a line and then moving the scan to another line and so on. Electron guns produce the electrons. There is a series of electromagnetic lenses which form the electron probe and reduce the crossover of the electron beam. The scan coils move the electron beam over the specimen. Secondary electrons and backscattered electrons are produced as the electron beam interacts with the sample. Finally, detector detects the electron emitted from the specimen which is amplified and used to create images.

One of the most significant disadvantages of the SEM is that the sample is needed to be dry. As the sample is subjected to high vacuum, it should not contain free water. To overcome this and make the analysis of the in-situ samples, ESEM is required which can work under wet sample conditions as well.

2.12.3 Energy Dispersive Spectrometer

The beam electrons can interact with the atoms in the sample and eject out the inner electrons thus leaving the atoms ionized for a fraction of second which returns to low energy state through the transition of outer-shell electrons to the inner-shell vacancy. This energy released during transition can be either used in ejecting another electron and forming auger electron or the energy difference can be released as characteristics x-ray. The characteristic x-ray can be detected with the help of detector. The characteristic x-ray is peculiar for each element and hence is used to identify the elements in the sample. The EDS uses Si (Li) detector. The p-n junction consists of the electron-hole pairs. As the electron-hole pairs are separated, the charge pulse of electrons can be measured at the rear contact. The number of electron-hole pairs created is directly proportional to the energy of the incoming X-ray. So, we can measure the energy used to separate the electron-hole pair and find the element associated with the energy.

2.12.4 Sample preparation for SEM imaging

Scanning electron microscope can be a great aid in micro analysis of soil or soil behavior. With the resolution as high as 1 nm, it is possible to see underlying structure and co-relate the science of fabric of the soil to its behavior under external factor such as load, moisture, temperature etc. However, a great attention is required to prepare good samples for SEM imaging. The fabric of the soil is subject to change with sample preparation techniques. Method of removal of moisture from the specimen affects fabric of the soil. When there is disturbance to the soil, open fabric is broken resulting orientation of the

particles in more closed configuration. Some of the techniques for sample preparation are discussed in the following sections.

2.12.4.1 Air drying

Samples can be prepared by air drying the samples. This is one of the quickest and simplest methods of preparation of sample. Both the ground sample (powder sample) and small cubic intact sample can be air-dried before scanning. However, it leads to considerable shrinkage in sample well above shrinkage limit. Greater shrinkage can lead to greater fabric damage. Shrinkage results in decrease in basal spacing of the minerals in the soil and hence the in-situ condition of the soil cannot be preserved. Other methods like freeze drying, if properly controlled can control the considerable shrinkage of the sample (Gillot, 1969).

2.12.4.2 Freeze Drying

Freeze drying technique involves freezing of the pore water. The freezing of the pore water is usually achieved by dipping the sample in the liquid nitrogen (LN₂) which changes the pore water into ice instantaneously. The ice is then converted into vapor through sublimation in a freeze drier at certain vacuum pressure. Freeze drying can control considerable shrinkage of sample after drying as surface tension due to pore water vanishes. However, freeze-drying can be difficult process to control the melting of ice during sublimation. Fabric damage may also result from freezing and recrystallization of ice glass. There are also chances of failure of freezing of monolayers of water near the surface. The sample after drying should be handled with care as the sample becomes fragile following the drying process.

2.12.4.3 Critical Point Drying

The temperature and the pressure above which the physical properties of the liquid and its vapor are same is defined as the critical point. So if the critical point can be

established for the pore liquid, the boundary layer no longer exists which means that surface tension vanishes (Anderson, 1950); (Gillot, 1969). Hence, if the moisture in the sample is reduced above critical point, the fabric damage should be reduced. The shrinkage can be controlled which increases the chances of sample observations at its true fabric conditions. The limitation of critical point drying however is the difficulty in establishing the critical point as true critical point is difficult to assess.

2.12.4.4 Impregnation Techniques

Impregnation techniques involve replacement of moisture rather than only removal of moisture from the pores of the sample. This method will minimize the shrinkage of the sample and fracture through cementing action. However, impregnation techniques are tedious and may take up to weeks to complete. Impregnation are done with the water-soluble compounds like Carbowax 6000, gelatin, Durcupan Fluka, Vestapal, dispersible epoxy resin (W.D.E.R) etc. Brewer (1964) and Smart (1967) provides details on use of these impregnation compounds. However, impregnation techniques cause some disturbance in the sample. Cracks might occur during impregnation. Gelatin and dispersible epoxy resin (W.D.E.R) were reported to produce good results (Barden & Sides, 1971).

2.13 Stabilization of Expansive Soil

Stabilization of expansive soil generally refers to the improvement of soil properties. The purpose of stabilization is to make the soil ideal for the construction such that it meets the present requirements. Stabilization is achieved through mechanical or chemical modification of the soil. Mechanical stabilization agglomerates the soil particles, reduce void moisture content and help in achieving substantial improvement in strength properties. In mechanical stabilization, soil is modified through physical exchange and process like soil blending which can be also be aided with the use of barrier in or on the

soil (Onyelowe & Okafor, 2012). Compaction is one the easiest ways for achieving mechanical stabilization.

Chemical stabilization of soil can be done using chemicals and additives that will alter the properties of soil in suitable ways. Chemical additives help in flocculation of the soil particles thereby enhancing densification of the soil (Alhassan & Olaniyi, 2013). Expansive soils can be stabilized by reduction of swell-shrink potential and increase in strength of soil using chemical stabilizer. Moreover, stability of expansive soil is associated with structure and mineralogy of the clay particles, clay-water interactions, clay particles' ionic exchange capacity and clay-organic or clay-inorganic interaction (Ali & Tatt, 2003).

2.13.1 Traditional and Non-Traditional Stabilizers

Tingle et al. (2007) broadly categorized stabilizers into two groups: traditional stabilizers and non-traditional stabilizers. Traditional stabilizers include lime, cement and fly ashes. Soil is mixed with low amount of lime and cement to change the physical and chemical properties of soil (Chittoori et al., 2013). Even small number of additives might be efficient to alter the properties of soil.

However, there are some demerits of traditional stabilization of expansive soil. The use of lime especially in sulfate-rich soil leads to intensive swelling and heaving. This is because of the formation of a complex mineral called ettringite. The sulfate and alumina in the natural soil react with the calcium in the stabilizer. The result is the formation of expansive minerals ettringite and thaumasite (Rauch et al., 2002). Apart from inefficiency in high sulfate soils, there are some other demerits of traditional stabilizers. High energy is required for the production of lime and cement and the production cost is very high. They are not environment-friendly as well. 1 ton of carbon dioxide is produced per ton of cement.

Non-traditional stabilizers can be an excellent alternative to the traditional stabilizers. Non-traditional stabilizers are generally non-calcium based. They are available

in low cost, and the application process is also cost effective; hence, they are marketed as viable solutions to replace use of quality construction materials (Tingle et al., 1989). The transportation cost is also lower. Tingle grouped non-traditional stabilizers into several categories: ionic, enzymes, lignosulfonates, salts, petroleum, resins, polymers and tree resins. Ionic stabilizers consist of acids, enzyme stabilizers consist of organic catalyst and polymer stabilizers consist of organic and inorganic catalysts.

Non-traditional stabilizers work through variety of mechanisms such as cation exchange, encapsulation of clay minerals, breakdown of clay minerals with expulsion of water from the clay (Katz et al., 2001). Non-traditional stabilizers are non-calcium based and hence can be used in high-sulfate soils. They are also environmental friendly. However, engineers are reluctant to use the traditional stabilizers despite some positive results are obtained in the lab. Following are the reasons:

- There is a lack of published independent studies on non-traditional stabilizers.
- Standard laboratory test methods haven't been introduced. Stabilizers alter the mineralogy of clay fines to the material with properties that can't be tested with the standard methods developed in laboratories. This puts the results from the laboratory into question if the results are good predictor of the field performance. (Laboratory tests are sometimes criticized for not being able to simulate the field conditions; hence the discrepancies).
- Lack of sufficient published data on the after-effects of treatment from either lab or field.
- Proprietary nature of the commercial non-traditional stabilizers often precludes the disclosure of actual constituents of the stabilizers and their proportion (Tingle et al., 1989).

- The manufacturers of the products often change the names, seem to disappear or appear with different names which make the products unreliable.
- Optimal rate of application of the products are defined in general i.e. it does not appear to be defined with the type of soil and application conditions. There is no logical background on its application rate.
- There are also evidences where effectiveness of the products is not well substantiated with independent field or laboratory studies unlike the claims made by manufacturers (Rauch et al., 2002).

2.13.2 Mechanism of Chemical Stabilization in Clay

Rock minerals Opaline and Gibbsite are formed as a result of complete hydrolysis of Feldspar which forms the basis of chemical stabilization process (Scholen, 1992). If the completion of hydrolysis is prevented due to the presence of alkali metals and/or ionized water at intermediate stage, clay minerals are produced. Chemical stabilizers remove the cations from the clay environment facilitating the hydrolysis of clay minerals into stable form. Complete hydrolysis produces stable crystalline residue. Stabilizers like ionic or enzyme stabilizers are highly electronegative to attract the cations from the clay minerals and ionized water (Mitchell, 1976). Once the lattice is free of cations, the lattice breaks down spontaneously into smaller pieces. It gets converted into amorphous or crystallized remains which eventually hardens as moisture drains out during the transformation. To achieve a cation-free environment, the chemical base should have a strong attraction to these cations and ionized water. The attraction should supersede the energy of clay lattice holding them. To prevent returning of these cations into the solution, the cations are either precipitated as salt or combined with organic molecules. The chemical base, however, only act as a catalyst to breakdown of minerals and do not become part of the clay minerals.

Reaction takes place only between stabilizer and clay mineral fraction of the soil (Scholen, 1995).

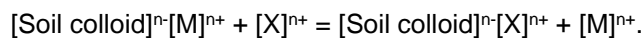
2.13.3 Ionic Stabilizer

Ionic stabilizers are a kind of non-traditional stabilizers which are composed of different type of strong ions. Ionic stabilizers are said to be effective in soil with clay percentage greater than 25% (Dong et al., 2004). Some ionic stabilizers are derivatives of combined organic Sulphur and buffered acids forming bi-sulfates (Alhassan, 2013). Ionic soil stabilizer can be used to treat expansive soil by reducing the thickness of water films (Xiang et al., 2010). According to ionic exchange mechanism, chemical compounds having stronger affinity to surface of clay particles than that of water are added to modify clay's original surface environment and render inertness to water by reducing its susceptibility (Ali & Tatt, 2003). Ionic soil stabilizer reduces the surface activity of the soil which will bring down the sensitivity of expansive soil to water. It reduces the plasticity index, free swell and shrinkage and increases the strength of the expansive soil. According to Alhassan & Olaniyi (2013), ionic soil stabilizers have enormous cationic exchange capacity. The enormous ionic exchange capacity activates the H^+ and OH^- ions and ionizes the water which then vigorously exchange its electrical charges with soil particles. The process removes the pellicular water in an irreversible phenomenon by breaking the electrochemical bond between water and soil and forcing it to remove through gravity or evaporation (Ali & Tatt, 2003). As the adsorbed water is released, diffused layer thickness decreases increasing the cohesion of the soil which imparts increased shear strength in the treated sample. Surface tension is also significantly reduced by hydrophilic and hydrophobic group of ISS (Xiang et al., 2010).

2.13.3.1 Mechanism of working of Ionic Stabilizer

The knowledge of underlying mechanism of stabilization using ionic stabilizers is limited as most laboratory and field experiments focus on effects and performance of the stabilizers rather than the mechanism (Tingle et al., 1989). Ionic stabilizers are hypothesized to work through cation exchange and flocculation of the clay minerals occurring because of change in electrolyte concentration of the pore fluid due to the ionic stabilizers (Scholen, 1992); (Scholen, 1995). They can also work through mechanisms like encapsulation of clay minerals, exchange of interlayer cations or breakdown of clay minerals with expulsion of water from the double layer. Clay minerals have negative charge on their surface and attract strong cation from ionic electrolyte pore fluid which transform the clay to more stable structure (Scholen, 1992).

(Xiang et al., 2010) explained a mechanism by which ISS reinforce the clay. ISS is a strong electrolyte which electrolyze quickly into cations $[X]^{n+}$ and anions $[Y]^{n-}$. Strong cations from the ionic soil stabilizer are exchanged with the cations from the clay particle surface.



The anions $[Y]^{n-}$ released through the reaction decrease the surface tension of the water film and transform the adsorbed water into free water. Ionic soil stabilizer thus helps to remove the easy hydration cations and instead replace them with small hydration and strongly felt cations (Chen & Dong, 1999). The reaction is not reversible, and the released cations and adhered water film cannot return back to the original clay-water system (Xiang et al., 2010). A schematic of the phenomenon is shown in the figure 2-15.

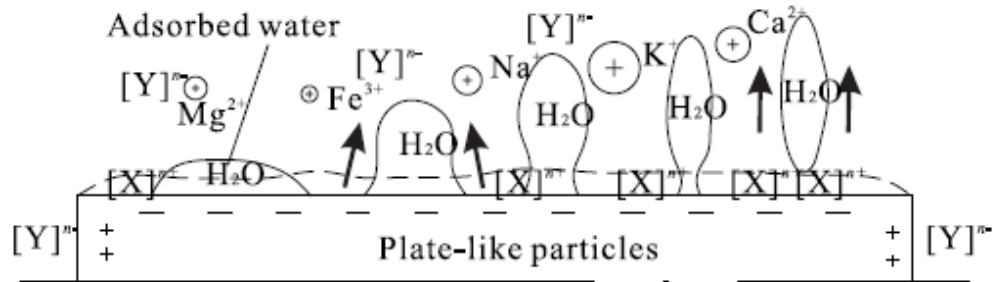


Figure 2-15 Reduction of water film in soil using Ionic Stabilizer

2.13.3.2 Effect of ionic stabilizer

Although, the studies on ionic soil stabilizers are limited, available studies done in the past have shown mixed results in effect of ionic stabilizer. Some literatures explain effectiveness of ionic soil stabilizer while other literatures do not show prominent effect in stabilizing expansive soil. Composition of ionic stabilizers differ from manufacturers to manufacturers and hence its efficiency. Ionic soil stabilizer is effective in reducing the plasticity index of the soil (Xiang, 2010). In this study done by Xiang (2010) on red clay and expansive clay, positive results of ionic soil stabilizer were observed. It was a brown-black water-soluble liquid. Ionic soil stabilizer reduced the thickness of the water films. It also reduced the plasticity index of the soil, increased the shear strength and reduced the shrinkage and free swell of the soil. The plasticity of soil was reduced by up to 27% when using the stabilizer at the doses varying from 1:50 to 1:350. However, when the doses were further increased, the index also increased. The cohesion value increased from 5.41 to 13.84 kPa but the friction angle was invariable. The free swell rate decreased from 59% to 18%. It was also effective in reducing the cation exchange capacity from 23.02 to 14.86 cmol/kg. The effectiveness also depends on the doses used for stabilization. In the study conducted by Xiang et al. (2010) correct proportion of the ionic soil stabilizer required for stabilization was determined from the plasticity index; use of ionic soil stabilizer decreases

the plasticity index of the soil, however, beyond certain amount it starts increasing the plastic limit of the soil.

In another study done by Lu & Xia (2015), showed the effect of curing days and increased unconfined compressive strength following the treatment with ionic stabilizer with different doses of treatment. Four curing periods were chosen in this study: 7, 14, 28 and 60 days in which strength was found to be increasing with the curing days. Study showed that maximum increase in strength occurred until 28 days and the rate of increase in strength slowed down beyond 28 days, suggesting maximum but all reaction has completed within 28 days of curing. The maximum increase in unconfined compressive strength was observed as 37%.

In the study done by Tavakoli (2016) at The University of Texas at Arlington, ionic stabilizer was effective in stabilizing the expansive soil from Caddo Mill, Texas. Different doses of stabilizer were tested and third ratio (10 ml of chemical per gallon of water) was very effective in reducing the PI of the soil. The PI of the control soil reduced from 58% to 26%. With treatment, the OMC of the soil was increased with the reduction in maximum dry density. The effect was seen on the unconfined compressive strength of the soil where the strength of the control soil increased from about 16 psi to 28 psi after treatment with third ratio and 28 days of curing. The stabilizer was also effective in reducing the swell potential of the soil from about 5% to below 1%. However, the tests were done only on one soil and proved to be effective with maximum rate of application of treatment.

(Liu et al., 2011) showed that ionic stabilizer was effective in reducing the water retaining ability and swell potential. However, no any major change in the mineral component was observed. On the contrary, some results have showed ionic stabilizer as less effective stabilizer. Studies by Sarkar et al. (2012) and Katz et al. (2001) have shown that in some clays there are only limited evidence for change in structure of the clays.

Investigation by Katz et al. (2001) led to conclusion that minor changes in mineralogy of expansive montmorillonite were observed with very high application rate of stabilizers while the doses suggested by manufacturers has almost negligible impact on it.

Stabilizers can also affect the compaction of the soil. Hence, separate OMC and MDD are determined for different treatment ratios. Lu & Xia (2015) observed decrease in optimum moisture content and increase in the maximum dry density after treatment with ionic soil stabilizer. However, according to Alhassan & Olaniyi (2013), there was sudden increase in the maximum dry density with certain proportion of ionic soil stabilizer which again decrease upon further increasing the rate of application.

In highway, shear strength is often characterized testing unconfined compressive strength. The data from UCS, however, might not be reliable as triaxial tests. Alhassan & Olaniyi (2013) observed increase in liquid limit with increase in the content of ionic soil stabilizers. The plastic limit however showed no consistent pattern corresponding to the increase in the doses of stabilizer. Significant changes in the properties of the soil are expected to be measurable in 7 days (Rauch et al., 2002).

Chapter 3

Methodology

The chapter presented here provides the details of the procedures followed for all the experiments observed in the laboratory to conduct the research. The soil was obtained as disturbed bulk samples from Carrollton Texas. The samples were transported to the laboratory after collecting in the buckets. The soil brought in the lab was reconstituted and tested in accordance to the standards set by American Society of Testing and Materials (ASTM), TxDOT or as per the guidelines set in the literature. The ionic soil stabilizer was obtained as a commercial product from market in concentrated form for the study. Another soil from Burleson, Texas was also obtained while continuing the research on soil from Carrollton, Texas. The results from this new soil is presented as case study in chapter 5.

3.1 Materials Used

The expansive soil from Carrollton Texas was obtained from the depth of 3 to 4 feet. All the samples were collected using the machine for digging. The samples were then transported to the laboratory in the buckets. Only disturbed bulk samples were obtained for the study.

Ionic soil stabilizer was used for stabilizing the expansive soil. The stabilizer consists of different acids. It contains sulfuric acid, phosphoric acid and citric acid. It was used in conjunction with the surfactant and the water. The surfactant was also provided by the manufacturer. The mixture of stabilizer and the surfactant was mixed with water and final solution was preserved to be used in the treatment. The proportion for mixing each component was calculated based on the application of the stabilizer in the field.

3.2 Field Application

The stabilizer is used in different lots of expansive ground before the construction to reduce the swell-shrink characteristics of the soil. Before applying the treatment into the

ground, the ground is first wetted with the water to near saturation by injecting water into the ground. The way this is achieved is by penetrating the ground and injecting water at a very high pressure using hollow rods with perforations around it using hydraulic system. The water is pumped into the rods which penetrate at different depths as per the requirements into the ground as shown in the figure 3-1. Five rods which are separated at approximately one foot to each other are hydraulically pushed into the ground. At each single pass, these rods can supply water to a depth extending up to 10 feet and a width extending up to 4 feet.



Figure 3-1 Pressure Injection of Ionic Soil Stabilizer in Field

After saturating the ground with water, it is allowed to swell for two to three days. The ground is then wetted with the chemical. Before injecting chemical into the ground, the product is mixed in a concentrated 250-gallon batch. The product is further mixed with water before application at the rate of 1:300 i.e. 1-part chemical per 300-parts water by volume. The process of injection of chemical is same as that of water i.e. pressure injection through the hollow perforated rods at different depths.

The swelling of the chemicals following the treatment is checked approximately after two to three weeks of the application of the chemicals. If the swell potential of the soil is not within the required limit, there may be the necessity to reinject chemicals into it.

The stabilizer used in the treatment is acidic in nature. However, it is diluted with water before application into the ground. So, the stabilizer is environment friendly and meets the safety standards for introduction into sewer system.

