A REFINED APPROACH TO IDENTIFYING AND QUANTIFYING CLAY AND NON-CLAY MINERALS IN EXPANSIVE SOILS

by

SPOORTHI REBALLI

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Abstract

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Spoorthi Reballi, M.S.

The University of Texas at Arlington, 2014

Supervising Professor: Anand J. Puppala

Expansive soils exhibit substantial swelling and shrinkage due to moisture content fluctuations. Predicting this volume change behavior of expansive soils has been a major challenge to the engineers in the past few years. One of the factors which play a vital role in understanding the behavior of the expansive soils is mineralogy. Chittoori (2008) had developed three models to quantify the clay minerals using Microsoft Excel Solver, Regression Equations and Artificial Neural networks. The previous models have only considered clay fraction, i.e. montmorillonite, kaolinite and illite. But the non-clay mineral contents like quartz and feldspar were not taken into consideration. Hence the main objective of this research is to include the quantification of both non-clay and clay minerals present in the soil. Also, the inclusion of potassium from feldspar and how it impacts clay mineralogy are studied.

In order to accomplish this research objective, seventeen natural soils samples were taken with different mineralogical contents. Basic soil classification tests like hydrometer, wet sieve analysis were performed on these soils to identify the type of soil and for the separation of silt and clay fraction along with greater than 75 microns samples. Mineralogy studies on the test soils include Cation exchange capacity (CEC), Specific Surface Area (SSA) and Total Potassium (TP). The results from these tests were analyzed using the regression model developed by Chittoori (2008) and also by the modified method which identifies the mineral feldspar and quartz. Results from both the methods were compared and the percent amount of minerals, kaolinite and illite has shown considerable change in the predicted percentages. This research paves way for future research studies to increase the accuracy of the mineral quantification process by quantifying the percentages of both the clay and non-clay minerals present in the soils, which in turn will provide better understanding of the expansive behavior of natural soils.

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Chapter 1

Introduction

1.1 General

Expansive soils, also known as swell- shrink soils are found in the arid and semi-arid regions of United States. These soils cover one-third of the earth's surface (Chen, 1988). Many parts of south western United States, South America, Canada, Africa, Australia, Europe, India, China and the Middle East have reported great distresses while constructing on expansive soils (Chen, 1988). Expansive soils experience volume change with change in moisture content which results in the swell and shrinkage movements in soils thereby causing severe damage to structures built above them (Nelson and Miller, 1992). The cost of damages to homes due to expansive soils was approximately $2 to $9 billion per year (Jones and Jones 1987).

Lot of research has been done in the past in order to understand the behavior of expansive soils. The common parameter which has been used for the characterization of expansive soils is using index properties. Recent studies by Pedarla (2013); Chittoori and Puppala (2011); Teresa et al. (2004) showed that mineralogy is one of the fundamental parameter governing the swell shrink behavior of any expansive soils. Also it was shown by Pedarla (2011) and Chittoori (2013) that the current stabilizing practices based on PI and gradation of soil is insufficient for selecting an effective stabilizer as soils having same PI act differently to stabilizers and due consideration should be given to mineralogy.

Soils in general contain various amounts of crystalline clay and non-clay minerals, non-crystalline matter and precipitated salts (Mitchell and Soga, 2005). There are many methods for the identification and quantification of clay minerals like X-ray diffraction, Vibration spectroscopy, Thermal analysis and atomic absorption spectrometry. Chittoori

(2008) developed models using the chemical properties for the identification and quantification of clay minerals based on the assumption that the soil has only the dominant clay minerals montmorillonite, kaolinite and illite. This method has been validated by Sarwanaj (2009).

A recent study by Chrysochoou (2013) has shown the presence of feldspar in soil. It also showed that unconfined compressive strength and the clay mineral content and the amorphous (Quartz, Feldspar and Micas) content all change logarithmically with time, yielding an overall linear relationship between UCS-kaolinite and UCS-amorphous.

Due to the concerns relating to the accurate quantification of minerals, the need for better research on quantification of clay minerals is crucial. This thesis works on improvising the model developed by Chittoori (2008) giving more accurate value of Illite, Montmorillonite, Kaolinite, Feldspar and Quartz.

1.2 Objectives

The main objective of this research is to develop an approach where both non-clay and clay minerals present in the soil. This work is developed based on original model developed by Chittoori (2008). Seventeen samples have been taken from different locations in United States with different mineralogical contents. For achieving the thesis objective, the following tasks were executed on the soil samples.

Basic soil characterization studies were conducted using standard techniques. With the help of wet sieve analysis and hydrometer the separate clay and silt fractions were collected and further analyzed for their mineralogy. The procedure developed by Chittoori (2008) was utilized to determine the clay minerals present in the test soil fractions. Mineralogy studies on the test soils include Cation exchange capacity (CEC), Specific Surface Area (SSA) and Total Potassium (TP). Chittoori's (2008) mineralogy method did not include non-clay mineral Feldspar in the quantification analysis. The new test procedure includes potassium sources in feldspar mineral which was determined by performing total potassium on silt.

The research tasks that has to be done is presented in the form of a flow chart in figure 1-1

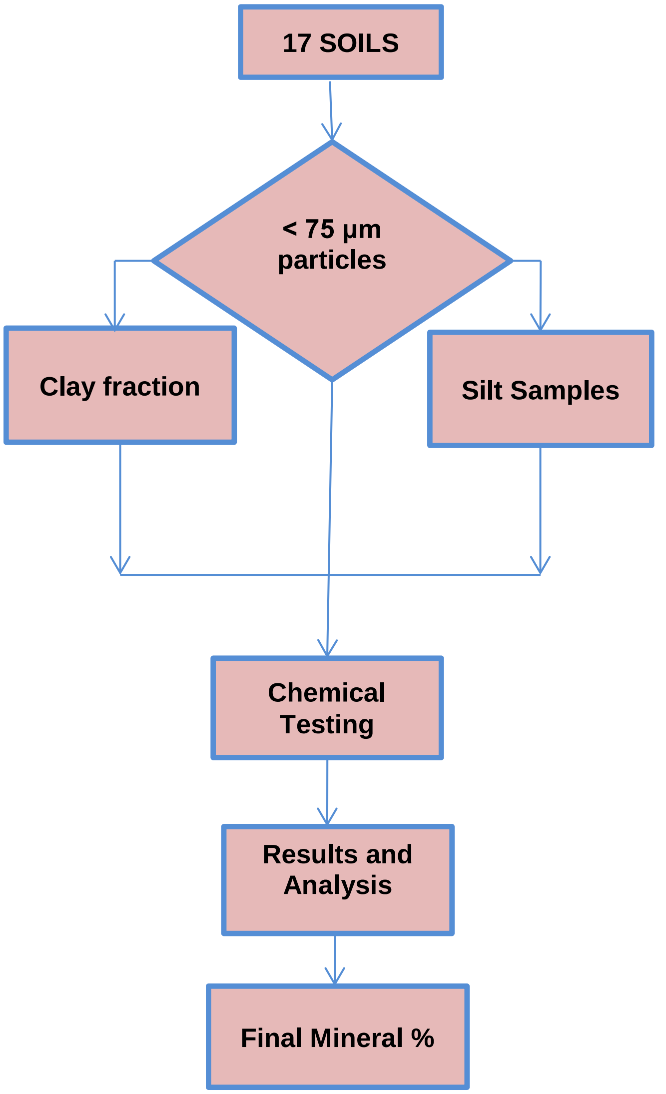


Figure 1-1 Flow Chart Representing the Research Tasks

1.3 Organization and Summary

This section will provide a brief overview of the contents of the following chapters.

Chapter 1 provides an introduction to expansive soils and how mineralogy plays a vital role in understanding its behavior, research objectives, and dissertation organization to provide a framework of the current research.

Chapter 2 provides a review of the literature available on expansive soils and the different clay and non-clay minerals present in soil. It has also given the brief review of the studies conducted by various researchers to identify and quantify them. It also provided the different methods that are currently in practice for the identification and quantification of minerals. Chemical tests that are used for the quantification are also presented in this chapter

Chapter 3 covers the basic soil classification. The experimental program includes basic Atterberg's Limit tests, Sieve analysis, Hydrometer analysis and chemical tests which are performed on soils. Test procedures for the Cation Exchange Capacity (CEC), Specific Surface Area (SSA), and Total Potassium (TP) are explained thoroughly.

Chapter 4 includes all the test results obtained from the samples. These results are used to analyze and quantify the percentage of each mineral present using the models developed by Chittoori and Puppala (2011). It includes a brief description of the model and the percentage of minerals derived using them. The mineral quantification using the modified method is also presented.

Chapter 5 provides the summary and conclusions of the research. It addresses the future research needs to improve the predictions and correlation of the data. It also addresses the possible reasons for the errors encountered in the data.

Chapter 2

Literature Review

2.1 Expansive Soils

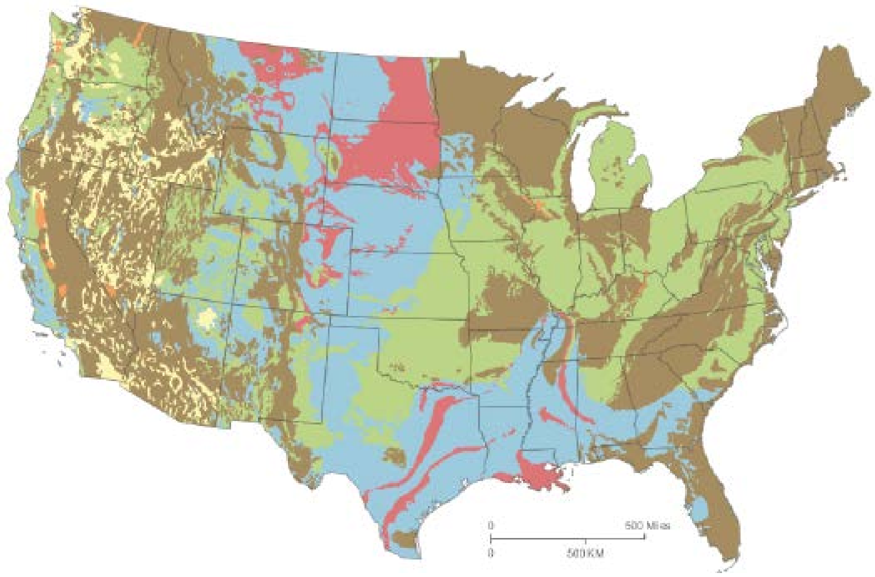
Soils which exhibit substantial swelling and shrinkage due to moisture fluctuations are termed as expansive soils. This swelling and shrinkage of expansive soil cause damage to the infrastructure built on them (Jones and Holtz, 1973, Puppala and Cerato, 2009). Each year, they cause billions of dollars of damage to the United States (Krohn and Slosson, 1980). Expansive soils are found in the arid and semiarid regions where the evapotranspiration exceeds the precipitation (Jones and Holtz, 1973). Expansive soil distribution is widely spread over many countries and more expansive soil regions will be discovered each year as the amount of construction increases.

There are many factors which influence the behavior of the expansive soils and these factors are mainly divided in three groups; soil characteristics (clay mineral, plasticity and pore connectivity), environmental factors (climate, groundwater, vegetation, soil suction, dry density and drainage) and the state of stresses (Nelson and Miller, 1992). Other factors such as stress history and current state of stress conditions will also influence the soil swell behavior patterns. Major factors, which help in understanding the behavior of these soils, are the type of mineralogy and pore size distribution (Pedarla, 2013).

The identification of expansive soils can be done in the lab by using their plastic properties. Generally Inorganic clays of high plasticity with liquid limits exceeding 50 percent and plasticity index over 30, usually have high inherent swelling capacity and are termed as expansive clay or fat clay. Examples of expansive clays are high plasticity index (high-PI) clays, over-consolidated clays rich with montmorillonite mineral and shales (Chittoori, 2008). The Expansion of these soils can also be measured in the lab directly, by immersing a remolded soil sample and measuring its volume change (Rogers et al., 1993).

The expansive soils can be classified in to three groups, the first comprises of the Sedimentary rocks that contain montmorillonite mineral, the second is rock sediment and the third is the product of weathering of rock (Ahmed, 1988).

These soils present in every state, cover one-fourth of United States (Puppala and Cerato, 2009). Figure 2-1 shows the distribution of expansive soils in United States.



🟥 Over 50 percent of these areas are underlain by soils with abundant clays of high swelling potential

🟦 Less than 50 percent of these areas are underlain by soils with clays of high swelling potential.

🟧 Over 50 percent of these areas are underlain by soils with abundant clays of slight to moderate swelling potential.

🟩 Less than 50 percent of these areas are underlain by soils with abundant clays of slight to moderate swelling potential.

🟫 These areas are underlain by soils with little to no clays with swelling potential.

🟨 Data was insufficient to indicate the clay content or the swelling potential of soils.

Figure 2-1 Distributions of expansive soils over the United States (USGS Publication)

Although expansive soils are present nationwide, certain areas like Colorado, Texas, North Dakota, South Dakota and Montana have very high concentrations of swelling soils. From the map we can see that Texas has soils with high swelling potential. This is due to the presence of dry weather conditions which results in the shrinkage of expansive soils resulting in cracks. These cracks when filled with water do not get closed and results in the soil swelling. As a result many damages are caused to the infrastructures built on these soils.

2.1.1 Problems Caused by Expansive Soils

Expansive soils in natural conditions are not suitable for the urban infrastructure. But the urbanization and the population growth led to the construction in these areas (Williams, 2003). As a result, the shrink and swell of expansive soils increase the damages to the engineering structures. It can be estimated that the annual cost of damage from these soils in the United States alone is $2 to $9 billion annually (Jones and Jones, 1987). This volume change or cyclic swell shrink behavior of expansive soils cause severe distress to engineering structures including foundations and pavement infrastructure. Some case studies with the problems caused due to the expansive soils are presented.

2.1.1.1 Building Foundation Failures

Many authors have conducted study on the building foundation failures. Jones and Jefferson (2012) reported the different types of failures caused to foundation due to the presence of expansive soils. Tree growth can also be another factor responsible for damage to foundation in expansive soils. This can cause both the physical disturbance as well as shrinkage of ground by removal of water. The failure of foundation results when volume is unevenly distributed. Al Rawas et al (2005) conducted a study on the potential problems associated with expansive soils on foundations in the Arabian Gulf.

Figure 2-2 shows the distress caused to the building by swell shrink characteristic of expansive soils.

The authors also investigated the mineralogical composition of expansive soils and rocks from this site using X-ray diffraction technique which results in the presence of smectite mineral. This can be reduced by taking the proper remedial measures before the construction of building.

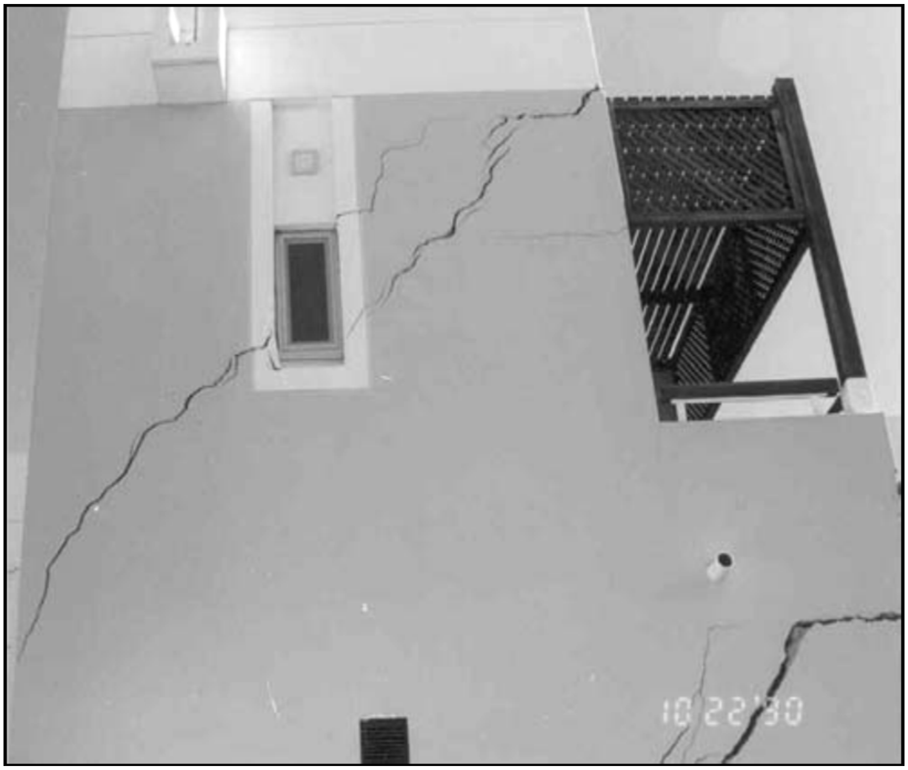


Figure 2-2 Structural Damage due to Foundation Failure (Al Rawas et al., 2005)

2.1.1.2 Pavement Failures in Texas

The shrinkage of expansive soils is the reason for the failure of pavements. The drying of the soil causes the shrinkage of soil which propagates through the pavement system causing longitudinal, transverse and fatigue cracking and rutting in the pavement surface. In addition to this, bumps caused due to heave can increase the roughness of pavements and result in poor riding comfort. Differential soil movements may induce large changes in moments and shear forces in the pavement structures. All these factors if not accounted in the original design, both rigid concrete and asphaltic pavements may experience severe distress in the forms of high roughness and cracking in both the longitudinal and transverse directions as shown in the Figure 2-3.

Figure 2-3 Pavement Failure (Al Rawas et al., 2005)

Overall, the magnitudes and extent of damages to pavement structures can be extensive, and it is often necessary to repair these pavements, thus increasing the pavement repair costs. Puppala et al. (2012) conducted studies on pavements constructed on expansive soils. Lime stabilization techniques utilized to mitigate the expansive behavior of these soils were successful. Below shown Figure 2-4 is a pavement distress caused by swell shrink characteristic of expansive soils.

Figure 2-4 Pavement Distress Caused by Swell Shrink Phenomenon

(Puppala et al, 2012)

2.1.1.3 Slope Failure

Slope failures occurred at Grapevine Dam in the state of Texas, USA was reported by McCleskey et al. (2008). This dam is built on expansive soil and is subjected to desiccation cracks during seasonal changes. During a rainfall event, water infiltrates into the soil through the desiccation cracks which increases pore water pressure which leading to reduction of shear strength triggering failure (Rahardjo et al., 2001; Cho and Lee, 2002). During dry season, the shrinkage cracks are formed and the water exerts hydrostatic pressure resulting in sliding of slope from the crack which is responsible for an increase of width of shrinkage crack. Figure 2-5 shows the slope failures associated with expansive soils. These failures are quiet detrimental and clearly shows the extent of damage.

Figure 2-5 Slope Failure Triggered by Desiccation Cracks of Expansive Soils

(Mccleskey et al., 2008)

Different Remedies have been used to meet the expansive soil challenges. Petry and Armstrong (1989) noted that it was more economical to perform initial stabilizations than performing remedial treatments later on with existing structures.

Stabilization of soils is the process of improving engineering behavior by changing one or more properties of the soil. It has been a topic of interest and discussion for all these years due to potential reduction in the construction and maintenance costs if infrastructure is built on problem grounds. There are two most commonly used soil stabilization methods they are by compaction or stabilization by chemical additives.

Chemical stabilization methods are widely used in the field to control soil heaving (Nelson and Miller, 1992; Puppala et al., 2003).

2.2 Chemical Stabilization

The Chemical stabilization is the process of mixing soils with stabilizing agents like lime and cement; usually in low amounts that changes both the physical and the chemical properties of the stabilized soil. The main properties that are altered by stabilization are Shear strength, Modulus, Resistance, Stability and Durability. In chemical stabilization additive refers to a manufactured commercial product that is added to the soil in proper quantities to improve the quality of the soil layer. The common stabilizers used are lime and cement, and sometimes-used in combination.

The current stabilization method used by the Texas Department of Transportation (TxDOT) is the Plasticity Index (PI) and gradation of soil to determine the kind of stabilizer (refer Figure 2-6). In some cases this method may not work due to the factors like different shrink/swell potential, plastic or liquid limit, the amount or kind of stabilizer used and the different mineral composition.

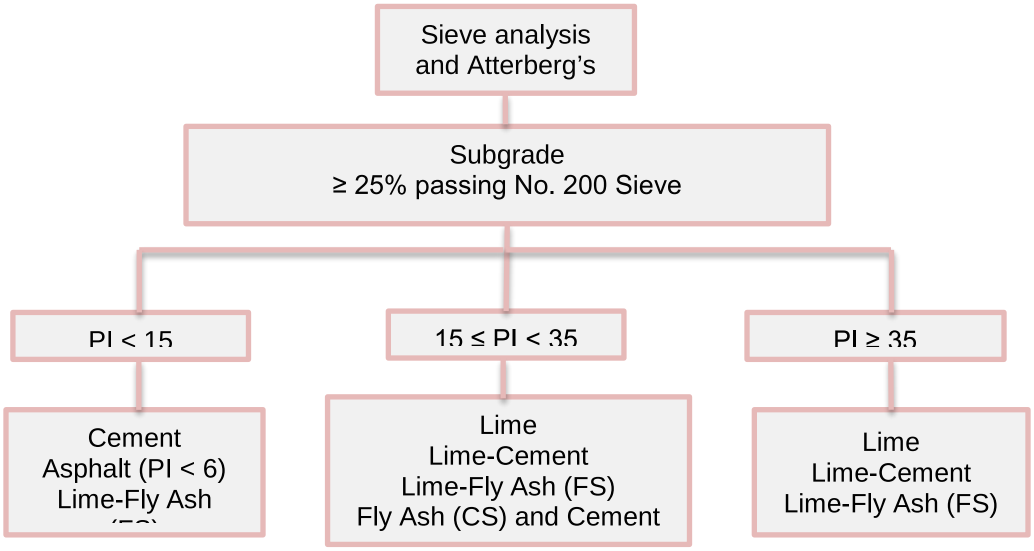


Figure 2-6 TXDOT Method of Additive Selection Criteria Using Soil Classification (TXDOT)

Studies conducted by Pedarla (2011) and Chittoori (2013) show that current practices based on PI and gradation of soil is insufficient for selecting an effective stabilizer as soils having same PI act differently to stabilizers and due consideration should be given to clay mineralogy. Hence in this regard, the next section focuses on the mineralogy of soils.

2.3 Soil Mineralogy

Soils in general contain various amounts of crystalline clay and non-clay minerals, non-crystalline matter and precipitated salts (Mitchell and Soga, 2005). The soils particles are mainly classified into 4 types based on the particle size. They are gravel (> 5mm), sand (between 0.74mm and 5mm), Silt (between 0.002mm and 0.074mm) and clay (< 0.002 mm). Similarly, the particles smaller than 2μm are not clay and greater than 2μm are non-clay particles. Majority of the soil is comprised of crystalline minerals which are primarily non-clay (Mitchell and Soga, 2005). The common non-clay and clay minerals present in the soils are feldspar, quartz, montmorillonite, kaolinite and illite. A brief review of these minerals has been given in the following sections.

2.3.1 Non-Clay minerals

The non-clays are mostly the rock fragments or mineral grains of common rock forming minerals. The non-clay minerals in soils are feldspar, quartz, pyroxenes and amphiboles, mica and other minerals. But of all these, the most common non-clay minerals are quartz and feldspar, which is explained in following section.

2.3.1.1 Quartz

Quartz is the most common mineral found on the surface of the Earth. It is estimated that about 12% of the mass of the Earth's crust is made of quartz and is considered as the most stable mineral under conditions at or near the surface. Quartz can occur in so many different geological settings and can also be associated with many different minerals. In igneous rocks and pegmatites, quartz usually occurs with potassium feldspars, muscovite, biotite and amphiboles. In sedimentary rocks or metamorphosed sedimentary rocks, quartz may be associated with potassium feldspar, calcite, dolomite or a variety of clay minerals

Quartz is composed of Silica tetrahedral grouped in such a way to form spirals, with all the tetrahedral oxygen bonded to silicon (refer Figure 2-7). There are many varieties of quartz that differ in color and form. But these different forms of quartz are classified into two groups (Schulze, 2002). First being the microcrystalline group which forms crystals or have microscopically crystalline structures. Second is cryptocrystalline group, which, does not show any visible crystals and have a dense structure like agate. The largest amount of quartz is usually found in igneous rocks when compared to metamorphic and sedimentary rocks (Ronov and Yaroshevsky, 1969).

