INVESTIGATION OF EFFECT OF ZEOLITE ON STRENGTH AND MICROSTRUCTURE DEVELOPMENT OF CEMENT STABILIZED CLAY

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ABSTRACT

Results of investigation of the effect of zeolite on the strength and microstructure development of cement stabilized clay soil are presented. Clay soil, collected from Giri Village, Gwagwalada area of Federal Capital Territory (FCT), Nigeria was stabilized with 0, 2, 4 and 6% cement, and admixed with 0, 3, 6, 9, 12 and 15% zeolite. Index properties tests were conducted on the natural soil, while Unconfined Compressive Strength (UCS) test was carried out on the natural soil and the soil treated with cement and zeolite. X-ray Fluorescence (XRF) was conducted on the soil, cement and zeolite, while X-ray Diffraction (XRD) and Scanning Electron Microcopy (SEM) tests were conducted on the natural soil and the soil treated with cement and zeolite. Results of the index properties of the clay indicated it classified under A-7-6 and clay of high plasticity (CH) based on AASHTO and Unified Soil Classification Systems respectively. The UCS increased from 71 kN/m^2 for untreated soil to a maximum value of 1746 kN/m^2 for the soil treated with 4% cement and 6% zeolite respectively. The XRF test result revealed silica, alumina and iron as the major oxides of the clay, while cement has quick-lime and silica, and zeolite composed of silica, alumina and sodium. XRD result showed that the clay composed of quartz, ankerite, orthoclase, montmorillonite and silicide minerals. Addition of 6% cement disintegrated the montmorillonite minerals to less active kaolinite mineral with introduction of calcite mineral. Addition of 4% cement and 6% zeolite further converted the montmorillonite mineral to kaolinite minerals with introduction of calcite and zeolite. The SEM result of the clay-cement-zeolite composite revealed a smooth, compact structure without pores, justifying the highest UCS value recorded at this mixture. 6% zeolite was therefore, observed to be the optimal zeolite for each of the cement contents considered in this study. The study also established that clay of high plasticity can be treated with 6% zeolite (and less cement content), to achieve the 1710 or 1750 kN/m^2, reported in the literature, as criteria for design cement content, considered adequate for soil-cement composite as base course material for highway pavements with light to medium traffic.

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ABBREVIATIONS, GLOSSARIES AND SYMBOLS

AASHTO	American Association for State Highway and Transportation Officials
ASTM	American Society for Testing and Materials
BS	British Standard
BSL	British Standard Light
CH	Clay of High Plasticity
CL	Clayey soil
Gs	Specific gravity
LL	Liquid Limit
M	Mass
m	Metre
MDD	Maximum Dry Density
M_s	Mass of compacted soil
OMC	Optimum Moisture Content
OPC	Ordinary Portland Cement
TBC	Tropical Black Clay
TRRL	Transport and Road Research Laboratory
PI	Plasticity Index
PL	Plastic Limit
UCS	Unconfined Compressive Strength
USCS	Unified Soil Classification System

Bulk density

Dry density

Natural moisture content

CHAPTER ONE

1.0 INTRODUCTION

1.1 Background to the Study

In construction of civil engineering structures, different types of soils are encountered. Some deposits of these soils, in their natural form, possess suitable engineering properties for construction purposes, while some others are unsuitable without treatment or modifications. In the latter case, such type of soils includes clay, some of which are described as a problematic. These types of soils need to be excavated and replaced, or their properties modified before they can effectively be used to sustain applied loads. Typical of problematic soils are the expansive soils, which are frequently observed due to their existence worldwide, except the arctic regions (Steinberg, 2000). These types of soils have caused significant amount of damages in road infrastructure, especially in the North Eastern part of Nigeria, where the largest deposit of a typical of these soils (the black cotton soil) is situated. This occurs due to its high susceptibility to volume changes as a result of changes in moisture content (Vera *et al*., 2019). The inherent volume change characteristics of expansive soils such as the Black Cotton Soils (BCS) are mainly as a result of their clay mineral content which is mostly montmorillonite. Expansive soils can be easily identified from their high plasticity, excessive heave, and high swell-shrink potential. Due to cost implication of total excavation of such soils, geotechnical engineers often prefer modifying the properties in-situ using different stabilization techniques.

Stabilization has been widely used to enhance engineering properties of soils such as strength, compressibility, hydraulic conductivity and density. Additives have been used to modify this soil through waterproofing, bonding or a combination of both (Chittoori, 2008). Puppala and Chittoori (2013) reported that these additives are divided into three main categories: traditional, non-traditional and by-product stabilizers. The traditional stabilizers include cement, lime bituminous materials and fly ash; non-traditional stabilizers include polymers, enzymes, ammonium chloride, while the by-product stabilizers comprise of cement kiln dust, iron ore tailings, bagasse ash and so on. Amongst these three categories, the traditional stabilizers still remain the most widely used for stabilization (NCHRP 11, 2009). For most highway construction, there is the need to treat the base, sub-base and subgrade materials to improve workability during compaction, to increase strength and stiffness of the foundation layers and to reduce shrink-swell characteristic due to moisture changes (Rauch *et al*., 2002).

The wetting and drying process of sub-grade layer composed of clay soils results to failure of pavements in the form of settlement and cracking. Therefore, prior to construction of roads on such subgrade, it is necessary to remove the existing soil and replace it with non-expansive soil or to improve its engineering properties by stabilization. To replace the existing soil is not a feasible option, so the best available approach is to stabilize the soil with suitable stabilizers (Onyelowe *et al*., 2012). Various stabilizers such as cement, lime and locally available materials like zeolite have been used for stabilization of expansive soils (Andreas, 2011). The selection of a particular type of stabilizer depends on the type of the sub-grade soil and availability of the stabilizers (Gregory, 2012). Several researchers have reported the benefits of stabilizers for modifying the engineering properties of soils. As the quality of a soil layer is increased, the ability of that layer to distribute load over a greater area is generally increased so that a reduction in the required thickness of the soil and surface layers may be permitted (Nazile, 2018). The most common improvements achieved through stabilization include better soil gradation, reduction of plasticity index or swelling potential, and increases in durability and strength. In wet weather, stabilization may also be used to provide a working platform for construction operations (Kennedy, 2012). These types of soil quality improvement are referred to as soil modification or soil stabilization.

Zeolite is a microporous, aluminosilicate minerals commonly used as commercial adsorbents and catalysts (Ceri, 2017). Zeolite occurs naturally but is also produced industrially on a large scale. In order to reduce the environmental impact caused by Portland cement in binders for soil stabilization, Zeolite is chosen to replace some percentages of Portland cement. A number of studies have been carried out to assess the mechanical behavior and compressive strength increase of cemented sands using added fiber, glass, fly ash, silica fume and nanoparticle (Consoli *et al*., 2007, Arabani & Azarhoosh., 2012; Choobbasti *et al*., 2015). However, there has been little effort on the use of natural zeolite documented in the literature.

Due to the urgent need to reduce global warming and preservation of the ozone layer which are of global concern, and the increasing scarcity of natural resources, over dependence on expensive construction materials like ordinary Portland Cement in many countries, researchers have over the years focused on the use of appropriate technology that encourages greenhouse technology and reducing carbon emissions using natural and available resources like zeolite for the stabilization of problematic soils. In contrast to using traditional quarried materials, this approach will ultimately lower carbon emission and other pollutants. This will lead to a more sustainable environment.

1.2 Statement of the Research Problem

Soil stabilization has proven to be economical as it provides affordable construction materials for subgrade, sub-base and base for road construction. Deficient soils improvement using Portland cement is one of such methods, which has proven to be effective especially in the case of sandy soil due to the ease of pulverization. The primary reaction of cement is with the water in the soil, which leads to formation of cementitious materials. Cement stabilized road bases, provide good support to the overlaying pavement layer thereby reducing stresses on subgrade soil. However, the enormous carbon emission and cost of using cement for improvement of deficient soils has led to the search for alternative low cost, naturally available, sustainable and low-carbon stabilization techniques or materials. Zeolite is one of such materials. Zeolites is an aluminosilicate member of the family of microporous solids known as "molecular sieves", consisting of Si, Al, O, and metals including Ti, Sn, Zn. This acts as a pozzolan to Cement clay-based mixture, which affects the mechanical properties of the problematic soils. Most research works focused on mechanical behavior that is mainly controlled by microstructure of the composite mix. The micro-structure is fabric i.e the arrangement of the particles, clusters and pore spaces in the soil as well as cementation (Mitchell, 1993). It is therefore important to understand the changes in engineering properties that result from the changes in the influential factors (water content, cement content, zeolite content, curing time, and compaction energy) on the engineering characteristics of cement, zeolite stabilized soils. This research work will attempt to investigate the microstructural changes in cement-zeolite stabilized clay to explain the different strength development according to the influential factors aforementioned.