3.3 Soil tests in the Laboratory

The soil was collected in bulk form and transported to the laboratory. The soil was identified as greyish fat clay by visual inspection and remolding a little amount in hand and mixing with water. The soil seemed like a weathered shale. Minus 200 tests carried out on soil revealed that about 85% of soil passed through number 200 sieve.

Various tests in the soil were carried out in the laboratory after collection of the sample and some processing. The soil was dried in the oven, crushed over the crusher and pulverized to process it. The soil was then sieved through number 40 sieve and the experiments were carried out in the minus number 40 soil. Most of the laboratory tests were carried out in accordance to either ASTM (American Society of Testing and Materials) or TxDOT standards. Some tests were carried out using the guidelines set in the literature. The tests were carried out in both the treated and untreated soil accordingly. The experimental procedures are described in detail in the following sections

3.3.1 Atterberg's Limit

Atterberg limit of the soil help in defining the consistency of the soil. They are used in several engineering classification systems to characterize fine fraction of the soil. Atterberg originally defined several limits of consistencies to describe the soil. Engineering practices today are usually focused on liquid and plastic limit of the soil. Sometimes shrinkage limit is also tested for the soil. These properties of the soil have been correlated

to various other important properties of soil like permeability, shrink-swell, compressibility and even to shear strength of soil. Various stabilizations of soil are also based on Atterberg's limit of soil. The correlation might be weak; however, the correlation based on these consistencies are important and can be informative at the initial stage of study.

ASTM D4318 "Standard Test Methods for Liquid Limit, Plastic Limit, and Plasticity Index of Soils" provides the detail about testing the liquid and plastic limit of the soil. Method A i.e. multipoint method for determination of the liquid limit was used. Accordingly, the test was performed in the minus number 40 soil i.e. in the soil passing number 40. 200 grams of representative soil was mixed with distilled water to bring to a consistency such that it would require 20 to 30 blows approximately to close the groove in the Casagrande's apparatus. The mixture was allowed to stand overnight in the humidifier. Following day, before beginning the test, the sample was thoroughly mixed and about 20 grams of soil was separated aside for plastic limit test. The test was performed in the Casagrande's apparatus where about 10 mm thick approximately flat soil mass was grooved with the grooving tool through its centerline and the brass cup was allowed to repeatedly fall through a height of 10 mm until the groove closed to a distance of 13 mm. The number of blows and the moisture content corresponding to these points was determined.

Plastic limit of the soil was determined using the plastic limit rolling device. After keeping the soil in the humidifier overnight, the soil was reduced to such a consistency at which it doesn't stick to hand using fan to dry it. Smooth unglazed rolling paper was attached to the device and a small ellipsoidal mass of soil was rolled and rerolled in between the gap of the device until the soil thread crumbled into pieces and couldn't be rolled. The moisture content of the soil mass was then determined by oven-drying the soil.

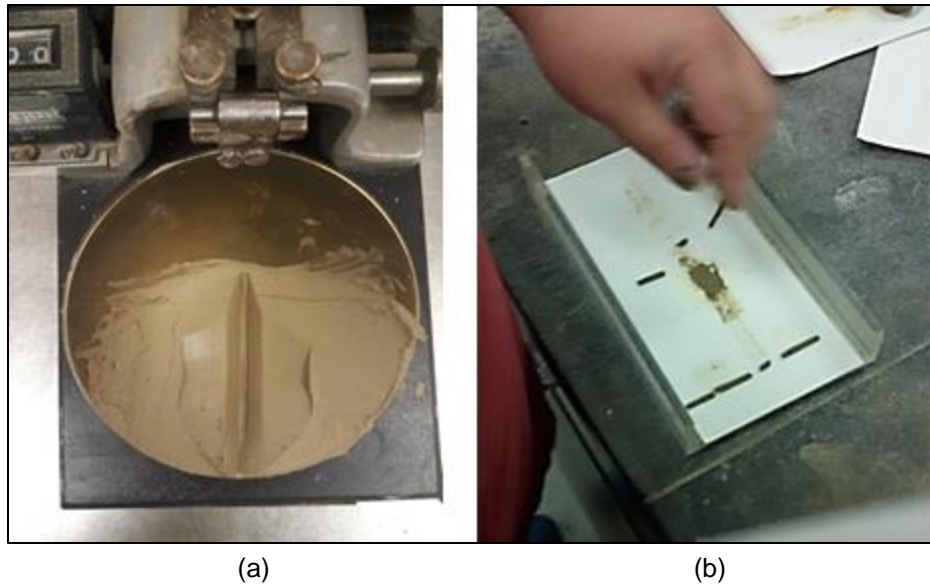


Figure 3-2 Atterberg's Limit of Control Soil a) Liquid Limit b) Plastic Limit

3.3.2 Bar Linear Shrinkage Test

TxDOT Tex-107-E guideline was followed to observe the bar linear shrinkage test. Soil mixture was prepared as explained for Atterberg limits test. The consistency of the soil required for bar linear shrinkage was achieved by mixing the soil with distilled water for untreated and liquid ionic stabilizer in a porcelain evaporating dish of 4.5-inch diameter. When the mixture flows immediately to close the groove made in the 0.5-inch-thick slurry in the porcelain evaporating dish, the soil was molded in the mold.



(a)

(b)

Figure 3-3 Bar Linear Shrinkage Test a) Before Drying b) After Drying

3.3.3 Hydrometer test of Soil

Quantitative determination of distribution of particles was done by performing the test in accordance to ASTM D422 "Standard Test Method for Particle Size Analysis of Soils". About 50 gm of air-dry soil passing number 10 was taken and was allowed to soak in 125 ml of sodium hexametaphosphate solution in a 250-ml beaker for about 16 hours. A mechanical stirring device capable of rotating the peddle at 10,000 rpm was used to disperse the mixture in the dispersion cup as shown in the figure 3-4 (a). Immediately after dispersion, the soil was transferred into sedimentation cylinder and hydrometer test was performed using ASTM 152H type hydrometer. Particle diameter can be found out using Stoke's law and the size of particle less than particular diameter can be known through hydrometer reading. Combined correction for zero reading and the meniscus was applied over the range of temperature recorded during the test. Figure 3-4 shows the test setup for the hydrometer test.



(a)

(b)

Figure 3-4 Hydrometer test a) Dispersion Cup b) Hydrometer Reading

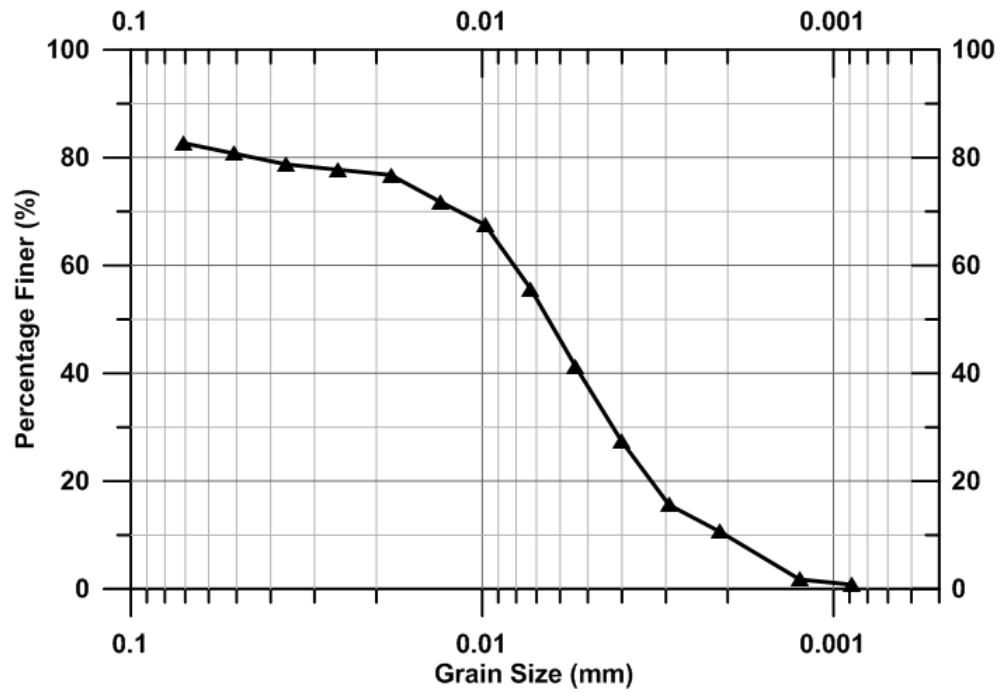


Figure 3-5 Grain Size Distribution Curve

Table 3-1 Properties of the Control Soil

Soil Properties	Control Soil
Liquid Limit	62.5%
Plastic Limit	20.72%
Plasticity Index	41.78%
USCS Classification	CH
Specific Gravity	2.71
Optimum Moisture Content	23%
Maximum Dry Density	96.5 psf
Cation Exchange Capacity	120.7 meq/100 gm
Specific Surface Area	181 m ² /gm

Figure 3-5 depicts the result of hydrometer test on soil. grain size distribution curve of the soil. More than 80% of the soil was finer than number 200 i.e. contained silt and clay fraction. However, the clay fraction of the soil by size was relatively low. It contained only 10% of the clay by size.

3.3.4 Standard Compaction Test

Standard compaction test is carried out to find out the relation between moisture and dry unit weight of the soil. The test was performed in accordance to ASTM D698 “Standard Test Methods for Laboratory Compaction Characteristics of Soil Using Standard Effort (12,400 ft-lbf/ft³ (600 kN-m/m³)). Five specimens of soil at different moisture content below and above of optimum moisture content (determined by visual inspection) were prepared. The soil was then compacted in the 4-inch diameter mold using mechanical hammer weighing 5.5 lbs. The hammer was allowed to fall freely through a height of 12 inch producing a compactive effort of 12,400 ft-lbf/ft³. The soil was compacted in three equal layers with 25 number of blows applied to each layer. After compaction, extra soil

bulging out of the mold was trimmed and weighed to determine the unit weight of the compacted soil. Finally, the soil was extruded, and moisture content of the sample was determined using oven.



Figure 3-6 Standard Compaction Test

3.3.5 One-dimension Swell Test

Swell potential of expansive soil can be determined with one-dimensional swell test. It is relatively easier to perform this test compared to three-dimensional volume change and expansive nature of the soil can be quickly identified using this test. ASTM 4546 "Standard Test Methods for One-Dimensional Swell or Collapse of Soils" discusses the method for determining one-dimensional swell potential of expansive soil. As the test was done on reconstituted soil, test method A was followed.

The soil sample passing number 40 sieve size was first mixed with the distilled water such that the final moisture content of the soil was equal to optimum moisture content. After thoroughly mixing the soil, it was kept in the humidifier overnight. Following

day, the sample was prepared by compacting the soil in a ring of diameter 2.5 inches and height of 1 inch. The mass of soil required for compaction can be computed from required density and volume of the ring. The sample was compacted statically as shown in the figure 3-7. The soil was compacted to 95% of the maximum dry density. After compaction, the sample was allowed to stand for some time to allow the time to rebound. The sample was then mounted in the oedometer with dry porous stones at top and the bottom. Filter papers were placed between the porous stone and the sample to prevent the intrusion of soil into the porous disc. The load frame was brought in the just touch condition over the top of the sample and small token load of 1 psi was applied over the sample. The weight of porous stone and the cap was also included in the calculation of 1 psi load. The dial gauge was zeroed after making sure that the applied load is not making movement of dial gauge due to compression of sample. Finally, the sample was inundated with distilled water and readings were taken immediately following the addition of distilled water at 15s, 30s, 1-min, 2-mins, 4-mins, 8-mins, 15-mins, 30-mins, 1-hr, 2-hrs, 4-hrs, 8-hrs, 24hrs and so on until dial reading was steady. The samples were then taken in the oedometer and small token load of 1 psi was applied to it vertically. The sample was inundated with the distilled water and the vertical displacement of the sample was recorded using dial gauge. The reading was taken until the dial gauge reading came to equilibrium.



Figure 3-7 Static Compaction of Soil and One-d Swell Test

3.3.6 Unconfined Compressive Strength Test

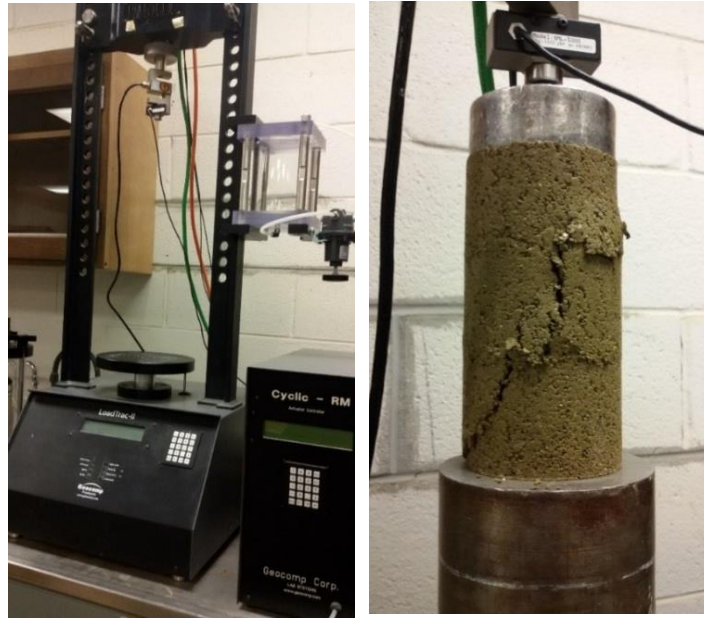
Unconfined compressive strength (UCS) test is a quick test to assess the strength of the soil. A cylindrical specimen of soil is made to fail in simple compression. The test was done to determine the unconfined compressive strength (q_u) of the reconstituted specimen using strain-controlled axial loading. The maximum load attained per unit area of the sample cross-section or load per unit area at 15% axial strain (whichever occurs earlier) is the unconfined compressive strength of the soil. The experiment was done in accordance to ASTM D2166 "Standard Test Method for Unconfined Compressive Strength of Cohesive Soil". The shear strength of the soil at failure is half the unconfined compressive strength of the soil.

Adequate mass of soil passing number 40 sieve size was taken. The required mass of soil corresponding to the 95% of maximum dry density and volume equal to the volume of cylindrical mold can be calculated. The soil was then mixed with adequate amount of distilled water such that final moisture content of the soil is equal to the optimum

moisture content. After thoroughly mixing the soil, it was kept in the humidifier overnight. Calculated mass of soil was then taken into the mold of diameter 2.86 inches and compacted in the static compaction machine. The compaction was achieved in three layers. Before compacting the second or third layer, previous layer was scarified so that there is proper bonding between the layers. The height of the cylindrical sample was maintained to be 5.8 inches. The sample was then allowed to rebound and placed in the humidifier after wrapping with a plastic bag. Cyclic triaxial load frame was used to perform the unconfined compressive strength test of the soil. The frame is equipped with the LVDT and load cell that records the load and the deformation. After overnight standing, the sample was mounted on the platen of the triaxial setup. The platen moves upward at the specified rate and exerts force on the load cell which is recorded and expressed as unconfined compressive strength at failure. The rate of strain was maintained at 0.5% per minute.



Figure 3-8 Compaction of UCS Sample



(a)

(b)

Figure 3-9 (a) Triaxial Machine for Unconfined Compression Test (b) Sample at Failure

3.3.7 Cation Exchange Capacity

Cations are adsorbed to the electronegative surface and edges of clay. Adsorbed cations can be measured with the help of cation exchange capacity test. The more is the adsorbed cations; the higher is the cation exchange capacity. Higher cation exchange capacity is also an indication of presence of expansive minerals like montmorillonite which will result in higher swelling in the soil (Chittoori & Puppala, 2011). One of the earliest methods for study of cation exchange capacity was given by Chapman 1965. In this method, the soil is saturated with ammonium acetate to replace the cation sites in the soil. The ammonium is again replaced by potassium by washing the soil with potassium chloride and the concentration of ammonium is measured which is then converted to cation exchange capacity of the soil. The flow chart for performing cation exchange capacity test is given below.

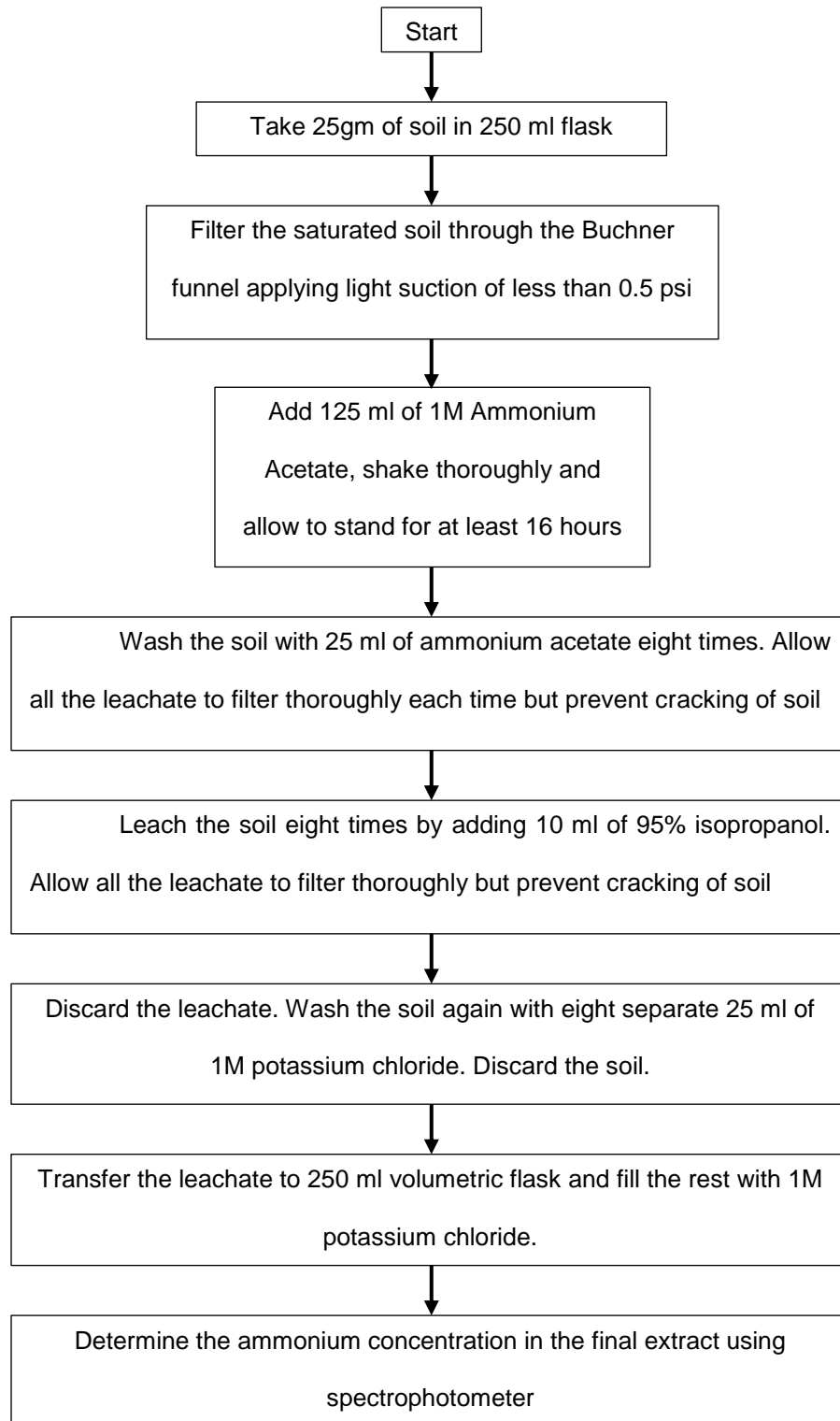


Figure 3-10 Flow Chart for CEC Test

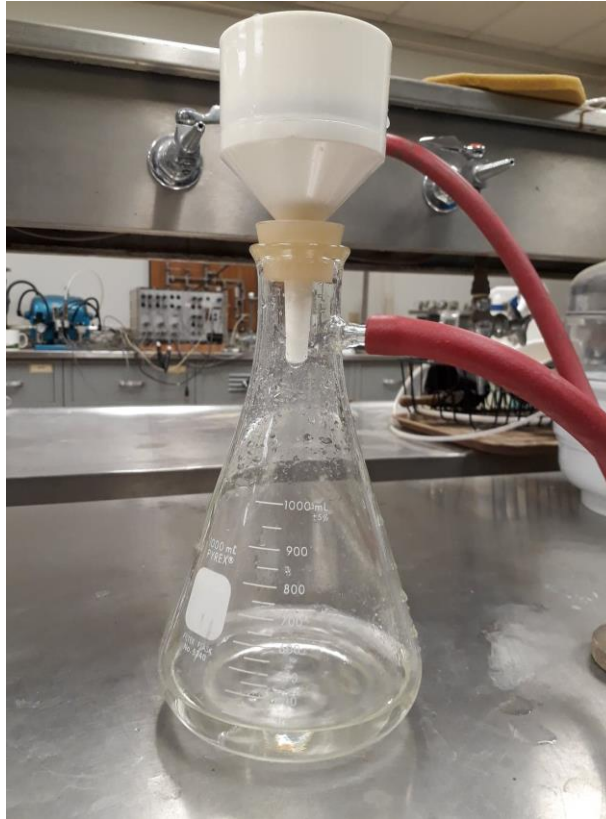


Figure 3-11 Extracting the wash from the soil for cation exchange capacity

3.3.8 Specific Surface Area

Specific surface area is another dominant factor in controlling the fundamental behavior of fine-grained soil (Cerato & Lutenege, 2002). It is defined as the total surface area contained in the unit mass of soil. With the increase in specific surface area of the soil, the surface activity of the soil increases which contributes to higher water holding capacity and greater swelling of the soil. The method for determining specific surface area was first introduced by Dyal and Hendricks (1950) which was then modified by Carter et al. 1965. The method suggests the use of Ethylene Glycol Monoethyl Ether (EGME) for wetting known weight of soil and then evacuating all the EGME in excess to the monolayer adsorbed on soil surface in the desiccator containing EGME-CaCl₂ solvate. The mass of

monolayer retained EGME by the soil in comparison to that retained by pure montmorillonite clay (which is assumed to have specific surface area of 810 m²/gm (Cerato & Lutenegger, 2002) gives the specific surface area of the soil. Specific surface area test was conducted by Cerato & Lutenegger (2002) over a large range of soils and concluded that it was suitable for wide range of mineralogy and is applicable to the range of soil having specific surface area between 15 to 800 m²/gm. The details of performing the test is presented below.