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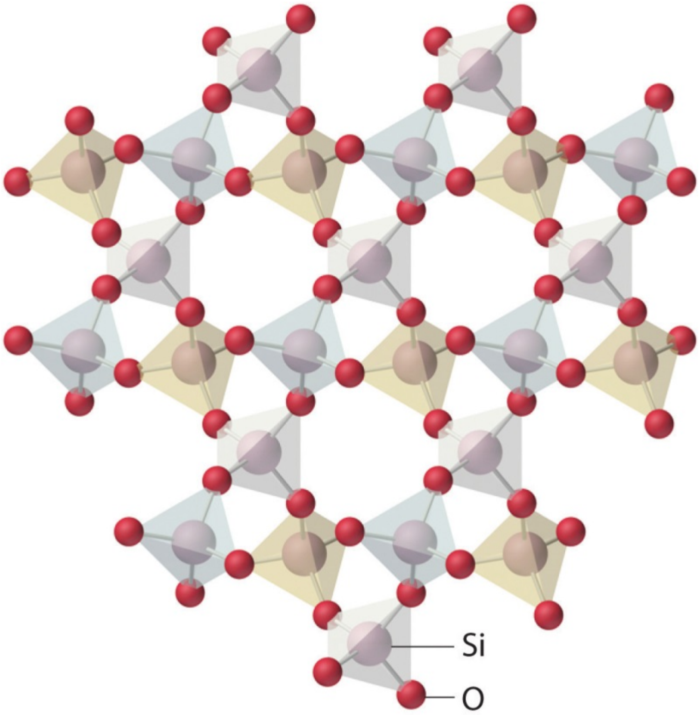


Figure 2-7 Crystal Structure of Quartz (from Principles of general Chemistry, 2012)

2.3.1.2 Feldspar

Feldspars are the most common rock-forming mineral (about 60% of the earth's crust) (Kauffman and Van Dyk, 1994). They crystallize from magma as veins in both intrusive and extrusive igneous rocks and are also present in types of sedimentary and metamorphic rocks. They are an important component of many building stones. Feldspar minerals are composed of aluminum-containing silicates and are composed of three-dimensional framework of strongly bonded TO_4 tetrahedra formed by the sharing of oxygen atoms between tetrahedra with T being dominated by Al^{3+} and Si^{4+} in natural feldspars (Mitchell and Soga, 2005) (Refer Figure 2-8). These minerals are usually white or very light in color with a hardness of 6 on the Mohr's Scale of Hardness. The chemical formula of Feldspar is $xAl(Al,Si)_3O_8$ (where x – Na, K, Ca). These minerals are further divided in to two groups. They are plagioclase feldspars, and Orthoclase (Sparks, 2003). The plagioclase is different from orthoclase in terms of the metals, plagioclase having calcium or sodium and orthoclase having potassium.

Plagioclase minerals are hard, non-metallic minerals that exhibit two cleavage directions that meet at nearly right angles. The name ‘plagioclase’ refers to this cleavage pattern, coming from the Greek words for ‘oblique’ (*plagios*) and ‘to break’ (*klasis*). Apart from pegmatites, the plagioclase minerals seldom occur as well-defined isolated crystals, but instead occur as blocky to prismatic cleavable masses that may comprise most of an igneous rock’s volume. They are harder than glass; most often white to gray in color, and have well-developed cleavage planes that often exhibit fine parallel grooves or striations. Color is variable however, and in mafic igneous rocks, the more calcium-rich plagioclase may be dark gray to almost black (Kauffman and Van Dyk, 1994). In general, the presence of striations is a more useful way to distinguish plagioclase minerals from the potassium feldspars with which they are most easily confused, although crystal twinning and compositional zonation in some potassium feldspar varieties may mimic striation.

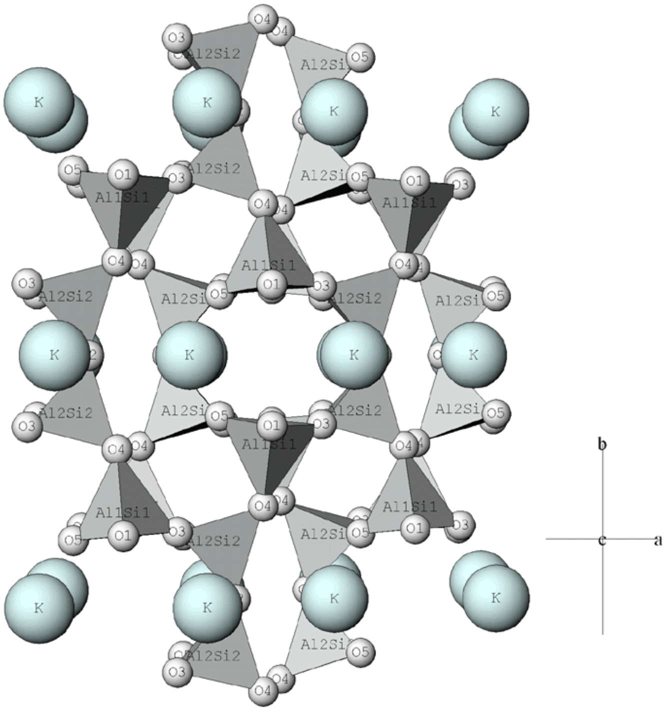


Figure 2-8 Crystal structure of Feldspar

(Source: https://staff.aist.go.jp/nomura-k/english/itscgallary-e.htm)

The potassium feldspars are most significantly used as aggregate in concrete and asphalt. They are differentiated from the plagioclase minerals by the lack of fine parallel striations, which are usually present on some cleavage surfaces in the plagioclase minerals. In general, potassium feldspars commonly have pink to reddish hues, while the plagioclase feldspars tend to be white or gray, but both mineral groups may exhibit similar colors, so the presence or absence of striations is more diagnostic (Huntley and Baril, 1997). These both feldspar groups commonly exhibit twinning and their crystal structures are so similar that they may actually form as thin alternating bands within a single specimen. This mixed variety of feldspar, consisting of alternating bands of plagioclase and potassium-feldspar, is called perthite.

2.3.2 Clay Minerals

The naturally occurring inorganic component is called as clay. Clayey soils exist with different clay mineral compositions in nature. These minerals contribute to the physical and chemical properties of the soil. Clay is used as both a particle size and also to represent a family of minerals (Velde, 1995). When representing particle size, it indicates that the soil particles have their size less than 0.002 mm. As a mineral type it represents the minerals, which have

a) Small particle size,

b) A net electrical negative charge and

c) Plasticity when mixed with water.

Clay minerals are primarily hydrous aluminum silicates. The shape of these minerals are usually platy or in few cases needle shaped or tubular (Mitchell and Soga, 2005). Clay minerals like kaolin, smectite and palygorskite-sepiolite are among the world's most important and useful industrial minerals (Murray, 1999). They have their importance in various geological applications such as stratigraphic correlations, indicators of environments of deposition and temperature for generation of hydrocarbons (Murray, 1999).

Mineralogy of a soil controls its size, shape, physical and chemical properties. Based on the mineralogy, the particle size of soil varies from very large cobbles and gravel to very fine silts and clays (Mitchell and Soga, 2005).

Minerals occupy more than one-half of the volume of soils. Minerals are the indicators of the amount of weathering that has taken place and the presence or absence of certain minerals explains the mechanical and chemical weathering processes that result in the formations of soils (Schulze, 2002). Crystal structure and chemical composition both are necessary to define a mineral (Schulze, 2002).

The percentage of crystalline clay minerals in a given soil is relatively low. However, the influence of these clay minerals on the properties of the soil is far more than their abundance. Clay minerals in soils belong to a family known as phyllosilicates or layered silicates. According to Brindley and Pedro (1972) "Clay minerals contain continuous two-dimensional tetrahedral sheets of composition Si_2O_5, Al_2O_5, Be_2O_5 etc. with tetrahedra linked by sharing three corners of each, and with the fourth corner pointing in any direction. The tetrahedral sheets are linked in the unit structure to octahedral sheets, or to groups of coordinated cations, or individual cations" (Bailey, 1980).

The clay minerals are usually divided into three main groups they are Kaolinite, Illite and Montmorillonite. Kaolinite is a common phyllosilicate mineral in subgrades and is most abundant in soils of warm moist climates. Illite exhibits moderate swelling and Montmorillonite are responsible for the swelling behavior of the soils. In all these minerals Kaolinite has weak bonding between the layers when compared to that of illite and montmorillonite. This due to the presence of positively charged metal ions like potassium in the case of illite, and calcium and sodium in the case of montmorillonite (Refer Figure 2-9). A brief overview of these common clay minerals is given in detail.

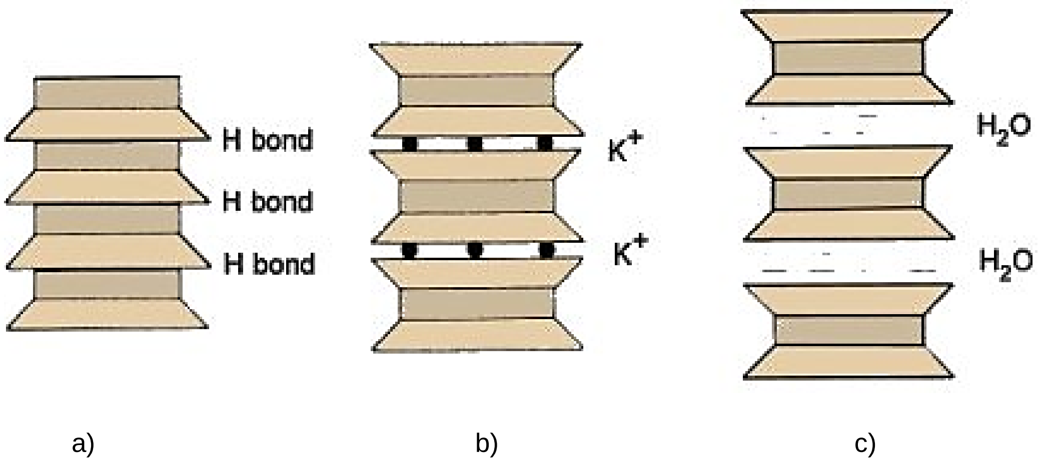


Figure 2-9 Structure of the main clay minerals: (a) Kaolinite, (b) Illite and (c) Montmorillonite

2.3.2.1 Montmorillonite

Montmorillonite is a member of the smectite family which includes other dioctahedral minerals beidellite, and nontronite, and the trioctahedral minerals hectorite (Li-rich), saponite (Mg-rich), and sauconite (Zn-rich). Montmorillonite is formed by the crystallization of the solution high in soluble silica and magnesium. It has a 2:1 layer structure in the form of an octahedral sheet along with two tetrahedral sheets, in which oxygen atoms are shared (Borchardt, 1989). The basic structural unit is a layer consisting of two inward-pointing tetrahedral sheets with a central alumina octahedral sheet (refer Figure 2-10). The bonds between layers are strong compared to kaolinite and have excellent cleavage, allowing water and other molecules to enter between the layers causing expansion (Grim, 1953).

Smectite minerals have a very small size and are concentrated in the fine clay fraction of soils. Because of this small particle size and interlayer expansion montmorillonites have very high specific surface area values ranging from 600 m2 /g to 800 m2 /g and also the range of cation exchange capacity values for these are as high as

47 to 162 meq/g (Borchardt, 1989). Soils containing montmorillonites retain more water and drain more slowly than soils with Kaolinite or soils with little clay. This nature of the soils with montmorillonite leads to destructive landslides as well as creep.

Figure 2-11 shows a scanning electron microscope (SEM) photograph of the mineral Montmorillonite.

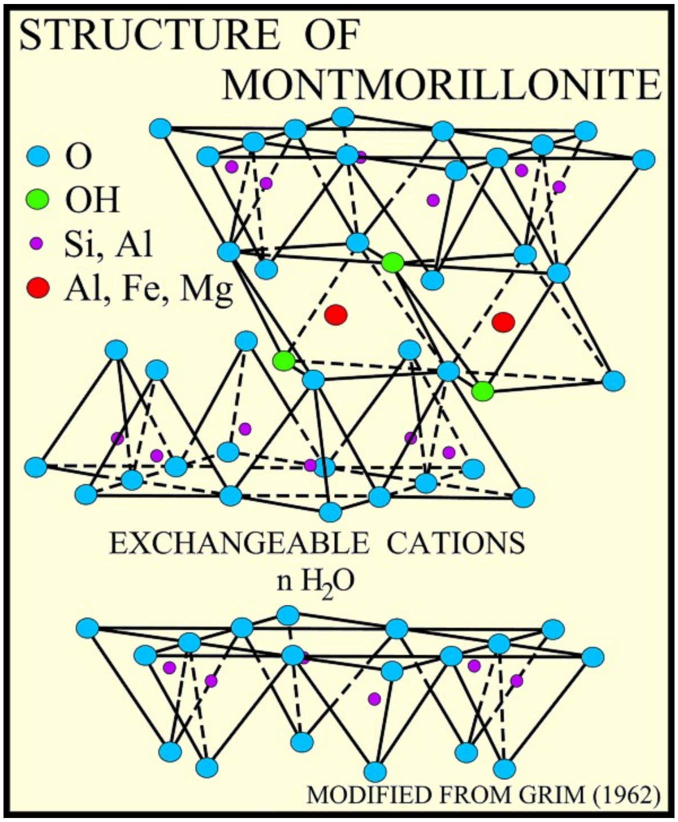


Figure 2-10 Mineral structure of Montmorillonite

(Source: http://pubs.usgs.gov/of/2001/of01-041/htmldocs/images/monstru.jpg)

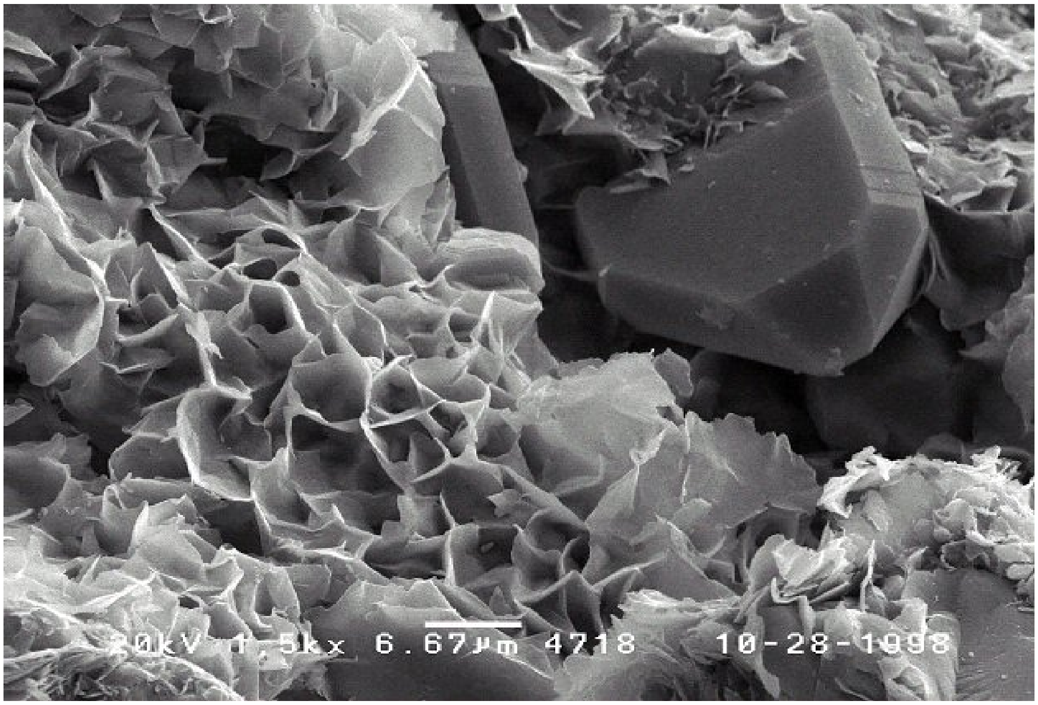


Figure 2-11 Photograph of Montmorillonite

(Source: http://www.webmineral.com/specimens/Smectite.jpg)

2.3.2.2 Kaolinite

Kaolinite is a common phyllosilicate mineral in subgrades; it is most abundant in soils of warm moist climates. The kaolinite group of minerals includes a lot of different kind of minerals including kaolinite, dickite, nacrite, and halloysite, and the trioctahedral minerals antigorite, chrysotile, and cronstedite. Out of all these minerals, kaolinite, dickite, nacrite, and halloysite have the same chemistry, but different structures. All four minerals are formed from the alteration (mostly weathering) of aluminum rich silicate minerals such as feldspars. The structure of kaolinite is composed of alternate silicate sheets (Si_2O_5) and aluminum oxide/hydroxide sheets ($Al_2(OH)_4$) called gibbsite sheets (See Figure 2-12). The silicate and gibbsite layers are tightly bonded together with only weak bonding existing between these silicate/gibbsite paired layers (called s-g layers)(The weak bonds between these s-g layers cause the cleavage and softness of this mineral. These minerals are also called as 1:1 minerals. Kaolinite is by far the most common and most clay deposits contain at least some Kaolinite.

Kaolinite has low value for the cationic exchange capacity (CEC), its value ranges from 1 to 5 meq/gm (White and Dixon, 2002). It is desirable to have the kaolinite in soil as it reduces the influence of more reactive minerals like smectite by lowering the water holding capacity and plastic properties. According to Di Maio and Fenelli (1994) Kaolinite is unaffected by exchangeable cations and so the Atterberg limits are less sensitive to CEC (Anson and Hawkins, 1998). A soil dominated by Kaolinite has particle arrangement which is regulated by liquid limit values (Sridharan et al., 1988).

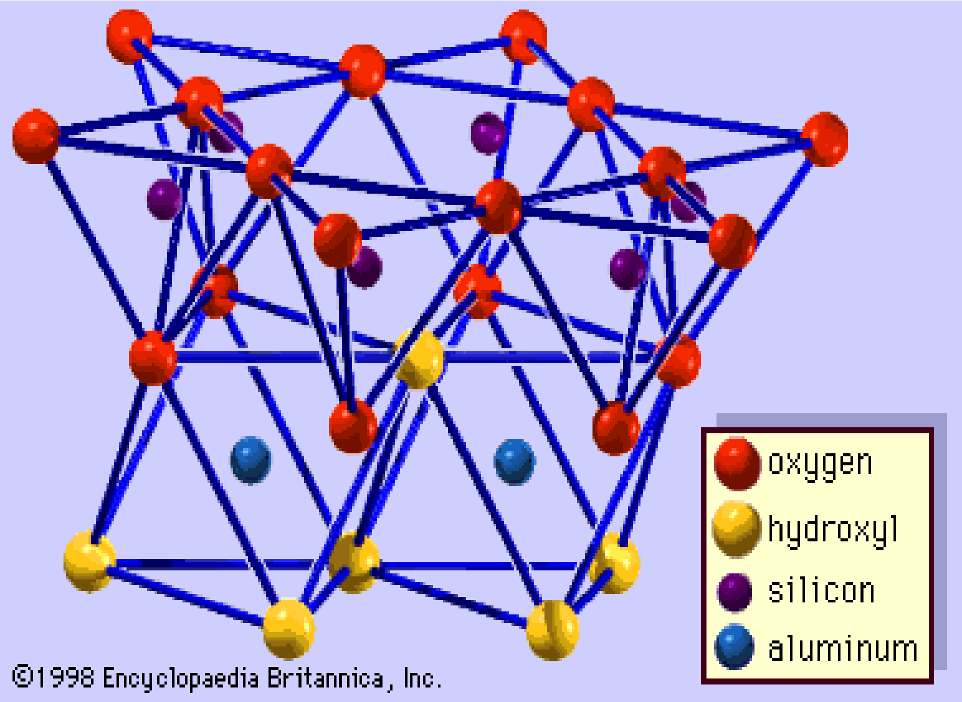


Figure 2-12 Mineral Structure of Kaolinite

(Source: http://media-2.web.britannica.com/eb-media/76/2676-004-3893834B.gif)

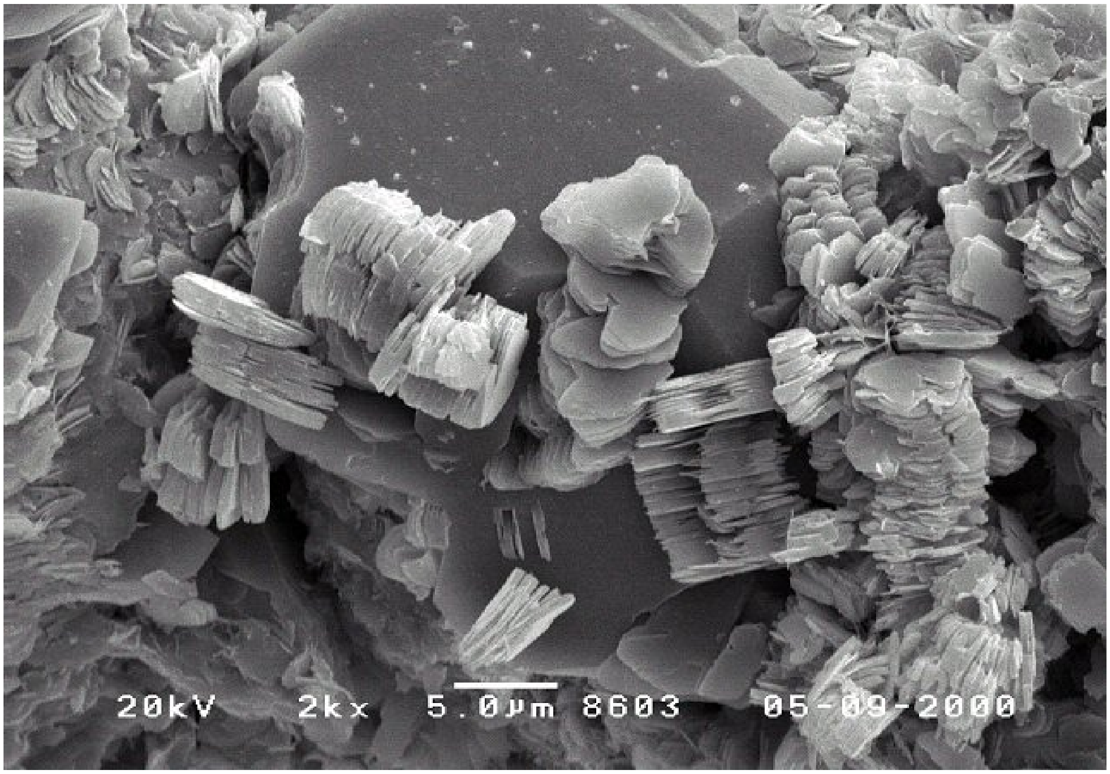


Figure 2-13 SEM photograph of the Mineral Kaolinite

(Source: http://www.uni-kiel.de/anorg/lagaly/group/jose/Kaolinite.gif)

2.3.2.3 Illite

Illites are part of mica family and they are non-expanding, clay-sized, dioctahedral, micaceous minerals. The structure of illite has a layer composed of two inward-pointing silica tetragonal sheets with a central octahedral sheet. They have cations between their interlayer, which balances a high layer charge. The most important cation is potassium. Figure 2-14 and Figure 2-15 show the mineral structure and scanning electron micrograph or SEM photograph of the mineral Illite. The weaker interlayer forces caused by fewer interlayer cations in Illite also allow for more variability in the manner of stacking (Grim, 1962).