1.3 Aim and Objectives of the study

The aim of this research was to investigate the microstructure of the cement-zeolite stabilized clay. This was achieved through the following set of objectives;

1. Determination of index properties of the natural clay soil.
2. Determination of the moisture–density relationship of the natural clay soil using British Standard light compaction effort.
3. Determination of the strength characteristic of the cement-zeolite stabilized clay.
4. Assessment of the microstructure of the optimal zeolite-cement stabilized clay.

1.4 Justification of the Study

Expansive soil is one of the problematic soil groups encountered on all continents except Polar Regions (Richard *et al.*, 2017). Problems caused by their heaving and shrinking behavior, particularly to light structures, have been reported from different countries. These have place large financial burden on developers. For this reason, many techniques have been developed and applied to prevent and/or remediate the damages caused by these soils. Soil stabilization using traditional chemical additives, especially the ordinary Portland cement, has been applied successfully since ancient times (Ali *et al.*, 2017). But global cement production has resulted to emission of CO_2 with intensity of 0.8 tone-CO_2/t-cement, which is 5% of total anthropogenic CO_2 emissions (Ernst *et al.*, 2001). These emissions are split roughly equally between CO_2 emitted from the calcination process (B52%) and emissions from combustion of fuel to fire cement kilns (B48%). This shows that the use of cement is not environmentally sustainable, therefore there is the need to minimized or replace its use with other natural/abundant resources like zeolite, which is sustainable and encourage greenhouse technology which reduce the overdependence on conventional construction materials.

1.5 Scope of the Study

The study focused on micro-structure study of cement- zeolite stabilized clay soil. The scope of the study includes: Sourcing and preparation of samples of the clay soil and testing for moisture content, Atterberg limits, sieve analysis and moisture-density relationship (Compaction). Unconfined Compressive Strength (UCS) on the cement-zeolite stabilized clay soil, at 0,3,6,9,12 and 15 percentages of Zeolite and ordinary Portland cement and at 1,7,14,28 and 60 days curing period, was also carried out. Investigation of the microstructures of both the constituent materials (clay soil, Cement and zeolite) and the cement-zeolite stabilized soil was also carried out.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Preamble

Problems related to construction sites for which soil properties are unable to meet the basic required project specifications are encountered by Geotechnical Engineers on almost daily basis. Consequently, many methods and techniques have been researched, developed and applied to improve soils in-situ in order to meet the basic specifications for the intended purpose. This literature review focuses on the problems caused by expansive soils, various common techniques applied for their improvement/stabilization and some emerging techniques of using zeolite for treatment of problematic soils. This section also reviews identification of these soils, factors influencing swelling and shrinking potentials, as well as the associated problems. As indicated by the appellation, expansive soils are soils that are prone to large volume changes that are directly related to changes in water content, which leads to heaving and settlement problems. Researchers have come up with definitions of expansive soils with a common factor of significant volume change due to existing conditions. Snethen *et al*. (1975) defined expansive soils as natural earth materials which, because of their inherent and environmental characteristics, undergo volume change due to changes in environmental conditions. According to Steinberg (2000), expansive soils are soils which are characterized by volume change due to moisture content variations. This section reviews the identification of those soils, factors influencing the swelling and shrinking potential as well as the associated problems. The ancient tools that allows designers to deal with in-situ soils that do not satisfy the basic design requirements and specification are the ground improvement techniques. Even though there are various techniques that have been applied to modify expansive soils, there is need to introduce new techniques and materials.

A large volume of literature has accumulated over the last two decades on surface modification and stabilization of clay soils. Through ion–exchange reactions with cationic surfactants, clay minerals can be modified to have hydrophobic surfaces by lowering the surface of energy. These clays pose very specific problems to pavement engineering field as their response to changes in moisture content and the platelet structure of the material frequently causes damages and defects, such as transversal and longitudinal cracks, or other faults (James, 2016). However, most soils used in construction are sufficiently stiff in the dry state but loose this stiffness when saturated with water, resulting to reduction in strength and bearing capacity with a corresponding increase in compressibility which may lead to excessive settlement and subsequent failure of structures on them (Moayed & Allahyari, 2012).

Clay minerals are a group of hydrous aluminosilicates that are characteristically found in most tropical soils. They range in dimensions less than 2µm (colloidal dimensions), hence they are prone to swelling when moist, shrinking on drying and flocculating in water suspension (Gidigasu, 1976). They are phyllosilicates which comprise of layers of tetrahedral coordinated silicon atoms fused to an edge-shared octahedral sheet of Alumina or Magnesia. Compared to other rock forming minerals, clay minerals possess a relatively large surface area arising from their micro particle size and due to isomorphous substitutions. The surfaces of clay particle possess a net negative charge. This results in sorption of cations on the surface and attraction of water molecules. In the presence of water, the surface charges result in formation of a strongly bonded layer of water molecule leading to the formation of a double diffuse layer of water around the clay particle (Van-Olphen, 1963).

2.2 Soil

Soil is an essential and most abundant component of the earth crust. Almost all human activities are directly or indirectly related to land. Soil is very essential to humans in diverse ways which includes; natural water filtration system, absorbing minerals, constructing buildings and roads, and providing food through agriculture (Soil Functions, 2020).

Soil is regarded as a mixture of materials such as organic compounds, water particles, very small rocks, microorganisms and decomposed dead matter. The combination of all of these makes soil. Soil serves as covering of the top layer of the earth. It is normally brown in color and provides base for plantation and also foundation for different civil engineering structures (Surendra *et al*., 2017). Soil has a porous structure and can allow water to pass freely through it. This passage due to porosity is the reason plants' roots get their water from soil (Soil and water Relationship,2001). Soil can be consolidated as well removing most air pockets. Soil varies depending on sizes of the constituent primary rock minerals in them, such as gravel, sand, silt and clay. While gravel and sand have relatively larger particle sizes, silt and clays have the finest particles (Yolcubal *et al*., 2004). Soil provides bases for foundations of most civil engineering construction on earth, therefore understanding the properties of soil is very important. Soil properties are usually influenced by its constituent primary rock minerals (gravel, sand, silt and clay) (Surendra *et al*., 2017). Properties of soil, whose primary rock minerals are predominantly of gravel and sand, depend mostly on gradation and density, while soils consisting predominantly of silt and clay have their properties depending on their interaction with water. In the latter case, especially in the case of clay soils, their mineralogical composition plays a significant role (Winterkorn *et al*., 1991).

2.2.1 Clay mineral

Many classification systems described clay minerals as particles which have effective diameter of two microns or less (Chen, 1975). However, it has been found that all particles whose diameter is less than two microns are not necessarily clay minerals (Sarsby, 2000). For this, the grain size does not suffice to describe clay mineral, hence the necessity to study the mineralogical composition, which is probably the essential property of fine-grained soils (Chen, 1975). Das (2010) defined clay minerals as aluminosilicate compounds, resulting from the combination of two basic units, namely silica tetrahedron and aluminium octahedron. Each tetrahedral unit is formed by a silicon atom surrounded by four oxygen atoms and combination of identical units results in the formation of a silica sheet in which neighboring tetrahedra shares three oxygen atoms at the base of each tetrahedron (Das, 2010). On the other hand, an octahedron consists of an aluminum atom surrounded by six hydroxyls. The combination of octahedral units leads to the formation of octahedral sheet also called gibbsite sheet. When the aluminium atom is replaced by magnesium, the octahedral sheet is called brucite sheet (Das, 2010). According to Chen (1975), Sarsby (2000) and Das (2010), there exist a number of clay minerals among which the important ones are kaolinite, illite and montmorillonite. All of them consist of a combination of silica-gibbsite sheets and are characterized by high specific surface (the surface area per unit mass). According to Snethen *et al*. (1975), the small grain size and resulting large surface are due to the clay minerals' origin by either weathering or diagenetic alteration of pre-existing minerals. Das (2010) describes the three important clay minerals as follows:

i. Kaolinite is composed of a combination of repeating layers of elemental silica gibbsite sheets held together by hydrogen bonding in 1:1 lattice. The estimated specific surface of kaolinite is 15m^2/g.
ii. Illite is composed of a gibbsite sheet sandwiched between two silica sheets bonded together by potassium ions which are balanced by the negative charge left by the substitution of aluminum for some silicon in the tetrahedral sheets. This phenomenon consisting in substitution of an element by another one without alteration of crystalline form is called isomorphous substitution. The specific surface of illite particles is estimated to 80m^2/g.
iii. Montmorillonite has the same structure as illite with attracted water molecules between layers instead of potassium ions. The specific surface is estimated to 800m^2/g.

In addition to these important clay minerals, other common once are chlorite, halloysite, vermiculite and attapulgite (Das, 2010).

Hunt (2007), described clay minerals as hydrous aluminium silicates that are categorized into different groups based on their chemistry and crystal structure. Based on their stability, Hunt (2007) divided clays into two main categories: common groups comprised of kaolinite, halloysite, illite, and montmorillonite, and less common categories includes vermiculite and chlorite which is easily transformed to other types.