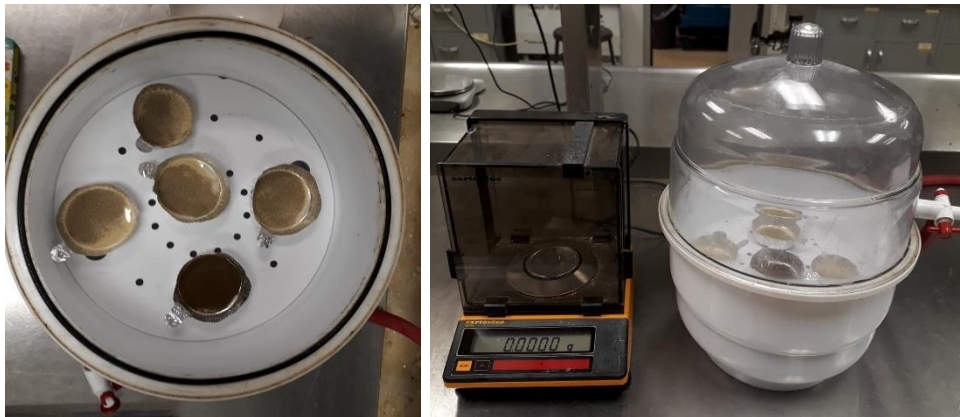


Figure 3-12 Specific surface area test

3.3.9 Mineralogy of soil

The important and dominant minerals in clays are montmorillonite, illite and kaolinite. The percentage of these minerals in the soil was found out using the correlation given by Chittoori (2008). The correlation are presented in the following equations which are based on cation exchange capacity, specific surface area and total potassium of the soil. The percentage of total potassium was obtained from energy dispersive spectrometer test which is presented in the later section.

$$\%M = -2.87 + 0.08 * SSA + 0.26 * CEC$$

$$\%I = \left[\frac{TP}{6} \right] * 100$$

$$\%K = 100 - \%I - \%M$$

According to the correlation, the percentage of montmorillonite in the soil was found to be 43%. As illite is the only mineral associated with the potassium, its percentage was found its correlation with total potassium percentage. The average potassium content for the control soil was 2.06%. Therefore, the illite percentage was obtained as 34%. The percentage of kaolinite mineral was obtained as 23% from the correlation. However, these correlations are crude estimate of the major minerals in the soil and assumes only three dominant minerals are present in the soil. In reality, the minerals may be present as mixed phase and hence these results need to be taken for crude estimation and qualitative idea only.

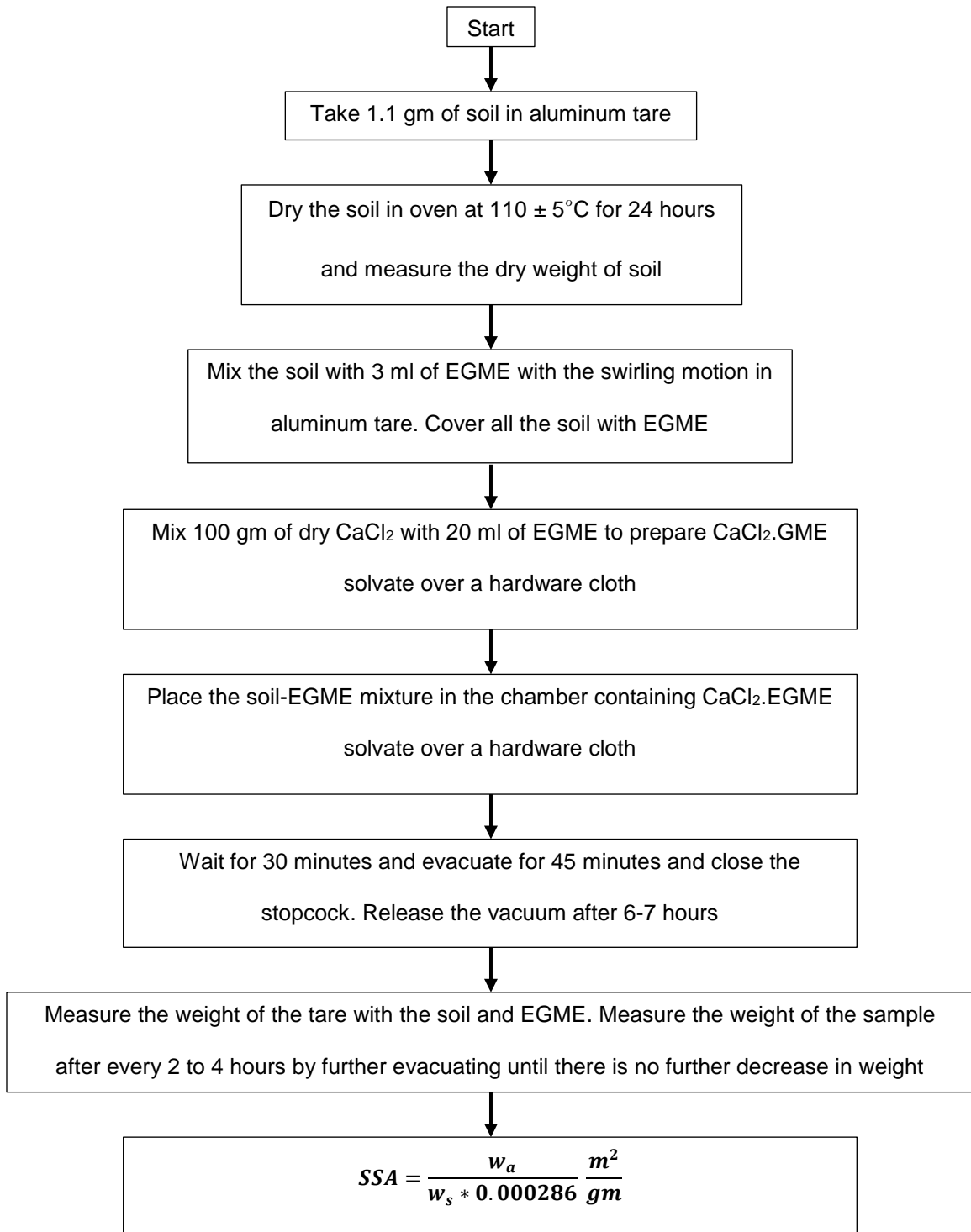


Figure 3-13 Flow Chart for SSA Test

3.3.10 Scanning Electron Microscope Imaging

SEM imaging is a powerful imaging technique that can be used for morphological examination of solid objects. It has a very high resolution of about 1 nm. SEM imaging of the sample was done in order to see any morphological changes brought by the treatment into the sample. Imaging requires special sample preparation as the technique involves direct reflection of electrons from the surface of the soil (Sides & Barden, 1970). Else, there are chances for erroneous interpretations of the false voids, fracture and damages in the structure.

Different instruments are involved in the SEM imaging. The instruments used during the process are described in the following sections.

3.3.8.1 CRC-100 sputtering machine

SEM samples are generally coated with silver, gold or platinum to improve the quality of the images observed. SEM operates with the magnetic lenses that control the deviation of the electron beams ejected from the source and focus it on to the raster of the object being observed. As the electron beam ejected from the source object travels under high velocity to hit the sample object, some of the electrons are ejected out of the object. As the sample object loses its electron, it becomes positively charged. In clays, it is known as local charging of clay particles. The charge appears as an unwanted brightness in the image as the object is scanned. It may also result in damage of the sample. There are some SEMs which can operate in lower vacuum mode. These SEMs have gas available in the system. When electron beam travels from the source to the object, it ejects some electrons out of the gas as well. These electrons ejected out of the gas help in neutralizing the positive charge of the object and prevent the glaring brightness effect in the image of the object. The image is somewhat better. However, with high vacuum mode, unwanted

brightness affects the quality of the image. To prevent this to happen, the surface of the object is given a fine conducting coat of silver, gold or platinum to a thickness of few angstroms. Thus, the quality of the final imaged is enhanced.

To enhance the quality of the image of soil, the soil sample was coated with silver using CRC-100 sputtering machine as shown in the figure 3-12. Samples of soil were prepared and mounted on the aluminum stub using double sided carbon tape. It was then mounted in the sputtering machine. The coating was performed at a pressure of 5 to 10 millitorr in presence of Argon gas. After mounting the sample on to the sputtering machine, the cylinder was closed, and cooling system was turned on. The vacuum system was then turned on to reduce the internal pressure to 0.001 millitorr or below. Once the pressure was achieved, Argon gas was turned on and the internal pressure was maintained between 5 to 10 millitorr. Coating mode was then switched on and the coating was performed for 2 minutes. Whole process takes up to about 20 to 25 minutes. However, it also depends upon the type of sample. After the process is completed, the sample is now ready for SEM scan.



Figure 3-14 CRC-100 Sputtering Machine

3.3.8.2 Scanning Electron Microscope

S-3000N Scanning electron microscope and Hitachi S-4800 Field Emission Scanning Electron Microscope were used to scan the image of the soil samples. The electron beam is generated by the thermionic source. S-3000N SEM uses hairpin filament of tungsten whereas the S-4800 FE-SEM uses LaB₆ filament as a source of electron beam. LaB₆ produces about 5 to 10 times higher brightness than tungsten filament. It has small tip and provides higher resolution. SEM uses a series of electromagnetic lenses to control the beam of electrons by reducing the crossover of beam. It also has apertures to limit the divergence of the electron beam and a beam deflection system which generates raster by moving the electron beam over the object. The electrons emitted from the specimen are collected by the detector, amplified and used to construct the image. A diagrammatic sketch of scanning electron microscope is shown in the figure 3-11.



(a)

(b)

Figure 3-15 (a) S-3000 N Scanning Electron Microscope (b) Hitachi S-4800 Field

Emission Scanning Electron Microscope

3.3.8.3 Preparation of Sample for Imaging

Sample preparation is one of the major tasks in SEM imaging. The quality of final image largely depends on the sample preparation method of the image. On the other hand, the results might be inconclusive or misleading if the sample preparation method is not good. With the advanced instruments like TEM (Transmission Electron Microscope), magnification up to 500k is possible with is even the smallest clay particles can be resolved (Barden & Sides, 1971). Despite such sophistication of the instrument, the studies in this area of clay is relatively less and still at developing phase. The main reason behind it is the difficulty in preparation of the sample. The techniques such as impregnation and prior substitution of pore water are delicate and time consuming. Presence of high sand and silt content in the natural clays causes difficulty in microtoming ultrathin sections and forming replicas (Barden & Sides, 1971). Different methods were followed to prepare the sample for SEM imaging. Bentonite clay consists of montmorillonite, a mineral from smectite group which is flaky in structure. It is used as a lubricant in drilling mud Different samples of bentonite were prepared with different preparation methods and the image generated was compared with the standard montmorillonite image. They are described in the following sections.

Powder method: In this method, powder of bentonite was directly scanned under the electron microscope. A small amount of bentonite enough to be held on the aluminum stub with double-sided tape was taken for imaging process. The sample taken was relatively dry as the scanning electron microscope doesn't handle the wet specimens. The powder sample was scanned under field emission scanning electron microscope.

Air-drying method: Wet specimens are rejected by the scanning electron microscopes as they are not sophisticated enough to handle the wet specimens. Some electron microscopes like E-SEM known as environment scanning electron microscope are modified to handle the wet specimen which facilitate the observation of the sample in its natural condition without drying. However, with scanning electron microscope, the samples should be dried enough to be accepted by the evacuation chamber of electron microscope.

Air-drying method was employed to dry the specimen of bentonite. Bentonite was first compacted at its optimum moisture content and maximum dry density. The optimum moisture content and maximum dry density relation was obtained using Harvard miniature compaction test. A sample of diameter 2.5 inches and height 1 inch was prepared by compacting the specimen with static compaction machine. A small sample was then cut out of the compacted specimen and was dried in air. The sample was more or less flat so that it could stick to the aluminum stub properly. The sample was then coated with silver in the sputtering machine and observed under electron microscope.

Drying in desiccator: Drying in desiccator is a slow drying process where the moisture from the sample is removed very slowly. Slow removal of moisture from the sample helps in preserving its microstructure by removing the pore fluid delicately out of the sample. The sample of bentonite prepared by compaction was flattened at its base and placed in the desiccator with the desiccant. Light suction was applied to the sample to facilitate the desiccation. The weight of the sample was monitored over time to check the loss in moisture. There was some improvement in the quality of the image. However, the drying process is extensively slow and took almost a month to remove the moisture from the sample.

Freeze drying Method: The drying involved in the previous procedures result in considerable shrinkage of the wet samples especially the samples like clays. Dimensional

changes are maximum with the air-drying method. It can be minimized with this method; however, can't be entirely eliminated. Freeze drying method used drying using liquid nitrogen and involves less shrinkage (Gillot, 1969). In the freeze-drying method, ice is formed by rapid cooling of the sample. The ice is then removed by sublimation under vacuum. As the ice changes to vapor without changing into liquid water, the shrinkage that would result due to surface tension is prevented. However, dimensional change can't be fully prevented using freeze-drying method. The freezing of pore water results in increase in volume pore water. Recrystallization of the ice glass and failure to freeze pore water close to the surface may result in the damage of fabric of soil (Gillot, 1969).

Consolidated specimen of bentonite was freeze-dried by dipping it in the liquid nitrogen. The sample was cut to small size appropriate enough for the SEM imaging using a sharp knife as it was dipped in the liquid nitrogen for freeze-drying. The process freezes all the pore water. Once the sample was freeze-dried, it was recovered from liquid-nitrogen and dried in the freeze-drier for about 24 hours. The vacuum pressure for drying was maintained to be 2 mbar. After 24 hours, the sample was recovered from the freeze-drier and the base of the sample was flattened to make it adjustable to the aluminum stub. The exposed surface was peeled with sellotape for more than 50 times. The peeling was done after all the methods of drying the specimen. Sample preparation involves cutting of sample which might result in sticking of debris of broken sample on the surface. Broken debris, if not removed are the major sources of misleading information in the SEM imaging process. Hence, these debris were removed with the help of sellotape by applying it more than 50 times.

3.3.11 Energy Dispersive Spectrometer

Energy dispersive spectrometer is helpful in identifying the elemental composition of the soil. Soil consists different types of element. Most predominantly soil is composed o

silicon and oxygen. EDS was used to identify major elements of the soil. EDS is usually equipped with SEM and hence the concentration of various elements within the selected region can be found out using EDS.

Usually, uncoated sample was used for EDS. However, when the silver coated sample was used, silver was removed from the possible element list in the spectrum. Similarly, carbon was also removed from the possible element list as carbon tape was used to stick the sample on aluminum stub. Total potassium present in the soil was observed using EDS in percentage and the basic minerals were quantified using the correlation provided by (Chittoori, 2008).

3.3.12 X-ray Diffraction

X-ray diffraction is one of the most popular methods used for identification of fine-grained soil minerals and crystal structure. We can identify the crystal phases of the compounds through X-ray diffraction. Each peak in the diffractogram represents the diffraction from a certain crystallographic plane. The position of the peaks at different 2θ angles and the relative peak intensities can be compared with the standard diffraction intensity of different crystallographic planes obtained from powder diffraction file. The comparison of the diffractogram with the pdf enable us to identify the crystalline structure in a specimen from which we can get the idea about the composition of the sample. X-ray diffraction technique was employed to identify the changes caused by the ionic stabilizer in process of stabilization of the expansive soil. With the treatment, there should be the reduction in the intensity of the montmorillonite peaks as well as change in 2θ angle indicating reduction in the basal spacing.

Bruker D-8 advanced X-ray machine was used for the identification of the soil as shown in the figure 3-14. The scan type employed was locked-couple scan. In this arrangement, the sample is fixed while the incident beam and the detector rotate. The X-

ray is always on. However, shutter is open only at the time of operation. The machine shuts automatically if any error occurs to protect from the X-ray hazard. The glass used in the chamber is X-ray proof.



Figure 3-16 Bruker D-8 X-ray Diffraction Machine

The soil was prepared in the powder form. The soil after treatment was crushed in the mortar with the pestle to prepare the powder sample. Powder sample was used in the larger holder after cleaning it with ethanol and the surface was leveled with the help of glass plate. Powder sample increases the probability of randomness in orientation of the crystal phases. The 2θ range was taken from 10° to 90° in steps of 0.02 for the first scan and the ranges were shortened by eliminating the 2θ where peaks were absent for the further scans. The diffraction peaks were analyzed using the Jade5 and MATCH! software.

Chapter 4

Results and Discussion

4.1 Atterberg's Limit

Atterberg's limit was performed on the untreated and the treated soil. The ratio of treatments used for the tests are listed below.

Table 4-1 Composition of treatment

Treatment	Volume of Chemical (ml)	Weight of Surfactant (gm)	Volume of Water (Gallons)
1:300	12.5	0.144	1
1:150	25	0.288	1

The liquid limit, plastic limit and linear shrinkage results are presented in the table below.

Table 4-2 Atterberg's Limit and Linear Shrinkage of Soil

Treatment	Liquid Limit	Plastic Limit	Linear Shrinkage
Untreated Soil	62.89%	21.46%	22.57%
1:300	65.51	21.90	23.10%
1:150	59.94	20.89	23.35%

4.1.1 Effect of treatment on Atterberg's limit and linear shrinkage

As seen in the results, the treatment does not have consistent effect on the Atterberg's limit and linear shrinkage of the soil. Plastic limit is more or less the same for soil before and after the treatment. Slight changes are seen in the liquid limit of the sample. The changes however are not consistent. There is increase in liquid limit of the soil after the treatment with 1:300 ratio. The liquid limit has decreased after the treatment with higher

ratio (1:150). However, compared to control soil, the decrease is insignificant. Similarly, the linear shrinkage of the soil has slightly increased after the treatment. The effect of treatment on plasticity index of soil is shown in figure 4-1 below.