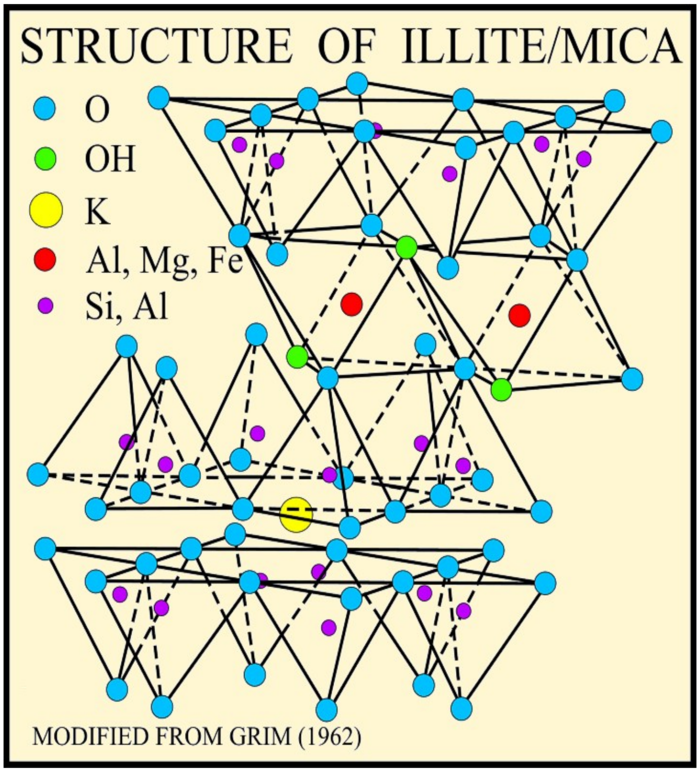


Figure 2-14 Mineral structure of Illite

(Source: http://joidesresolution.org/node/1395)

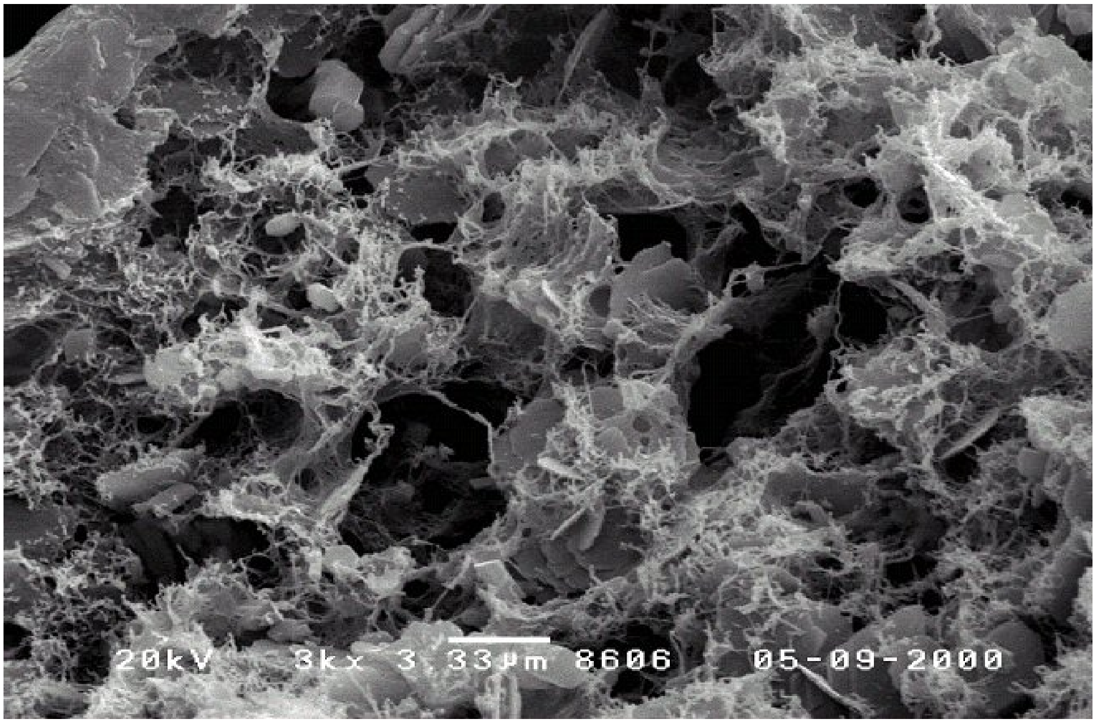


Figure 2-15 SEM photograph of the mineral Illite

(Source: http://www.uni-kiel.de/anorg/lagaly/group/jose/Illite.gif)

Illites are the dominant clay minerals in argillaceous rocks and are formed by the weathering of silicates (primarily feldspar), through the alteration of other clay minerals, and during the degradation of muscovite. Formation of Illite is generally favored by alkaline conditions and by high concentrations of Al and K. The number of inter particle contacts is less in micas and hence, the cohesive forces between the crystallites are weak (Thompson & Ukrainczyk, 2002). The degree to which Illite crystals contact adjacent grains is a function of soil water content as well as particle size, shape and flexibility.

2.4 Method for Identification of Minerals

Soil minerals are identified primarily by their elemental composition and structure. Other distinctive properties such as color, thermal behavior, and solubility are also used.

This section is aimed to provide an overview of various methods that are currently in practice for the identification of minerals, they are vibrational spectroscopy, thermal analysis, atomic absorption spectrometry and X-ray diffraction.

X-ray diffraction alone often provides enough information, but in many instances, two or more analytical techniques are needed to confirm mineral identity (Amonette, 2002).

2.4.1 X-Ray Diffraction (XRD)

Clay minerals have long range structure that repeats itself for hundreds of nanometers to millimeters. These minerals are characterized to be crystalline in nature. These kinds of minerals can be identified by X-ray diffraction studies because `each crystal contains planes of atoms separated by constant distance which is due to the periodic nature of the standard spacing of the atoms (Whittig & Allardice, 1986).

When an X-ray beam falls on equally spaced atoms of a crystalline mineral they are transmitted, absorbed or scattered (Cullity, 1978). When scattering, they can be scattered coherently (without loss of energy) or incoherently (with loss of energy). The coherently scattered light will form an interference pattern when the scattering centers are arranged in a regular array and the distance between scattering centers is comparable to the wavelength of the light and this phenomenon is called diffraction (Amonette, 2002). When the incident beams of X-rays are diffracted a detector captures the beam and converts the analog signal into digital data, which can be plotted. This data is used to measure the distances between the planes of the atoms using Bragg’s law.

The basis for the identification of crystals using X-ray diffractions is that, they have different intensities i.e. no two minerals will have similar inter atomic distances in three dimensions. This intensity pattern is compared with standard patterns for known materials. Extensive X-ray diffraction data for clay minerals and other soils minerals are given by Grim (1953), Whittig and Allardice (1986), and Moore and Reynolds (1989). A detailed analysis of X-ray patterns is given by Mitchell and Soga (2005).

Powder diffraction method is generally used for these soils because of the small size of the clay particles which makes it difficult to study single crystals. In this method small sample of particles, containing all possible orientations is kept under the X-ray beam. This method works because of the large number of particles which will insure that some will be oriented in the right way to produce the desired reflection (Whittig & Allardice, 1986).

2.4.2 Thermal Analysis (TA)

The mineral identification in thermal analysis is achieved by evaluating the weight loss and enthalpy changes in the soil sample with temperature. Many researchers have used this method for the identification of the clay minerals in soils (Benham, 1990; Karathanasis and Harris, 1994; Wunderlich, 1990). The most common methods used in TA are thermal gravimetric analysis (TGA) and differential scanning calorimetry (DSC) (Beck, 2004). A detailed explanation of these methods is given by Karathanasis and Harris (1994). In TGA, with the constant rate of changing of temperature the weight of the soil sample is monitored. Many researchers (Barshad, 1965; Dish and Duffy, 1990) had used the TGA method to identify and quantify soil minerals. In DSC method the energy required to maintain the soil sample and the reference material at same temperature during heating is measured and evaluated. A detailed explanation of this technique is given by Karathanasis and Harris (1994) and Beck (2004).

2.4.3 Atomic Absorption Spectrometry (AAS)

Atomic Absorption Spectrometry (AAS) has been used for the identification of elements from quite long time. As the name implies this method depends on the measurement of atomic species. This technique depends on the ability to consistently atomize the element of interest in a reproducible manner in both samples and standards (Baker and Suhr, 1982).

When an atom is excited by thermal energy or other energy sources they emit radiation by dropping down to less energetic states or to the ground states as atoms always try to be in ground state at all times. This can be given by the following equation (Baker and Suhr, 1982):

$$M + hv \rightarrow M' \rightarrow M + hv$$

Where M = Neutral atom, h = Planks constant, and v = Frequency of the irradiating light.

This method relies on the absorption of a photon having the exact energy needed to convert an atom or ion in the ground state to an excited state. For each element to be determined, a lamp producing light of appropriate energy irradiates the atomized sample, and the loss of intensity in this light is used to determine the amount of element present (Amonette, 2002).

There is also another method called Atomic Emission Spectrometry (AES) similar to AAS. The major difference between these two methods is that in former the loss in the intensity is measured and in latter the intensity of the light that is emitted is measured.

Majority of non-destructive soil/clay testing depend on the excitation of atoms in the specimen to produce X-ray characteristics of each element present based on the energies and the intensities of the X-ray produce elements are identified and quantified. X-rays are produced in two stages. In the first stage a vacancy is created in the inner shell of an electron and then in the second stage that vacancy is filled by a higher energy electron coincident with the release of an X-ray photon. The vacancy is created when the atom absorbs the energy from an energy source in the form of a photon or a charged particle. X-rays produced by absorbing the energy from photons are called X-ray fluorescence (XRF) and that by charged particles are called X-ray emission (XRE). The energy source for XRF can be X-rays from an X-ray tube or gamma rays from nuclear decay. For the XRE a variety of high energy charged particles can be used, although electrons and protons are the most common (Amonette, 2002).

2.4.4 Vibrational Spectroscopy (VS)

Vibrational spectroscopy gives the most definitive means of identifying the surface species generated by molecular absorption as well as surface reactions. In principle, including infrared spectroscopy and Raman spectroscopy methods any technique that can be used to obtain vibrational data from solid state or gas phase samples can be applied to study soil surfaces.

Only two techniques are routinely used for vibrational studies of molecules on surfaces. They are INFRARED Spectroscopy and RAMAN Spectroscopy. Infrared spectroscopy involves the direct measurement of infrared light absorbed or emitted by a specimen. Raman spectroscopy on the other hand, is an incoherent-scattering technique in which the loss or gain in energy by the interaction of light with the atoms in a bond is measured (Amonette, 2002). Vibrational spectroscopy is particularly well suited for characterization of minerals containing hydroxyl, carbonate or sulfate groups and for studies of organic molecules associated with minerals surfaces. The use of mid-infrared attenuated total reflectance (ATR) spectroscopy enables direct measurement of nitrate concentration in soil pastes (Linker et al., 2005). But their accuracy depends on the soil type with varying contents of carbonate, whose absorbance band overlaps the nitrate band.

The study done by Kariuki et al. (2006) indicated that spectroscopy can be used in classifying soils however; high organic matter and the presence of moisture were found to affect area and depth intensities and would require consideration in such applications.

2.3.5 X-ray Absorbance Spectroscopy (XAS)

X-ray absorption spectroscopy is an element-specific probe of the local structure (short range) of elements in a sample (XAS). Interpretation of XAS spectra commonly uses standards with known structures, but can also be accomplished using theory to derive the structure of a material. X-ray absorption spectroscopy is commonly divided into two spectral regions; the first is the X-ray absorption near edge structure (XANES) spectral region and the second is termed the extended X-ray absorption fine structure (EXAFS) region (Amonette, 2002).

XANES spectra are unique to the oxidation state and speciation of the element of interest, and consequently are often used as a method to determine the oxidation state and coordination environment of materials. EXAFS spectra are best described as a series of periodic sine waves that decay in intensity as the incident energy increases from the absorption edge (Amonette, 2002). These sine waves result from the interaction of the elected photoelectron with the surrounding atomic environment. As such, their amplitude and phase depend on the local structure of excited atom. Since this interaction is well understood, theory is sufficiently advanced that the local structure of the excited atom can be determined by matching a theoretical spectrum to the experimental spectrum. This fitting yields many types of information, including the identity of neighboring atoms, their distance from the excited atom, the number of atoms in the shell, and the degree of disorder in the particular atomic shell (as expressed by the Debye-Waller factor) (XAS). These distances and coordination numbers are diagnostic of a specific mineral or adsorbate-mineral interaction; consequently, the data are useful to identify and quantify major mineral phases, adsorption complexes, and crystallinity.

2.5 Methods for Quantification of Minerals

The soil minerals can be quantified in many ways using the XRD method which have been developed by many researchers. There is also other method called chemical balance equation which is used to quantify the minerals present in the soil (Hughes et al., 1994; Salyn & Drits, 1972; Smith, 1989; Jones, 1989). Both these method together can also be used in determining the mineral quantification. The brief review of these two methods is explained in the following sections.

2.5.1 XRD Based Methods

There are several different types of XRD tests. In XRD using the relative intensities of the peaks the concentration of the mineral species present in the test specimen can be estimated (Whittig & Allardice, 1986).However, there are other factors such as crystal perfection, chemical composition, variation in sample packing, crystal orientation and presence of amorphous substance that influence the diffraction peaks (Jackson, 1969).

A different scheme using XRD data was developed by Smith et al (1987) which is based on the analysis of the whole diffraction pattern from 2°- 50° 2Ɵ through comparisons with a reference database of diffraction traces obtained from a set of carefully purified reference clay samples run under the same conditions. Quantitative determinations of the amount of clay minerals based on simple comparison of the diffraction peak heights or areas are uncertain due to many factors like differences in absorption coefficients, particle orientations, crystallinity and others (Mitchell & Soga, 2005). Considering all these differences many other methods are developed and are currently in use (Alexaides & Jackson, 1966; Hodgson & Dudeney, 1984; Johnson et al., 1985; Randall et al., 1994). There is another full pattern modeling method known as the

Arquant model developed by Blanc et al. (2006) which has been successfully applied for clay mineral quantification in soils and rocks.

Chrysochoou et al. (2010) also showed that XRD coupled with Rietveld quantitative analysis allows for direct quantitative mineralogical comparison. RQA first involves the qualitative identification of crystalline constituents in an XRD pattern. The production of X-ray reflections of each constituent can then be mathematically predicted. Rietveld (1969) used this principle to build a mathematical model that constructs a synthesized diffraction pattern from the identified compounds and seeks to minimize the difference between the synthesized and experimental patterns through an optimization scheme. The relative weights of the modeled phases are then calculated on a percent basis based on the observed intensities and the mineral properties. RQA therefore provides a relative quantification of the identified crystalline compounds of an XRD but cannot detect the amount of amorphous compounds. Hence to estimate the amorphicity of a specimen and obtain true phase quantification, the XRD powder sample is spiked with a known mass of a substance of very high crystallinity, an internal standard whose dilution is linked to the amorphous content (Jones et al., 2000; De La Torre et al., 2001; Whitfield and Mitchell, 2003). In this way, RQA enables the direct comparison of mineral quantities in different samples and materials, as well as a function of time in reactive systems.

The other general approaches of XRD that are commonly used for the quantification of minerals in soils are Method of Known Additions, Absorption Diffraction, Full-pattern fitting, Mineral Intensity factors (Khale et al., 2002).

2.5.2 Chemical Mass Balance method

Chemical mass balance alone can be used to quantify clay minerals in soils. Many researchers (Alexaides and Jackson, 1966; Hodgson and Dudeney, 1984; Johnson et al., 1985) have already used elemental mass balance techniques to asses each mineral percentage in the soil samples. In these methods amount of each element is measured with the help of laboratory chemical analysis of the soil sample and based on this information simultaneous equations are formulated which can be solved to obtain the percentage of the minerals in soils.

Randall et al. (1994) compared four such methods of clay mineral quantification using elemental mass balance methods and highlighted their corresponding strengths and weaknesses. A brief description of those methods is given here. These methods primarily are solving a system of simultaneous linear equations which are formulated using the elemental information of the soil specimen and the minerals for which they are being analyzed.

2.5.3 Previous Quantification Methods Using Chemical Tests at University of Texas at Arlington

Due to the difficulty of solving the simultaneous equations with absolute error, models were developed by Chittoori (2008) for predicting the different clay mineral percentages. They are Regression analysis and artificial neural network or ANN based models. Details of these techniques are given below.

2.5.3.1 Regression Analysis

Regression analysis is a statistical tool to establish a relationship between two or more variables. Regression equations were formulated to predict the three percent clay minerals using each of the soil or chemical properties (Chittoori, 2008). The equations used to predict the mineral percentages are

$$\%I = \left[\frac{TP}{6}\right] \times 100 \qquad (2.1)$$

$$\%M = -2.87 + 0.08 \times SSA + 0.26 \times CCE \qquad (2.2)$$

$$\%K = 100 - \%I - \%M \qquad (2.3)$$

Where,

%I = Percentage of the mineral Illite

%M = Percentage of the mineral Montmorillonite

%K = Percentage of the mineral Kaolinite

TP = Total potassium

CEC = Cation exchange capacity

SSA = Specific surface area

Equation (2.1) gives the percentage Illite present in the soil based on the TP of the soil. Equation (2.2) is the regression model developed to obtain the percentage Montmorillonite in the soil and finally equation (2.3) gives the percentage Kaolinite present in the soil sample. The predictive performance of the multiple regression models is measured through the coefficient of determination (R^2). The coefficient of determination (R^2) for equation (2.2) is found to be 0.95, which indicates that the model can predict very well.

2.5.3.2 Neural network model

Artificial Neural Networks (ANNs), in general, simulates the biological structure of human brain by means of their architecture. ANN technique is an emerging technique that has been applied to many geotechnical engineering applications successfully by many researchers (Shahin et al., 2001). The Transportation Research Board (TRB) has summarized the state-of-the-art ANN applications in geotechnical engineering (E-C012, 1999). The model is used to predict the relationship between the model input(s) and corresponding output(s).

The model development database is divided into three subsets, a training set and a testing set, to develop the neural network model and then a validation set to check the accuracy of the predictions (Chittoori, 2008). Both the training set and the validation set contain all the patterns to represent the entire data set.

The data is preprocessed and in the next step the number of hidden layers and their corresponding nodes in each hidden layer were determined. According to Hornik et al. (1989), a network with a single hidden layer provided with enough connection weights can be used to approximate any continuous function. Accordingly, the network with a single hidden layer having eight nodes was developed. The input layer has 3 nodes, the hidden layer has 8 nodes and the output layer has 3 nodes in the particular network as shown in Figure 2-16. After this step the model undergoes the training stage and then was ascertained with the validation data.

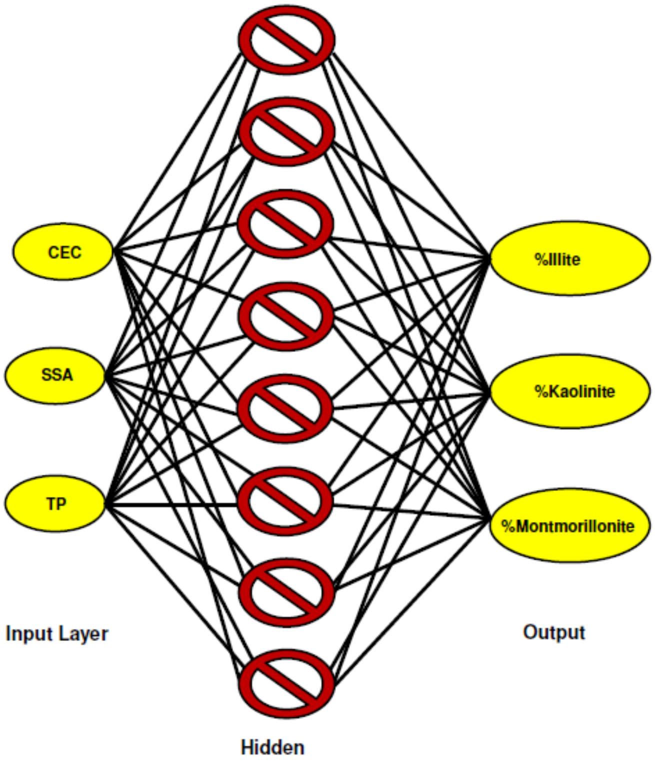


Figure 2-16 Optimized Network Architecture (Chittoori, 2008)

More detailed explanation of the regression model and neural networking model were given by Chittoori (2008). As mentioned, these models were developed using chemical mass balance equation method which was based on properties like Cation exchange capacity, Specific surface area and Total potassium. Each of these properties is explained in the following sections.

2.5.3.3 Cation Exchange Capacity (CEC)

The cation exchange capacity (CEC) of a soil is simply a measure of the amount of readily exchangeable cations neutralizing negative charge in the soil. CEC refers to the quantity of negative charges in soil existing on the surfaces of clay and organic matter (Camberato, 2002). In cation exchange capacity the positively charged cations attracts to negative ions. Soil CEC is normally expressed in units of charge per weight of soil. CEC is a good indicator of soil reactivity with the chemical species.

The negative charges in the soil are obtained from the following sources and reactions (Rhoades, 1982):

(a) Isomorphous substitution within the structures of layer silicate minerals

(b) Broken bonds at mineral edges and external surfaces

(c) Dissociation of acidic functional groups in organic compounds

(d) The preferential adsorption of certain ions on the particle surfaces.

The first of these charges is permanent and is independent of pH and the rest are dependent on ph. CEC is not independent of the conditions under which it is measured hence it is necessary to measure the soils capacity to adsorb cations from an aqueous solution of the same pH, ionic strength, dielectric constant and composition as that encountered in field.

There are numerous methods for determining CEC and many will give quite different results. Rhoades (1982) has given the following four methods that can be used for CEC determination. They are the summation method, direct displacement method, radioactive method and the displacement after washing method.

There is a significant variation in the results obtained by the above four methods. This is due to the complicating interactions between saturating, washing, and extracting solutions. Also, CEC is not an independent and a single valued soil property (Rhoades, 1982).

Camberato (2001) says the primary factor determining CEC is the clay and organic matter content of the soil. Higher quantities of clay and organic matter beget higher CEC. Different types of clays have different CECs. Stewart and Hossner (2001) reported unusually high cation exchange capacity (CEC) values relative to clay content for lignite overburden and mine soils. On an average, the CEC values are found to be greater than 100meq/gm.

2.5.3.4 Specific Surface Area (SSA)

Total surface area contained in a unit mass of soil is considered as specific surface area of that soil. Soils with high specific surface areas have high water holding capacities, more adsorption of contaminants, and greater swell potentials. Specific surface is closely tied to particle size distribution. This phenomenon is explained by Campbell (2005) with a simple thought experiment in which a $1cm^3$ Cube with a density 1 gm/cm^3 is considered. This cube has a specific surface area of $6cm^2/g$. Now, if this cube is divided into smaller cubes of 1 mm on the side, the resulting 1000 cubes would have the same mass of material, but its specific surface area will be 60 cm^2/g, similarly if the cube were to be divided into 10^{12} cubes of 1 um on a side, the surface area would be 6 x 10000 cm^2/g hence it could be understood that within the same mass, presence of smaller particles will result in higher specific surface area.

Various approaches have been used to measure specific surface area, including adsorption of nitrogen and other gases on the soil (Yukselen and Kaya, 2006). The most commonly used method is the adsorption of ethylene glycol monoethyl ether (EGME) (Carter et al. 1986). This involves saturating prepared soil samples, equilibrating them in a vacuum over a $CaCl_3$-EGME solvate, and weighing to find the point when equilibrium is reached. The specific surface is then determined from the mass of retained EGME in comparison to the amount retained by pure Montmorillonite clay, which is assumed to have a surface area of 810 m2/gm (Carter et al. 1986). The measurement typically takes around two days to complete. Soil is typically in a hydrated state, and surface area measurements should apply to that state. It would therefore be ideal if water could be used as the probe to determine the specific surface area.

Quirk (1955) reviewed such measurements and concluded that water clusters around cation sites, and can therefore lead to errors in the measurements. Recent work which uses more modern methods for measuring the energy state of the water in the soil, have shown promise as simple methods for determining specific surface of soil samples. A comprehensive evaluation of the EGME method for geotechnical usage was done by Cerato and Lutenegger (2002). They concluded that the method is applicable to a wide range of mineralogy and is capable of determining specific surface area ranging from 15 to 800m^2/g.

2.5.3.5 Total Potassium

Potassium is an element which can be used to detect the presence of the mineral illite and feldspar in the soil sample. Potassium belongs to the alkali metals in the periodic table that are characterized by a single electron in the outer most shell. This electron is easily lost and they readily form stable monovalent ions (Knudsen et al., 1982). The method proposed by Knudsen is widely used compared to all the other methods.

Potassium is the inter layer cation in the clay mineral illite and illite is the only clay mineral to have potassium in its structure (Mitchell and Soga, 2003). The test procedure formulated by Knudsen et al. (1984) was followed to obtain the amount of total potassium present in the soil. The method involves a double acid digestion technique developed by Jackson (1958) which uses two acids (Hydrofluoric acid and Perchloric acid) to break the mineral structure of the soil and extract the potassium ions from the structure. Once the potassium is extracted, its concentration in the solution can be obtained with the help of a spectrophotometer or any other suitable device.