2.2.1.1 Characteristics of a typical clay mineral

Clay particles have some typical characteristics which include cohesion, adhesion, plasticity, consistency and activity. Hunt (2007) described these properties as follows:

i. Cohesion of clay is defined as its ability to stick to itself. It results from a bond, developing at the contact surfaces of clay particles, caused by electrochemical attraction forces. Cohesion of clay particles is as a result of two important elements namely high specific surface area (surface area per unit weight), and electrical charge on the basic silicate structure, resulting from ionic substitutions in crystal structure of the clay particle.

ii. Adhesion of clay is its ability to stick to another material to which it comes into contact. The value of adhesion (a) is usually considered as a fraction of soil cohesion c.

iii. Plasticity is the ability of a material to undergo a change in shape without undergoing change in volume, as its moisture content remains constant.

iv. Consistency is referred to as the relative ease with which a soil can be deformed. It involves cohesion and adhesion as well as its ability to resist deformation and rupture. In other words, soil consistency refers to the description of the resistance of a soil at various moisture contents to mechanical stresses or manipulations. The consistency of soils is generally illustrated at three soil moisture levels: wet, moist and dry. With decreasing moisture contents, clays pass from fluid state (very soft) through a plastic state (firm), to a semisolid state, and finally to a hard brick-like state. The moisture contents at the transitions between these various states are defined by the Atterberg limits, which vary with clay type and its purity.

v. Activity is defined as the ratio of the plasticity index to the percent by weight finer than 2μm. Some important clay minerals characteristics are summarized by Hunt (2007) is presented on Table 2.1.

Depending on the clay mineral content, soils can be classified as competent, deficient or problematic.

Table 2.1: Common Characteristics of clay minerals

Mineral	Origin	Activity	Particles
Kaolinite	Chemical weathering of feldspars Final decomposition of micas and pyroxenes in humid climates or well drained conditions Main constituents of clay soils in humid-temperate and humid tropical regions	Low. Relatively stable material in the presence of waters	Platy but lumpy
Halloysite	Similar to kaolinite, but from feldspars and mica (primarily sialic rocks)	Low, except properties are radically altered by intense drying. Process not reversible	Elongated rod like units, or hollow cylinders
Illite	Main constituent of many clay shales, often with montmorillonite	Intermediate between kaolinite and montmorillonite	These plates
Montmorillonite (smectite)	• Chemical decomposition of olivine (mafic rocks) • Partial decomposition of micas and pyroxene in low rainfall or poor drainage environment • Constituent of marine and clay shales • Alteration of rock during shearing by faulting • Volcanic dust	• Highly expansive and the most troublesome of the clay minerals in slopes and beneath foundations • Used as an impermeabilizing agent	Under electron microscope, appears as a mass of finely chopped lettuce leaves

Source: Hunt, 2007

2.2.2 Deficient or problematic soils

Deficient or problematic soils are those that pose problem when constructing civil engineering structures on them. These types of soils include expansive soils, collapsible soils, and sanitary landfill (Richard & Sabry, 2017). Problematic soils are generally difficult to deal with during and after construction on them (Manish, 2016). These soils exhibit certain characteristics (excessive movement) which are undesirable to any foundation. Deficient soils such as expansive and collapsible soils can cause high differential settlement, whose consequences are dire to structures. Almost same issues arise for foundations on sanitary landfill. Such soils must be identified in the field before construction is planned. Nevertheless, not all problems related to deficient soils can be solved, therefore, preventive measures can be taken to ameliorate the extent of damage to structures built on them.

Collapse susceptible soils also known as metastable soils are defined as soils that are stable under load and when they undergo large decrease in bulk volume virtually instantly, when in dry condition and become wet to near saturation under the same load (Sandra, et al., 2001). Problems caused by these soils are encountered worldwide. According to studies by Steinberg (2000), introduction of 'soil mechanics' by Karl Terzaghi, paved the way for great efforts, at the beginning of twentieth century, at understanding deficient soils,. The first studies focused on the destructive behaviour of expansive materials and their impact on highways as well as the efforts needed to control the impact. These studies revealed that a lot of damages that were initially attributed to structural design shortcomings or faulty construction were actually caused by problematic soils (Steinberg, 2000).

Problematic soils (especially expansive soil) cause damage because of their volume change due to water content fluctuation (Manish, 2016). When soil absorbs water, it swells creating swelling pressure, (defined as the maximum force per unit area that needs to be placed over a swelling soil to prevent its volume increase) (Sabat, 2012). Swelling pressure is the most important indicator of the damaging potential of a swelling soil (Sabat, 2012). If the swelling pressure of a soil is very high the damaging potential of the soil will also be very high and vice versa. The most affected civil engineering structures are the light types, which include low-rise buildings, retaining structures, channel and reservoirs linings, utility lines, railways and roads (Kayabali and Demir, 2011). These volume changes result to settlement problem. The first indicator of settlement problem, are cracks in building walls and pavements as observed by (Williams *et al*., 1985). However, not all cracks in wall buildings result from swelling soils. Chen (1975) found that, the vertical and horizontal cracks in basement walls are mainly due to earth pressure on walls and sometimes caused by earth moving equipment such as backhoe. It was also discovered that, cracks caused by swelling movement are most of the time diagonally located below windows and above doors Chen (1975).

Based on the study carried out by Williams *et al*. (1985), the difficulties arise, not because of a lack of adequate engineering solutions but largely because of failure to recognize, during the early stages of a project the existence of expansive soils. The most critical challenge is that, it is possible to determine how much soil will swell through laboratory testing but not how much the soil volume will change beneath a particular structure (Holtz and Gibbs, 1956). This situation becomes more complicated when the climatic conditions are taken into consideration for swelling and shrinking prediction.

The damages caused by the settlement of deficient soils have been reported from different countries around the world. Jones and Holtz (1973) estimated damages caused by expansive soil in USA to stand at $2,255 million per year. The affected structures and corresponding average annual loss are shown on the Table 2.2.

Table 2.2: Estimated damage attributed to expansive soils

Construction category	Estimated average annual loss (millions of dollars)
Single-family homes	300
Commercial buildings	360
Multi-story buildings	80
Walks, drives, parking areas	110
Highway and streets	1,140
Underground utilities and service	100
Airports	40
Urban landslides	25
Others	100
TOTAL ($)	2,255

Source: Jones and Holtz (1973)

Jones and Holtz (1973) stated that the cost of problems caused by expansive soils in United States is evaluated to be more than $2 billion each year. It was also stated that the cost associated to damage caused by expansive soil is twice the cost of damages caused by flood or landslides and, more than twenty times the cost of damages caused by earthquake. Williams *et al*. (1985) observed that damages caused by problematic soil to buildings and light structures, in USA and UK, are estimated at $1,000 million and $100 million, respectively. In South Africa, a study carried out by (Williams *et al*., 1985) estimated the cost of repair of houses built or to be built on swelling clay, over a period of 20 years, to R1, 000 million. In China, the damage to railroad has been estimated at 12million dollars per year. There are not documented costs associated with damages caused by expansive soil in Nigeria.

2.2.3 Treatment of expansive soils

Often times, some in-situ soils do not meet construction requirements. Many solutions to this problem are available and the choice depends on various parameters such as type of structure, cost, etc. Some of those solutions are (Gregory *et al*., 2003).

i. Relocation of the facility,
ii. Replacement of the challenging in-situ soil,
iii. Special design to overcome the suspected problems and
iv. Soil treatment, etc.

The first solution can only be applied to only some projects, requiring relatively small space, and availability of another site. For some other project (linear facilities such as highways, pipelines, tunnels; and big projects like airports, harbour), requiring large spaces, this alternative becomes impracticable. For this reason, and coupled with the ever-increasing demand for land, this solution cannot be considered adequate. The second solution may be found impracticable or uneconomical depending on the extent of the constraint and the project (Richard *et al.,* 2017). The third and fourth solutions are mostly considered in practice.

Due to global distribution of expansive soils and the increasing losses they cause, many technical methods have been developed for their stabilization before and after construction of structures on them (Nelson and Miller, 1992; Chen, 1975). Preliminary investigations and evaluation of the soils' properties must be carried out to identify the extent of the constraint and the probable factors which may affect the shrink-swell of such soils, and other parameters needed to determine the appropriate soil treatment method (Charles, 2008). These parameters are: the project nature and size; cost comparison of different alternative treatment methods (Nelson and Miller, 1992). The treatment methods, required for the treatment of problematic soils to support pavements may differ from those required for buildings. Several methods have been applied successfully to treat deficient soils (Thomas, 2002). These methods include soil replacement, surcharge loading, pre-wetting, moisture fluctuation control and chemical treatment (Nelson and Miller, 1992). Chen (1975) stated that compaction is another common method used to minimize swelling potential of expansive soils.

2.2.4 Removal and replacement by suitable soil

Replacement of problematic soils with non-problematic or non-swelling soils is one of the techniques applied to adequately control the constraints caused by expansive and shrinking behavior of the former (Chijoke and Chimobi, 2019). Depending on the thickness of soillayer and the extent of the project, the deficient strata may be entirely or partially removed. However, in some cases, the problem soil layer may be too deep to economically allow complete removal and replacement (Nelson and Miller, 1992). In this case improving or stabilizing the existing soil is considered.