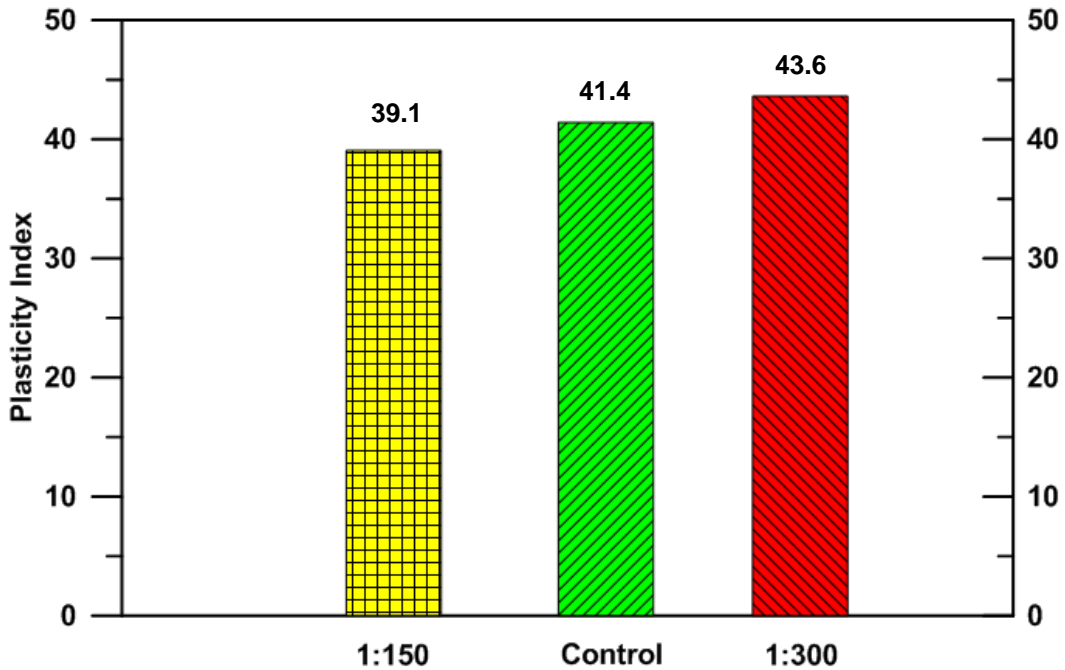


Figure 4-1 Effect of ionic stabilizer in plasticity index of soil

4.2 Standard Compaction Test

The moisture-density relationship of treated and control soil was determined through standard compaction test. Soil was prepared at five different moisture contents and was mixed thoroughly to distribute the moisture homogenously throughout the soil mass. A time of about 16 hours was allowed for the treated soil to stand before compaction.

4.2.1 Effects of treatment on OMC and MDD

Figure 4-2 shows the relationship between moisture content and dry density of the soil for treated and control specimens. It indicates that the optimum moisture content of the soil increased after the treatment and the maximum dry density decreased. The optimum moisture content increased from 23% to about 26% after the treatment. The optimum

moisture content for both the treated soils with different ratios of treatment are almost same. The maximum dry density of the control soil is 96.5 pcf. After treatment, it decreased to 91.10 pcf for the 300:1 ratio of treatment and 92.5 for the 150:1 ratio of treatment.

The increase in OMC and decrease in MDD can be attributed to the modification of the internal clay structure. This is the change in fabric of the soil owing to the reaction with the stabilizer. The face to face arrangement of the clay particles changes to edge to face structure as a result of reaction with the stabilizer. As the clay particles are arranged edge to face, more voids are created within the structure providing room for additional water to enter the structure and increasing the optimum moisture content. However, when the clay particles are arranged edge to face, the resistance to the applied load also increases. Face to face clay particles slip against each other in presence of water when external load is applied whereas edge to face clay particles offer reaction through resistance to compaction. Consequently, there is less amount of soil in the given volume and hence the dry unit weight of the soil decreases with the treatment. Water also plays important role in the acceleration of the reaction of the stabilizer with the soil. As the doses of stabilizer increases, more water is required to assure the completion of the reaction.

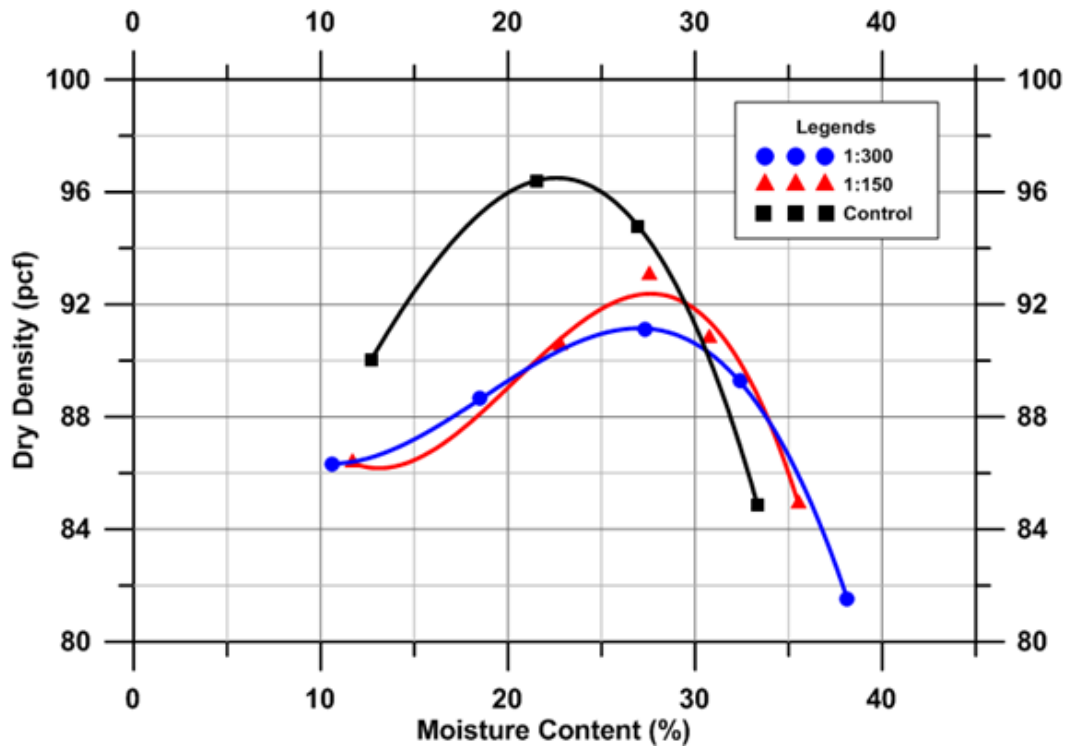


Figure 4-2 Standard Compaction Curve

Table 4-3 OMC and MDD of control and treated soil

Treatment	OMC (%)	MDD (pcf)
Control	23	96.5
1:300	27	91
1:150	27	92.5

Table 4-4 pH after Treatment

Treatment Ratio	pH
1:150	5.53
1:300	6.50

4.3 One-dimensional Swell Test

The test procedure for performing the one-dimensional swell test is discussed in chapter three. The soil sample prepared at respective optimum moisture content and 95% of maximum dry density were tested for one-dimensional swell test. The samples were kept in the humidifier for 7 days and 28 days before performing the test.

4.3.1 Effects of treatment on vertical swell

Figures 4-3 to 4-4 show the effect of different curing time and different treatment doses on the swell potential of the soil.

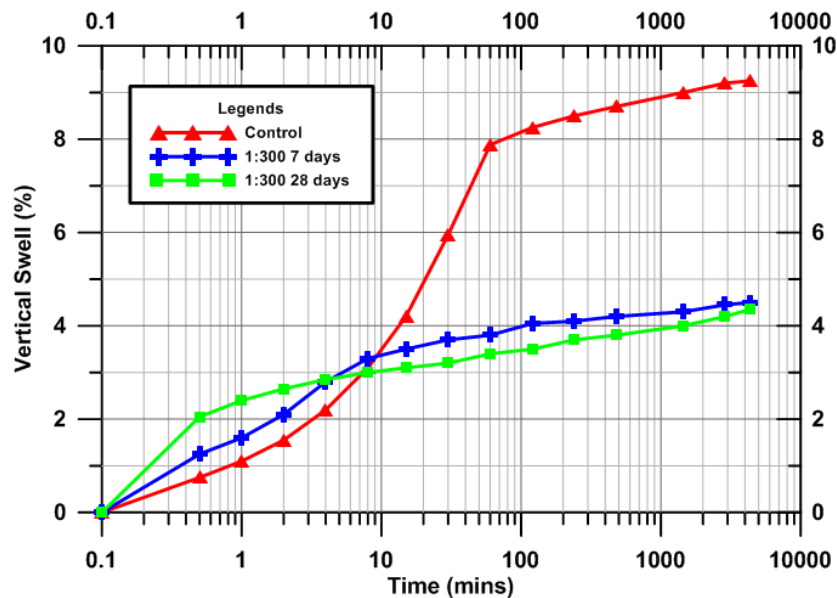


Figure 4-3 Comparison of swell before and after treatment (1:300) at their respective OMC and MDD

The figure 4-3 shows that there is a reduction in the swell potential of the soil with the treatment. The dose of stabilization used for the treatment is 1:300. The result shows that the control soil has swelling potential of 9.25% when allowed to swell under a token load of 1 psi. However, after the treatment the swell potential has reduced. The graph shows that the swell potential is reduced to a value of 4.5 % after the treatment and 7 days of curing and to a value of 4.35% after the treatment and 28 days of curing. About 5% reduction in swell has been observed after the treatment. However, there is insignificant difference in the swelling potential observed after 7 and 28 days of curing. This can hint to the possible reasoning that reaction is almost complete within 7 days of treatment. As the ionic soil stabilizer reacts with the soil in presence of water, it ionizes quickly and reduces the surface charge density of the soil particles through cation exchange. The affinity of clay particle to water is reduced. This results in loss of electronegativity of the soil which causes the swell potential to be reduced.

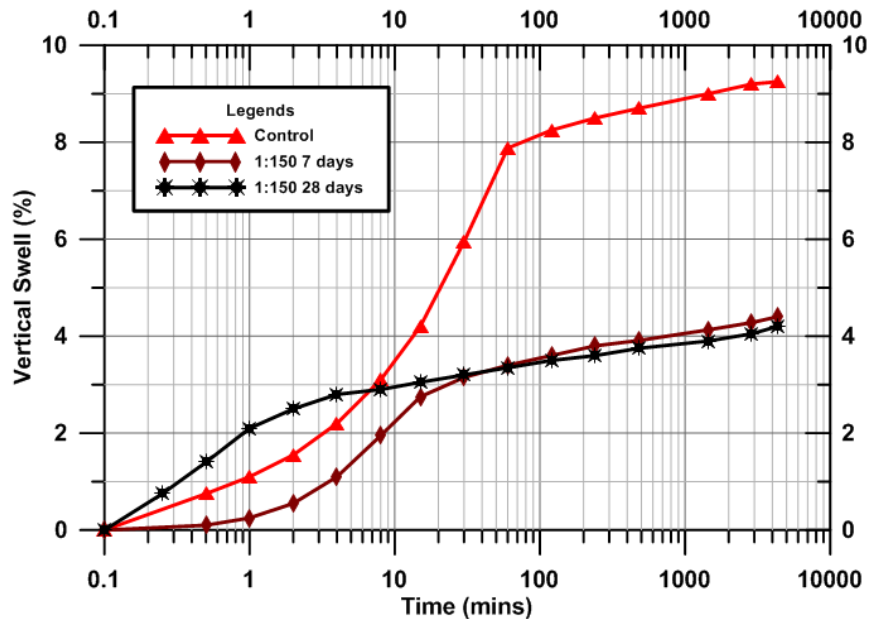


Figure 4-4 Comparison of swell before and after treatment (1:150) at their respective OMC and MDD

The figure 4-4 just like figure 4-3 shows the reduction of swell potential of the soil with higher application ratio of treatment. While doubling the dose of application of treatment, the swell potential reduced to a final value of 4.4% after 7 days of curing and 4.2% after 28 days of curing. The treatment with 1:150 dose of application is effective in reducing the swell potential of the soil by about 5%. However, there is only a slight reduction in swell with increasing number of days of curing. The reduction is not significant. Also, with the increasing dose of application of treatment, no significant reduction in swell was observed. Comparison of effect of curing and doses of treatment are shown in figure 4-5.

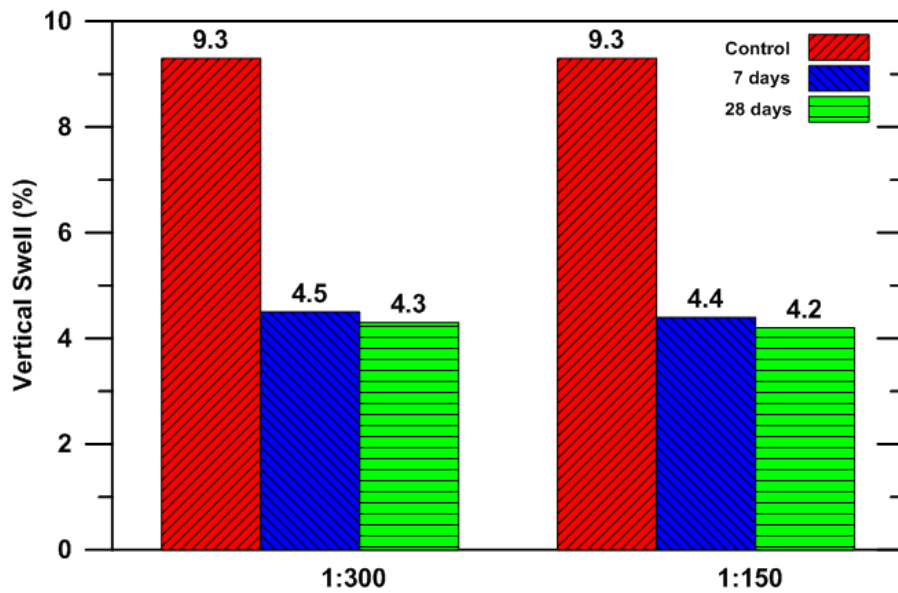


Figure 4-5 Effect of curing days on vertical swell of stabilized soil

Even though, the treatment doses were able to reduce the swell potential, none of the treatment dose was able to reduce the swell potential below 2%. International building code (IBC) requires the expansion index of the soil under the foundation to be below 20. There is no exact conversion between expansion index and vertical swell percentage. However, expansion index is measured in thousands and the sample should be 50%

saturated. Roughly converting, it may be equal to 2% vertical swell. In Texas, the suitability of foundation is checked with respect to PVR. The PVR should be less than 4 inches and differential movement should be less than one inch in twenty feet within the foundation. However, for PVR, the information about all the subsurface layers in the active zone should be known usually up to 10 to 15 feet. The swell for each layer is calculated and converted into potential vertical rises in inches. Though the treatment was able to reduce the soil, it was not sufficient enough to completely stop the swelling of the soil or reduce it to the acceptable limit.

4.4 Unconfined Compressive Strength Test

The manufacturers of ionic soil stabilizer use the stabilizer solely for the reduction of swell potential so as to bring the swelling of soil under the acceptable limit for foundation construction of residential building. However, for the research, the impact of stabilizer on the strength of soil was also assessed. The modification of internal clay structure may impart greater strength on the soil along with the reduction of swell potential. For this, unconfined compressive strength test was done. As discussed in previous chapter, the samples were prepared at 95% of MDD and OMC for testing its strength.

4.4.1 Effects of treatment on Unconfined Compressive Strength

Figures 4-6 to 4-8 show the result of unconfined compressive strength test done on different samples with different doses of treatment. At least two samples were tested for each condition. The average unconfined compressive strength of the control soil is 48.0 psi. Maximum strength obtained was 51 psi and minimum strength obtained was 44.9 psi for control soil. The strength of the soil has decreased after the treatment. As seen in the figure 4-6, the strength of the soil after 1 day of treatment with 1:300 ratio has decreased to 30.7 psi from 48.0 psi. This corresponds to about 36% loss in strength with the treatment. Similarly, the strength after 7 and 28 days of treatment is 36.0 psi and 36.2 psi. There is

increase in the strength from 1 day to 7 days of curing and 28 days of curing. However, even after 28 days of curing, the strength is less than the untreated strength of control soil. The increase in strength from 7 days to 28 days is not significant.

The effect of treatment with higher ratio also yields similar result. There is net decrease in strength of the soil with the treatment with 1:150 ratio. The strength has decreased from 48.0 psi of control soil to 39.0 psi of treated soil after 1 day of curing corresponding to about 19% loss in strength of the soil. The strength of soil after 7 days and 28 days of curing is 42 psi and 42 psi respectively. The result as in the previous case shows no significant change in compressive strength of the soil with curing after 7 to 28 days. Slight increase in seen from 1 day to 7 days.

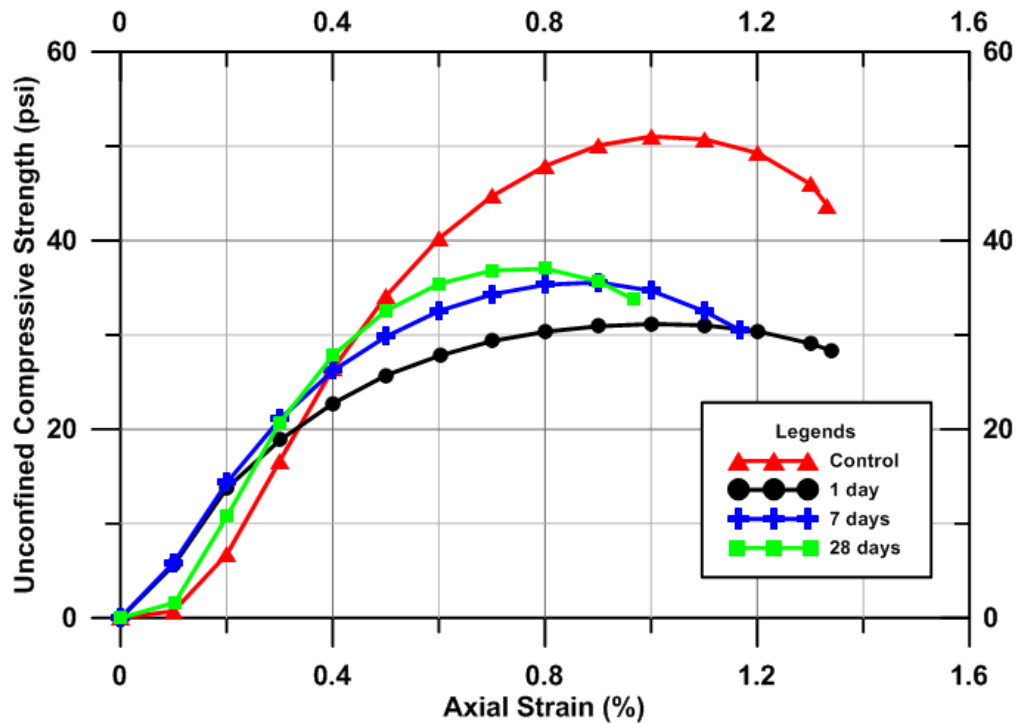


Figure 4-6 Effect of treatment (1:300) on Unconfined Compression of Soil

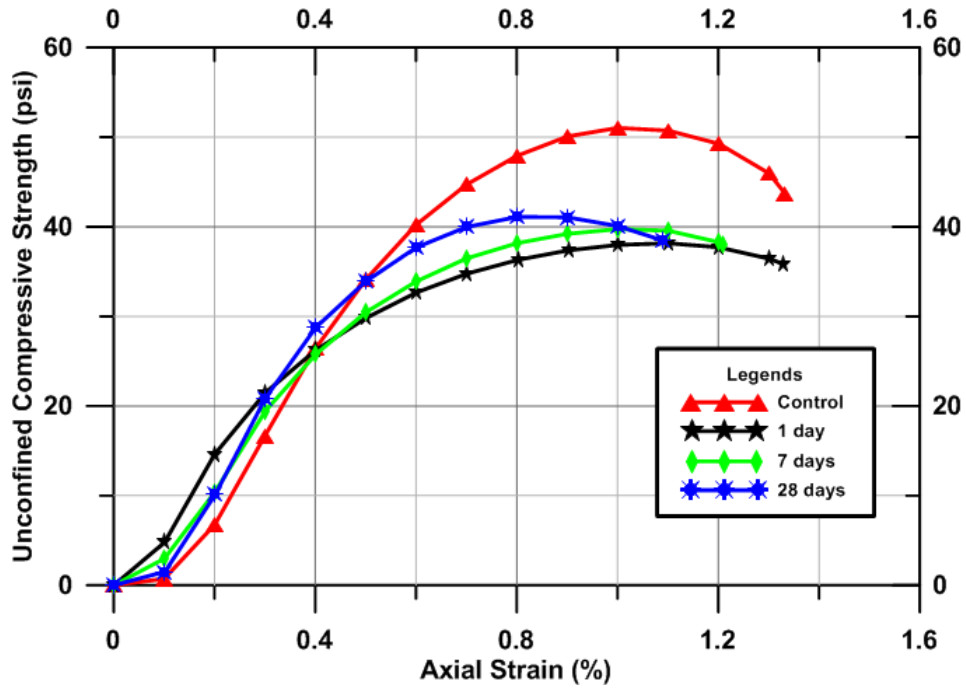


Figure 4-7 Effect of treatment (1:150) on Unconfined Compression of Soil

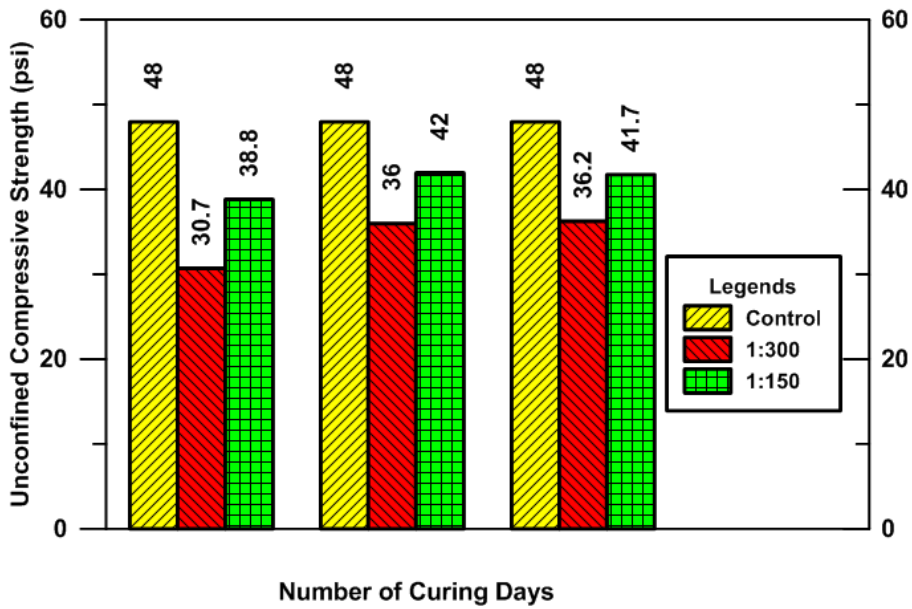


Figure 4-8 Comparison of average unconfined compressive strength

4.5 SEM/EDS and XRD Results

The behavior of treated soil showed anomalous behavior as in one hand swell potential of the soil was decreasing with the treatment and on the other hand the strength of the soil was also decreasing. The aim of stabilization was to decrease the swell potential of the soil without decreasing the strength of the soil. However, results from the laboratory were otherwise. To further investigate the behavior of the soil, microanalysis of the soil was carried out. It included imaging of sample under scanning electron microscope, energy dispersive spectrometer analysis and x-ray diffraction analysis of the soil before and after the treatment.