These methods developed by Chittoori (2008) are based on the assumption that the soil has only the clay minerals montmorillonite, kaolinite and illite. But studies by Chryoschoou (2013) showed that in the evaluation of the treatment approaches for soil, the contaminant speciation is important. For example, the type and amount of clay in a soil is important as an immobilization mechanism for heavy metals as the Al-source in treatment design to ensure sufficient pozzolanic reaction. The amount of fines in a soil does not necessarily correspond to the amount of clay present, as other minerals (micas and feldspars) may contribute to the fine fraction. This suggests that more work has to be done in the models developed for giving more accurate values of the clay minerals.

2.6 Summary

This chapter presented the available literature on expansive soils and mineralogy. From the literature review it is clear that mineralogy plays a vital role in the behavior of expansive soils. A brief review of different types of minerals (both clay and non-clay minerals) and their Identification methods like X-ray Diffraction Testing (XRD), Thermal Analysis (TA), Atomic Absorption spectrometry (AAS), X-ray Absorbance spectroscopy(XAS), Vibrational spectroscopy (VS) are reviewed.

The different quantification methods for determining the mineral contents in soils are discussed. The two models developed by Dr. Chittoori using the chemical tests like Cation Exchange Capacity (CEC), Specific Surface Area (SSA) and Total Potassium (TP) are also presented in this chapter.

In the next chapter, soil selection and basic soil classification results are provided. Methodology of all the chemical tests opted for soil samples to determine the mineral percentage is also studied.

Chapter 3

Experimental Program

3.1 Introduction

The present research focuses mainly on the identification and quantification of minerals present in soil. In nature soils exists with different mineral compositions which in turn show variation in soil behavior. As stated before, mineralogy of a soil controls its size, shape, physical and chemical properties. Based on the mineralogy, the particle size of soil varies from very large cobbles and gravel to very fine silts and clays (Mitchell and Soga, 2005). There are methods like vibrational spectroscopy, thermal analysis, atomic absorption spectrometry and X-ray diffraction for identification and quantification of soil minerals. But these methods require expensive equipment's and trained analysts (Whittig and Allardice, 1986).

In this research, a new procedure developed by Chittoori and Puppala (2011) to identify clay minerals using chemical properties like cation exchange capacity (CEC), specific surface area (SSA) and total potassium (TP) was improvised. The experimental program followed including the various laboratory tests and the testing procedures to meet the desired research objectives is presented in this chapter.

3.2 Soil Selection

Expansive unsaturated soils cover one-fourth of the United States (Puppala and Cerato, 2009). In the present research 17 soil samples with different mineral contents and topographical conditions were selected. The samples were collected from Oklahoma, Colorado, San Diego and Texas.

The basic soil classification details of the soils being tested in this study are presented in Table 3-1. Sieve and hydrometer analysis were conducted per ASTM D 422 and Atterberg's Limit Tests (Liquid Limit and Plastic Limit) were conducted per ASTM D

4318 to find the_plasticity index of the soil. Soil classification was based on Unified Soil Classification System (USCS)

Table 3-1 Soil Classification test Results of the 17 Soils

Soil No.	Samples	Clay %	Silt %	Sand and Gravel %	PI	Soil Type based on USCS
1	Dallas	68	32	0	45	CH
2	Sherman	50	38	12	42	CH
3	Burleson	41	49	10	37	CH
4	Wilcox	34	26	40	30	CL
5	Riverside	31	37	32	24	CL
6	Eagle ford	51	39	10	32	CH
7	Joe pool	41	30	29	34	CH
8	Woodbine	39	13	48	0	SM
9	Stephenville	48	33	19	22	CL
10	Colorado	32	34	34	42	CH
11	San Diego	38	38	24	28	CL
12	Austin	35	50	15	13	CL
13	Grayson	48	34	18	49	CH
14	Rolling Hill	39	51	10	24	CL
15	Keller	35	45	20	11	CL
16	Anthem	32	29	39	27	CL
17	Oklahoma	41	49	10	21	CL

3.3 Methodology

For attaining the research objective the tasks were executed in three phases. Initially, dry sieve analysis was performed on the test soils for the separation of particles retained and passed through 75µm sieve. Later in first phase, X–Ray diffraction testing was performed on powdered test soils for the identification of minerals present in it. Using this data the concentration of each mineral was also be found.

In second phase, all the chemical tests CEC, SSA and TP were performed on particles passed through 75µm sieve. These Particles have both silt and clay. The clay minerals Montmorillonite, Kaolinite and illite were quantified using these results using the Chittoori and Puppala (2011) model. The flow chart below shows step wise procedure for clay mineral for Chittoori model.

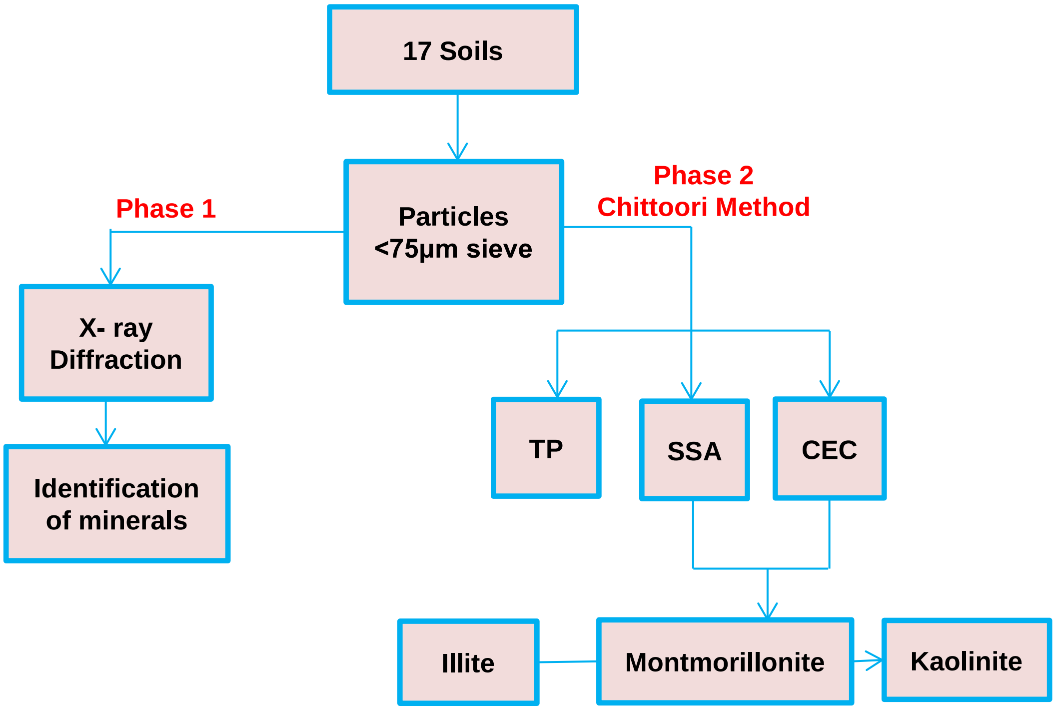


Figure 3- 1 Flowchart showing the step wise procedure for Chittoori

In third phase, silt and clay fractions were separated using the hydrometer setup explained in the following section. Wet silt collected from hydrometer cylinder was washed and dried. Later chemical test was performed on these samples for total potassium. The total potassium for clay fractions was found using the total potassium results for 75μm particles and for silt fractions. Figure 3-2 shows the separate silt and clay fractions obtained using the hydrometer setup.

Figure 3- 2 Picture showing the silt and clay fractions of the Keller soil

In this phase, clay and non-clay minerals montmorillonite, kaolinite, illite, quartz and feldspar present in the soil were quantified using the part of the data from Chittoori method and from the Total potassium results of separate silt and clay fractions. This step wise procedure in phase 3 is represented in the form of a flow chart (refer figure 3-3).

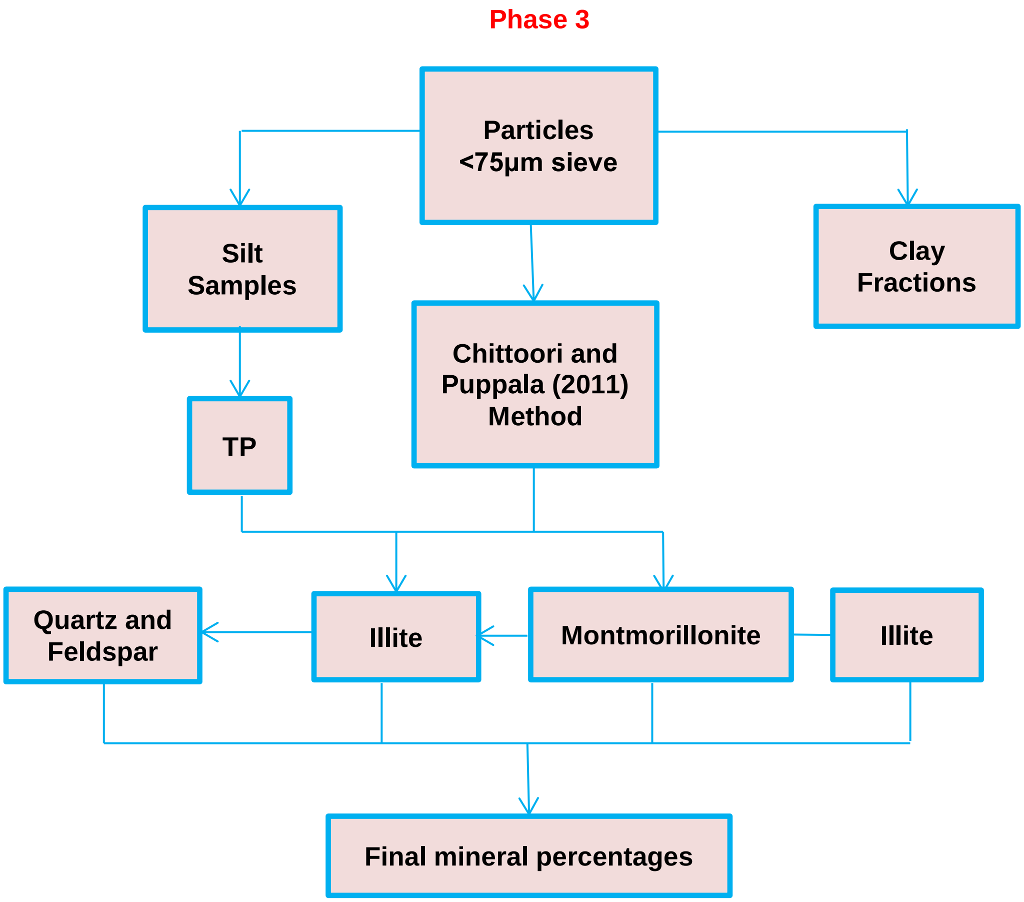


Figure 3- 3 Flowchart showing the Experimental Program for Phase 3

3.4 Separation of Silt and Clay Fractions Using Hydrometer Setup

For this test, 50 grams of 75 microns sample was mixed with 125 ml of sodium hexameta phosphate and allowed to stand about 12 hrs. After the end of soaking period the soil mixture was transferred in to the sedimentation cylinder and the distilled water was added up to 1000ml mark on the cylinder. The solution was allowed to stand until the diameter of the particles reached 0.002mm.

The time taken for each test soil to reach the 0.002mm diameter was estimated using the hydrometer data and is given in table 3-2. From table, it can be observed that the average time taken for all the test soils for the collection of clay particles was 695 min. In this research a wait time of 720 min has been taken for the collection of silt and clay fractions. Figure 3-4 and 3-5 shows the time estimation plot from hydrometer setup for soils Dallas and Riverside.

Table 3-2 Estimated Times for Collection of Clay for 17 Soils

Soil No.	**Samples**	**Estimated time (Min)**
1	Dallas	600
2	Sherman	630
3	Burleson	680
4	Wilcox	720
5	Riverside	740
6	Eagle ford	720
7	Joe pool	700
8	Woodbine	680
9	Stephenville	640
10	Colorado	720
11	San Diego	720
12	Austin	720
13	Grayson	640
14	Rolling Hill	720
15	Keller	720
16	Anthem	720
17	Oklahoma	680

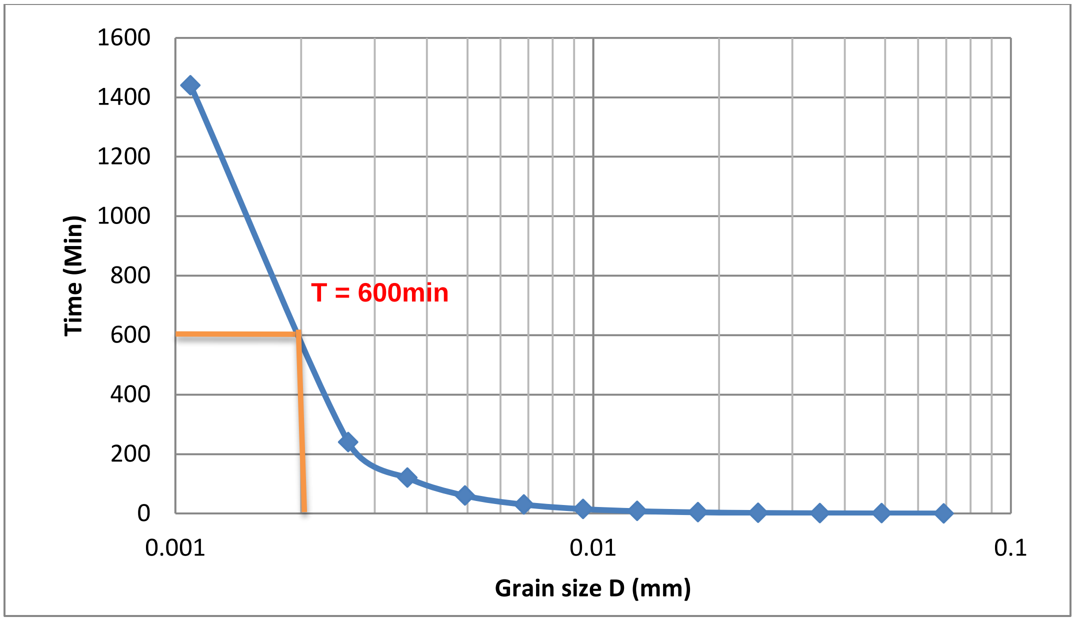


Figure 3-4 Plot for Time estimation in Dallas soil

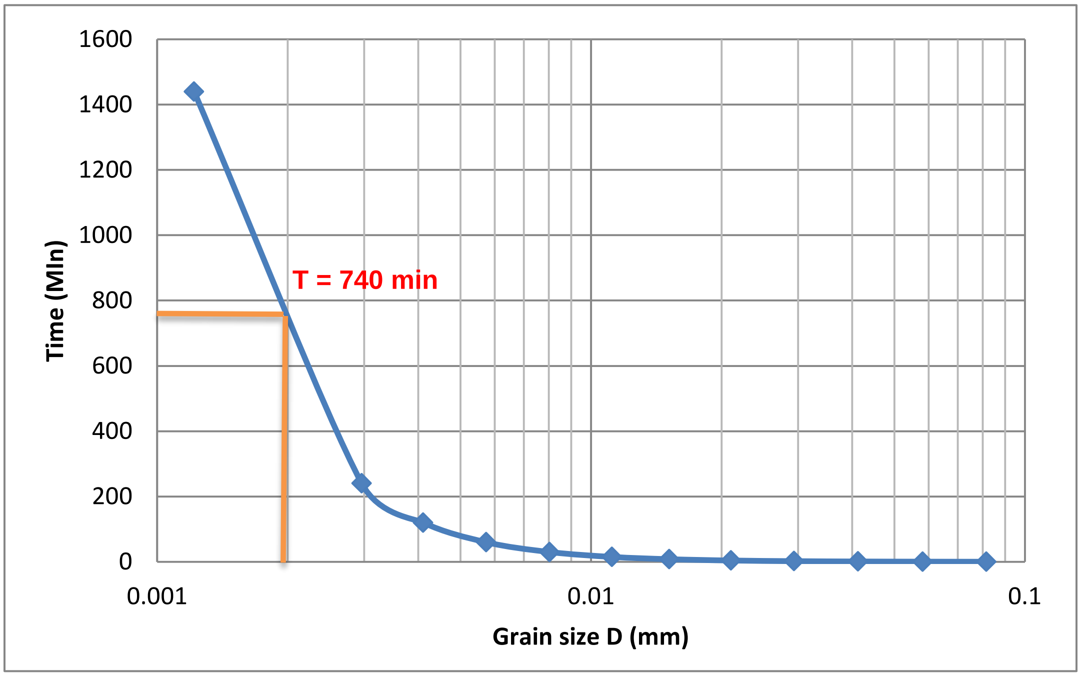


Figure 3-5 Plot for Time Estimation in Riverside Soil

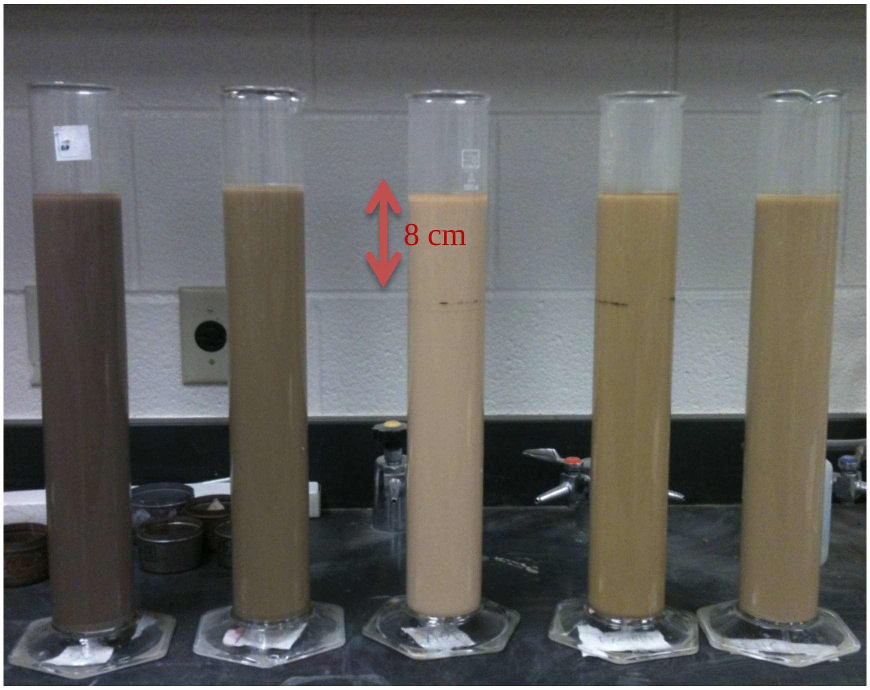


Figure 3-6 Typical Hydrometer Setup for Separation of Silt and Clay Fractions

Finally after allowing the solution to stand for 720 min in hydrometer cylinders, the clay fractions were collected by taking out the top 8cm of the solution using the syringe as shown in the figure 3-6. Later the leachate was discarded and the silt fractions were collected by discarding the top 2cm of the settled down particles. These particles were then washed to get the pure form of silt.

3.5 Chemical Testing Procedures

The three chemical properties that are used to determine the mineral content are:

1. Cation Exchange Capacity (CEC)
2. Specific Surface Area (SSA)
3. Total Potassium (TP)

3.5.1 Cation Exchange Capacity (CEC)

The Cation Exchange Capacity (CEC) is an estimate of the soils ability to attract, retain and exchange cation elements. The higher the cation exchange capacity higher will be the amount of expansiveness in soils. This expansiveness is due to the presence of the clay mineral Montmorillonite. Likewise, a low CEC value indicates the presence of kaolinite or illite.

The common method in practice for the determination of CEC of soil was proposed by Chapman in 1965.In this method, a saturating solution is added to the sample and then an extracting solution is used to remove the adsorbed cations. Ammonium acetate (NH_4OAc) at pH 7 is used as the saturating solution for this research and the extracting solution used is potassium chloride (KCl).

The natural soil sample has to be treated for organics using 30% Hydrogen Peroxide (H_2O_2). After the treatment, 125 ml of ammonium acetate (NH_4OAc) is added to a 25g sample. This mixture is shaken for about half an hour on the shaker and is allowed to stand for 16 hrs. This is done to ensure that all the exchange locations are occupied by ammonium ions (NH_4+).

The solution is then filtered using a Buchner Funnel, while applying a light vacuum, and washed with 5 separate 25ml additions of NH_4OAc. This is done to filter out all the cations replaced by the NH4+. Any excess NH_4OAc is washed out by 8 separate 10ml additions of 2-propanol. Now the sample is washed with 8 separate 25ml additions of 1 molar potassium chloride solution. The potassium ions substitute the ammonium ions and these are leached out into the solution. The amount of ammonium ions can be determined by using a spectrophotometer. Therefore, the concentration of ammonium ions in the KCl extract gives the CEC of the sample. The step wise procedure followed to determine the CEC is given in figures 3-7 and 3-8.