2.3 Soil Stabilization

Soil stabilization refers to any physical, chemical, mechanical, biological or combined technique of transforming a natural (mostly deficient) soil to meet basic specification and requirement for engineering purposes (Winterkorn *et al.*, 1991). Improvement gains during stabilization include increased bearing capacity, tensile strength, and overall performance of the in-situ soil (Vega *et al.,* 2018). Bitumen emulsion, which is used as a binding agent for road construction, is traditionally and generally accepted as a technique of soil stabilization. However, bitumen is not environmentally friendly and becomes brittle when dried. Portland cement has also been long used for soil stabilization. However, this is often expensive and its production is not environmentally friendly. Cement fly ash, lime fly ash (separately, or with cement or lime), bitumen, tar, Cement Kiln Dust (CKD), tree resin and ionic stabilizers are all commonly used stabilizing agents. Other stabilization techniques include using in-situ materials such as sub-soils, sands, mining waste, natural stone industry waste (Gutiérrez *et al.*, 2019) and crushed construction waste.

Recently, there are new innovations and renewable technologies used in stabilization which includes enzymes, surfactants, biopolymers, synthetic polymers, co-polymer based products, cross-linking styrene acrylic polymers, tree resins, ionic stabilizers, fiber reinforcement, calcium chloride, calcite, sodium chloride, magnesium chloride, zeolite and more . Some of these new stabilizing techniques create hydrophobic surfaces and mass that prevent road failure through prevention of water from penetrating or heavy frosts. Soil can also be stabilized mechanically with geosynthetics, for example, geogrids or geocells, and a 3D mechanical soil stabilization technique (Thirumalai *et al.,* 2016). Stabilization is achieved via confinement of particle movement to improve the strength of the entire layer (Gregory *et al*., 2012).

Generally, all types of soil stabilization can be classified into two groups: mechanical and chemical stabilization. In mechanical stabilization, grading of a soil is changed by mixing it with other types of soils of different grading (Anjan, 2019). Through this, a compacted soil mass can be achieved. On the other hand, chemical stabilization is associated with modification of soil properties by the addition of chemically active materials. In soil stabilization, it is very important to understand the material properties involved in the mixture and the outcome after mixing (Gregory, 2012). At the same time the effects of the process on the nearby structures and surrounding need to be evaluated. Accordingly, decisions can be taken on selection of materials and the corresponding doses. In addition to the selection of materials and the doses there are many other factors governing effectiveness of this method, for example, mixing and spreading, selection of roller, compaction layer thickness, compaction effort, sequence of operation, curing, environmental and climatic conditions.

2.3.1 Chemical/mechanical stabilization

Soil stabilization is defined as chemical or mechanical treatment, designed to increase, improved or maintain the stability of a soil mass or its engineering properties (Gregory, 2012). According to Grogan *et al*., (1999), mechanical stabilization is achieved by compaction or blending soil with other materials to improve the engineering properties without chemical reaction. Addition of aggregates to fine soil to improve gradation and the mixture of soil with asphalt cement are some of the cases of mechanical stabilization. When asphalt is added to soil, the soil particles are coated, resulting to improving the soil properties (Grogan *et al*., 1999). On the other hand, chemical stabilization occurs when the added materials either react with the soil or react on their own (Kezdi, 1979). These reactions lead to formation of new cementing compounds, which play a great role the stabilization process (Grogan *et al*., 1999). Many additives such as salt, polymers, surfactants, cement, lime, fly ash, have been used to stabilize soils (Al-Mukhtar *et al*., 2012).

2.3.2 Soil stabilization additives/agents

Soil stabilization is mainly to improve strength and durability properties of deficient soils often relies on cement, lime, fly ash, and asphalt emulsion (Gregory, 2012). These materials are relatively easy to apply, and provide benefits to many different soil types (Ali, *et al.,* 2017). However, there are varieties of non-traditional soil stabilization/modification additives available from the commercial sector such as polymer emulsions, acids, lignin derivatives, enzymes, tree resin emulsions, zeolite and silicates (Jeb and Rosa, 2003). These additives may be in different forms, liquid or solid and are often touted to be applicable for most soils (Ali, *et al.,* 2010*)*. Studies have demonstrated that many soil stabilization additives have little to no benefit for silty, sandy soil types. Sandy soils are problematic for stabilization and often require cement and/or asphalt emulsion to provide cohesion. Generally, lime works well with most clay soils, and cements and asphalt emulsions can be used for a wide range of soils. For clay soils, the clay fraction may often be altered through chemical reaction or ion exchange in a similar way with lime (Gregory *et al.*, 2003).

Soil stabilization using polymer emulsion is a straightforward process, in that the liquid is simply diluted to the proper amount (Site and Gurmel, 2014*)*. The dilution amount is selected to achieve the target additive quantity at the desired moisture content required for the most efficient compaction of the soil.

2.3.3 Lime stabilization

According to Petry and Little (2002), stabilization of clay aims at controlling volume change of the material, improving its workability and strength. Stabilization of deficient soil has been achieved successfully by using lime to reduce volume change and improve workability as well as plasticity of the deficient soils (Nelson and Miller, 1992). Lime improves the strength of expansive soils. While improvement of workability follows immediately after addition of lime to the deficient soils, the enhancement of strength increases with time when the added lime reacts with silica and alumina to form silicates and aluminates according (US Department of the Interior Bureau of Reclamation, 1998). It has also been observed that workability enhancement results from various reactions that follow the addition of lime to the deficient soils. These reactions include reduction in soil plasticity, agglomeration of colloids to form larger particles, disintegration of clay clods and reduction in moisture content due to hydration of the lime which results in soil drying (James and Ralph, 1979*)*.

According to the National Lime Association (2004), lime can be used in three distinct forms, that is, as quick lime or calcium oxide (CaO), hydrated lime or calcium hydroxide [$Ca(OH)_2$] and lime slurry consisting of suspension of hydrated lime. Additionally, dolomitic lime is also used for soil stabilization.

Different reactions such as cation exchange occur when lime is added to soil, resulting to flocculation, agglomeration, carbonation, and pozzolanic reaction (Nelson and Miller, 1992). These reactions are responsible for the modification of the soil properties. Cation exchange, flocculation and agglomeration contribute to change of plasticity, shrinkage and workability characteristics whereas strength enhancement is mainly as a result of lime soil pozzolanic reactions, with a minor influence of lime carbonation. Water is the most essential factor for soil-lime reaction but the soil-water mixture is not itself cementitious McNally (1998). In the presence of water, lime reacts with silica and alumina from clay minerals to produce bonding agents as in the case of Portland cement (McNally, 1998).

The basic pozzolanic reactions are as follows:

$Ca^+ + OH^-$ soluble clay silica → calcium silicate hydrate (CSH)

$Ca^+ + OH^-$ soluble clay alumina → calcium aluminate hydrate (CAH)

Another effect of lime on deficient soil is decrease in Maximum Dry Density (MDD) and the corresponding increase in Optimum Moisture Content (OMC). According to Khattab *et al*. (2008), reduction in MDD is attributed to the formation of cementitious products due to flocculation and agglomeration which lead to the formation of more open structure thus resulting in decreased MDD. The author found that increase in OMC involves different phenomena such as water consumption during lime-soil reactions, lubrication of new formed materials and evaporation of water due to high temperature released during soil-lime reactions.

2.3.4 Cement stabilization

Cement has for a long time been used for treatment of deficient soils. Cement stabilization of soil occurs in the presence of water, when its calcium silicates and calcium aluminates hydrate react to form cementicious compounds of calcium silicate hydrate and calcium aluminate hydrate (Micheal and John, 1999). This reaction releases calcium hydroxide which plays a great role in cement-soil stabilization, in the same way as for lime-soil stabilization (Gregory, 2012). Stabilization of soil with cement produces the same effects as lime, which includes reduction in liquid limit and plasticity index, increases shear strength and shrinkage limit, resulting reduction in the soil expansion (Nelson and Miller, 1992). Treatment of soil with cement results in development of cementitious bonds between the calcium silicate (tricalcium silicate) and aluminate hydration products of cement hydration and the soil particles.

The choice between lime and cement as a soil stabilizer depends highly on the soil characteristics Lime can be used alone for high plastic soils, whereas stabilization of high plastic soil with cement requires prior soil modification by lime (Ali *et al.,* 2010). Prior addition of lime to these soils reduces the plasticity and improves the workability, which facilitates the soil stabilization process with cement (Puppala and Chittoori, 2010). Although, cement can be used for stabilizing various soil types it is more effective with granular materials (Puppala and Chttoori, 2010).