4.5.1 SEM imaging of bentonite clay

Scanning electron microscopic images of samples were developed in the laboratory. The images of the samples were taken for control soil and for the treated soil. Preparation of the sample plays important role in the quality of the image. Different preparation methods were used to find the suitable preparation method. For this, samples of bentonite were prepared as per different sample preparation methods. The sample preparation methods included powder method, air-drying method, desiccation-drying method and liquid-nitrogen drying method. Bentonite is rich in montmorillonite mineral. Hence, the images obtained from the different methods for bentonite sample were compared with standard image of montmorillonite to find the suitable image preparation method.

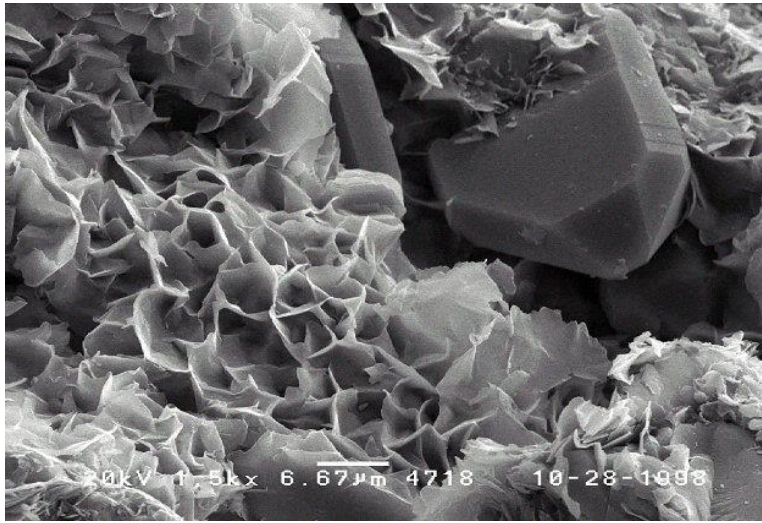


Figure 4-9 Montmorillonite observed under Scanning Electron Microscope

(Source: OMNI Laboratories, Inc)

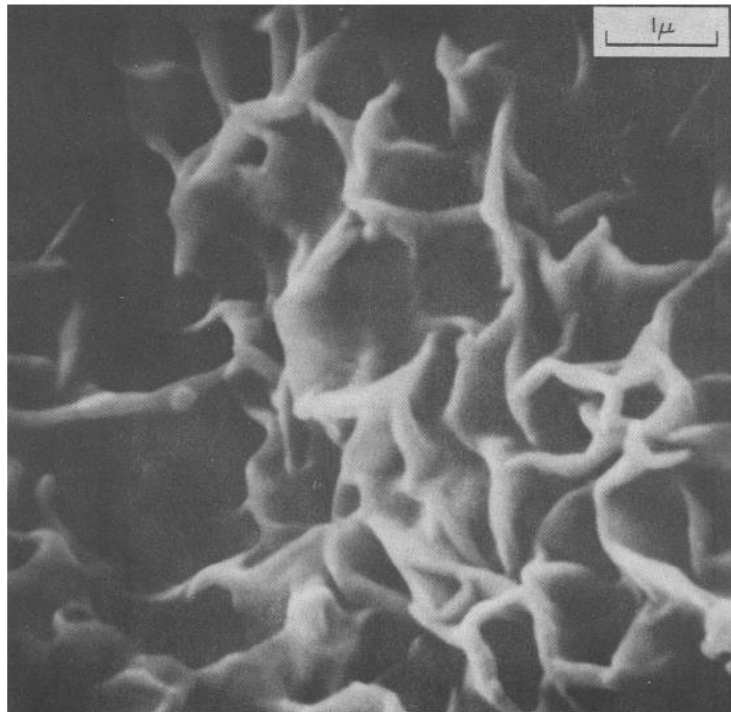


Figure 4-10 Montmorillonite observed under Scanning Electron Microscope

(Source: Bohor, 1971)

4.5.1.1 Air-drying method

Air-dry sample of bentonite in powder form was taken for the observation. The images were taken at different magnification ranging from 2.0k to 30k at 15kv-25kv voltage. The working distance was maintained close to 8 mm for FE-SEM image and 15mm SEM image. The SEM image shown in figure 4-11 displays lots of voids in the sample. The degree of disturbance is higher in the powder sample and more voids will be reflected which are not a part of clay structure. These voids are not the natural voids in the sample. When powder sample is used, there is no interlocking among the particles which appear as false voids in the image as shown in the figure 4-11. The image shows some irregular wavy edges of the particles.

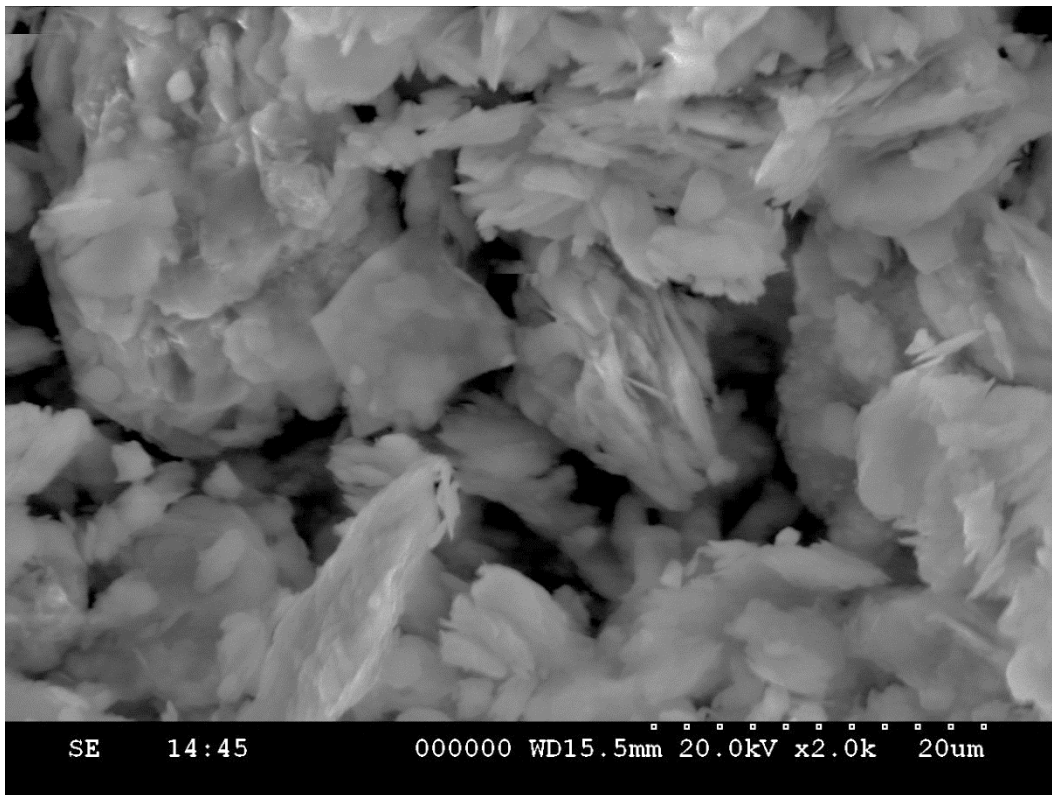


Figure 4-11 SEM image of powder bentonite clay at 2k magnification

Figure 4-12 shows the compacted sample of bentonite. The sample was compacted statically in a consolidometer ring and small sample was cut out of it. The sample was air-dried and prepared for SEM observation. The image shows the platy structure rather than the flaky edges of montmorillonite. The edges are smooth compared to the image of montmorillonite. At higher magnification (5K) as shown in the figure 4-13, the platelets are not seen. False voids as in powder sample have been reduced. During sample preparation i.e. cutting out small specimen from sample, the edges are broken at the point of cut. The broken pieces remain at the surface as debris and occlude the original morphology of the sample. The image rather shows the particles as broken debris and disorganized which do not reveal the actual features of bentonite clay.

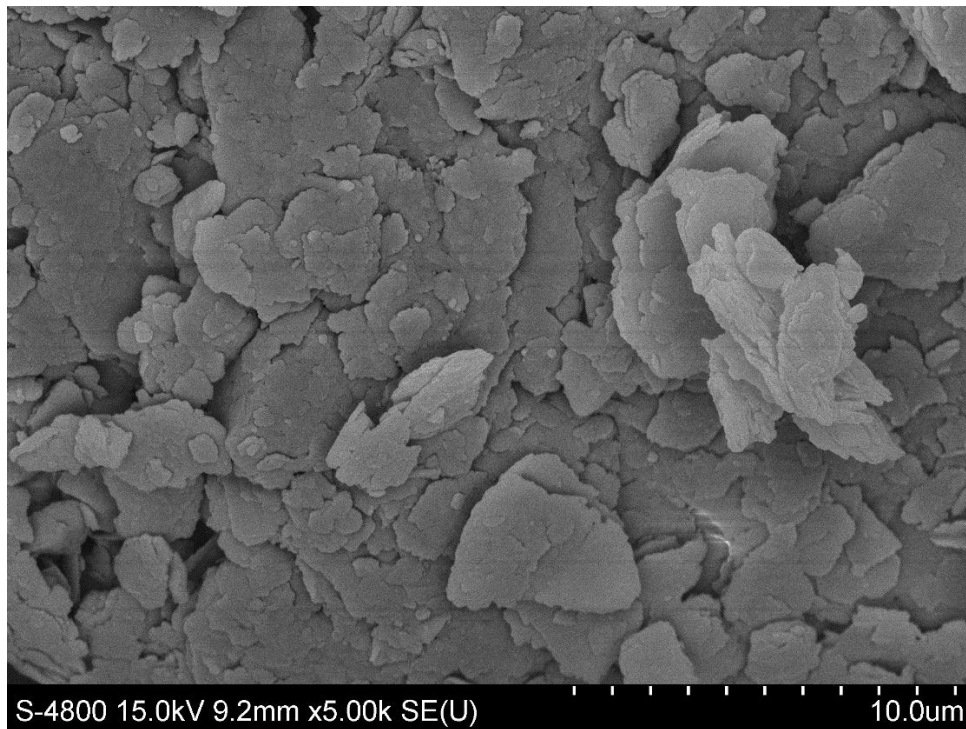


Figure 4-12 FE-SEM image of bentonite at 5k magnification

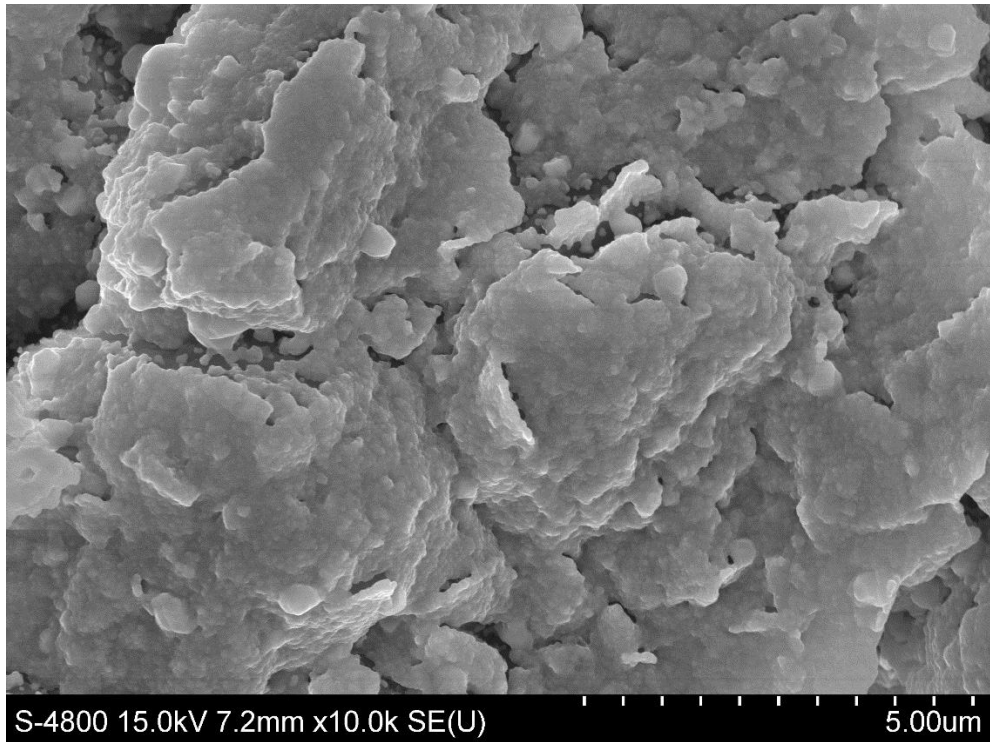


Figure 4-13 FE-SEM image of bentonite at 10k magnification

4.5.1.2 Desiccator drying method

The desiccator drying method allows sample to dry slowly. The sample of bentonite was first compacted statically at its OMC and MDD in a consolidometer ring. The sample was kept in a relative humidity chamber at 50% relative humidity for about two weeks and allowed to dry. A small cubical specimen was cut out of it and kept in a calcium chloride desiccator to further dry for further two weeks. The surface was then smoothed by applying tape for about 50 times to remove broken debris on the surface of the sample.

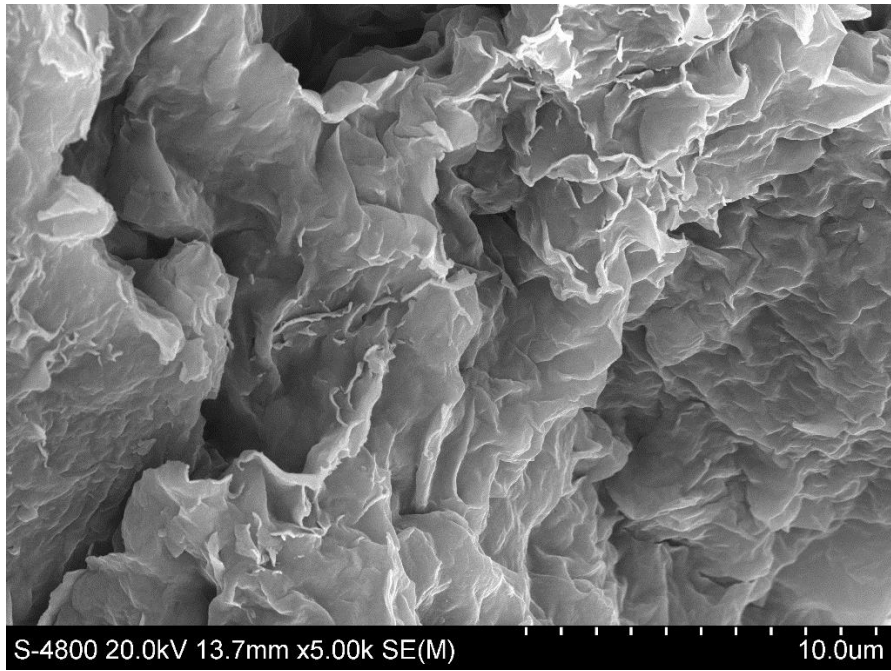


Figure 4-14 FE-SEM image of bentonite after drying in desiccator (5k magnification)

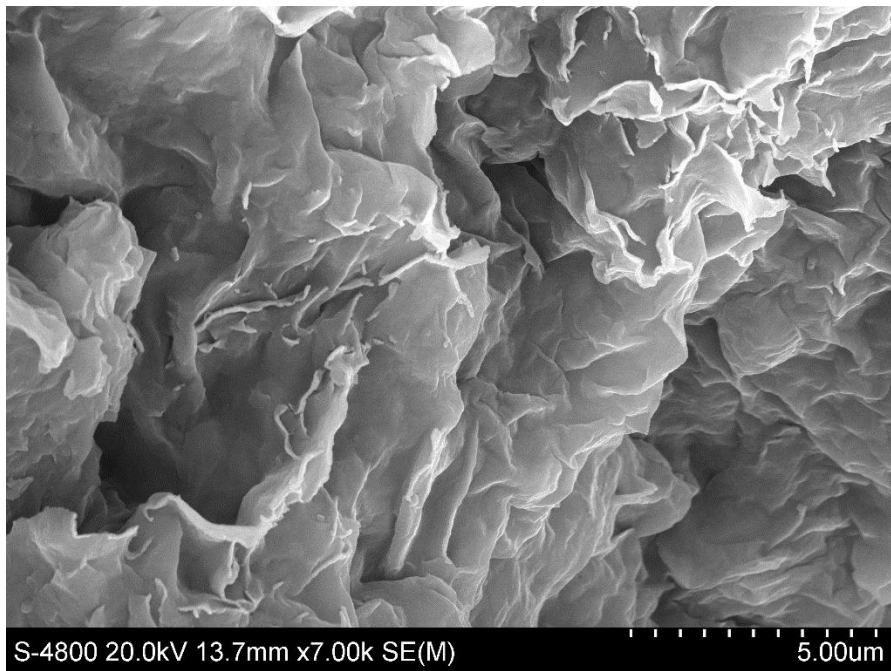


Figure 4-15 FE-SEM image of bentonite after drying in desiccator (7k magnification)

Figure 4-14 and 4-15 shows the image of the sample after drying in desiccator and further processing with tape to remove debris. The image has improved quality and depicts the features of montmorillonite. The wavy and flaky edges can be clearly seen. The curly ridges depicting the crinkly and honeycomb-like texture is typical of the smectite group. The crinkly texture might have developed as a result of the shrinkage owing to drying of sample (Bohor & Hughes, 1971).

4.5.1.3 Freeze-drying method

Specimens of freeze-dried bentonite were prepared for observation under scanning electron microscope. Slurry of bentonite was prepared by mixing with water and allowed to self-consolidate. Slurry was then frozen in liquid nitrogen and the frozen sample was allowed to dry in vacuum pressure to allow the ice to sublimate.