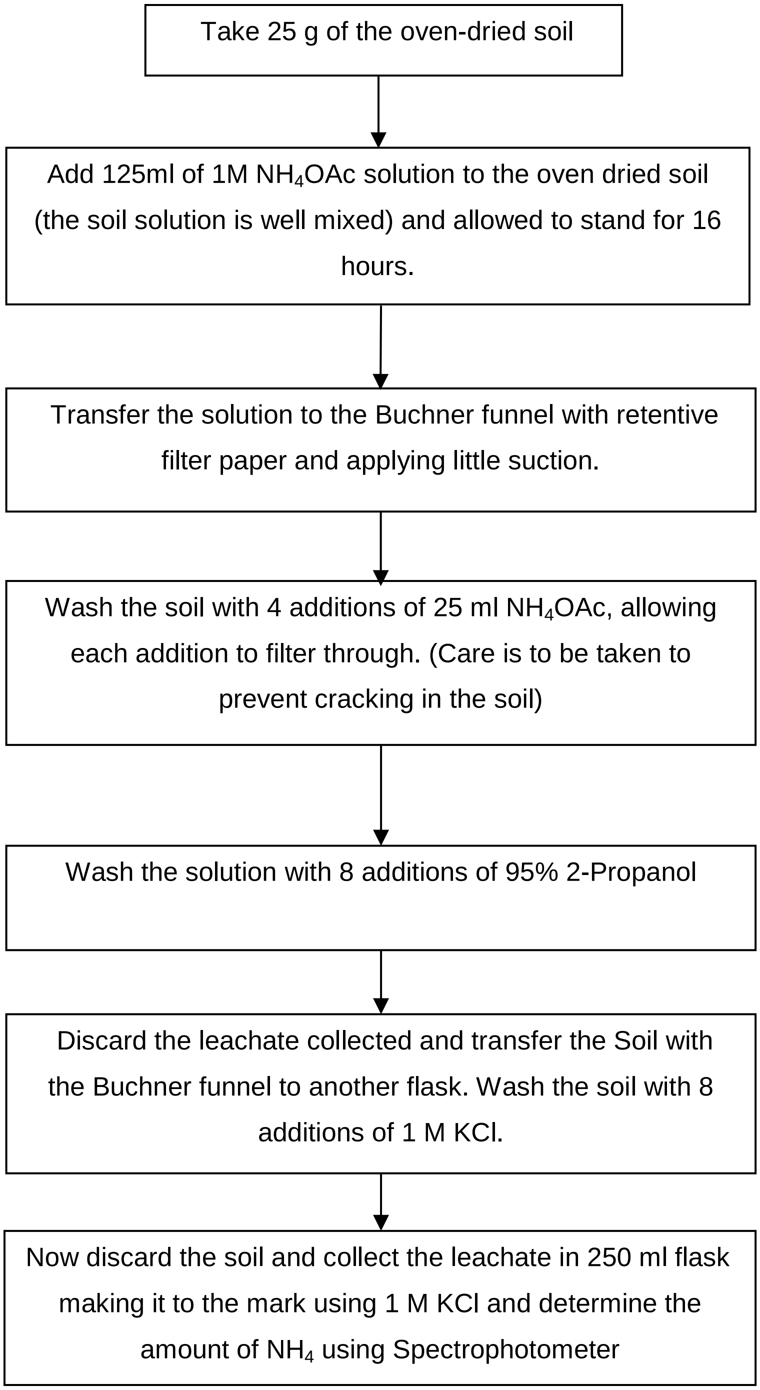
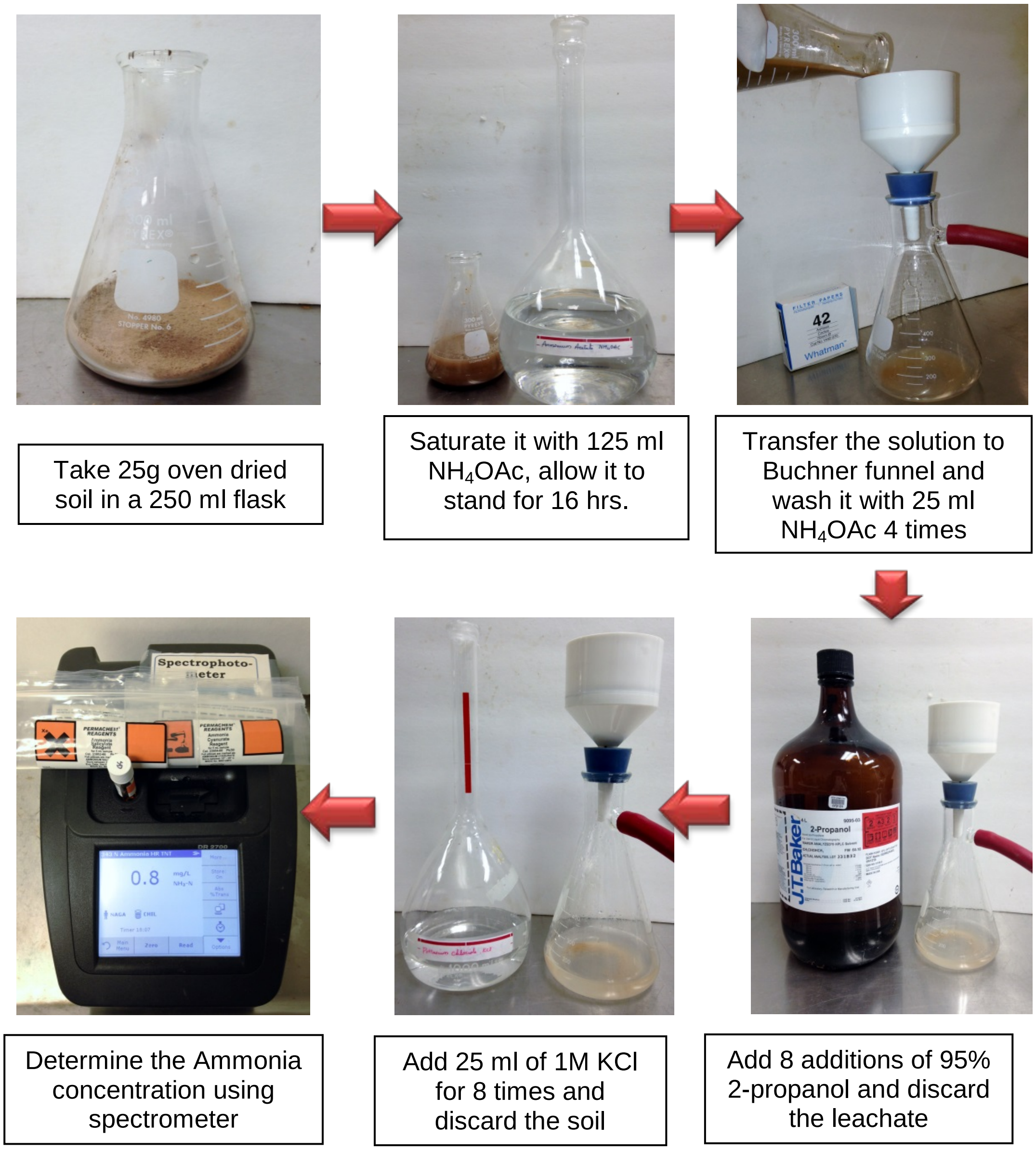


Figure 3- 7 Flow Chart showing the stepwise procedure for determining CEC



Take 25g oven dried soil in a 250 ml flask

Saturate it with 125 ml NH_4OAc, allow it to stand for 16 hrs.

Transfer the solution to Buchner funnel and wash it with 25 ml NH_4OAc 4 times

Add 8 additions of 95% 2-propanol and discard the leachate

Add 25 ml of 1M KCl for 8 times and discard the soil

Determine the Ammonia concentration using spectrometer

Figure 3-8 Photographic illustrations of steps for CEC

3.5.2 Specific Surface Area (SSA)

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

The traditional methods used are N_2 gas and adsorption of ethylene glycol-monoethyl ether (EGME). But the common method used in the field is EGME because it measures the total surface area unlike the other method where only external surface area is measured. Also it doesn’t require special instrument. The samples in this method cannot be used for multiple times. This method involves saturating the soil sample with EGME, then equilibrating them in vacuum over a calcium chloride solvate, and weighing to find the point when equilibrium is reached. Specific surface is then determined from the mass of retained EGME in comparison to the amount retained by pure montmorillonite clay, which is assumed to have a surface area of 810 m^2 /g (Carter et al., 1986). Test procedure typically takes two days to complete. This method was fully evaluated for geotechnical usage by Cerato and Lutenegger (2002) and concluded that the method is applicable to a wide range of mineralogy and is capable of determining specific surface area ranging from 15 to 800 m^2 /g. A detailed procedure was given in thesis of Chittoori (2008). Stepwise procedure of this method is presented is Figure 3-9 and 3-10.

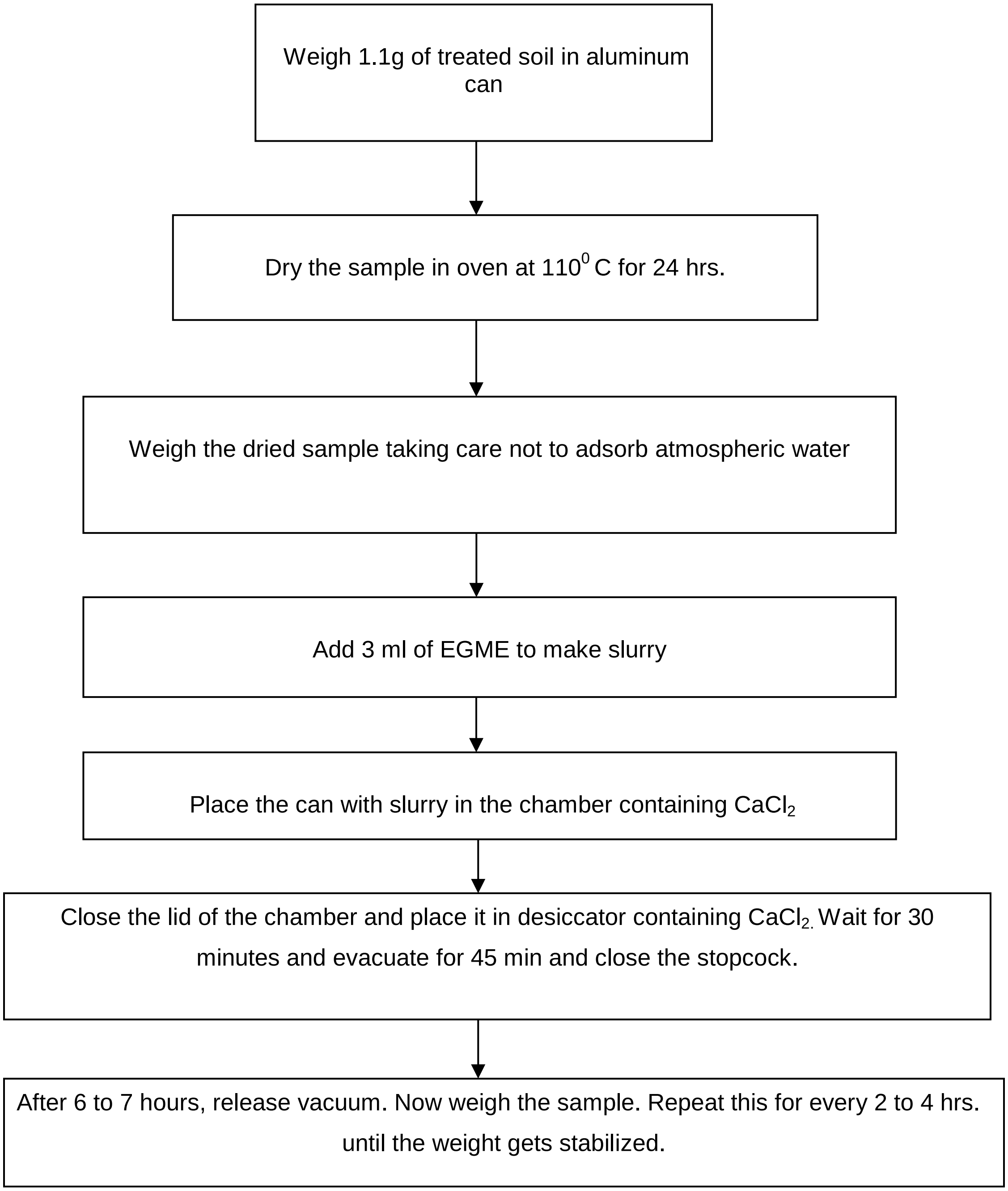
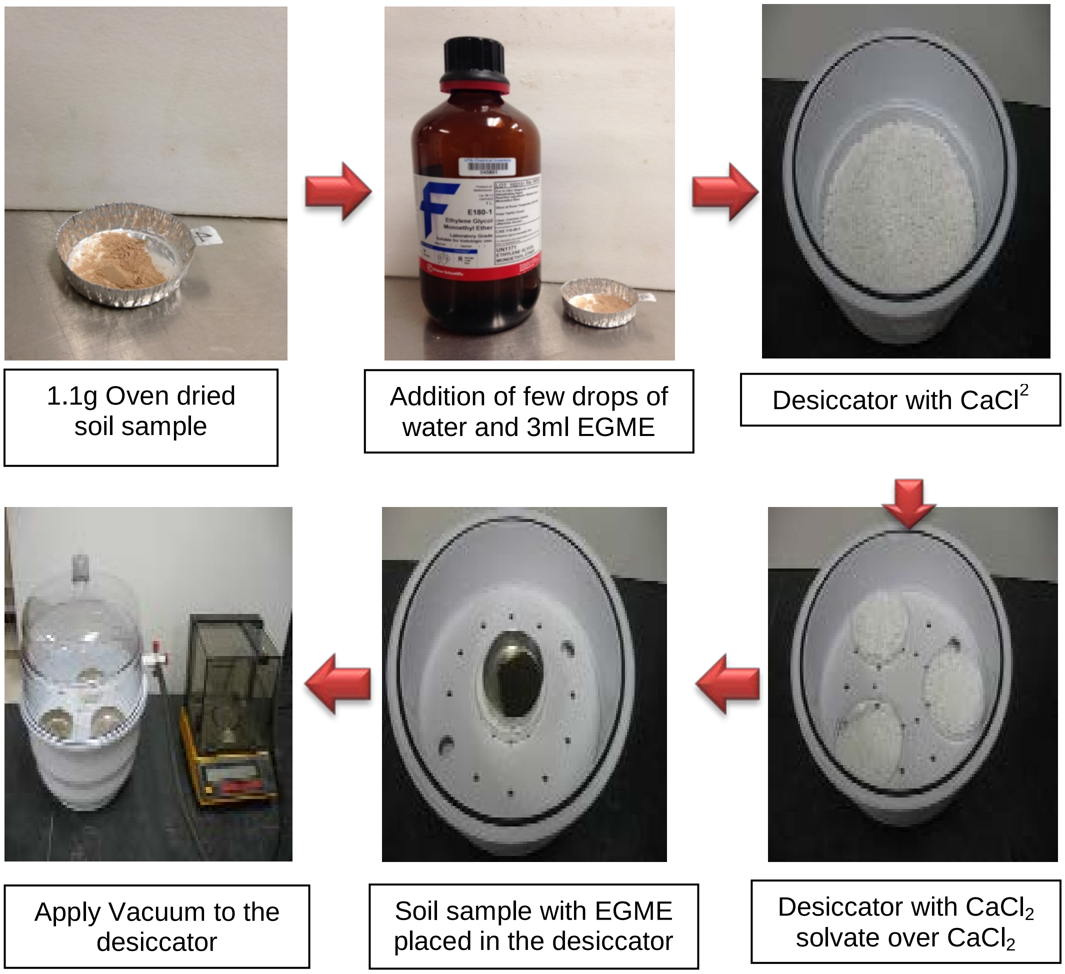


Figure 3-9 Flow Chart showing the stepwise procedure for determining SSA



1.1g Oven dried soil sample

Addition of few drops of water and 3ml EGME

Desiccator with $CaCl^2$

Apply Vacuum to the desiccator

Soil sample with EGME placed in the desiccator

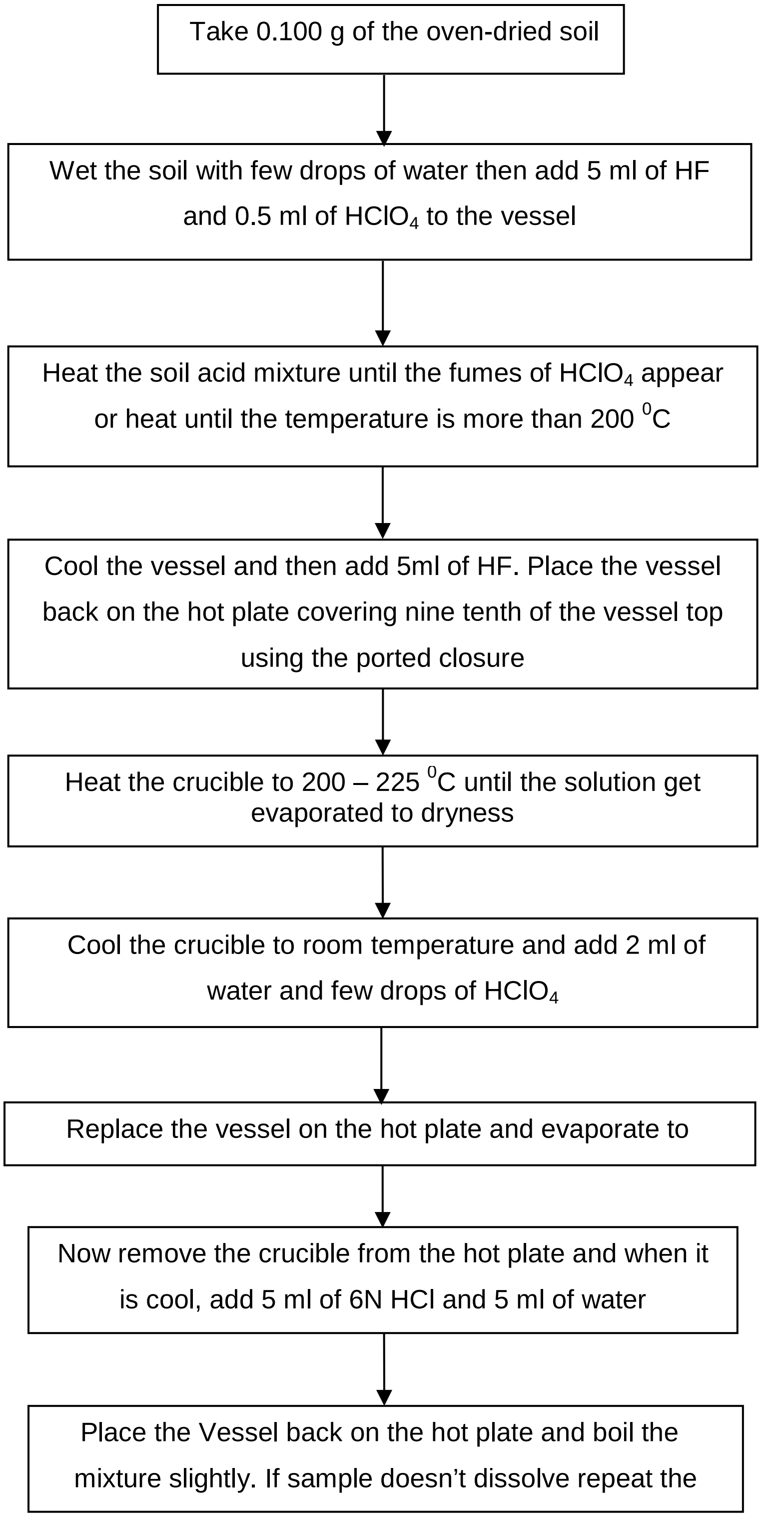
Desiccator with $CaCl_2$ solvate over $CaCl_2$

Figure 3-10 Photographic illustrations of steps for SSA

3.5.3 Total Potassium (TP)

Illite is the only clay mineral to have potassium in its structure (Mitchell & Soga, 2005). The non-clay mineral which has potassium is feldspar. In this research the test procedure formulated by Knudsen et al. (1984) was followed to obtain the amount of total potassium present in the soil. The method involves a double acid digestion technique developed by Jackson (1958), which uses two acids (Hydrofluoric acid and Perchloric acid) to break the mineral structure of the soil and extract the potassium ions from the structure.

In this test 0.1g of soil is taken in a Teflon digestion vessel. The Teflon vessel are used as an alternative to the platinum vessel because of it cost and resistance to high temperatures. To the 0.1g soil 5 ml of hydrofluoric acid and 0.5 ml of perchloric acid are added and is placed on the heat pan until the fumes of $HClO_4$ appear. Hydrofluoric acid dissolves the silicate mineral structure and releases the interlayer cations. Perchloric acid is used as an oxidizing agent to oxidize the organic matter in the soil sample. The solution is cooled to room temperature. Again 5 ml HF is added and reheat the soil acid mixture until dryness. Cool the vessel; add 2ml of water and few drops of $HClO_4$ again heat the solution to dryness. This is done to make sure all the interlayer cations are released. Then finally add 5ml of water and 5ml of 6N HCl. The amount of potassium in this solution is obtained by using a spectrophotometer. This step wise procedure is presented in the pictorial form and in flow chart for better understanding (refers Figure 3-11 and 3-12).



Take 0.100 g of the oven-dried soil

Wet the soil with few drops of water then add 5 ml of HF and 0.5 ml of $HClO_4$ to the vessel

Heat the soil acid mixture until the fumes of $HClO_4$ appear or heat until the temperature is more than 200 ^{0}C

Cool the vessel and then add 5ml of HF. Place the vessel back on the hot plate covering nine tenth of the vessel top using the ported closure

Heat the crucible to 200 – 225 ^{0}C until the solution get evaporated to dryness

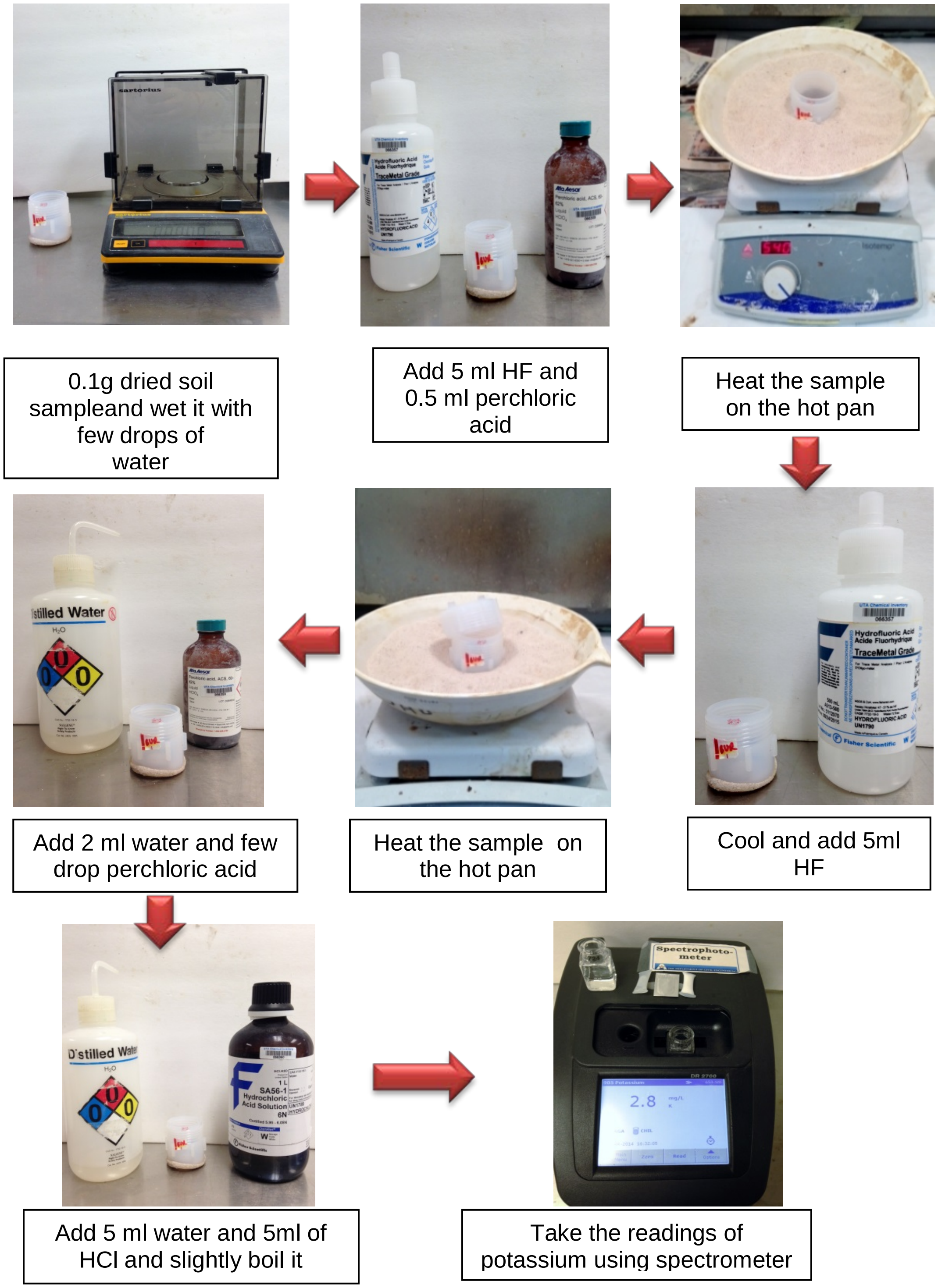
Cool the crucible to room temperature and add 2 ml of water and few drops of $HClO_4$

Replace the vessel on the hot plate and evaporate to

Now remove the crucible from the hot plate and when it is cool, add 5 ml of 6N HCl and 5 ml of water

Place the Vessel back on the hot plate and boil the mixture slightly. If sample doesn't dissolve repeat the

Figure 3-11 Flow chart showing the stepwise procedure for determining TP



0.1g dried soil sampleand wet it with few drops of water

Add 5 ml HF and 0.5 ml perchloric acid

Heat the sample on the hot pan

Cool and add 5ml HF

Heat the sample on the hot pan

Add 2 ml water and few drop perchloric acid

Add 5 ml water and 5ml of HCl and slightly boil it

Take the readings of potassium using spectrometer

Figure 3-12 Photographic illustrations of steps for TP

3.6 Summary

This chapter summarized the various laboratory tests conducted to achieve the research objective. The procedure for collecting the different grades of soil and for separation of silt and clay fraction was explained in detail. The time estimation plots and data for all the test soil for collection of clay particles was also presented. Furthermore, the chemical test procedures covered in this chapter include Cation Exchange Capacity (CEC), Specific Surface Area (SSA) and Total Potassium (TP). These tests were presented with the photographic illustrations and flow charts showing the step by step procedure.

The test results obtained by chemical testing procedures like CEC, SSA and TP are presented in the next chapter. This is followed with a thorough analysis and discussion on these results.

Chapter 4

Results and Discussions

4.1 Introduction

The focus of this research was to accurately quantify non-clay minerals feldspar and quartz along with the clay minerals Montmorillonite, Kaolinite and Illite. A total of 17 natural soil samples were collected from Colorado, Texas, Oklahoma and San Diego and tested. Basic soil characterization studies were conducted using standard techniques and later subjected to chemical testing.

The chemical tests include Cation Exchange Capacity (CEC), Specific Surface Area (SSA) and Total Potassium (TP). These results were first used to quantify both clay and non-clay minerals using the quantification model developed by Chittoori and Puppala (2011). The drawbacks of this model were highlighted and a new model was developed that predicts the percentages of both clay and non-clay minerals with greater accuracy. This chapter presents the laboratory test results along with their analysis and discussion.