2.3.5 Bio-enzymes

Bio-Enzyme is a natural, non-toxic, non-flammable, non-corrosive liquid enzyme formulation, fermented from vegetable extracts that improves the engineering properties of soil, facilitates higher soil compaction and increases strength (Janaki, 2018). Enzymes catalyze the reactions between clay and the organic cat-ions and accelerate the cat-ionic exchange process, resulting to reduced absorbed layer thickness (Sanjeev and Sapna, 2013). For other types of chemical stabilization, the additives are mixed with the soil, which is difficult to mix thoroughly, but Bio-Enzyme is easy to use as it can be mixed with water at optimum moisture contents and then sprayed over soil and compacted (Selvakumar, 2016). By altering the physical and chemical characteristics of soil, materials treated with Bio-Enzyme retain higher performance levels and extended life span (Sravan and Nagaraj, 2019). Bio-Enzyme may be used to increase the MDD and UCS values of a soil material to achieve specified standards for a base course.

Soil materials that have been treated with Bio-Enzymes as trials, include sandy clay, silty clay, sandy silt, plastic and non-plastic clay, sandy loam, fine loam, and loam mixed with clay. The dosage levels of the Bio-Enzymes vary from 1 to 5 litres for 5 m^3 of soil, depending on the soil type, soil characteristics, and product concentration (Tanveer and Mohd, 2015). Also the amount of dilution water has also been known to depend on in-situ moisture content of soil (Ferguson and Graham, 1984).

In clay-water mixtures positively charged ions (cations) are present around the clay particles, creating a film of water around the particle that remains attached or absorbed on the surface. The absorbed water or double layer gives the clay particles their plasticity (Nazile, 2018). In some cases the clay can swell and the size of the double layer increases, but it can be reduced by drying (Gens and Sanchez, 2004). Therefore, to truly improve the soil properties, it is necessary to permanently reduce the thickness of the double layer using cation exchange processes (Yu-Jun *et al*., 2013). Also utilizing fermentation processes, specific micro-organisms can produce stabilizing enzyme in large quantities. These soil-stabilizing enzymes catalyze the reactions between clay and the organic cat-ions and accelerate the cationic exchange without becoming part of the end product (Puneet and Suneet, 2015).

2.3.6 TerraZyme

TerraZyme is a bio-Enzymatic soil stabilizer and a natural, non-toxic liquid, formulated using vegetable extracts (Sravan and Nagaraj, 2019). Apart from being an accepted concept as a sound and resourceful road building practice, which completely replaces the conventional granular base and sub base, it emphasizes on strength, performance and higher resistance towards deformation. Organic enzymes come in liquid form, they are perfectly soluble in water, brown in colour and smell of molasses, with specific weight similar or equal to that of water (Ravi, *et al*., 2009). Their pH level is between 4.3 and 4.6, and have aroma that has no effect. Neither gloves nor masks are required during handling but they have been known to cause irritation to eyes. If they are to be stored for long periods of time without losing any of their properties, it is necessary to maintain a temperature of 55°C with no risk of decay. They react with oxidizing agents and are specially formulated to modify the engineering properties of soils (Hans and Hsai-Yang, 1991). They require dilution in water before application, and act to reduce the voids between soil particles and minimize absorbed water in the soil for maximum compaction, thereby decreasing swelling capacity of soil and reducing permeability (Szilvasszy, 2019). Application of TerraZyme enhances weather resistance and increases load bearing capacity of soils (Puneet and Suneet, 2015). These features are particularly evident in fine-grained soils such as clay in which the formulation affects the swelling and shrinking behavior (Nazile, 2018). The formulation has the ability to change the matrix of soil so that after compaction the soil loses its ability to reabsorb water, therefore the mechanical benefits of compaction are not lost even after water is reapplied to the compacted soil. Once the enzyme reacts with soil, the change is permanent and the product is bio-degradable (Puneet and Suneet, 2015).

2.3.7 Fiber

Generally, there are three types of fiber, Polypropylene Fibers (FP Fibers), Nylon Fibers and Polyvinyl Alcohol (PVA) Fibers. Polypropylene is a common material used for fiber reinforcement of soils, and it is manufactured in two forms: monofilament and fibrillated (Bayasi and Mcintyre, 2002). Monofilament fibers are individual, cylindrical fibers, while fibrillated fibers are flat, tape-like fibers that can be described as a latticework of “stems and webs” as the fibers break apart during mixing and compaction.

Nylon fibers are used as reinforcement in concrete to increase ductility, durability, and toughness (Song *et al*., 2005). When used in concrete, they can absorb water, allowing curing of the concrete from the inside out. This absorbed water also contributes to adhesion between the fibers and concrete (Lim, 2011). Although, there are not too many reports as regards to the use of nylon fibers with soil, these fibers may mechanically and chemically stabilize soil, especially when combined with cement.

PVA fibers are typically not used for soil stabilization, but they are used as reinforcement in concrete to increase ductility, durability, and toughness because hydrogen bonds form between the hydroxyl groups of the PVA fibers and cement particles (Thong, 2016). However, clay has been stabilized with PVA solution instead of PVA fibers, where hydrogen bonds have also formed between the hydroxyl groups of the PVA molecules and the silicate sheets of the clay (Silvia and Eladio, 2004). Combining these two findings, the hydroxyl groups of the PVA fibers should theoretically form hydrogen bonds with the silicate sheets of the clay and could be effectively used in stabilizing clay soil both chemically and mechanically. If the soil is also treated with cement in addition to PVA fibers, the fibers may bond better to a clay-cement mixture than clay alone, since bonding between fibers and cement has been verified (Mehdi *et al*., 2018) Occasionally, the hydrogen bonding between the PVA fibers and concrete is so strong that the PVA fibers rupture instead of pulling out of the cement matrix. If the PVA fibers do rupture, this fiber reinforced concrete may be too brittle for a particular application (Suvash *et al.,* 2020). To counteract this phenomenon, some PVA fibers are coated with oiling agent to allow pulling out of the cement matrix instead of rupturing (Li *et al*., 2012).

2.3.8 Polymer

Polymers and biopolymers have long been recognized as viable soil conditioners, because they stabilize soil surface structure and increase pore continuity (Maghchiche *et al., 2010*). In the past decade water-soluble polymers were identified as high effective erosion preventing and infiltration enhancing polymers (Maghchiche *et al.,* 2010). Polyacrylamide was effectively used in enhancing the stability of soil aggregates and increase infiltration in some areas, especially in sandy loam soils (Birhanu *et al*., 2020). Polyacrylamide, which is a long-chain synthetic polymer, acts as a strengthening agent, binding soil particles together and holding them in place, but they cannot alone remediate poor soil structure (Maghchiche *et al.,* 2010).

Acid-hydrolyzed cellulose micro fibrils, which is as an alternative to Polyacrylamide for soil stabilization was tested and showed promises (William *et al*., 2007). The study was aimed at producing series of low costs polymers and biopolymers to quantify their effectiveness on soil retain water, decrease evaporation, improve soil aggregate stability and other structural properties. The uses of Polyacrylamide-cellulose blend appear promising for reducing the labor cost of irrigation at arid and Semi-arid soils, and offers a safe environmentally friendly, inexpensive materials (Abdelhak *et al*., 2010).

2.4 Zeolite

Zeolites are microporous, like clays, naturally occurring hydrated alumina and silica (aluminosilicate) minerals, having potentially suitable characteristics for ground improvement applications (Nazife *et al*., 2017). Zeolites occur naturally but are also produced industrially on a large scale (Eva, 2018). Many large deposits of Zeolite tuffs occur worldwide as sedimentary units, must times have a thickness of more than 100m, making their use as bulk commodities economically feasible (Cornelius *et al.,* 2019). Zeolites have a wide range of commercial applications, including uses in catalytic cracking, commercial detergents for water softening, wastewater treatment and soil benefaction to control soil pH, moisture content and manure malodour. Synthetic Zeolites are used as an additive in production process of warm mix asphalt concrete (Milan *et al*., 2017). They help to decrease the temperature level during manufacturing and placement of asphalt concrete, resulting in lower energy consumption, thereby reducing emission of carbon dioxide, aerosols, and vapours . The use of synthetic zeolites in hot mixed asphalt leads to easier compaction and, to a certain degree, allows cold weather paving and longer hauls (Milan *et al*., 2017). When added to Portland cement as a pozzolan, they can reduce chloride permeability and improve workability. They reduce weight and help moderate water content, while allowing for slower drying which improves break strength, when added to lime and lime-metakaolin mortars, synthetic Zeolites pellets can act simultaneously as pozzolanic material and water reservoir. In recent years Zeolites have been added to binder materials, which blended with PC and offers enhanced strength as well as durability over PC alone in concrete, based on the following:

(a) Zeolite produces finer pore matrix in cement paste, reducing the permeability; and

(b) Zeolite has a higher pozzolanic activity than commonly used pozzolans, offering much improved durability.