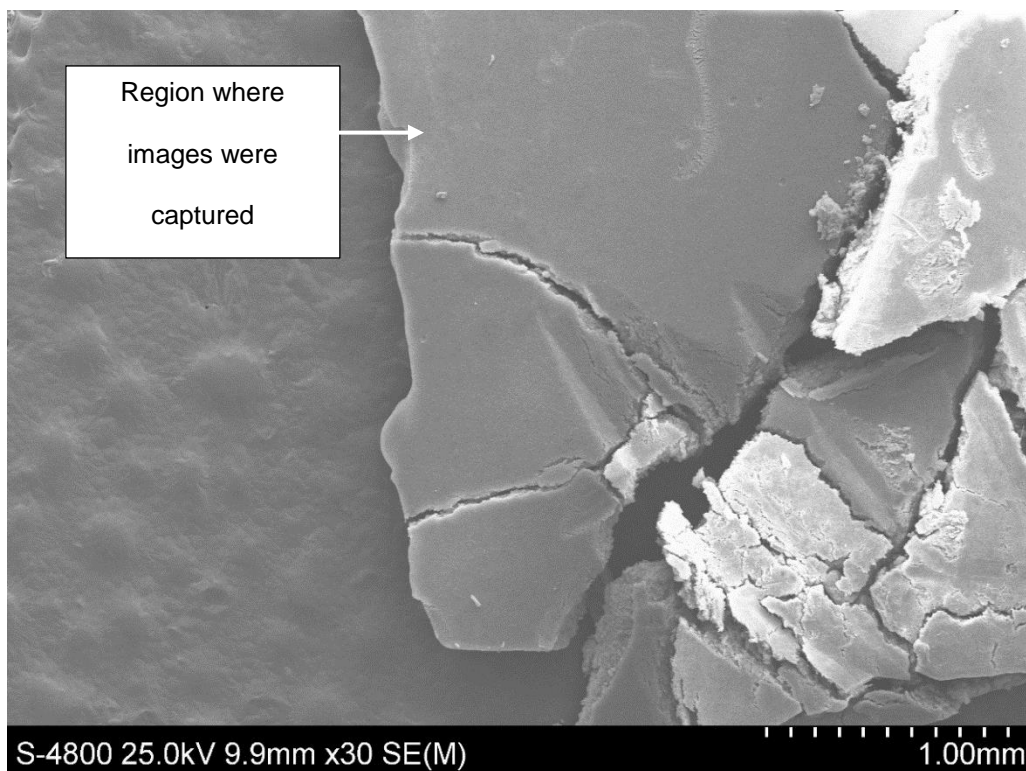


Figure 4-16 FE-SEM image of freeze-dried bentonite slurry at 30 times magnification

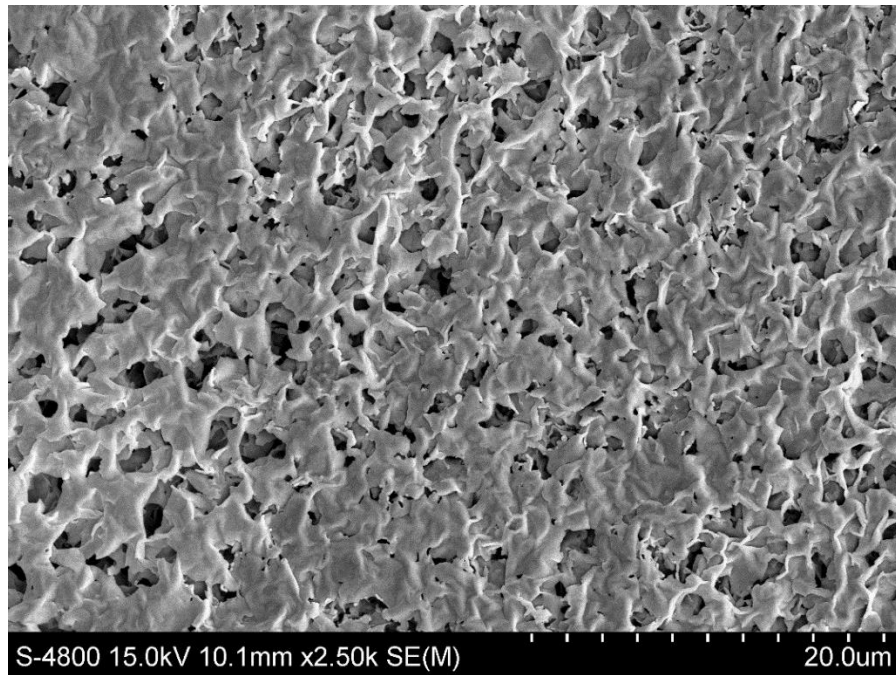


Figure 4-17 FE-SEM image of freeze-dried bentonite slurry at 2.5k magnification

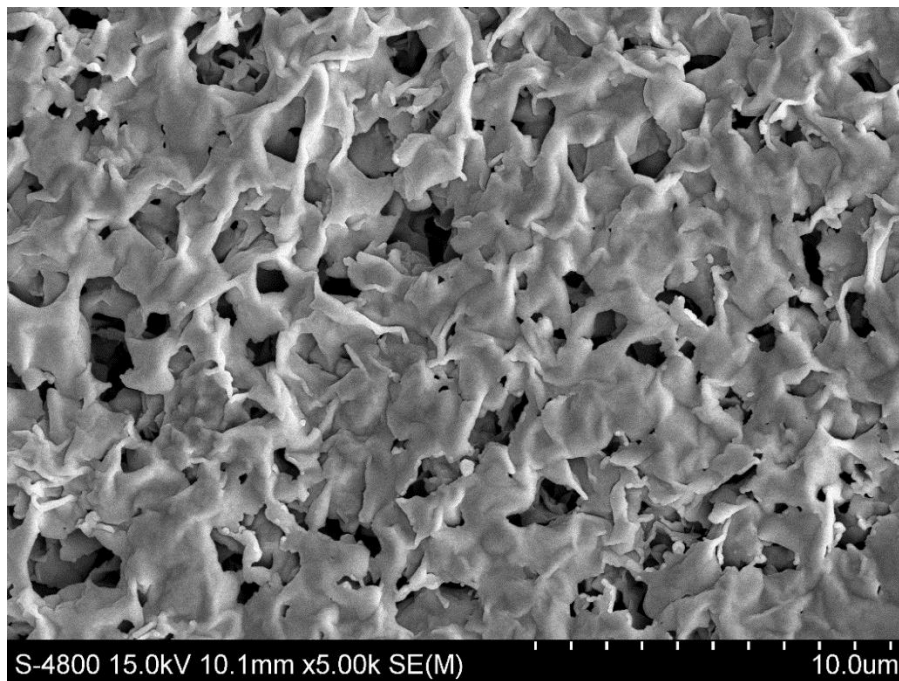


Figure 4-18 FE-SEM image of freeze-dried bentonite slurry at 5k magnification

Figure 4-17 and 4-18 show a lot of voids in the image. The slurry of bentonite when subjected to freeze-drying by dipping in liquid-nitrogen gets converted into the frozen ice. When the ice is removed by sublimation, the voids are preserved which can be seen in the image. It also shows the water affinity of montmorillonite. It can hold large amount of water compared to its size resulting swelling in the soil. The quality of the image is enhanced with this preparation method. Curly ridges, crinkly and honeycomb-like structure typical of smectite group can be clearly observed in the image.

Freeze-drying method is very useful method especially for electron microscope which do not handle wet specimens. Clays are tricky and difficult to deal with when they are wet. The true fabric of clay when they are wet at their natural condition can be observed with freeze-drying method with minimum disturbance to the sample. While preparing samples with other preparation method, the pore water is removed through drying. As the pore water decreases, the sample shrinks, and in-situ fabric of the sample is destroyed. With freeze-drying method, the water phase of pore water is eliminated by first converting it to ice and sublimating it without converting it to water. So, sample disturbance compared to other method is minimum. However, it cannot be eliminated completely.

4.5.2 SEM imaging of control and treated sample

SEM images of samples before and after treatment were taken. Images of control sample were taken with all the methods. However, the images of treated sample were taken with few methods only.

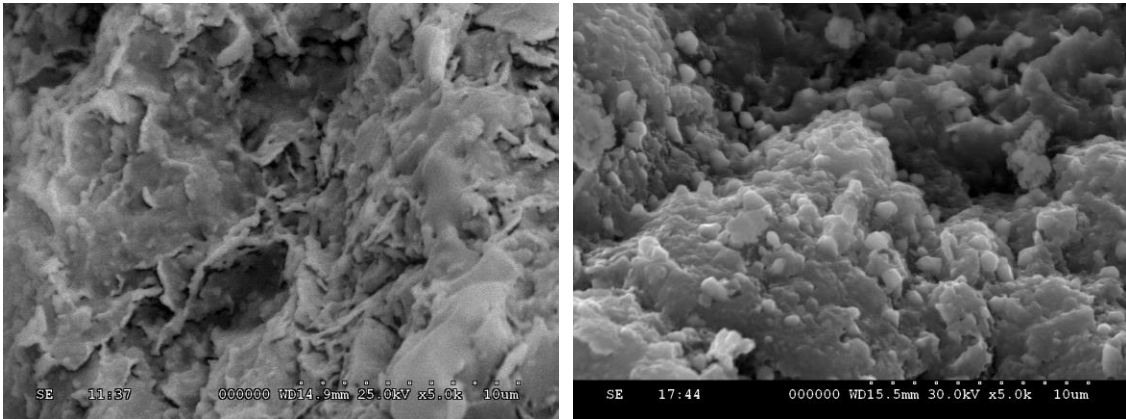


Figure 4-19 SEM image (a) Control sample (b) Sample treated with 1:300 ratio

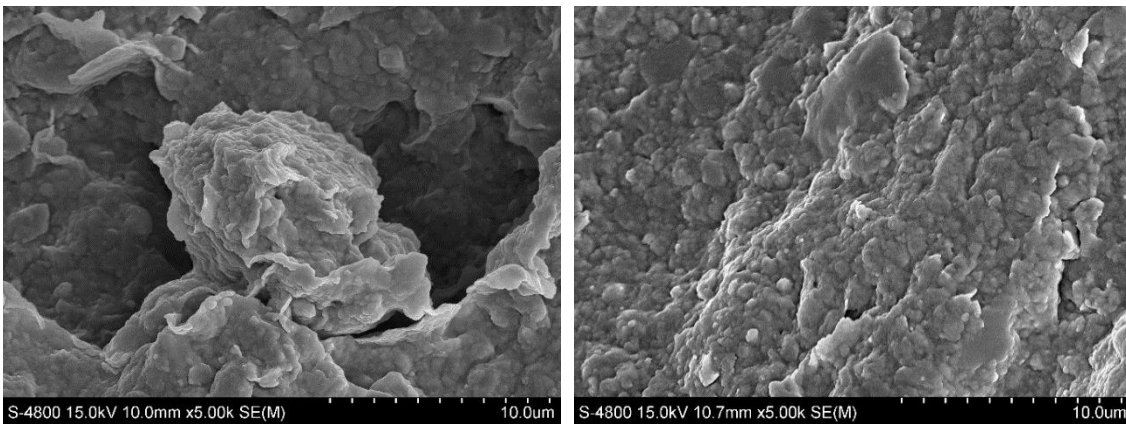


Figure 4-20 FE-SEM image (a) Control sample (b) Sample treated with 1:150 ratio

There are some changes in the image before and after the treatment with 1:300 ratio. The flaky observations before treatment has vanished in the second picture and there is some bead-like appearance in the soil after treatment. Similarly, treatment with 1:150 ratio also shows some changes in the morphology before and after the treatment. The particles seem to come closer with the treatment. Somewhat flaky edges can be seen on the image before treatment which is not seen after the treatment. But the features of images weren't consistent in all the images. Some images revealed modification of structure while

other revealed no modification. SEM image interpretations are tricky and can't be solely relied upon until the results are validated through other tests like EDS and XRD.

4.5.3 Energy dispersive spectrometer of control and treated soil

Energy dispersive spectrometer of soil was used to identify the elements in the control and the treated sample. Figure 4-21 to 4-23 shows the typical EDS spectrum of control and the treated soil.

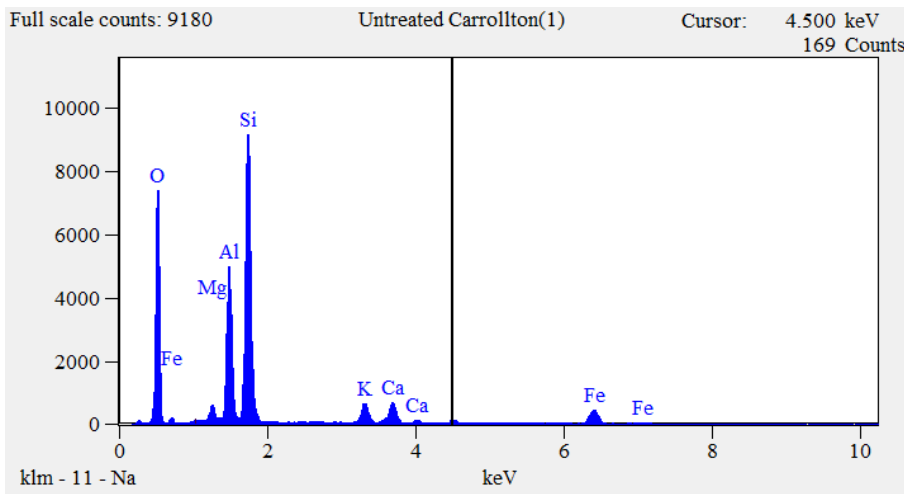


Figure 4-21 Typical EDS of Control Soil

Table 4-5 Major Element Percentage for Control Soil

	O (%)	Si (%)	Al (%)	Mg (%)	K (%)	Ca (%)	Fe (%)
Trial 1	55.42	24.52	11.06	0.79	2.06	2.32	3.84
Trial 2	58.59	21.88	9.92	0.71	1.62	3.84	3.44
Trial 3	58.77	21.77	10.47	0.72	1.76	2.66	3.85
Average	57.59	22.72	10.48	0.74	1.81	2.94	3.71

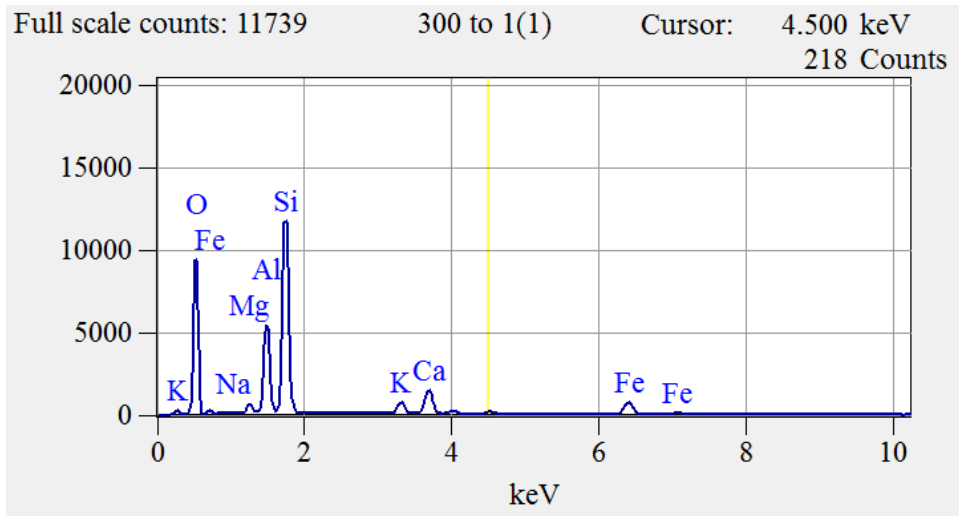


Figure 4-22 Typical EDS of treated soil (1:300)

Table 4-6 Major Element Percentage for treated soil (1:300)

	O (%)	Si (%)	Al (%)	Mg (%)	K (%)	Ca (%)	Fe (%)
Trial 1	55.05	23.63	9.38	0.73	1.84	4.28	4.86
Trial 2	52.17	22.24	9.75	0.75	2.14	4.69	7.85
Trial 3	42.91	30.76	12.93	0.70	4.42	3.71	4.30
Average	50.04	25.54	10.69	0.73	2.8	4.23	5.67

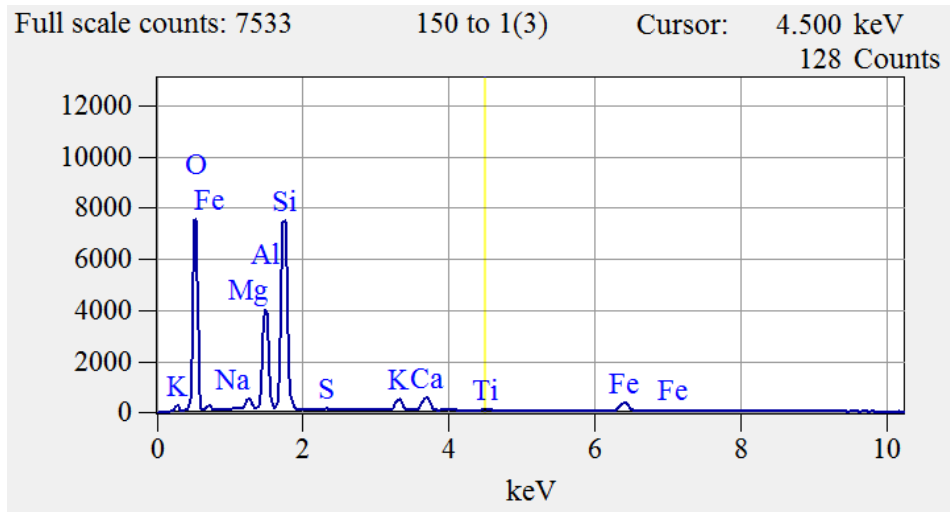


Figure 4-23 Typical EDS of treated soil (1:150)

Table 4-7 Major Element Percentage for treated soil (1:150)

	O (%)	Si (%)	Al (%)	Mg (%)	K (%)	Ca (%)	Fe (%)
Trial 1	58.46	22.30	10.15	0.77	1.62	2.33	3.49
Trial 2	54.10	22.15	11.60	0.57	2.13	3.66	4.98
Trial 3	54.58	29.43	10.02	0.66	1.51	1.11	2.44
Average	55.71	24.63	10.59	0.67	1.75	2.37	3.64

As seen in the spectrum, the soil comprises of elements like oxygen, silicon, aluminum, magnesium etc. The EDS result of all the samples were obtained at a magnification of 1000. Table 4-1 shows the percentage of major elements in the treated and untreated samples by their weight.

Table 4-8 Composition of elements in the soil sample before and after treatment

	O (%)	Si (%)	Al (%)	Mg (%)	K (%)	Ca (%)	Fe (%)
Control	57.59	22.72	10.48	0.74	1.81	2.94	3.71
1:300	50.04	25.54	10.69	0.73	2.8	4.23	5.67
1:150	55.71	24.63	10.59	0.67	1.75	2.37	3.64

The soil also consisted of few other elements like titanium and sodium in very trace amount. The composition of elements in the soil before and after the treatment do not reveal much change. Percentage weight of the elements is almost the same before and after the treatment. The Al:Si ratio is a good indicator of weathering of silica mineral. In the natural weathering process, silica is released from the clay and hence the Al:Si ratio increases. On the other hand, if Al is selectively extracted from the clay, the Al:Si ratio decreases (Rauch et al. 2003). The suggested mechanism of cationic exchange would mean selective extraction of aluminum followed by the decrease in Al:Si ratio. The Al:Si ratio was determined using the average weight percentage result of the scanned matrix from EDS which can be an indicator of chemical structure change due to stabilizing effects of ionic stabilizer. The decrease in aluminum to silica ratio means an increase in silica or decrease in aluminum due to transfer of Al^{3+} cations to stabilizer (Rauch et al., 2003). The result above shows slight decrease in Al:Si ratio from 0.46 to 0.42 and 0.43 after the treatment with 1:300 and 1:150 ratio treatment.