4.2 Chemical Testing Results

The chemical tests were performed on the soils following the methodology in Chapter 3. For the consistency of the results, repeatability of the chemical tests was important. Previous studies by Chittoori (2008) and Sarwanaj (2009) indicated that tests have very good repeatability with a very low standard deviation between the results.

Before performing the tests, sieve analysis was performed on the test soils to separate the particles retained and passed through 75μm sieve. In the next step, particles that passed through 75 microns sieve were further separated into silt and clay fractions following the procedure in Chapter 3. These samples were chemically tested in three different phases. In first phase, X-ray diffraction test was performed on particles passed through 75μm sieve for the identification of minerals. In second phase, cation exchange capacity (CEC), specific surface area (SSA) and total potassium (TP) tests were performed on the particles passed through 75µm sieve. This phase is further divided into two sub phases 2(a) and 2(b) where clay and silt fractions were tested separately for total potassium. Finally all the results will be analyzed for quantification of minerals.

Results for CEC, SSA and TP obtained in the phase 2 are presented in the table 4-1. The results from this table were used for the quantification of minerals using regression model.

From the table 4-1 it was observed that Eagle ford soil has high CEC and SSA values indicating that the soil is dominant with expansive clay mineral montmorillonite. It was also noted that Dallas soil has high total potassium content indicating the presence of high percentage of minerals Illite when compared to other soils.

Table 4-1 CEC, SSA and TP Results from Phase 2

Soil No.	Soil Samples	Cation Exchange Capacity (CEC) meq/100g	Specific Surface Area (SSA) m^2/gm	Total Potassium (TP) %
1	Dallas	104.1	238.9	3.36
2	Sherman	156.0	133.9	1.9
3	Burleson	100.1	132.4	1.17
4	Wilcox	75.0	449.4	1.28
5	Riverside	114.7	185.2	1.36
6	Eagle ford	170.0	368.0	1.69
7	Jeopool	169.8	115.0	1.68
8	Woodbine	58.4	129.4	1.28
9	Stephenville	124.9	203.0	0.94
10	Colorado	91.6	185.0	2.1
11	San Diego	87.6	91.4	1.51
12	Austin	25.2	248.1	1.31
13	Grayson	116.8	221.9	1.43
14	Rolling Hill	54.9	197.6	0.84
15	Keller	60.0	115.0	1.1
16	Anthem	71.7	118.5	1.46
17	Oklahoma	64.1	76.0	4.2

In phase 3, the chemical analysis was performed for total potassium on separate fractions of silt and clay in soil samples. The tests results for total potassium performed on these samples from each soil are presented in the table 4-2. The data from this table along with the part of data from table 4-1 were analyzed in modified method for mineral quantification.

Table 4-2 Total Potassium for Silt and Clay Fractions

Soil Samples	**TP for particles passed 75μm sieve**	
	Silt Fractions %	**Clay Fractions %**
Dallas	0.09	3.27
Sherman	1.66	0.235
Burleson	0.11	1.065
Wilcox	0.09	1.19
Riverside	0.35	1.01
Eagle ford	0.15	1.545
Jeopool	0.34	1.335
Woodbine	0.1	1.175
Stephenville	0.14	0.8
Colorado	0.22	1.885
San Diego	0.42	1.09
Austin	0.72	0.585
Grayson	0.15	1.285
Rolling Hill	0.14	0.705
Keller	0.11	0.99
Anthem	0.29	1.175
Oklahoma	0.53	3.67

Finally, these results are analyzed for quantifying the minerals using the regression equation model developed by Chittoori and Puppala (2011) and the modified model. The details of this analysis are presented in later sections.

4.3 Identifying Soil Minerals

Siemens D500 X-ray powder diffraction system was used in this research for the identification of minerals present in the soil. The sample preparation for XRD includes the pulverization of soil into powder fine enough to mount in the focal plane of the diffractometer. The planar upper surface of the powder has been placed precisely in the instrument to get the accurate values of d- spacing. The commonly used radiation for the soil mineralogical analysis is CuKα (λ=1.54^{o}A). The results of XRD are plotted as 2θ angle on x- axis and intensities on y- axis. From this plot, the peaks were identified with their corresponding d-spacing and intensity values using the JADE software.

JADE software was also used for the identification of minerals by comparing d-spacing and intensity values of soil peaks with that of pure minerals like Montmorillonite, Kaolinite, Illite, K- Feldspar and Quartz. Finally the minerals were identified. Table 4-3 and 4-4 shows the identified mineral in Anthem and Keller soils.

In the table, the stars marked below each mineral column indicate that the particular mineral is present at that peak d-spacing and intensity. It was observed from the table that Anthem soil has more stars in K-feldspar column indicating the presence of high percentage of that mineral whereas the low percentage of K-Feldspar was found in the Keller soil. Similarly the minerals for all the test soils were identified and the corresponding tables are presented in Appendix A. Table 4-5 gives the summary of matched peaks for K-feldspar in test soils.

Table 4-3 Identification of minerals in Anthem soil using peak d-spacing and Intensity values

Peak d-Spacing (^{o}A)	**Peak intensity %**	**Montmorillonite**	**Kaolinite**	**Illite**	**K- Feldspar**	**Quartz**
10.541	4.7			★		
8.5038	2.9					
6.9607	2.7					
6.435	7				★	
4.4911	5	★			★	
4.2728	20.4					
3.8791	4.2					★
3.67	5.4					
3.3567	100					★
3.2007	14.3			★		
3.1651	3.5					★
3.0383	33.4	★				★
2.834	2.6		★			
2.4968	4.8		★			
2.4601	5.8				★	
2.2852	12.6					
2.2396	3.6					
2.13	4.7		★			
2.0933	6.3					
1.9828	4.2					
1.9109	4.4				★	
1.8753	5.8					
1.8204	10.7					
1.8058	2.4				★	
1.6748	4.2					
1.6598	3.1					
1.6244	2.3					
1.6039	3.3					
1.5427	11.2					
1.5056	2.6			★		★
1.3844	4.1				★	★
1.373	8.1				★	

Table 4-4 Identification of minerals in Keller soil using peak d-spacing and Intensity value

Peak d-Spacing (°A)	Peak Intensities %	Montmorillonite	Kaolinite	Illite	K-Feldspar	Quartz
4.3352	31.6			★		
4.0979	8.7					
3.9296	8.7		★			
3.6396	9.5	★				
3.387	100					★
3.069	49.8	★		★		
2.5132	12.9					
2.4796	17.9		★			
2.303	19.4					★
2.2561	8.4		★			
2.1417	17.1					★
2.11	7.2				★	
1.9912	16		★	★		
1.9216	9.1					
1.8855	14.4					
1.8315	16.7					
1.6794	11				★	
1.6692	6.5					
1.611	7.6					
1.5497	16.3					
1.4592	6.5					
1.4282	6.8					
1.3877	9.5					★
1.3773	16					

Table 4-5 Number of matched peaks with Potassium feldspar Mineral for all the test soils

Soil No.	Samples	No. of matched peaks
1	Dallas	2
2	Sherman	7
3	Burleson	3
4	Wilcox	4
5	Riverside	5
6	Eagle ford	3
7	Joe pool	4
8	Woodbine	4
9	Stephenville	3
10	Colorado	6
11	San Diego	4
12	Austin	6
13	Grayson	4
14	Rolling Hill	4
15	Keller	2
16	Anthem	7
17	Oklahoma	4

4.4 Mineral Quantification Using Chittoori and Puppala (2011) Model

As mentioned in chapter 2 there are many methods for the identification and quantification of clay minerals like X-ray diffraction, Vibration spectroscopy, Thermal analysis and atomic absorption spectrometry. Some of these methods rely on XRD peaks to quantify the amount of minerals present based on the intensity of the peaks. Other methods rely on the use of chemical analyses to predict the quantity of minerals present. This research focused on chemical mass balance analysis for quantifying the soil minerals. The results obtained from chemical tests CEC, SSA and TP are analyzed using the regression model developed by Chittoori and Puppala (2011). The mineralogical compositions of different soils can be estimated using the following set of three simultaneous equations:

$$\%M \times CEC_M + \%K \times CEC_K + \%I \times CEC_I = CEC_{soil} \qquad (1)$$

$$\%M \times SSA_M + \%K \times SSA_K + \%I \times SSA_I = SSA_{soil} \qquad (2)$$

$$\%M \times TP_M + \%K \times TP_K + \%I \times TP_I = TP_{soil} \qquad (3)$$

Where,

CEC_M, CEC_k, CEC_I = Cation exchange capacity of Montmorillonite, Kaolinite and Illite

SSA_M, SSA_k, SSA_I = Specific surface area of Montmorillonite, Kaolinite and Illite

TP_M, TP_k, TP_I = Total Potassium of Montmorillonite, Kaolinite and Illite

All these equations are based on the assumption that the soil has only three clay minerals Montmorillonite, Kaolinite and Illite. Based on this assumption the regression equation model was developed.

4.4.1 Regression Equation Model

Solving the simultaneous equations is a very tedious process and involves numerous iterations. A simpler regression equation model using CEC, SSA and TP for quantification of minerals was developed by Chittoori and Puppala (2011). The equations which are used to predict the mineral percentages are

$$\%I = \left[\frac{TP}{6}\right] \times 100 \qquad (4)$$

$$\%M = -2.87 + 0.08 \times SSA + 0.26 \times CCE \qquad (5)$$

$$\%K = 100 - \%I - \%M \qquad (6)$$

Where,

%I = Percentage of the mineral Illite

%M = Percentage of the mineral Montmorillonite

%K = Percentage of the mineral Kaolinite

TP = Total potassium

CEC = Cation exchange capacity

SSA = Specific surface area

These equations give the percentage of each clay mineral Montmorillonite Kaolinite and Illite. These results are presented in the table 4-6. From the table, it can be observed that the Eagle ford soil has high montmorillonite and illite percentages and the Keller soil has less percentage of these minerals. The mineral kaolinite is dominant in Rolling Hill soil and is found less in Dallas soils

Table 4-6 Percentage Minerals Using the Regression Model by Chittoori and Puppala (2011)

Soil Samples	Montmorillonite %	Kaolinite %	Illite %
Dallas	29.45	0.47	38.08
Sherman	24.20	10.01	15.79
Burleson	13.84	19.17	8.00
Wilcox	17.87	8.90	7.23
Riverside	12.95	11.03	7.03
Eagleford	36.09	0.53	14.38
Jeopool	20.70	8.86	11.45
Woodbine	8.84	21.87	8.29
Stephenville	22.00	18.48	7.52
Colorado	11.44	9.36	11.20
San Diego	10.34	18.09	9.56
Austin	8.24	19.15	7.61
Grayson	21.72	14.84	11.44
Rolling Hill	10.61	22.93	5.46
Keller	7.68	20.91	6.42
Anthem	8.08	16.13	7.79
Oklahoma	8.15	4.15	28.70

4.5 Limitation

One limitation for regression equation model developed by Chittoori and Puppala (2011) was that it does not consider non-clay minerals in the analysis and hence couldn't identify minerals such as feldspar and quartz. Also, the potassium content determined was from both silt and clay particles. In this regard, present research focus mainly on the scope of finding the accurate method for quantification of the clay minerals present in the finer soil by giving the percentage of amorphous contents like feldspar and quartz.

4.6 Modified Mineral Quantification Model

The basic assumption of modified mineral quantification model is that the soil has common minerals Montmorillonite, Kaolinite, Illite, Quartz and feldspar.

In this method, the percentages of clay minerals were calculated using the regression equation model. The Modification includes calculating the percentage of illite by considering the total potassium from clay fractions. The calculated mineral percentages show the amount of mineral present in the clay of soil. Percentages of clay minerals present in the finer part of the soil were obtained by multiplying the percentage clay present in the soil with each clay mineral percentages.

In next step, the percentage of non-clay mineral quartz and Feldspar were calculated. The percentage of non-clay minerals were found by deducting the amount of clay minerals from 100% which represents the percentage of clay and non-clay minerals in soils.

$$\%Q + \%F = 100 - (\%M + \%K + \%I) \qquad (8)$$

The total potassium from the silt fractions of test soils can give the approximate percentage of K-feldspar. More research has to be done for the accurate quantification of non-clay mineral feldspar because more the amount of feldspar there are chances of lesser clay mineral illite in the soil. The results using modified quantification method were presented in the table 4-7.

Table 4-7 Percentages of Minerals Using the Modified Method

Soil Samples	**Montmorillonite %**	**Kaolinite %**	**Illite %**	**Quartz and Feldspar %**
Dallas	29.45	1.49	37.06	32.00
Sherman	24.20	23.84	1.96	50.00
Burleson	13.84	19.89	7.28	59.00
Wilcox	17.87	9.38	6.74	66.00
Riverside	12.95	12.83	5.22	69.00
Eagle ford	36.09	1.77	13.13	49.00
Jeopool	20.70	11.18	9.12	59.00
Woodbine	8.84	22.52	7.64	61.00
Stephenville	22.00	19.60	6.40	52.00
Colorado	11.44	10.51	10.05	68.00
San Diego	10.34	20.75	6.90	62.00
Austin	8.24	23.35	3.41	65.00
Grayson	21.72	16.00	10.28	52.00
Rolling Hill	10.61	23.80	4.58	61.00
Keller	7.68	21.55	5.78	65.00
Anthem	8.08	17.65	6.27	68.00
Oklahoma	8.15	7.77	25.08	59.00

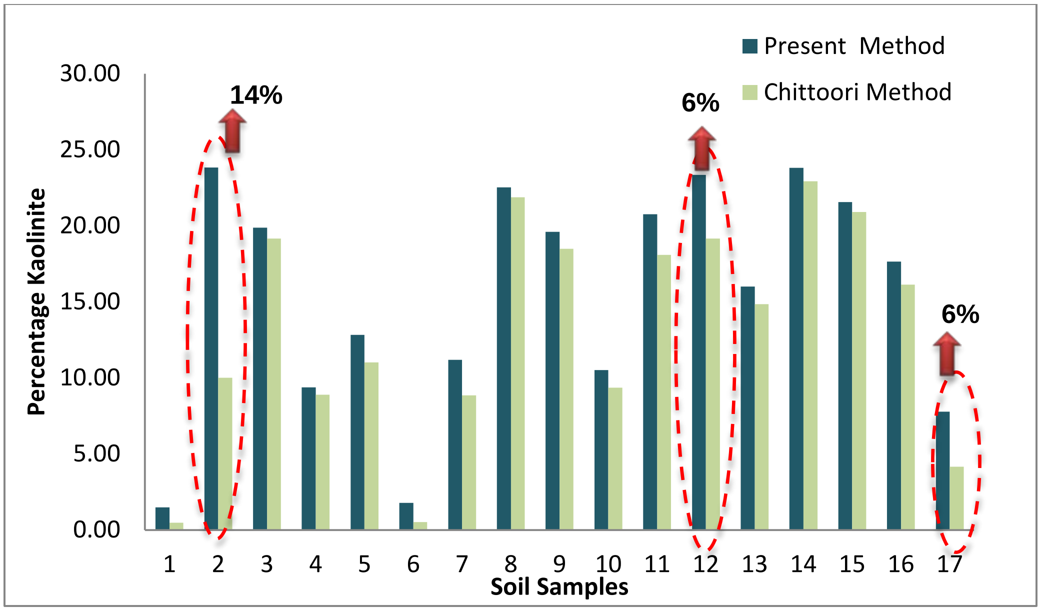
From the table; it was observed that the percentage Montmorillonite has remained same in both the method. The other minerals Kaolinite and illite have changed compared to previous method.

4.7 Comparison of Modified Quantification Model with Chittoori Model

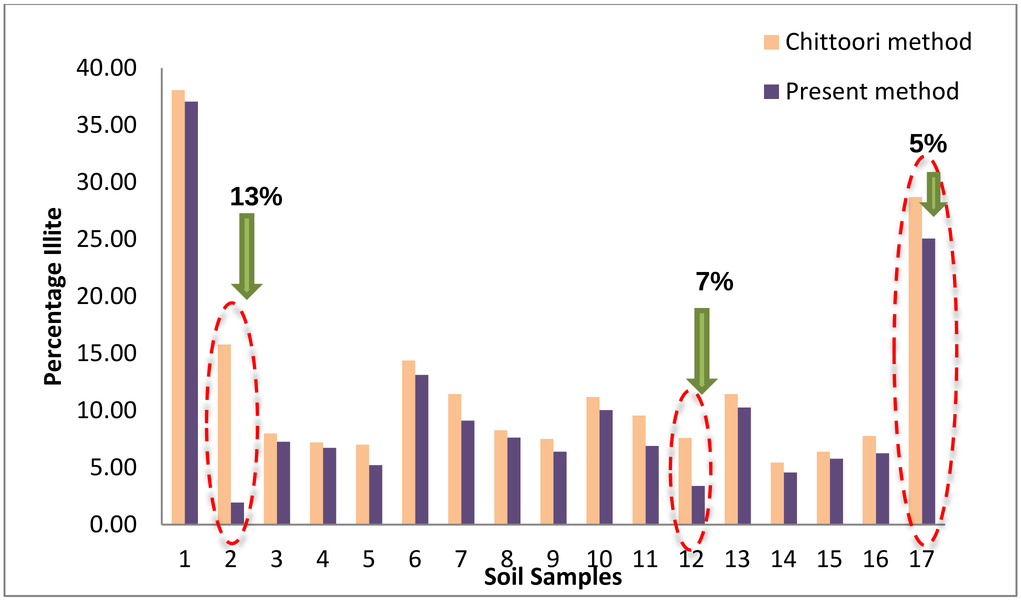
The modified quantification model developed could identify and quantify both the clay and non-clay minerals present in the soil. The major finding of this method is giving the accurate percentage of clay minerals montmorillonite, Kaolinite and Illite by the identification and quantification of non-clay minerals quartz and feldspar. The results from Chittoori and Puppala (2011) method and the modified quantification method were compared in figures 4-1.

From the 4-1(a), it was observed that the kaolinite percentage for the Sherman soil calculated using the Chittoori method predicted low percentages compared to the modified quantification method. The difference is as high as fifteen percent. This high difference in the mineral value could change the behavior of the entire soil. Similarly the difference of up to 5% was observed in two more samples Austin and Oklahoma.

From the plot 4-1(b), soils it can be observed that the Illite percentage predicted using the modified method is very less compared to Chittoori method for soils Sherman, Austin and Oklahoma. In Chittoori method, it was assumed the total potassium for particles passed through 75μm sieve is from illite. Even though the total potassium coming from particles passed through 75μm sieve is very high, most of it is from silt fractions of the soil indicating the presence of non-clay mineral K-feldspar.



a)



b)

Figure 4.1 Comparisons of Results between the Present Method and Chittoori Method

a) Kaolinite and b) Illite

4.8 Summary

In this chapter, the minerals were quantified using the regression equation model by Chittoori and Puppala (2011) and modified quantification model. First, the data obtained from the chemical tests CEC, SSA and TP performed on seventeen soil samples for the quantification of minerals was presented. The clay mineral percentages were obtained by analyzing the chemical test results with regression equation model. The results from the X-ray diffraction testing performed on the test soils for identification of minerals were also presented which indicated the presence of minerals K-feldspar and quartz. Finally the results for percentages of both clay and non-clay minerals in the finer soil of 75μm using the modified model was also presented.

Furthermore, Modified quantification method results were compared to that method developed by Chittoori and Puppala (2011). With this comparison it can be concluded that, In soils with higher percentage of feldspar the Chittoori's method over-estimated the amount of swell mineral illite and lesser percentage of mineral kaolinite. The present method could accurately quantify the amount of clay and non-clay minerals present in finer part of soils.

The summary, conclusions and future recommendations for the quantitative validation of this modified method will be discussed in the next chapter.

Chapter 5

Summary Conclusions and Recommendations

5.1 Introduction

Soils which exhibit substantial swelling and shrinkage due to moisture fluctuations are termed as expansive soils. This swelling and shrinkage of expansive soil cause damage to the infrastructure built on them (Jones and Holtz, 1973, Puppala and Cerato, 2009). Several research studies have been attempted in the past to understand the behavior of expansive soils. Basic soil properties are adopted by researchers but the mineralogical content has always been neglected for understanding the behavior of soils.

Recent studies have shown that mineralogy is one of the fundamental parameter governing the swell shrink behavior of any expansive soils. Also it was showed by Pedarla et al. (2011) and Chittoori (2013) that the current stabilizing practices based on PI and gradation of soil is insufficient for selecting an effective stabilizer as soils having same PI act differently to stabilizers and due consideration should be given to mineralogy. There are many methods for the identification and quantification of clay minerals like X-ray diffraction, Vibration spectroscopy, Thermal analysis and atomic absorption spectrometry. Chittoori and Puppala (2011) developed models using the chemical properties for the identification and quantification of clay minerals based on the assumption that the soil has only the dominant clay minerals montmorillonite, kaolinite and illite. This method has been validated by Sarwanaj (2009).

This present study focuses on the scope of finding the accurate method for quantification of the clay minerals present in the soil by giving the percentage of amorphous contents like feldspar and quartz using the regression model developed by Chittoori (2008). In order to achieve this goal the chemical testing like CEC, SSA and TP was performed on samples with different mineralogical content. For the study seventeen soils from different parts of United States were selected. Chemical tests were performed on different grades of test soils.

From the study, several significant conclusions were developed. The analysis showed that the test soils have considerable amount of feldspar and quartz along with clay minerals montmorillonite, kaolinite and Illite. The results from both the methods were compared and showed that there was a considerable change in the mineral Kaolinite and Illite whereas the mineral montmorillonite remained constant. The modified method should be followed when XRD results predict more concentration of K-feldspar in the soil.

5.2 Conclusions

Based on the results obtained from the chemical analysis for quantification of non-clay and clay minerals, the following conclusions can be deduced.

X-ray diffraction analysis performed on particles passed through 75μm sieve in each test soil has shown the presence of non-clay minerals quartz and potassium feldspar. In some soils there are few traces of k-feldspar but in soils like Sherman, Anthem, Austin and Colorado, more traces of potassium feldspar are detected.

It was observed that the regression model developed by Chittoori and Puppala (2011) for quantification of minerals assumed that the soil has only three dominating clay minerals Montmorillonite, kaolinite and illite. In this method, Illite was calculated using the total potassium coming from particles passed through 75μm sieve (has both silt and clay). The total potassium coming from silt is from the non-clay mineral K-feldspar and this is not considered in the analysis.

The modified quantification model developed in this research accounted for this correction. This method has also provided the percentage of non-clay minerals including feldspar and quartz. From the comparison of both the methods, it was observed that the percentage of kaolinite has shown high values when compared to old method whereas the percentage of mineral illite has been decreased. The non-clay fraction quantification will assist engineers for better characterization of subsoils.

5.3 Future Recommendations

1 X-ray diffraction analysis should be performed especially on soils coming from igneous rocks where feldspar is the prevalent mineral. If the soil has high traces of K-feldspar then the modified method should be followed.

2 The method developed could predict the non-clay minerals quartz and feldspar along with the dominating clay minerals montmorillonite, kaolinite and illite. Future studies should focus on determining the percentages of quartz and feldspar, separately.