Zeolite is of natural clinoptilolite kind with particle size smaller than 75µm which are referred to as fine aggregates. It is non-plastic and classified as silt according to the Unified Soil Classification System (USCS), and has specific gravity 2.2 (Ramu, 2017). Zeolite has important characteristics which includes high specific surface area and cation exchange capacity as well as ability to store heat between hydration and dehydration cycles (Colella *et al*., 2001). Zeolite, which is a softer material than Portland cement, increases fineness of the ground material and reduces grinding time (Canpolat *et al*., 2004). Replacement of Portland clinker by zeolitic tuff reduces workability and increases water demand as cement–water mixture contacts with zeolite minerals, the alumino-silicate framework of the zeolite starts decomposing, under the attack of OH- in a high-pH solution (Sersale, 1995). Depolymerised species, such as $[SiO(OH)_3]$- and $[Al(OH)_4$]- , enter the solution and react with Ca_2+ , forming hydrated calcium silicate and calcium aluminate compounds, very similar to those formed during hydration of cement**.** Table 2.2 present chemical composition of Zeolite.

Application of Zeolites, which ranges from petroleum refining, soil improvement, to waste treatment, has led to increase in production (Okoro, 2015). Unlike the conventional stabilizing agents like cement and phosphates, which contribute to eutrophication of aquatic life, Zeolite has remarkable potentials of being more Eco friendly. One of the main hindrances in its application is the high cost of production (Yili *et al*., 2017). This has necessitated the diversification of more economic or very low-cost ways of producing Zeolite on large scale which has gone a long way to reduce the high cost and encouraged its utilization (Okoro, 2015).

Natural zeolite has been investigated for use as cement and concrete improver (Poon *et al*., 1999; Perraki *et al*., 2003). Based on XRF results by Poon *et al*. (1999), natural zeolite In stabilization, effects of some influential factors (water content, cement content, curing time, and compaction energy) that affect strength development of stabilized expansive soil have been extensively researched (Kamon and Bergado, 1981; Miura *et al*., 2001; Horpibulsuk *et al*., 2004; Horpibulsuk *et al*., 2005; Horpibulsuk *et al*., 2006). Based on the available compression and shear test results, many constitutive models were developed to describe the engineering behavior of stabilized clay (Suebsuk *et al*., 2010). These investigations have mainly focused on the mechanical behavior, which is controlled by the microstructure and has to do with the arrangement of the particles, clusters and pore spaces in the soil, as well as cementation (Mitchell, 1993). It is therefore, very essential to understand the changes in engineering properties that results from the changes in the influential factors from the microscopic scale.

Kaolin clay as a major source of natural zeolite offers a unique and sustainable route towards manufacturing of Zeolite (Abdulahi, *et al*., 2017). This has ultimately been a factor in an effort to reduce the cost of production. Kaolin clay is a naturally occurring mineral, and it has been found in commercial deposit in many places in Nigeria including Ahoko, kogi state, Nigeria (Tatabu, 2015), which is considered as the most promising local sources of Zeolites in Nigeria (Okoro, 2015). The total reserve of kaolin in Nigeria is estimated to be about three billion metric tons in at least 45 known deposits, distributed in some state of the country (Alabi and Omojala, 2013).

Based on the oxide composition of Zeolite analysis presented in table 2.2, Zeolite is pozzolanic as the summation of oxide composition of silica (67.44%), alumina (10.8%) and iron oxide (0.84%) is <70% oxide. Which satisfy the requirement of a material to be pozzolanic.

Table 2.2: Chemical composition of zeolite

Chemical Composition of Zeliote (%)	
SiO_2	67.44%
$Al_2 O_3$	10.8%
$Fe_2 O_3$	0.84%
CaO	1.24%
pH	7.1

Source: AKSHAR EXIM CO.PVT.LTD

The applications of Zeolite in sustainable binders for soil stabilization, and reports that to decrease the environmental impact caused by Portland cement in binders for soil stabilization, Zeolite can replace some percentage of the cement. In the study, UCS was used as a recognized parameter for assessing the performance of the stabilized soil. The UCS values of the Zeolite-PC mixes in three binder dosages (2.5, 5.0 and 10%) and at three curing periods (7, 28 and 90 days) were for two model soils:gravel sand and a clayey silt. results from the experiment demonstrated that the highest UCS value 1.28MPa, of Zeolite-PC mixes was obtained at 90 days, in a 10% content binder dosage, indicating that it exceeded the current USA guideline of 350kPa, comparing all of the UCS values for all dosages and all curing periods of the clayey silt. The highest UCS value of 7.65MPa for Zeolite-PC mixes was achieved at 90 days and in a 10% content binder dosage in gravel sand. In comparison, the PFA (Pulverised Fuel Ash)-PC mixes presented lower UCS values than the Zeolite-PC mixes with the same content ratio and the same testing time.

Turkoz (2017) investigated the effect of cement and natural zeolite additives on the characteristics of dispersibility and swelling potential of clay soils. In the study, a constant percentage of cement (3%) plus different percentages of natural zeolite (1, 3, 6, 10, 15 and 20%) were mixed with four different clay soil samples. Physical and chemical properties of
Turkoz (2017) investigated the effect of cement and natural zeolite additives on the characteristics of dispersibility and swelling potential of clay soils. In the study, a constant percentage of cement (3%) plus different percentages of natural zeolite (1, 3, 6, 10, 15 and 20%) were mixed with four different clay soil samples. Physical and chemical properties of
the soil samples were first determined, and swell percentage, swell pressure, crumb, pinhole and UCS at different curing times were performed on samples with and without the additive by compressing the sample to achieve particular compaction characteristics. It was discovered that significant strength increases depended on curing time, and the properties were improved with the mixture of cement and zeolite additives, depending on the Sodium Adsorption Ratio (SAR) and Exchangeable Sodium Percentage (ESP) values of clay soil samples with different plasticity characteristics that exhibit dispersive and swell properties. The author stated that the study did not only showed that mixture of cement and zeolite additives improved the dispersive and swell properties of clay soil samples, but also significant increases in strength values were observed.

Ismail *et al.* (2014), in their research discusses the improvement of soil materials by synthesizing zeolite from bentonite as low-cost adsorbent materials. The evaluation approach includes efficiency of the studied raw materials and their synthesis products for the improvement of engineering parameters of clay rich materials. The synthetic zeolite was investigated by powder X-ray Diffraction (XRD) analysis, Scanning Electron

Microscope (SEM) which was attached with EDX unit (Energy-dispersive X-ray spectroscopy). The stabilization and modification of the clay soil materials were detected as swelling and shrinkage limit decrease by addition of various proportions of the synthetic zeolite.

Hossein *et al*. (2016), in a study, based on experimental program, effects of zeolite on the characteristics of cemented sands were investigated. Stabilizing agents used in the study includes Portland cement of type II and zeolite. Results of the study showed improvements UCS and failure properties of cemented sand when the cement is replaced by zeolite at optimum proportion of 30%, after 28 days. The rate of strength improvement was approximately between 20 and 78%. They reported that the efficiency of using zeolite increases with increase in cement content and porosity. The concluded that power function of void-cement ratio and zeolite content demonstrated to be an appropriate method to assess UCS of zeolite-cemented mixtures.

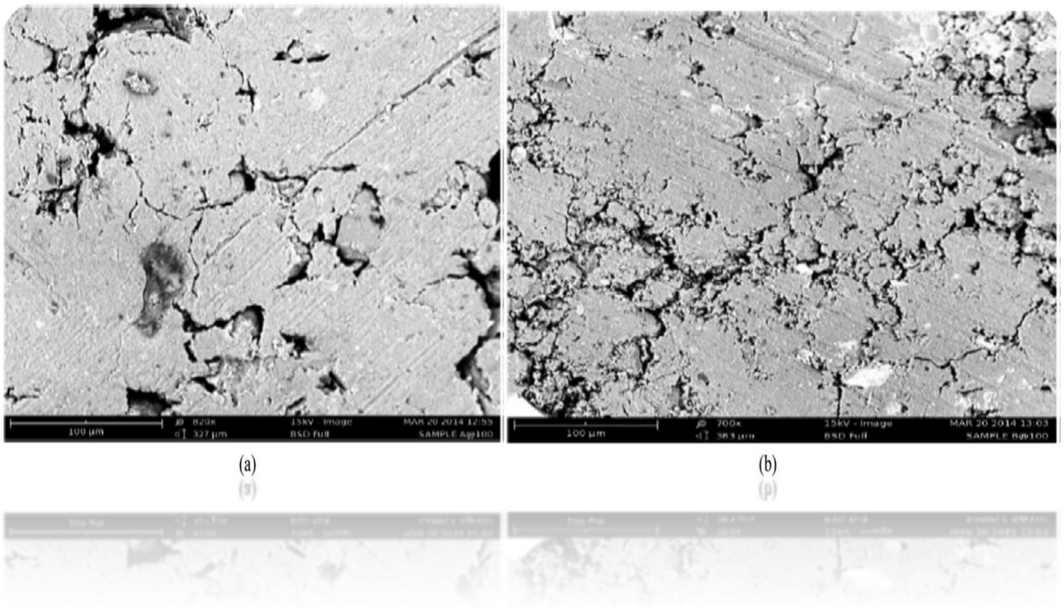
2.5 Microstructural Investigation of Stabilization Process

The knowledge of physicochemical changes of stabilized soil is crucial to the engineers, Microstructural analysis provides a convincing explanation on the physicochemical changes that occurs during stabilization process (Chijoke *et al*., 2019). Such analysis can be presented in the form of qualitative digital image analysis, and so on, SEM, or quantitative charts as in X-ray diffraction (XRD), they help to clarify the impact of stabilization on the soil constituent, microfabric and pore structure (Rose and Lyn, 2014).