4.5.4 X-ray diffraction comparison of control and treated soil

X-ray diffraction analysis data shows the peak of quartz as a distinct peak. Quartz is a good scatterer of x-ray and hence the peaks of quartz are significantly larger in the plots shown in figure 4-24. The 2θ values at approximately 20, 29- and 35-degrees match

with the montmorillonite spectra in the database. The peaks located at 2θ of approximately 21 and 26 degrees is the peaks for the quartz. The result shows no any changes in the position of the montmorillonite peaks after the treatment. The usual trend for the effective treatment is the shift in the peak of the position towards the right side. When there is shift in the position of the peaks towards the right side, the 2θ angle increases. As a result, the d-spacing of the mineral decreases according to Bragg's Law.

$$n\lambda = 2d_{hkl}\text{Sin}\theta$$

Similar results were obtained in the study by Katz et al. (2001). The montmorillonite peak at 29 degree was not seen after the treatment which might have resulted as a result of change in orientation and shift in the peak was observed at around 7 degree. However, the shift was not significant enough to conclude that significant weathering has occurred as the d-spacing was above 10 Angstrom even after the treatment. No any significant changes or shift of peaks were obtained in the study of stabilization effect of ionic stabilizer on expansive soil as shown in figure 4-25.

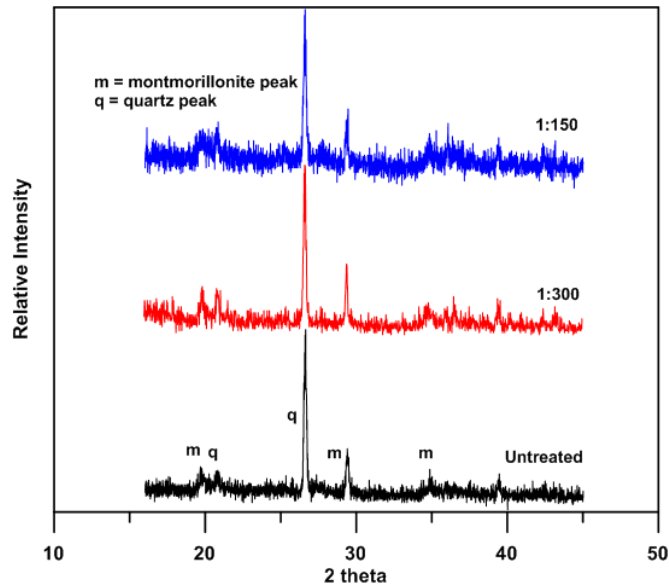


Figure 4-24 XRD pattern of treated and untreated soil

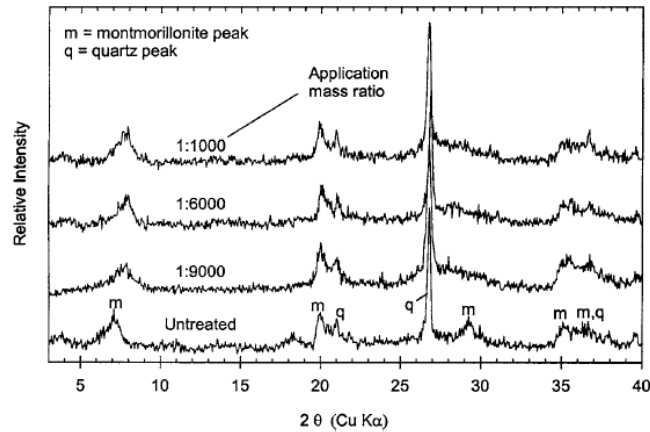


Figure 4-25 X-ray diffraction patterns for composite clay samples treated with the ionic stabilizer at different application mass ratios (Katz et al. (2001))

4.6 Effect of OMC and MDD on 1-D swell and unconfined compression

After analysis of all the results from one-dimensional swell test, unconfined compressive strength test and micro analysis of the soil, it was evident that apart from swell results, other results did not demonstrate the efficacy of ionic stabilizer in demonstrating the stabilizing effect on soil. Loss in strength after the stabilization and especially the XRD results which portrayed no any structural change in the soil cast doubt on the performance of the stabilizer. The behavior of stabilized soil especially in unconfined compression where loss in strength was observed after treatment hinted that the hypothesized reaction supposed to occur between the soil and stabilizer in the presence of water and surfactant occurred in a very trace amount or did not take place at all. If this happened, then it would mean that stabilizer remained as an inert component in the soil while increased water content led to the decrease in strength of the soil. To validate the results, samples were prepared again for unconfined compressive strength test. However, this time, the treated samples were prepared at lower moisture content and higher dry density both equal to the OMC and MDD of untreated soil. Similarly, for the swell test, control sample was prepared

at higher moisture content and lower dry density both equal to OMC and MDD of treated soil (1:150 ratio). Again, treated sample was prepared at lower moisture content and higher dry density equal to OMC and MDD of untreated (control) soil for one-dimensional swell test. The results are presented in the following sections.

4.6.1 Effect of Changed Moisture Content and Density on Unconfined Compressive Strength of Soil

Figure 4-26 and 4-27 show the effect of changing the moisture content and density of compaction on the treated soil as compared to the untreated soil. The result shows increase in strength as compared to the previous results where decrease in strength was noticed. The strengths now are comparable to original strength of control soil. The peak strength is occurring at slightly lower strain and there is also slight effect of curing as the strength in 28 days is slightly higher than strength is 7 days.

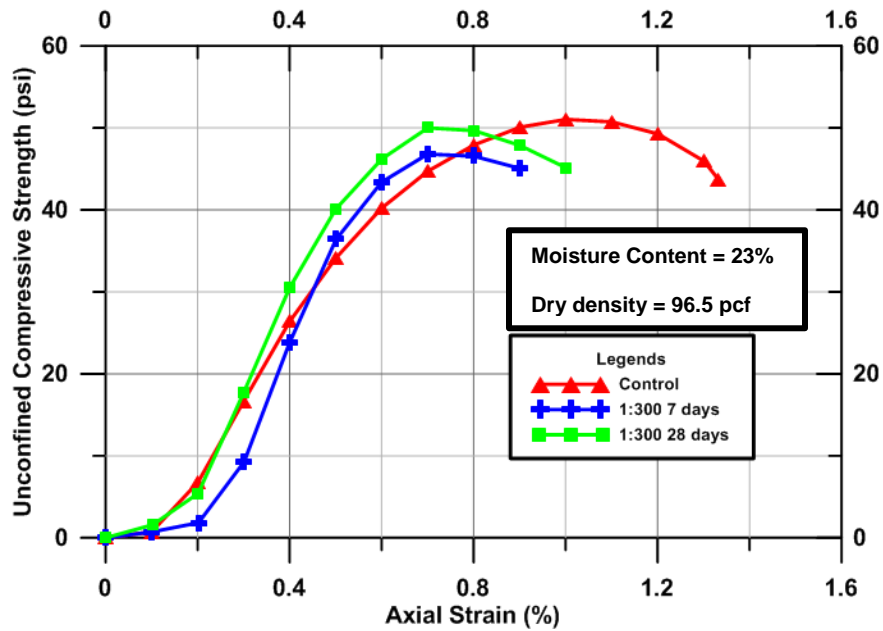


Figure 4-26 Effect of increasing moisture and decreasing density on UC strength of soil after treatment with 1:300 ratio

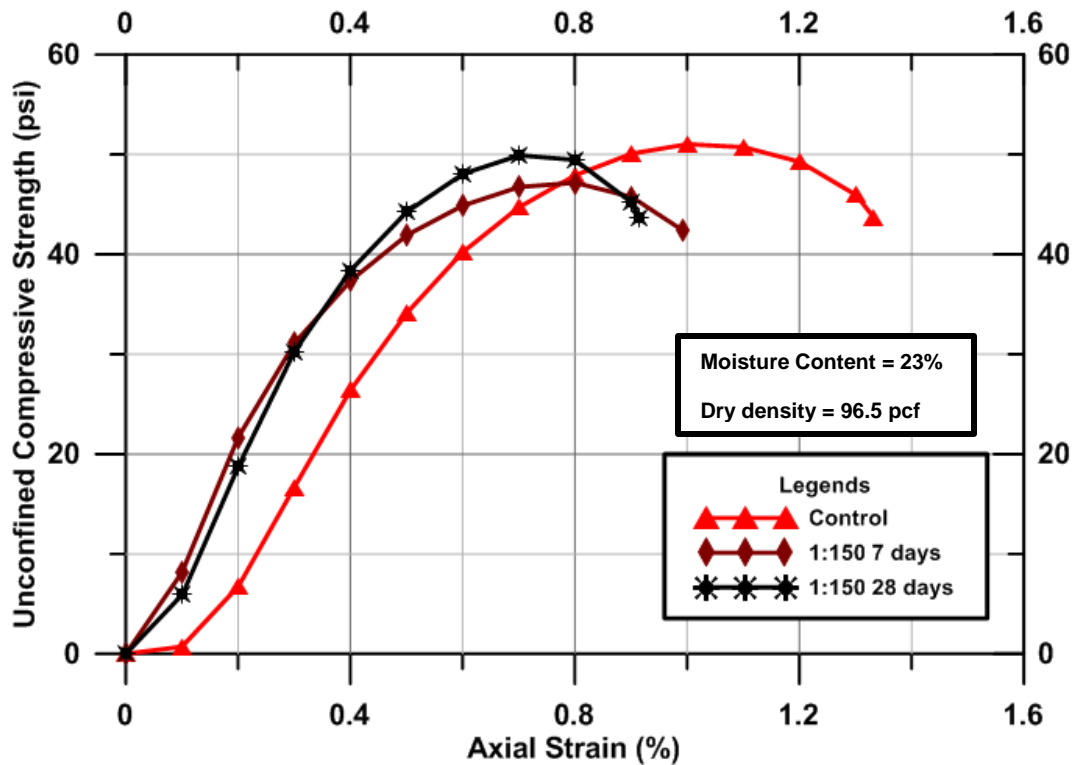


Figure 4-27 Effect of increasing moisture and decreasing density on UC strength of soil after treatment with 1:150 ratio

4.6.2 Effect of changed moisture content and density on one-dimensional swell potential of soil

As the treated samples were prepared at lower moisture content and higher density and untreated samples were prepared at higher moisture content and lower density, surprising results were obtained. Figure 4-28 shows the effect of changing moisture and density in the treated and untreated sample.

Figure 4-28 shows that the swell potential of the soil even after treatment has risen to 9.38% as opposed to its initial swell potential of 4.4% after 7 days of curing when prepared at its OMC and MDD. The swell potential of 9.38% is almost equal to the swell potential of control soil which is equal to 9.25%. Again, when the swell potential of control

soil was checked at increasing moisture content and decreasing density, the swell potential of control soil decreased to 5.35%. The initial moisture content and density of compaction plays important role in the swelling of the soil. When the initial moisture content is lower, the soil absorbs more water and swells more. The graph shows that the swell potential was reduced in the previous case because they were prepared at different initial moisture content and density. The effect of stabilizer is less pronounced, and the overall swell potential seem to reduce only because of the higher initial moisture content and lower density.

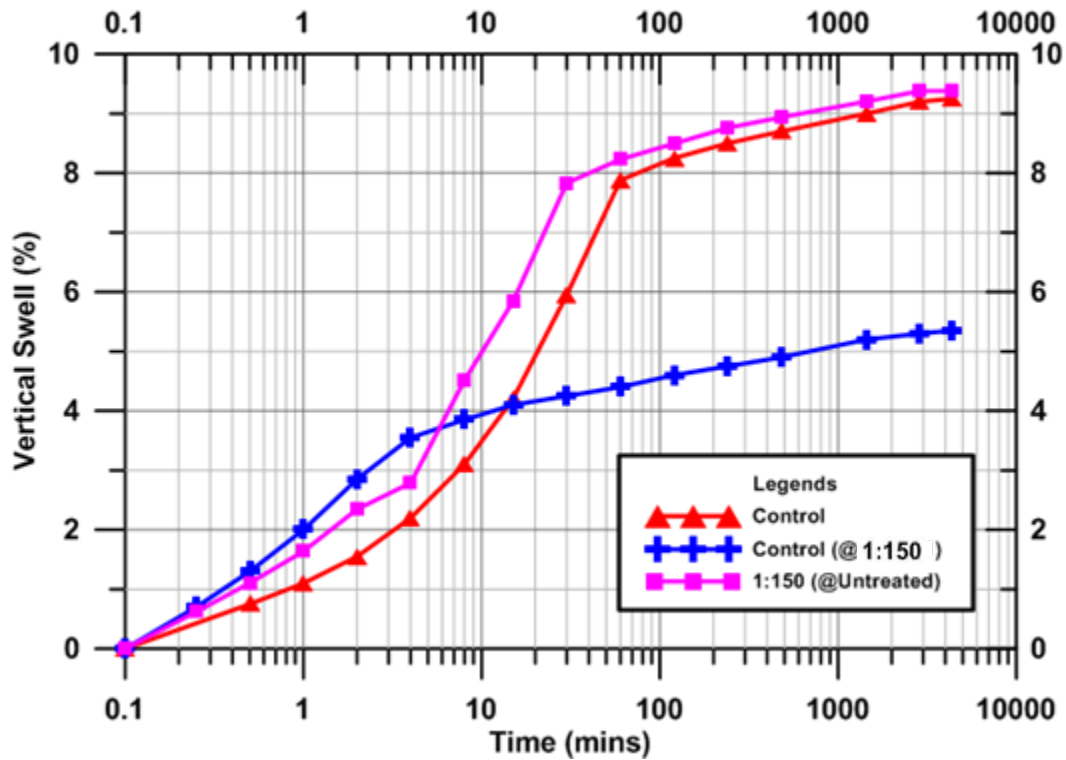


Figure 4-28 Effect of initial moisture content and density of compaction in vertical swell

Chapter 5

A Case Study of Field Application in Burleson, Texas

5.1 Introduction

Ionic stabilizer is used in stabilization of expansive soil under the foundation of residential building. The method of application of stabilizer in the field is completely different than what is practiced in the laboratory. Because of this reason, laboratory results sometimes are different than that is observed in the field. This chapter includes the results from the samples collected from the different site in Shelby tube both before treatment and after the treatment. and compares the result with the samples treated in the laboratory as well.

5.2 Site Selection

A residential home construction site of Burleson, Texas was chosen for the study. The site was stabilized using liquid ionic soil stabilizer. Samples were collected before and after the treatment in the field. Samples were collected in Shelby tube and bulk form. The depth of treatment extended up to 10 feet. Shelby tube samples were obtained up to the depth of 10 feet. However, bulk samples were obtained from 2 to 4 feet due to limitation of equipment for excavation. Figure 5-1 shows the site stabilized with the ionic stabilizer.



Figure 5-1 Site for soil stabilization

5.3 Materials and Experimental Methods

The soil obtained for the study was characterized as high plastic expansive clay.

Basic properties of the soil are listed in the table below.

Table 5-1 Basic properties of control soil

Soil Properties	Control Soil
Liquid Limit	59.4%
Plastic Limit	26.2%
Plasticity Index	33.2%
USCS Classification	CH
In-situ Moisture Content	20.9
Dry Density	96.5 pcf

The ratio of the treatment used by manufacturers is 1:300 which means one part by volume of chemical is diluted by 300 parts by volume of water and applied in the field.

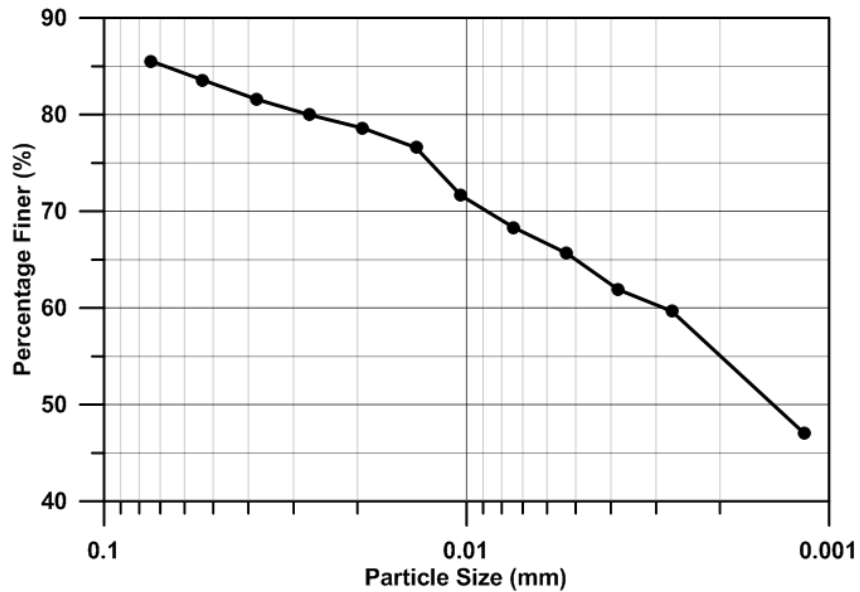


Figure 5-2 Grain size distribution curve for Burlinson soil

The laboratory experiments mainly focused on the swell test of the treated and untreated specimen from the field. Basic tests like grain sized distribution, minus 200, Atterberg's limit and linear bar shrinkage were also performed. The bulk soil treated in the field was mixed with distilled water in the lab to obtain the properties and do further test. However, the bulk untreated soil from the field was mixed with ionic stabilizer in the laboratory to do the further tests. Further, results from hand-mixed sample were also compared. Percentage passing number 200 was found to be 90.72%. The clay percentage for the soil was 55% as shown in the figure 5-2.

Finally, the strength comparison between control and treated sample by observing unconfined compressive strength. However, due to limitation of Shelby tube samples, unconfined compression test was done on hand-mixed samples only.

5.4 Results

The results obtained from the laboratory tests are discussed in the following sections.

5.4.1 Atterberg's Limit

Liquid limit and plastic limit tests were carried out in accordance to ASTM standards. Laboratory results showed some positive effect in the liquid limit of the soil. However, plasticity index of the soil remained unaffected. The results are shown in the figures 5-3 and 5-4.

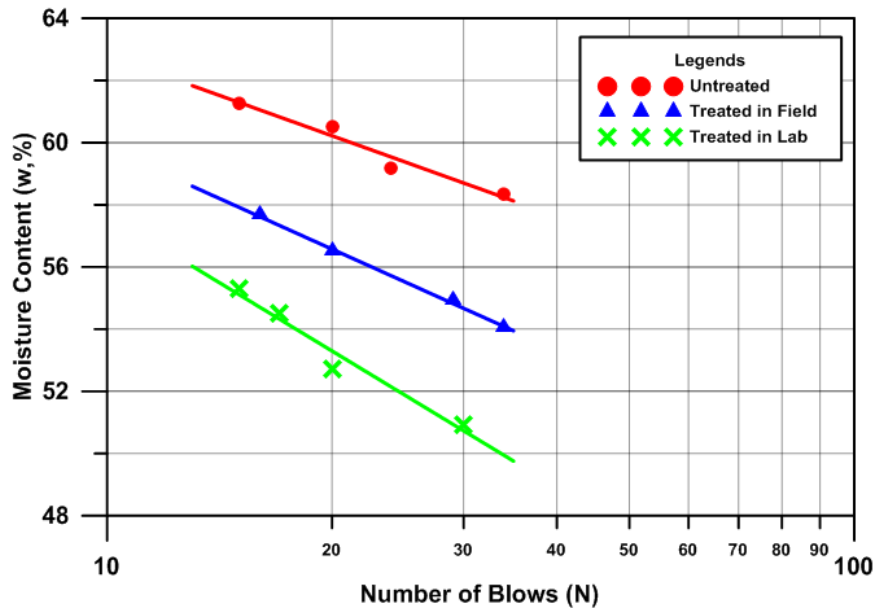


Figure 5-3 Flow curve of Burleson soil treated in different ways

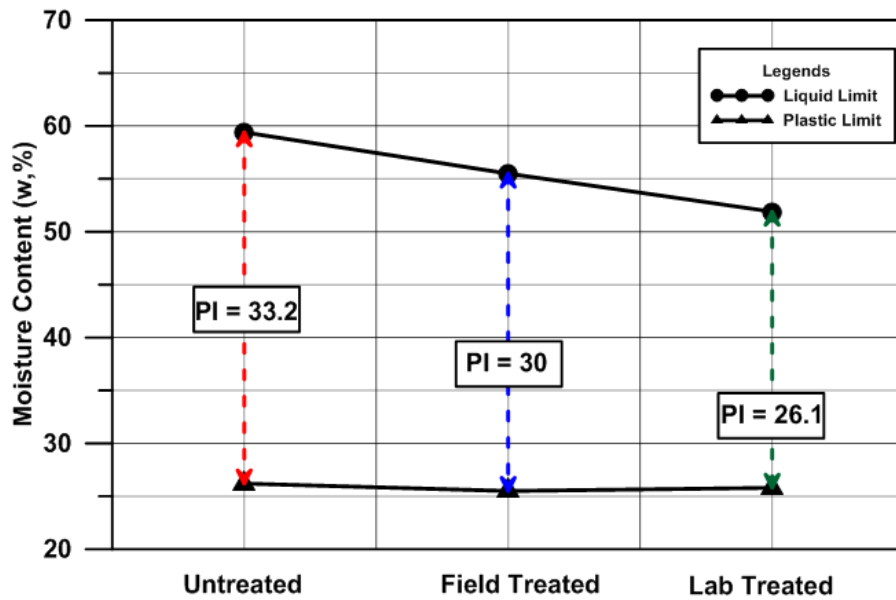


Figure 5-4 Atterberg's Limit of control/treated Burleson soil

Figure 5-3 shows the reduction of liquid limit with treatment. The liquid limit of control soil is 59.4% and the corresponding plasticity index is 33.2. After treatment in the

field, the PI reduced to 30% and after treatment in the lab, the PI reduced to 26.1%. The soil treated in the field was mixed with distilled water to obtain the Atterberg's limit. However, soil treated in the lab was mixed with chemical solution in the lab itself.