Appendix A

X-ray Diffraction Results

Table 1 Identification of minerals in Dallas soil using peak d-spacing and Intensity values

d(A)	I%	Montmorillonite	Kaolinite	Illite	K-Feldspar	Quartz
22.4968	14.7					
4.5189	20.8	☐	☐			
4.294	15.8					
3.3667	100			☐		☐
3.0444	12.1	☐		☐		
2.6141	10.2			☐	☐	
2.5964	17.4	☐				
2.5759	15.5	☐				
2.4738	14.3		☐			
2.2896	10.9					
1.9865	8.3		☐			☐
1.825	16.2					
1.6746	9.4			☐		☐
1.5454	10.2					
1.5098	9.4					
1.4998	10.6			☐	☐	☐

Table 2 Identification of minerals in Sherman soil using peak d-spacing and Intensity values

d(A)	I%	Montmorillonite	Kaolinite	Illite	K-Feldspar	Quartz
15.9348	9.8					
15.1172	10.8	☐				
7.778	7					
7.3246	5		☐			
5.1366	3.6					
4.5048	16.6			☐		
4.4079	9.6					
4.2854	20					
3.8676	4.1				☐	
3.3616	100			☐	☐	
3.0705	7.1		☐			
3.0404	29.5					☐
2.6038	5.5	☐				
2.5761	9.9	☐				
2.5564	8.3				☐	
2.4615	8.8	☐				
2.2864	8.8					
2.2447	4.8					
2.1366	7.9		☐		☐	
1.9814	4					
1.8794	4.8		☐			☐
1.8204	10.9					
1.6736	6.3				☐	
1.5434	9.8					
1.4994	4.6					
1.4946	5					
1.4876	3.8					
1.3838	5.1				☐	☐
1.3772	8.3					
1.3738	6.1			☐	☐	

Table 3 Identification of minerals in Burleson soil using peak d-spacing and Intensity values

d(A)	I%	Montmorillonite	Kaolinite	Illite	K-Feldspar	Quartz
4.5505	10.1					☐
4.4787	12.8	☐	☐			
4.2891	17.4				☐	
3.879	12.3					☐
3.3522	100					
3.0483	83.3					☐
2.5718	8	☐				☐
2.4958	10.9		☐			
2.4599	5.6					
2.2885	23.2					
2.2401	5.3			☐		
2.1329	4.8					
2.0981	18.8			☐		
1.9836	6.3		☐		☐	☐
1.933	7.2					
1.9169	18.4					
1.8784	19.8					
1.8218	13					
1.6251	6.3					
1.6024	7.2					
1.545	8.5			☐		☐
1.5256	5.8					
1.5198	8.2				☐	
1.4417	6.3					☐

Table 4 Identification of minerals in Wilcox soil using peak d-spacing and Intensity values

d(A)	I%	Montmorillonite	Kaolinite	Illite	K-Feldspar	Quartz
6.0051	3.3	☐				
4.3229	22.9					☐
3.912	4.1					
3.3793	100					☐
3.0606	11.5	☐				
2.7574	3.6		☐			
2.4717	8.7	☐		☐		
2.2987	12					☐
2.2499	4.9		☐	☐		
2.1388	6.5				☐	
2.1026	2.8					☐
1.9903	3.5		☐			
1.9216	3.8			☐		
1.8848	3.5				☐	
1.8266	21.6					
1.6788	5				☐	
1.6686	3					
1.5478	12					
1.4593	2.8				☐	☐

Table 5 Identification of minerals in Riverside soil using peak d-spacing and Intensity values

d(A)	I%	Montmorillonite	Kaolinite	Illite	K-Feldspar	Quartz
13.5069	3	☐				
8.9437	3.2					
4.5828	3					☐
4.5238	6.1					☐
4.3942	3.4					☐
4.3141	18.4					☐
4.0829	3.5					
3.7058	2.3		☐			
3.3742	100				☐	☐
2.6005	6.1	☐				
2.468	9.3			☐		
2.2951	7.6		☐			
2.2518	5.3					
2.1387	5.4				☐	
1.991	5.8		☐	☐	☐	☐
1.8246	15.2					
1.6781	5.4				☐	
1.6658	2.6			☐		
1.5469	12.3					
1.4558	2.7				☐	
1.3852	5.1					
1.3759	9.8					

Table 6 Identification of minerals in Eagle Ford soil using peak d-spacing and Intensity values

d(A)	I%	Montmorillonite	Kaolinite	Illite	K-Feldspar	Quartz
15.5527	9.7	☐				
4.5185	10.8	☐				☐
4.474	11.7	☐	☐			
4.4025	4.1					
4.2888	20.7					
3.3568	100					☐
3.0461	18.3	☐				☐
2.5972	4.1	☐				☐
2.5717	8					
2.556	5.9			☐	☐	
2.5114	4.3					
2.4638	10.4			☐		
2.2885	13.5					
2.2415	4.9			☐		
2.1329	6				☐	
2.0971	3.2			☐		
1.9828	8		☐			
1.9169	3.7					
1.8774	4					
1.8225	19.4					
1.6746	5.1					
1.6608	2.9					
1.6063	4.3					
1.5445	15.4					
1.4554	2.7				☐	☐
1.382	15.1					
1.3734	5.7			☐		

Table 7 Identification of minerals in Jeopool soil using peak d-spacing and Intensity values

d(A)	I%	Montmorillonite	Kaolinite	Illite	K-Feldspar	Quartz
4.5556	4.3					
4.5226	4	☐				☐
4.3343	17.3			☐		
3.3866	100					
3.0671	8.1					☐
2.608	3					☐
2.5893	5.4	☐				
2.478	8.5					
2.2976	7.3		☐		☐	
2.2513	3.5			☐		
2.1436	6.7					
2.1075	3					☐
1.9943	5.9		☐			☐
1.923	3.9				☐	
1.8267	12					
1.6815	3.7				☐	
1.6659	2.8			☐	☐	

Table 8 Identification of minerals in Woodbine soil using peak d-spacing and Intensity values

d(A)	I%	Montmorillonite	Kaolinite	Illite	K-Feldspar	Quartz
4.5009	4.3	☐				
4.4563	4					☐
4.2697	27					☐
3.3543	100				☐	☐
2.5662	1.9		☐			
2.4599	7.7					
2.2853	7.9					
2.2405	5.7		☐	☐		
2.132	10				☐	
1.9827	3.9					
1.8205	12.5			☐		
1.6748	3.9				☐	
1.6604	2.2					
1.5436	14.1					
1.4911	1.5					
1.4534	1.5					
1.3833	5.3					☐
1.3747	7.8				☐	

Table 9 Identification of minerals in Stephenville soil using peak d-spacing and Intensity values

d(A)	I%	Montmorillonite	Kaolinite	Illite	K-Feldspar	Quartz
4.5099	6.7	☐				☐
4.478	6.7	☐	☐			☐
4.2976	15.1			☐		☐
3.8687	9.7				☐	
3.3593	100					☐
3.0503	81.5	☐				
2.853	6.3					
2.5775	4.2	☐				
2.5024	15.5					
2.4676	11.7			☐	☐	
2.2918	26.2					☐
2.2439	5.8		☐			
2.1348	6.7					
2.0997	20.6		☐			
1.9829	7.5					
1.9286	6.2					
1.9177	16.3					
1.879	22.2					
1.8245	15.5					
1.676	4.6				☐	
1.6068	6.9					
1.545	10.9			☐		
1.5278	4.6					
1.4425	5.2					
1.4255	3.2					☐
1.4198	3.6					
1.3827	5.6					
1.3772	11.5					
1.3734	12.3		☐			

Table 10 Identification of minerals in Colorado soil using peak d-spacing and Intensity values

d(A)	I%	Montmorillonite	Kaolinite	Illite	K-Feldspar	Quartz
4.5707	5.6	☐				
4.3399	13.1			☐		☐
3.402	100					☐
3.0415	4.6	☐			☐	
2.5928	6.2	☐			☐	
2.4876	10.8				☐	
2.3044	9.2					☐
2.2638	4.4				☐	
2.1523	5.9		☐	☐		
1.9982	5.1		☐			☐
1.8328	15.1					
1.6826	6.9				☐	
1.5524	10.5					
1.5032	4.1			☐		☐
1.3888	7.7				☐	

Table 11 Identification of minerals in San Diego soil using peak d-spacing and Intensity values

d(A)	I%	Montmorillonite	Kaolinite	Illite	K- Feldspar	Quartz
6.4978	2.6					
4.5741	6.9	☐				☐
4.4932	4					
4.3012	13.1					
4.0753	8.3					☐
3.8913	3.3					☐
3.7974	8					
3.6939	4.5					
3.3931	5.2		☐			☐
3.3618	100					
3.2099	29			☐		
3.0446	12.8				☐	
2.9945	3.1	☐			☐	
2.6381	4					
2.5436	3.5					
2.4665	5					
2.2875	5.8					
2.1359	5.5		☐			
2.0963	2.6		☐			
2.0743	3.3				☐	
1.9853	3.4					
1.9172	4.1					
1.8796	3					
1.8231	4.7					
1.7867	2.1					
1.6753	5.4				☐	
1.6059	2.6					
1.545	4.6					
1.5059	2.4			☐		☐
1.3848	3					☐

Table 12 Identification of minerals in Austin soil using peak d-spacing and Intensity values

d(A)	I%	Montmorillonite	Kaolinite	Illite	K-Feldspar	Quartz
6.4759	4.2					
3.8954	9.3			☐		
3.3845	5.8	☐				
3.0646	100	☐				☐
2.8589	18.1				☐	
2.5118	14.9					
2.2985	23		☐		☐	☐
2.1054	15.9					☐
1.9371	10.7				☐	
1.9217	17.7		☐			
1.887	18.3					
1.6299	4			☐	☐	
1.6109	9.1					
1.5312	6.9				☐	
1.522	3.8					
1.5135	3.8				☐	
1.4778	3					☐

Table 13 Identification of minerals in Grayson soil using peak d-spacing and Intensity values

d(A)	I%	Montmorillonite	Kaolinite	Illite	K- Feldspar	Quartz
15.478	11.9	☐				
10.161	7.1			☐		
6.5337	10.2				☐	
5.05	6					
4.4746	22.4	☐	☐			
4.4216	14.8					
4.2772	33.6					
3.5205	7.9	☐				☐
3.3568	100					☐☐
3.0748	6.2					
3.0407	20.7	☐				
2.6077	11					
2.5833	16.2				☐	
2.5661	14.5		☐			
2.4974	8.3		☐			
2.4611	12.6			☐		
2.2906	12.6		☐			
2.248	8.1			☐		
2.1435	6.2					
2.1348	7.9					
1.8218	16					
1.6764	8.8					
1.546	9.3			☐		
1.5027	6.2					☐
1.498	6.2					
1.4918	8.1					
1.3861	6.2				☐	☐
1.384	6.4					
1.3772	9.8					
1.3748	9.8				☐	

Table 14 Identification of minerals in Rolling Hill soil using peak d-spacing and Intensity values

d(A)	I%	Montmorillonite	Kaolinite	Illite	K-Feldspar	Quartz
4.5054	3.2	☐				☐
4.4613	3.3					
4.2732	6					
3.8614	10.2				☐	☐
3.3422	21.9	☐		☐		☐
3.0381	100					☐
2.8354	2.7		☐			
2.4968	14.1		☐			
2.4623	2.4				☐	
2.2829	22					
2.0942	21.1		☐			
1.9286	6.9				☐	
1.9102	19.3					
1.8731	19.8					
1.8178	3.2					
1.6242	4.8					
1.6023	9					
1.5418	3.7			☐		
1.5256	5.5					
1.5207	5					
1.5085	3.1					☐
1.4377	5				☐	
1.4194	3.2					
1.3731	3					

Table15 Identification of minerals in Keller soil using peak d-spacing and Intensity values

d(A)	I%	Montmorillonite	Kaolinite	Illite	K-Feldspar	Quartz
4.3352	31.6			☐		
4.0979	8.7					
3.9296	8.7		☐			
3.6396	9.5	☐				
3.387	100					☐
3.069	49.8	☐		☐		
2.5132	12.9					
2.4796	17.9		☐			
2.303	19.4					☐
2.2561	8.4		☐			
2.1417	17.1					☐
2.11	7.2				☐	
1.9912	16		☐	☐		
1.9216	9.1					
1.8855	14.4					
1.8315	16.7					
1.6794	11				☐	
1.6692	6.5					
1.611	7.6					
1.5497	16.3					
1.4592	6.5					
1.4282	6.8					
1.3877	9.5					☐
1.3773	16					

Table 16 Identification of minerals in Anthem soil using peak d-spacing and Intensity values

d(A)	I%	Montmorillonite	Kaolinite	Illite	K- Feldspar	Quartz
10.541	4.7			☐		
8.5038	2.9					
6.9607	2.7					
6.435	7				☐	
4.4911	5	☐			☐	
4.2728	20.4					
3.8791	4.2					☐
3.67	5.4					
3.3567	100					☐
3.2007	14.3			☐		
3.1651	3.5					☐
3.0383	33.4	☐				☐
2.834	2.6		☐			
2.4968	4.8		☐			
2.4601	5.8				☐	
2.2852	12.6					
2.2396	3.6					
2.13	4.7		☐			
2.0933	6.3					
1.9828	4.2					
1.9109	4.4				☐	
1.8753	5.8					
1.8204	10.7					
1.8058	2.4				☐	
1.6748	4.2					
1.6598	3.1					
1.6244	2.3					
1.6039	3.3					
1.5427	11.2					
1.5056	2.6			☐		☐
1.3844	4.1				☐	☐
1.373	8.1				☐	

Table 17 Identification of minerals in Oklahoma soil using peak d-spacing and Intensity values

d(A)	I%	Montmorillonite	Kaolinite	Illite	K-Feldspar	Quartz
4.6214	12.7	☐				☐
4.3692	19		☐	☐		
3.4077	100					☐
3.24	11.8				☐	
2.6068	14.5	☐			☐	
2.3043	8.6					☐
2.1506	9		☐	☐		
1.8349	10.4					
1.5521	10.4					
1.3822	14.5				☐	
1.3784	9.5			☐	☐	