Mirzababaei *et at*., (2009) microstructurally investigated effect of polymers on soil microfabric. The polymer, furan, added at 3, 5 and 10% by weight of the soil, progressively reduced the free swell percentage of three expansive soils, compacted at OMC and MDD with an average maximum drop of about 83.5%. From the different image magnification ratios (500 and 1000), the natural soil mainly consisted of discrete granular in combination with sparse aggregations, few silt grains, and some connectors. The inter-assemblage pores between the discrete and sparse aggregations are obvious. However, on addition of furan at 5 and 10%, the soil fabric changes to dense aggregations with formation of intra-assemblage pores coming from formation of dense aggregations, with the inter-assemblage pores becoming less evident, indicating reduction in pore sizes. Also clothed silt grains became more obvious than that in the natural soil. The changes in the soil fabric and pore structure were responsible for the observed reduction in swelling properties of the soil. However, the micro-structural investigation conducted in this study was only qualitative.

In a study by Osinubi *et al*. (2015), changes in pore size, in addition to image observations are confirmed quantitatively, showing the variation observed in the soil fabric and pore sizes (Figure 2.1). This was done using fibremetric analysis (a statistical package incorporated in SEM used for pore ranges from 40 mm to 100 nm). It was inferred that a reduction in both soil fabric and pore sizes occurred can further be consolidated by observing the histograms. The fibre histogram showed reduction in the mixture fabric from 2.23 mm for the natural soil to 875.16 nm for the modified soil, while the pore histogram confirms a surface area reduction of the natural soil pores from 2.75 mm^2 to 0.84 nm^2 for the modified soil.

Similarly, many works (Nazile, 2015., Osinubi *et al*., 2015; Mousavi, 2017) provided microstructural analysis for estabilized expansive soils, using tests like Fourier Transform Infrared Spectroscopy (FTIR), Zeta Potential (ZP), environmental SEM, Transmission Electronic Microscopy (TEM), fibremetric analysis and others. These tests explain the physicochemical changes which modify the soil to produce desirable engineering properties.



(a) (b)

Figure 2.1: Microfabric of black cotton soil at 28-d curing: (a) Natural soil, and (b) Optimal modified soil (Osinubi *et al*., 2015)

CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1 Preamble

This work was carried out in accordance to British Standard codes (BS 1377 part 2,4, and 7 of 1990 and BS 1924 part 1 of 1990) to study the strength development and the stabilization effect of Zeolite on Cement-Zeolite stabilized soil. The Cement and Zeolite were added in varying proportions by weight of the dry clay soil. Samples were prepared based on the different mixed proportions. UCS tests were carried out on the prepared samples after 1, 7, 14, 28 and 60-days curing periods. Microstructural study was carried out on the optimal mixed composition of the Cement-Zeolite stabilized clay soil.

3.2 Materials

All the materials used in this research work were obtained from Niger State except for the clay, which was obtained from Gwagwalada, FCT-Abuja. The laboratory works were carried out at Civil Engineering Laboratory, Department of Civil Engineering, School of Infrastructure, Process Engineering and Technology, Federal University of Technology Minna, Minna, Niger State, Nigeria, while the microstructural study (SEM), elemental quantitative analysis (XRD), and elemental qualitative analysis (EDS and XRF) was carried out in South Africa. The materials used for this research work are clay soil, Cement, Zeolite and distilled water (Figure 3.1).

Figure 3.1: Materials used in the study: (a) Tropical Black clay, (b) cement, (c) zeolite, (d) distilled water, used for this study

3.2.1 Tropical black clay

The Tropical Black Clay (TBC) used in the study was collected from Gwagwalada area of FCT, Abuja, North Central, Nigeria. Gwagwalada is located at an elevation of 210m above sea level and on a latitude of $8^{o}56'\ 29''$ N, longitude of $7^{o}\ 5'\ 31''$ E on the Nigerian geographic map (FCDA, 2015) as shown in the google Earth map (Figure1). The soil sample was collected at depth of between 1.0 to 1.5m below the ground surface to avoid organic matter. Disturbed sampling method was used in the collection of the sample. The collected soil sample was wrapped in polythene bags to avoid loss of moisture and transported to Civil Engineering Laboratory, Federal University of Technology, Minna. In the laboratory, the natural soil sample was air dried and pulverized before further tests were conducted. The location of the tropical black clay is showed on the google map on Figure 3.2.

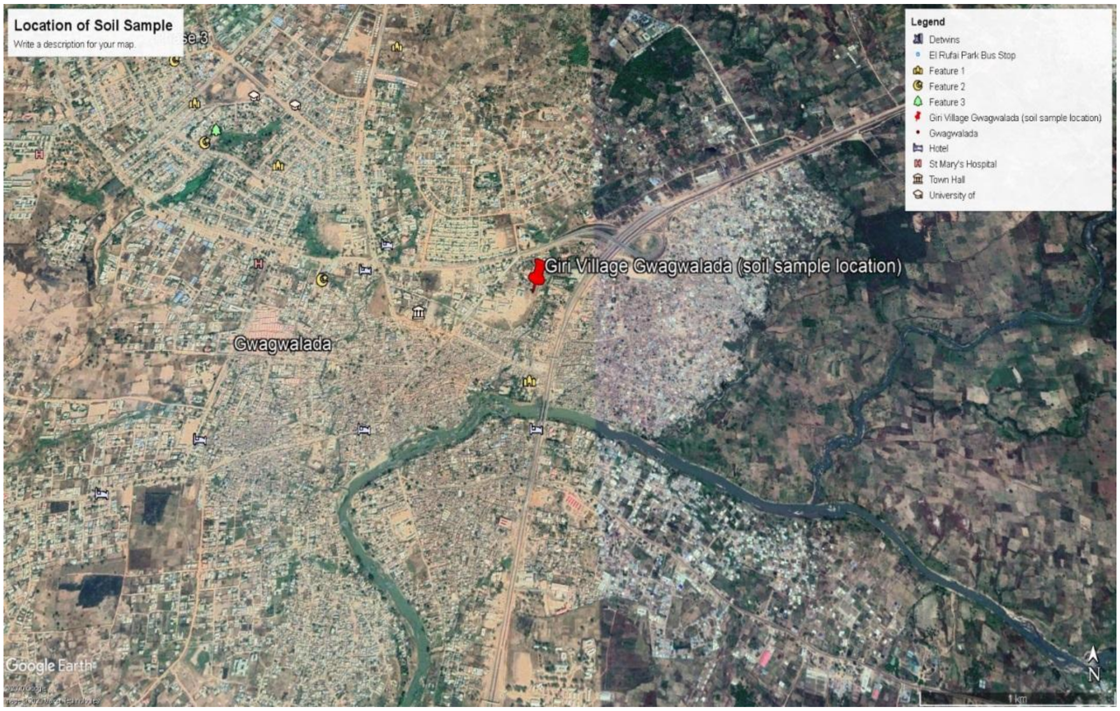


Figure 3.0: Location of sampling point (Source: Google Earth Maps)

3.2.2 Ordinary Portland cement (OPC)

The cement used for the study was Ordinary Portland cement, procured from a cement vendor at Minna building materials market. Dangote cement brand of grade 42.5R was used. The cement was properly stored under dry condition.

3.2.3 Zeolite

The zeolite used for the study was purchased from a vendor in Zaria, Kaduna State, Nigeria. It was produced by TM Media.

3.2.4 Water

The water used in this study is distilled water. The distilled water was purchased from a laboratory equipment/chemicals store, opposite Minna General Hospital, along hospital road, Minna, Niger state.

3.3 Experimentation

The laboratory tests performed on the natural soil in order to determine its engineering properties were in accordance with BS 1377 (1990) parts 2, 4 and 7, while on the stabilized soil, the tests were conducted in accordance with BS 1924 (1990). In accordance with the literature, the soil was mixed with 0, 2, 4 and 6% cement, and each was then admixed with 0, 3, 6, 9, 12 and 15% zeolite. Natural moisture content, specific gravity, particle size distribution, Atterberg limits (liquid limit, plastic limit and plasticity index), compaction at Standard Proctor energy level, Unconfined Compressive Strength (UCS) tests were performed on the natural soil, while on the stabilized soil, compaction and UCS tests were performed. Samples for UCS test were prepared at Maximum Dry Densities (MDD) and Optimum Moisture Contents (OMC), obtained from the respective compaction tests at respective mixtures of the soil and the additives (cement and zeolite). Table 3.1 presents the mix proportion used in the study.