The reduction in PI can be attributed to the change in affinity of the clay particles. Ionic stabilizers convert the hydrophilic clay to hydrophobic because of which their affinity to water is lost and correspondingly, the plasticity index is reduced. As the application mass ratio i.e., net chemical available to the solid soil particles are greater in the laboratory treated sample than in field treated sample, the plasticity index further reduced with the laboratory treatment method.

5.4.2 Bar Linear Shrinkage

Figure 5-5 shows effect of treatment in the linear shrinkage of the soil. The linear shrinkage of the soil has reduced from 21.55% to 16.88% after treatment in the field and 16.74% after treatment with the laboratory method. Unlike plasticity index, the difference between the linear shrinkage of the soil treated in the field and treated with laboratory method is insignificant. However, reduction in linear shrinkage shows stabilization impact of ionic stabilizer and its potential in limiting the shrinkage beyond certain limit.

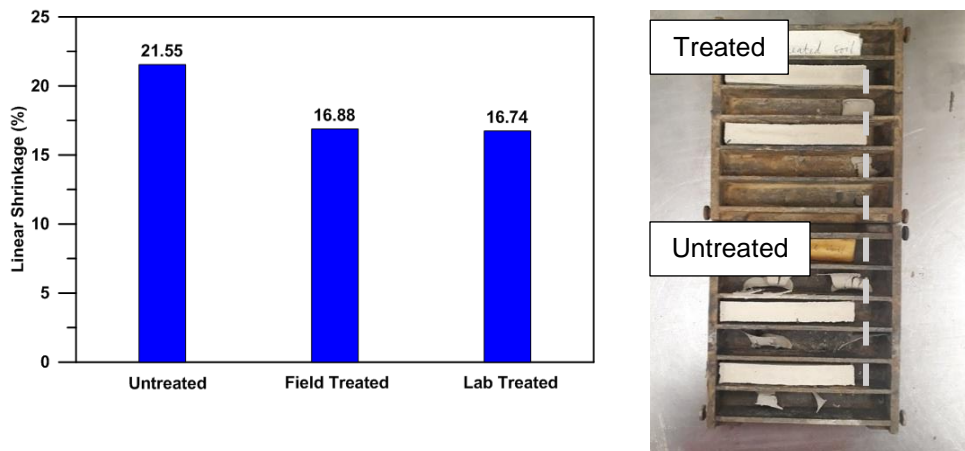


Figure 5-5 Linear shrinkage of control/treated soil

5.4.3 Standard Compaction Test

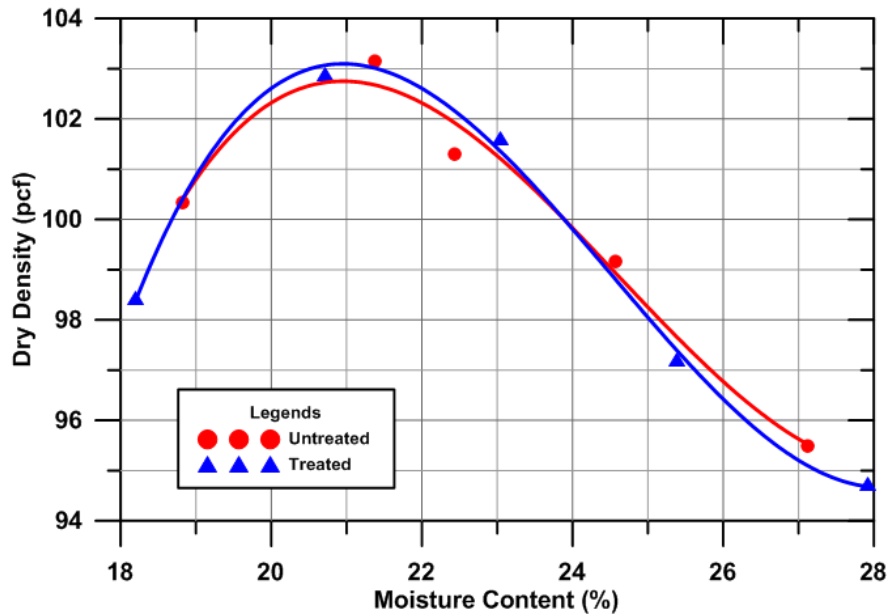


Figure 5-6 Standard Compaction Curve

Figure 5-6 shows the relationship between moisture content and dry density of the soil for treated and control specimens. Both the treated and untreated samples were prepared at different moisture content and were kept in the humidifier overnight before compaction. The graph indicates that there was no any pronounced effect on the compaction characteristics of soil before and after the treatment. The OMC is obtained at 21% and MDD is nearly equal to 103 pcf for control and treated samples.

5.4.4 Percentage of Major Minerals

The cation exchange capacity and specific surface area of the Burleson soil was quantified according to the procedures outlined in chapter 3. The cation exchange capacity of the Burleson soil was obtained to be 77.5 meq/100 gm and the specific surface area of the soil was found to be 127.21 m²/gm. Using the correlation provided by Chittoori B (2008), the montmorillonite percentage was found to be 27.5%. Total potassium for the Burleson soil was measured using Energy Dispersive Spectrometer (EDS). The average

potassium content for the Burluson soil was obtained to be 1.88%. Using the correlation provided by Chittoori (2008), the percentage of Illite was obtained to be 31.3% and the percentage of Kaolinite was obtained to be 41.2%.

5.4.5 One-dimensional swell test

Figure 5-6 shows the results obtained from one-dimensional swell test. The swell potential of the untreated sample is about 5 to 5.5 percentage. Higher swell potential is seen in the laboratory prepared sample than in the field treated Shelby tube sample. The swell potential dropped from 5% to 2.6% for the field treated sample. However, the drop of swell potential for lab treated sample is from 5.5% to 3.45%. which is 0.35% less than the Shelby tube specimen. There is almost 1% difference in the maximum swell potential obtained between field and laboratory treated specimen. In the laboratory, the target moisture content of the treated sample was kept equal to the in-situ moisture content (before treatment) of 20.9%. However, in the field, the moisture content was increased to 25.4 % after the treatment and pre-wetting.

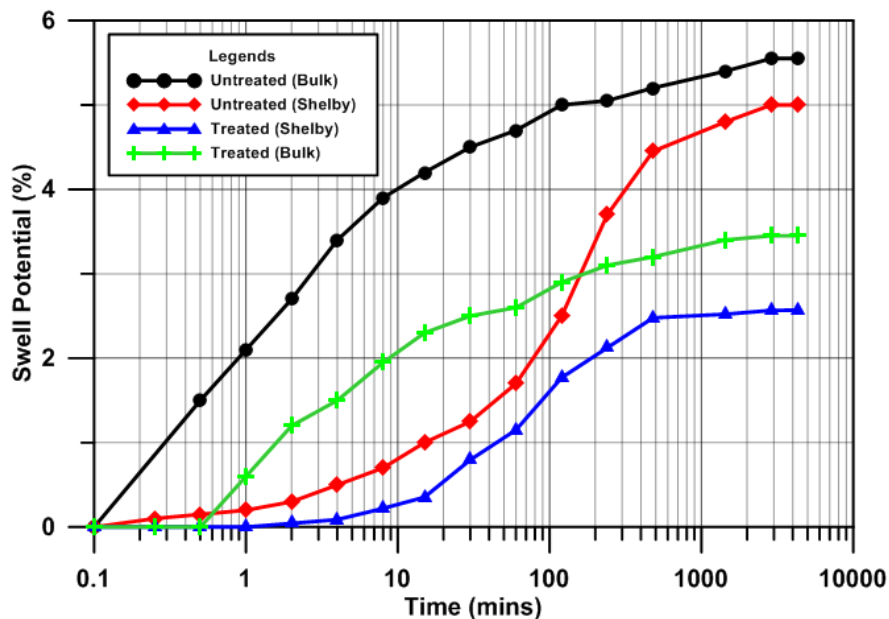


Figure 5-7 Effect of Ionic Stabilizer in Swell Potential of Bulk/Shelby tube Sample

5.4.6 Swell Pressure and Fatigue of Swelling

Swell pressure test showed there was a reduction in the swell pressure after the treatment with the stabilizer. Swell pressure test was done on the Shelby tube sample before and after the treatment. From figure 5-7 (a), it can be observed that without treatment the soil showed a large swell pressure of about 1,900 psf. However, after the treatment the swell pressure reduced to about 900 psf.

Similarly, fatigue of swelling test as in figure 5-7 (b) showed that the sample exhibited constant swelling after 5-6 cycles of wetting and drying. Final axial strain or swell potential was observed to be 2.04%. The swell was stable after 6 wet-dry cycles with the residual swell potential of 2.04%. However, the test could not be done for the laboratory-molded sample as it was too fragile after drying and lost its weight as the soil mass came off it.

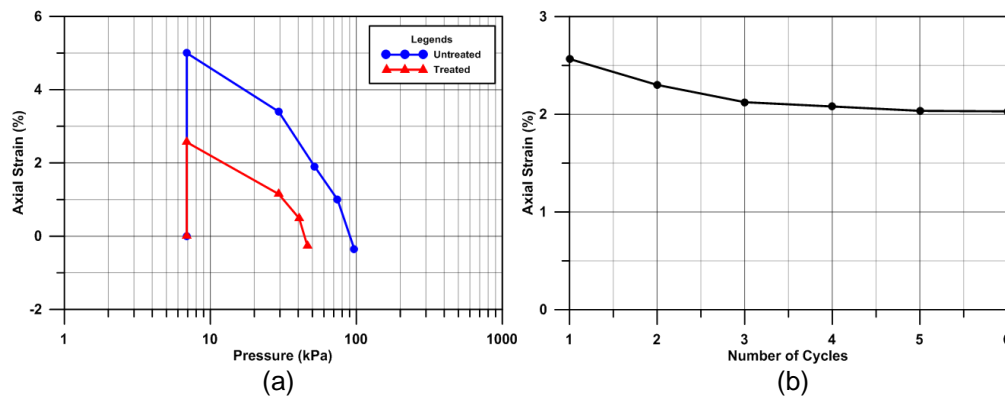


Figure 5-8 (a) Swell Pressure Comparison between Treated and Untreated Shelby tube Sample (b) Fatigue of Swelling for Filed Treated Sample

5.5 Conclusion

This case study compared the finding from both the samples treated in laboratory and treated at their in-situ condition. The result showed some promising influence of ionic stabilizer in reducing plasticity index and linear shrinkage. After treatment, the swell

potential and swell pressure was reduced indicating that the soil was transformed from hydrophilic to hydrophobic. Also, the results compared between Shelby tube sample and the sample prepared in the laboratory showed some discrepancies which is obvious given the disturbance subjected on the laboratory sample. However, the samples prepared in the laboratory was deemed viable to study the effects before and after the treatment.

Chapter 6

Summary and Conclusions

6.1 Summary

Stabilization of expansive soil using chemical additives has been practiced for a long time. Stabilization using ionic soil stabilizers is relatively a new topic in chemical stabilization of expansive soil. Although it has been claimed to be practiced for more than decades, there is a lack of solid documenting histories available for ionic stabilizers. This research has attempted to bridge the gap of the study of ionic soil stabilizers by documenting some of the findings from both the laboratory and field tests.

This study was focused on treating the expansive soil from two separate places from Texas viz. Carrollton and Burleson where the soil was stabilized using the ionic stabilizer. The treatment effect on Burleson soil was presented as a case study as Shelby tube samples from the treated site were obtained. The focus was to evaluate the efficiency of doses of stabilizer in reducing the vertical swell potential and strengthening the soil and validate these results through microanalysis of the soil. The microanalysis of the soil would aid in understanding the mechanism of stabilization and provide evidence to the hypothesis proposed by previous researchers.

The study can be divided into three parts. In the first part of the study, two doses of treatment were chosen and applied to the control soil from Carrollton and then the laboratory study was done on this soil to evaluate the performance of the stabilized soil in comparison to the control soil. First, the control soil was characterized through a series of experiments like Atterberg's limit, linear shrinkage, cation exchange capacity, surface area test and other basic tests in laboratory. Further, the control soil was treated, and the effect of treatment were measured mainly through the observation of swelling and strength at

different conditions of treatment which included doses of treatment, initial moisture condition and density of compaction at different curing days.

The second part of the study was focused on observing the changes of the structure of the particles at the micro level. The study was achieved through the aid of electron technology like scanning electron microscope, energy dispersive spectrometer and x-ray diffraction. The second part of the study required good sample preparation which was done referring to the available literature and modifying the procedure as per the available resources.

The third part of the study focused on the case study utilizing Shelby tube samples from the field. The study portrayed the results before and after stabilization in the field and the laboratory through a series of tests viz. Atterberg's limit, bar linear shrinkage, one-dimensional swell test, unconfined compression, and micro study.

6.2 Conclusions

The study on Carrollton soil especially the swell study revealed that the swell potential of the soil was reduced when compacted at their respective moisture content and dry density. So, in order for the stabilizer to be effective in reducing the swell, the soil is recommended to be compacted at its optimum moisture and density. This will help in reducing the swell potential of the soil. Following points can be concluded from the experiments on expansive soil from Carrollton.

- Liquid limit of the soil increased after the treatment with 1:300 ratio and decreased after the treatment with 1:150 ratio. However, the change in the liquid limit was not significant. Linear shrinkage of treated soil slightly increased after the treatment.
- The soil compaction curve was altered by the ionic soil stabilizer treatment. Optimum moisture content of the soil increased from 23 to 27% with the treatment

for both ratios while the maximum dry density decreased from 96.5 pcf to 91 pcf after treatment with 1:300 ratio and to 92.5 pcf after treatment with 91 pcf.

- One-dimensional vertical swell of the soil decreased with the treatment when samples were prepared at their respective OMC and MDD. The swell reduced from 9.3% to 4.5% after treatment with 1:300 ratio and 7 days of curing. Similarly, it reduced to 4.4% after treatment with 1:150 ratio and 7 days of curing. There was negligible reduction in swell after 7 days.
- The reduction of the swell cannot be completely attributed to the stabilizer as moisture content and density played a role in it. When preparing the treated samples at the OMC and MDD of untreated soil, the treated sample showed only small reduction in swell. The stabilizer was effective in reducing only about 1% of swell.
- Unconfined compressive strength of the soil decreased with the treatment when the samples were prepared at their respective OMC and MDD. The average strength decreased from 48 psi to 36 psi and 42 psi for 1:300 and 1:150 ratio of treatment respectively after 7 days of curing. The strength was almost constant after 7 days of curing.
- The treated soil when prepared at optimum moisture content and dry density of untreated soil showed increase in strength compared to its previous strength at respective OMC and MDD. The average strength of the treated soil at this condition was similar to untreated soil.
- SEM image of sample showed some changes in the soil fabric after the treatment. The result however, were not consistent and seen only in some pictures. EDS analysis did not reveal any significant change in composition of major elements.

The field case study presented in chapter 5 showed encourage results regarding reduction of the plasticity index of the soil and swell potential. The prewetting process inculcated in the field increases moisture content of the swell and might change the density of the soil as well. The application of treatment in the field is done after prewetting. So, it is important to segregate the impact of prewetting and treatment in the swell reduction. The Shelby tube samples utilized for swell test after the treatment had higher initial moisture content. There was reduction in swell potential. To make sure that the swell reduction occurred due to treatment and not because of the increased moisture content, treated laboratory samples were compacted at in-situ moisture content and density. Upon performing the test, the swell was reduced unlike Carrollton soil. In case of Carrollton soil, the effect of moisture and density variation were more pronounced than the treatment effect itself. However, in case of Burleson soil, with constant moisture and density between control and treated specimen, reduction in swell was observed. Following points can be concluded from the experiments on expansive soil from Burleson.

- Liquid limit of the soil decreased after the treatment. The results were consistent with both the Shelby tube samples and hand mixed samples in the laboratory. The liquid limit of the control soil was 59.4% and the plastic limit was 26.2. The plastic limit remained unaffected of treatment. However, liquid limit reduced to 56.2% and 52.3% respectively for soil treated in field and soil treated in lab.
- Bar linear shrinkage of the soil reduced from 22% to 17% after the treatment which suggests that stabilizer was efficient in controlling the shrinkage of the soil beyond certain limit.
- The swell potential of the sample reduced after the treatment. The results were consistent with both the field and laboratory treated samples. The swell reduced

from 5% to 2.6% for the sample treated in the field and to 3.5% for the sample treated in the laboratory. In the field, the injection procedure and wetting of the field to near saturation with chemical increases the application mass ratio of the stabilizer to the soil solid. However, in the laboratory the soil samples were prepared at in-situ moisture and density before treatment. So, the application mass ratio for the samples prepared in the laboratory is relatively lower compared to the field sample. Further, in the field after the treatment, the moisture of the soil increased to 25.4% which contributed to further reduction in swell in the field.

- The swell pressure also reduced after the treatment. The swell pressure for untreated soil in the field was about 1900 psf which reduced to 900 psf after the treatment.
- There was reduction in swell potential after each cycle of drying and wetting. The swell stabilized after 5-6 cycles with the residual swell potential of 2% which is relatively low value for expansive soil.

Based on the study from two soils, the response of both the soils were different to the stabilizer effect. It might be an important finding that stabilizer may not be compatible with every type of soil at all test conditions. There are some obvious differences between these two soils. Carrollton soil despite being expansive soil has very less amount of clay fraction in it. The clay fraction for Carrollton soil accounts only for 10 percentage. However, the clay fraction in the Burleson soil is very high which accounts for 55 percent of clay. These findings from the study of two soils align with the study from some literature. Ionic soil stabilizers are effective in the soil with clay percentage greater than 25% (Xiang et al., 2010). To make the maximum use of the stabilizer in the field, a pre-test in the laboratory to find out the optimum conditions and doses of use is necessary.

6.3 Recommendation for Future studies

The results presented in this thesis highlights two important things. Looking at the case of Carrollton soil, the swell potential of the soil was reduced at optimum conditions of moisture and density. However, this led to decrease in strength of the soil. With Burleson soil, the stabilizer proved to be effective in reducing swell even at the in-situ field condition before treatment unlike Carrollton soil. Pre-wetting itself is one of the techniques employed to reduce the swell potential of soil. Also, the inundation of field with chemical solution leads to increased amount of stabilizer available for soil solids. However, testing the soil at other moisture and density condition in the laboratory might be one reason why only small reduction in swell was observed for Carrollton soil. Hence, future study should focus on better simulating the field conditions by applying the injection technique in the laboratory itself at high pressure. Further, it is important to find the compatibility of ionic soil stabilizer with the soil. This can be done by performing laboratory tests before field injection. The optimum properties at which the stabilizer works without compromising much of its strength should be evaluated before field application and applied in the field accordingly.

The study in this thesis focused in evaluating the performance of soil after stabilization through treatment. The composition of the stabilizer was not known properly and the knowledge about its composition was based on manufacturer's statement. Future studies should focus on evaluating the composition of ionic stabilizer through chemical study of the stabilizer and synthesizing the same in the laboratory. Stabilization should be carried out in large number of expansive as well as high-sulfate soil to determine the suitability of ionic stabilizers and criteria of its use. Micro analysis of soil should be carried in the soil where ionic stabilizers prove to effective so that underlying mechanisms can be well understood.

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