Appendix B

Time Estimation Plots for Separation of Silt and Clay Fractions

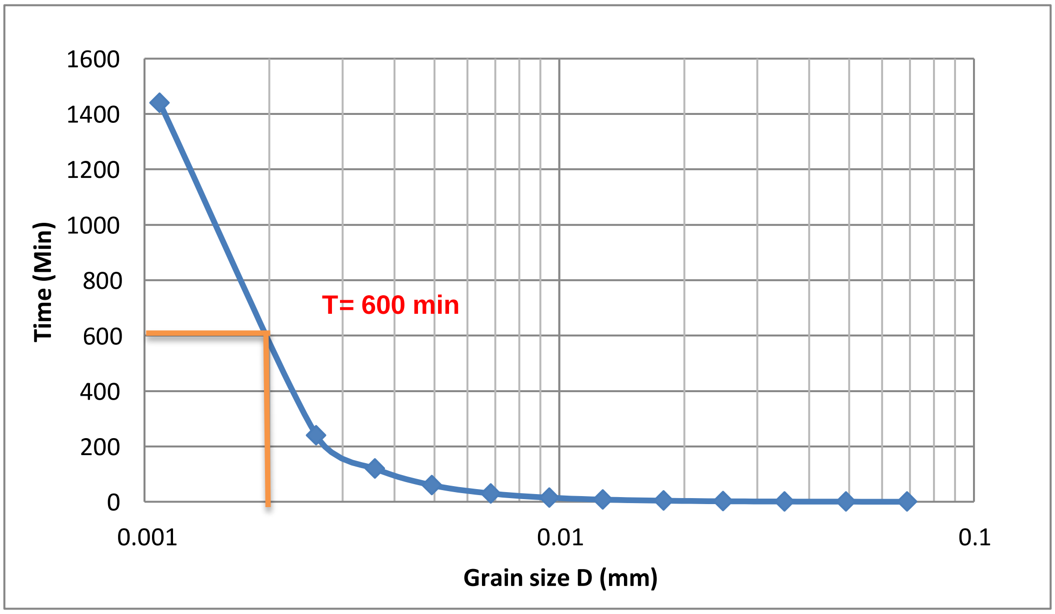


Figure 1 Time Estimation Plot for Dallas Soil

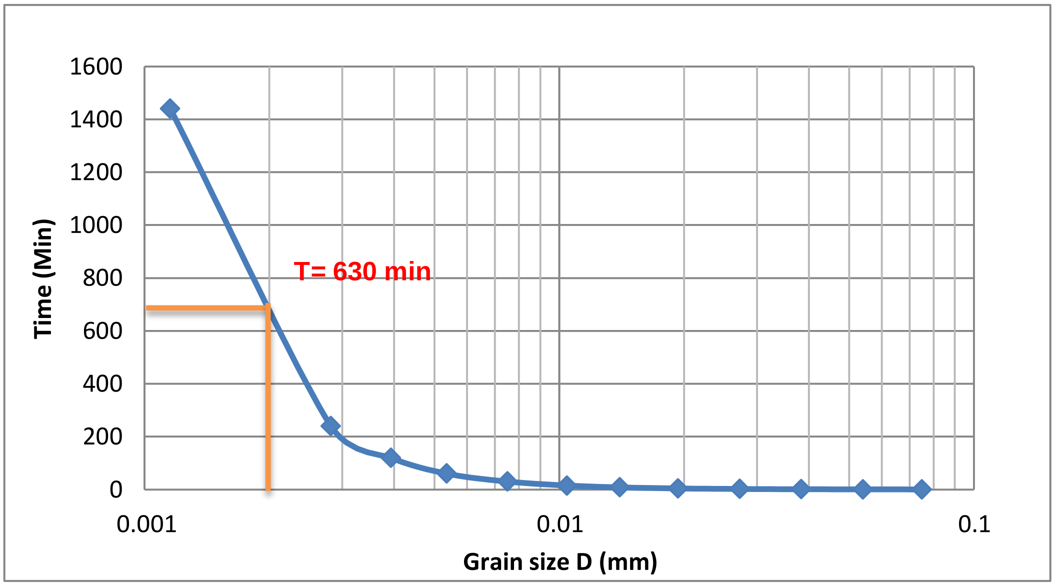


Figure 2 Time Estimation Plot for Sherman Soil

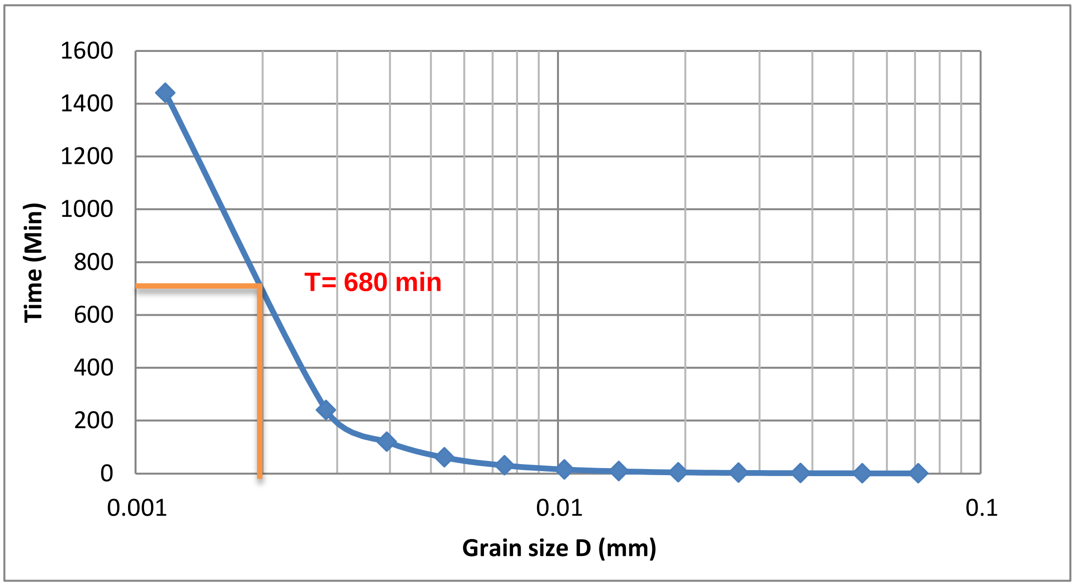


Figure 3 Time Estimation Plot for Burleson Soil

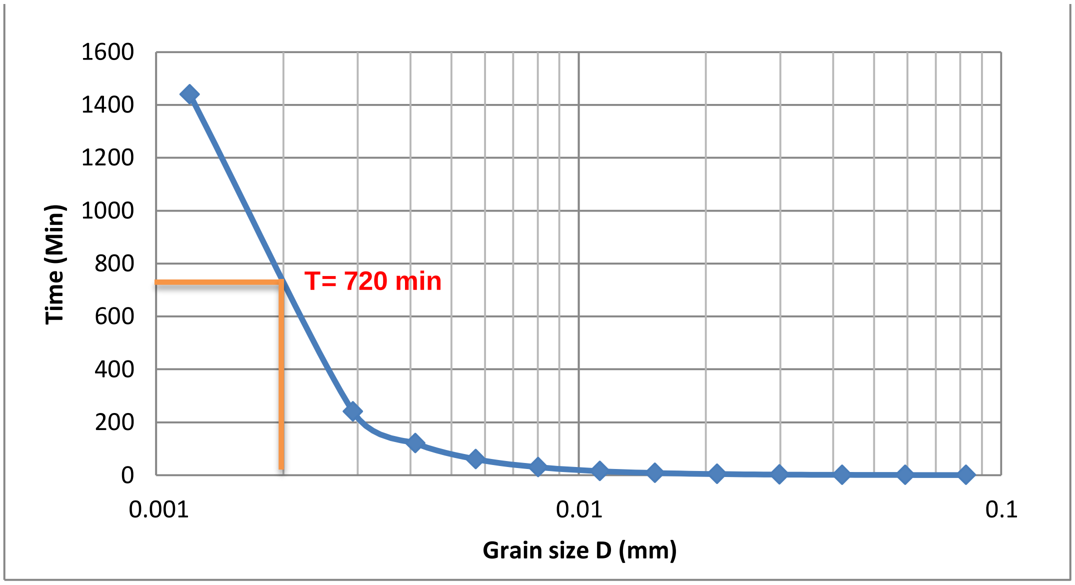


Figure 4 Time Estimation Plot for Wilcox Soil

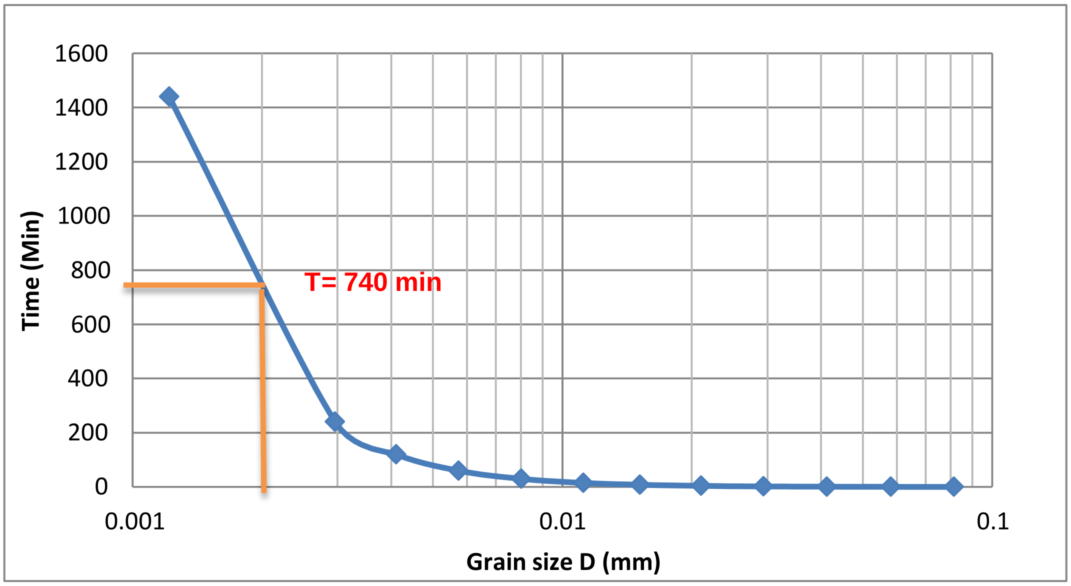


Figure 5 Time Estimation Plot for Riverside Soil

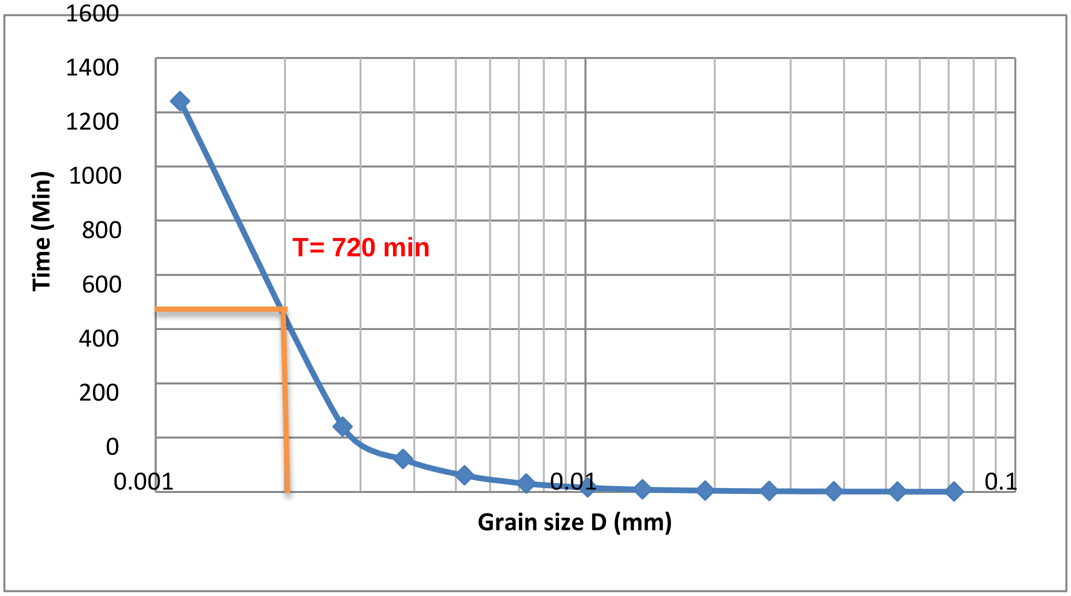


Figure 6 Time Estimation Plot for Eagle Ford Soil

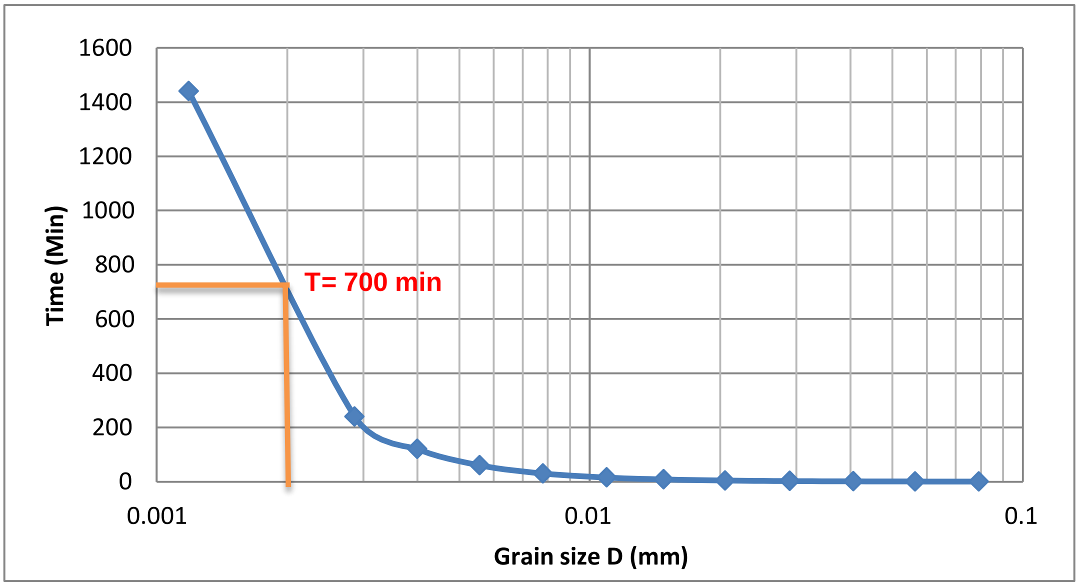


Figure 7 Time Estimation Plot for Jeopool Soil

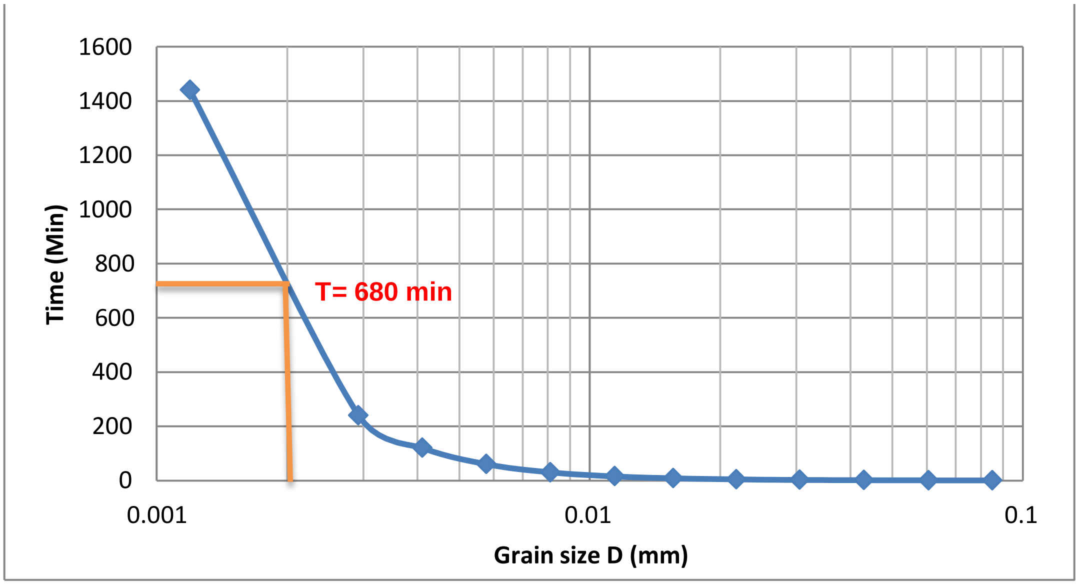


Figure 8 Time Estimation Plot for Woodbine Soil

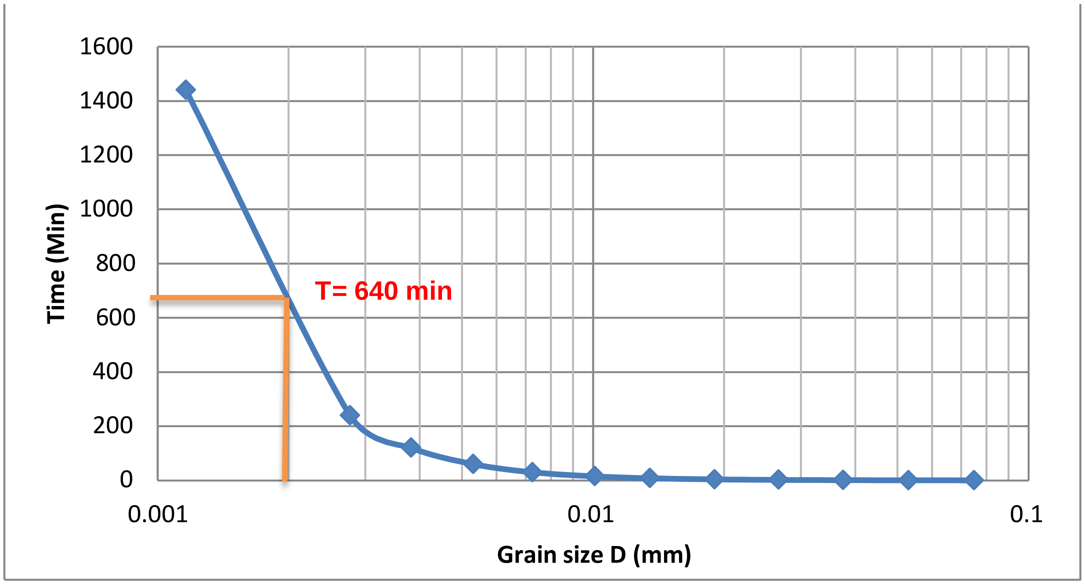


Figure 9 Time Estimation Plot for Stephen Ville Soil

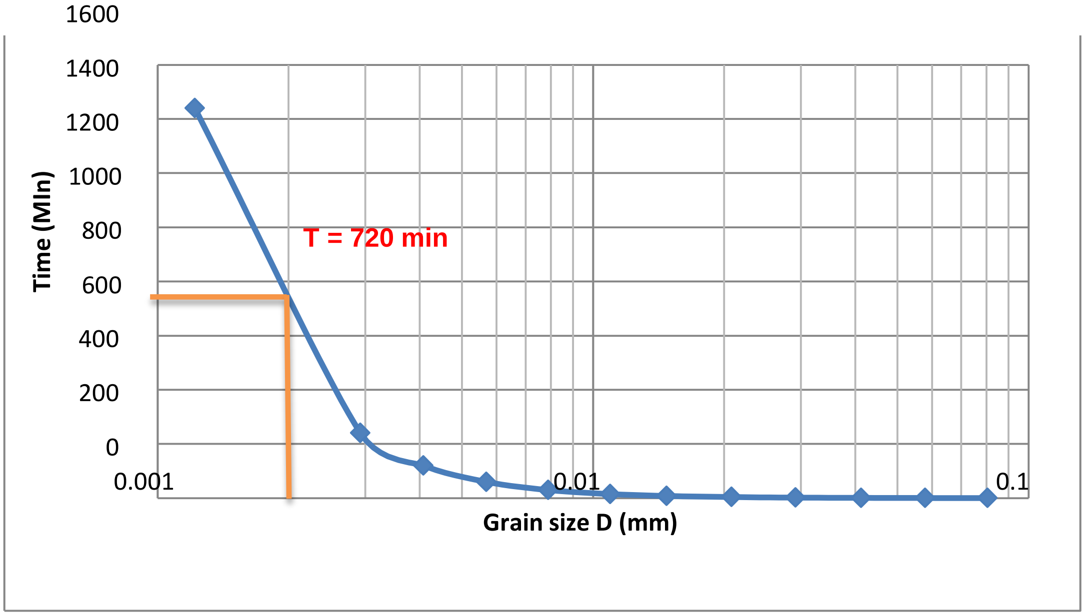


Figure 10 Time Estimation Plot for Colorado Soil

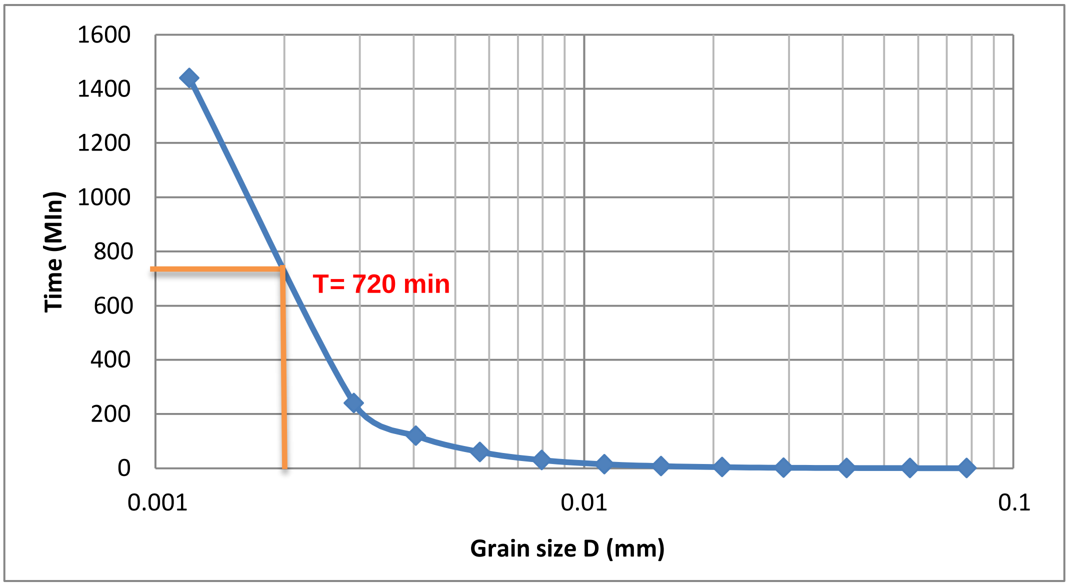


Figure 11 Time Estimation Plot for San Diego Soil

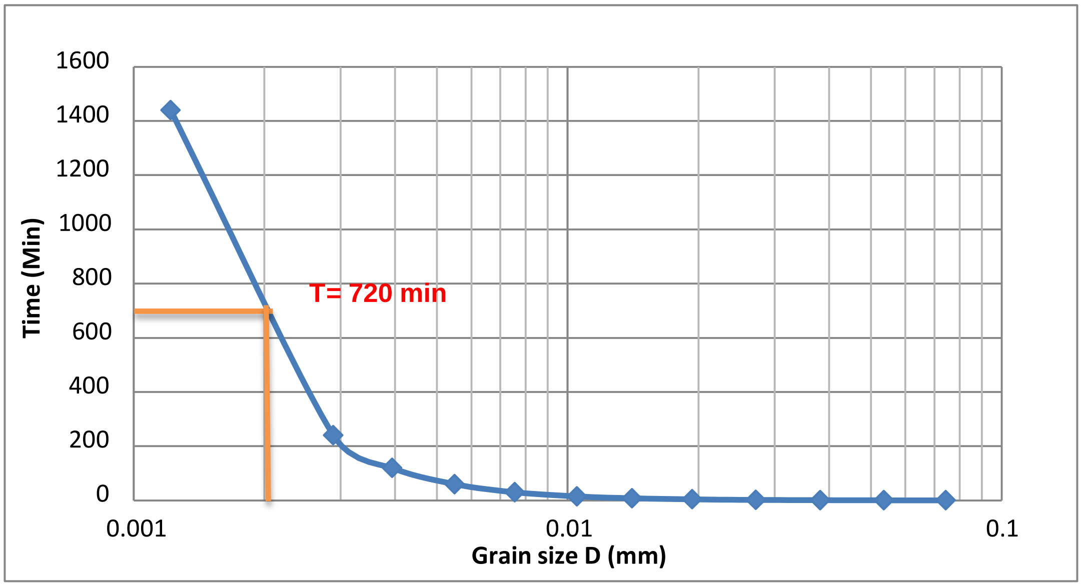


Figure 12 Time Estimation Plot for Austin Soil

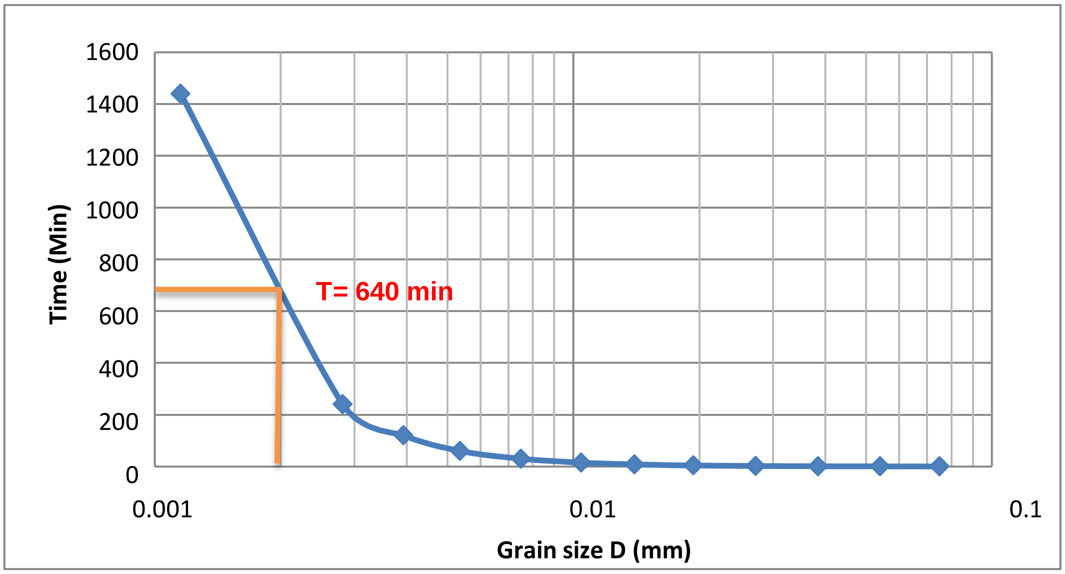


Figure 13 Time Estimation Plot for Grayson Soil

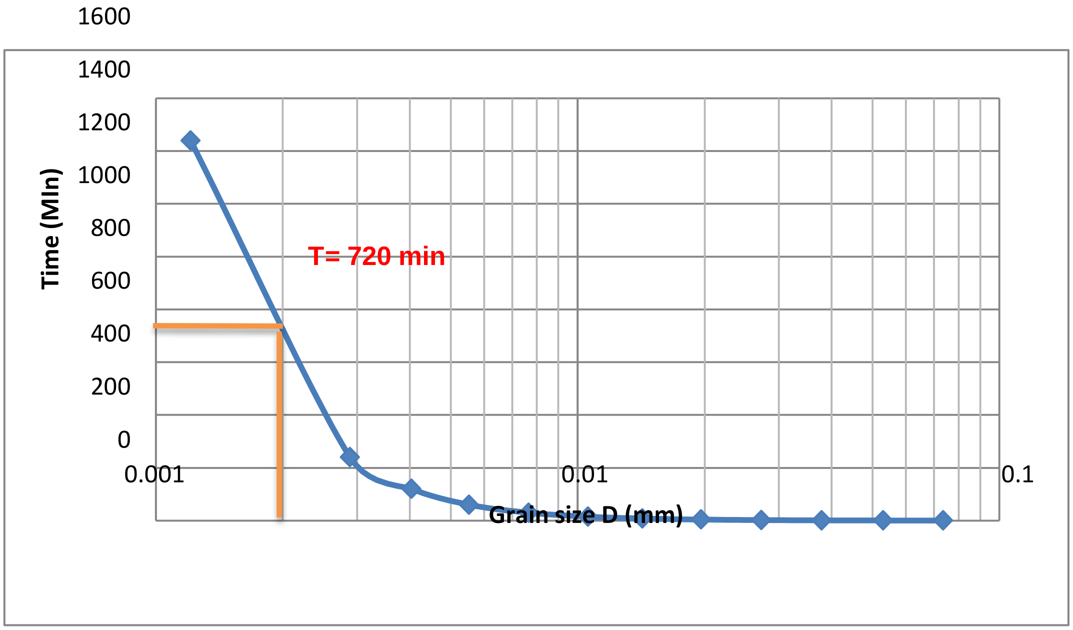


Figure 14 Time Estimation Plot for Rolling Hill Soil

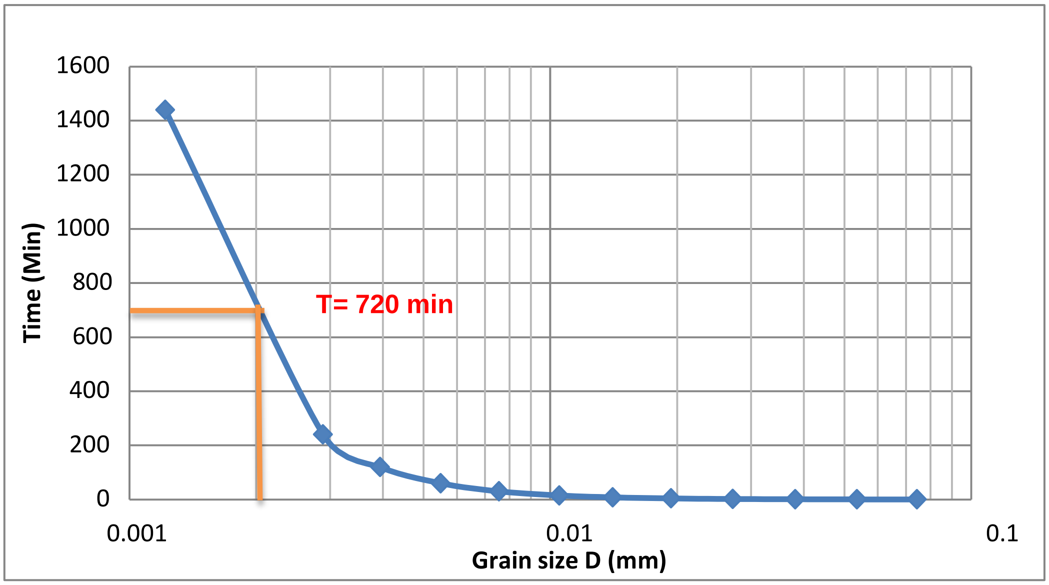


Figure 15 Time Estimation Plot for Keller Soil

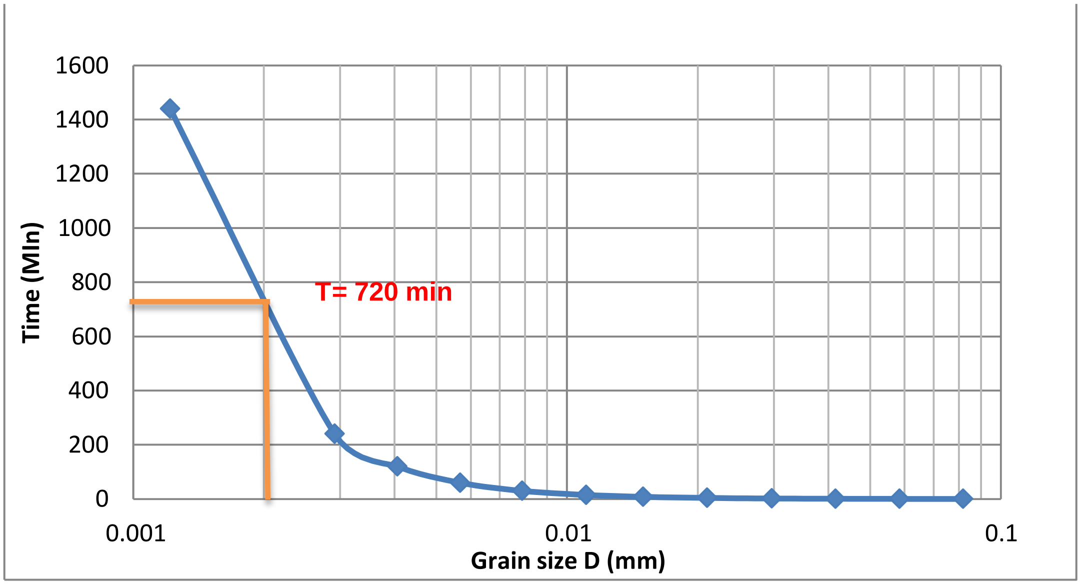


Figure 16 Time Estimation Plot for Anthem Soil

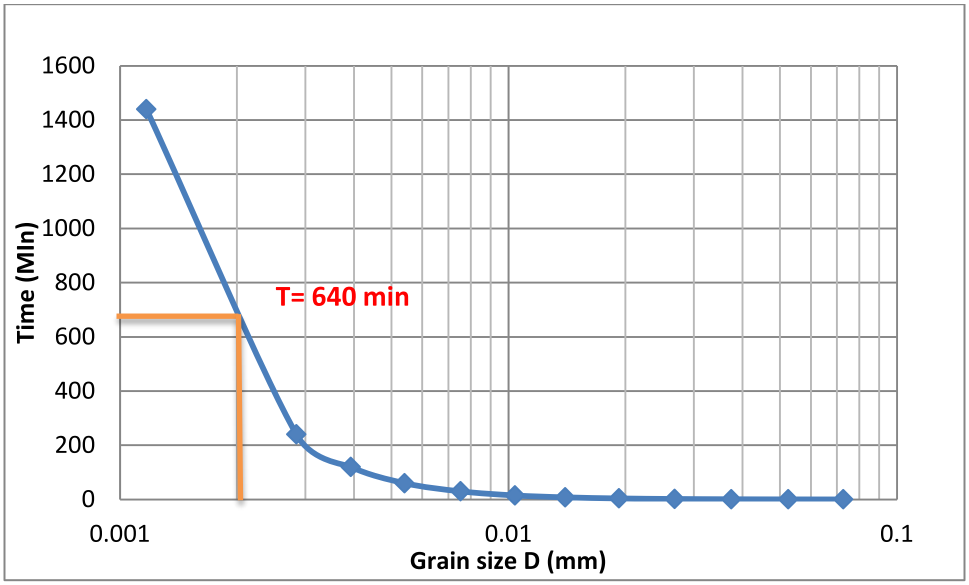


Figure 17 Time Estimation Plot for Oklahoma Soil

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Biographical Information

Spoorthi Reballi was born in Hyderabad, Andhra Pradesh, India on the 5th of January 1991. She received her bachelor's degree from the J.B Institute of Engineering and Technology which is affiliated to the Jawaharlal Nehru Technological University, India in April 2012. Author started her Masters in Civil engineering majoring Geotechnical engineering in fall (2012) from the University of Texas at Arlington. During the course of her study she worked as a graduate research assistant with Professor Dr. Anand J. Puppala. This has given the opportunity for the author to work in many lab tests like mineralogy testing, sulfate testing, Chlorides and organics testing and also worked on projects like mitigation of sulfate heaving, Integrated pipeline project and slope stability analysis. The authors present research is based on the quantification of clay and non-clay minerals present in soils.