Table 3.1: Mix proportions

0%Zeo. & 0% Cem.	0%Zeo. & 2% Cem.	0%Zeo. & 4% Cem.	0%Zeo. & 6 % Cem.
3%Zeo. & 0% Cem.	3%Zeo. & 2% Cem.	3%Zeo. & 4% Cem.	3%Zeo. & 6% Cem.
6%Zeo. & 0% Cem.	6%Zeo. & 2% Cem.	6%Zeo. & 4% Cem.	6%Zeo. & 6% Cem.
9%Zeo. & 0% Cem.	9%Zeo. & 2% Cem.	9%Zeo. & 4% Cem.	9%Zeo. & 6% Cem.
12%Zeo. & 0% Cem.	12%Zeo. & 2% Cem.	12%Zeo. & 4% Cem.	12%Zeo. & 6% Cem.
15%Zeo. & 0% Cem.	15%Zeo. & 2% Cem.	15%Zeo. & 4% Cem.	15%Zeo. & 6% Cem.

In preparing the samples for UCS tests, the mixtures were placed in a split mold of height 76mm and 38mm diameter and compacted at three layers with respective number of blows using 3.15kg rammer falling freely at the height of 30cm. The samples were then removed from the mould, excess soil trimmed, and stored in a properly labelled polythene bag. A total of 8 samples were produced per each mix proportion. The produced samples, stored in properly labelled polythene bags, were cured in moist river sharp sand and UCS test was carried out on the respective samples after 1, 7, 14, 28, 60 curing days.

3.4 Adopted Method of Research Work

The experimental work was conducted in accordance to BS 1377 (1990) parts 2 and 4. They tests were all carried out in Civil Engineering Laboratory of the Federal University of Technology Minna Except for the physio-chemical test which was carried out in South Africa. The tests carried out include those for determination of index properties of natural clay (natural moisture content, specific gravity, particle size distribution, Atterberg Limits); determination of moisture density relationship (optimum moisture content and Maximum dry density); determination of strength characteristic (unconfined compressive strength test); and physio-chemical test (SEM, XRF, EDS, and XRD).

3.5 Preliminary Laboratory Test on the Tropical Black Clay

Preliminary laboratory tests and analysis were carried out on the tropical black clay in order to determine its physical and geotechnical properties. The index properties provide parameters needed for classification of the soil. Index properties tests carried out were particle size distribution, specific gravity, moisture content, Atterberg limits. Compaction test was also carried out on the natural soil.

3.5.1 Natural moisture content

Moisture content of a soil is taken as the amount of water within the pore space between the soil grains which is removed by oven drying at specified temperature. Moisture content has a profound effect on soil behavior. The test was done in accordance to BS 1377 Part 2 (1990):

Oven drying techniques of at between 105 to 110°C was employ in this study. Some empty, cleaned and dried moisture content cans were marked and weighed before usage. The collected sample was crumbled and loosely placed in the containers. The containers with the samples were immediately weighed and recorded. They were then placed in electronic oven, set at a temperature of 110°C for 24 hours. After drying, the samples were taken out of the oven and allowed to cool before weighing. Moisture content of the soil specimen, *w*, expressed as a percentage of the dry soil mass was calculated using the equation (3.1):

$$w = \frac{\mathrm{M}2 - M2}{M3 - \mathrm{M}1}\, x\, 100 \tag{3.1}$$

where:

M_3 = mass of the container and dry soil (in g)

M_2 = the mass of the container and wet soil (in g)

M_1 = the mass of the container (g)

3.5.2 Particle size distribution

The procedure adopted involved soaking 300g of the dry soil for 24hr and then washing through sieves 2.0mm and 0.075mm. The washing was continued until the water from the washed soil became crystal clear. The retained samples during washing on sieve size 2.0 and 0.075mm were carefully collected and placed in a pan, which was in turn placed in oven at between 105 to 110°C for 24 hours. Set of sieves were measured empty and arranged sequentially with the sieve having the largest apertures on top and that with the lowest size below as follows; 5.0, 3.35, 2.0, 1.18, 0.85, 0.60, 0.425, 0.300, 0.150, and 0.075mm, and ending with the pan at the base. The oven dried samples were poured into the uppermost sieve and the set of the sieves placed on a mechanical sieve shaker, and allowed to shake for 10 minutes. The weight of each sieve with the retained soil was taken and recorded. The weights of empty sieves were subtracted to give weight of the retained soil on each sieve. The percentage of total sample passing each of the sieves was calculated

3.5.3 Specific Gravity

The procedure adopted is outlined in BS 1377 (1990). The density bottles with stoppers were washed dried and weighed empty with the stopper as M_1. About 50g of soil sample which pass through sieve size 2mm was poured into the density bottles. The density bottles and content together with the stoppers were weighed as M_2. Distilled water was added and covered and allowed to fully soak. After this, the stoppers were inserted, the bottles together with the content were shaken, and the stoppers were then removed and water was added to reach 250ml capacities. The bottles with the content and stoppers were weighed as M_3. The density bottles were emptied and thoroughly cleaned and oven dried at 105°C. The clean oven dried density bottles were filled with distilled water to 250ml capacities and stoppers were inserted and then weighed as M_4. The specific gravity of the sample was calculated from:

$$GS = \frac{M_2 - M_1}{(M_4 - M_1) - (M_3 - M_2)} \tag{3.2}$$

where;

M_1 = Weight of bottle

M_2 = Weight of bottle +dry soil

M_3 = Weight of bottle + soil + water

M_4 = Weight of bottle + water.

3.5.4 Atterberg limits

The test conducted includes Liquid Limit (LL) and Plastic Limit (PL). They were carried out in accordance with BS 1377 (1990).

3.5.4.1 Liquid limit test

Cone penetrometer method of liquid limit determination was used. Reasonable quantity of air-dried sample was pulverized and sieved through 425µm sieve. About 200g of the sieved sample was placed on a flat glass and mixed thoroughly with clean water using spatula until the soil mass become a thick paste. The paste was pushed into the cup with spatula, making sure air was not trapped, until filled. The top of the soil was trimmed with the top of the cut and placed beneath the cone. The cone was then lowered so that it just touched the surface of the soil. When the cone was in correct position, a slight movement of the cup gives a small mark on the surface of the soil and the reading of the dial gauge was recorded.

The cone was then released for a period of 1-5 seconds. After penetration, the dial gauge was lowered to the new position of the cone shaft and readings recorded. The difference between the readings at the beginning and at the end of the test was recorded as the cone penetration. Average of two penetrations was recorded. The cone lifted out and cleaned. A moisture content sample of about 10g was taken from the area penetrated by the cone for moisture content determination. The soil was then removed from the cup, remixed and the procedure outlined above was repeated using the same sample with more water added until penetration of about 20mm was recorded. The relationship between the moisture contents and cone penetration was plotted. From the plotted graph, moisture content at 20mm penetration was taken as liquid limit of the soil.

3.5.4.2 Plastic limit test

About 20g of the pulverized soil sample sieved through 425μm sieve was used for the test. The soil was thoroughly mixed with clean water. A small sample of soil ball was then rolled between the hand and glass plate. The rolling was continued until a thread that grumbles at about 3mm diameter was obtained. The crumbled soil was then gathered and placed in moisture can for moisture content determination. Plasticity Index (PI) was then determined from the result of liquid and plastic limits

3.5.4.4 Plasticity index

The plasticity index is the differences between liquid limit and the plastic limit. The expression used in calculating the plasticity index is given in the equation (3.3).

$$PI = LL - Pl \tag{3.3}$$

where

LL= Liquid Limit

PI = Plasticity index

PL= Plastic Limit

3.5.5 Compaction Characteristics

In this study, optimum moisture content and maximum density were carried out to determine the moisture density relation of the natural tropical black clay. Standard Proctor Compaction test was used to determine the mass of dry soil per cubic meter when the soil is compacted in a specified manner over a range of moisture contents. The dry density of the soil depends on the degree of compaction and its moisture content. The primary objective of compaction test is to determine the optimum moisture content and the maximum dry density of a soil.

The compaction energy effort used in the study is the British Standard Light (ASTM D4609-08, 2008). The procedure adopted is as outlined in BS1377) part 4 (1990. The mass of an empty mould was weighed, noted and recorded as M_1. Then, a 3kg of air-dried soil sample was thoroughly mixed with small amount of water. The mixed sample was compacted into a 940cm^3 cylindrical mould in three layers of approximately equal mass, with each layer receiving 25 blows of a 2.5kg rammer falling freely through a height of 300mm. after compacting the last (third layer), the collar was removed and the surface of the soil was trimmed level with the mould and then weighed as M_2. Specimen from top and bottom of the mould were taken for moisture content determination. The soil sample was demoulded and mixed together with the remaining sample on the tray. The above procedure was repeated at varying moisture content, until the mass decreased. The dry density, in each case was calculated and plotted against its corresponding moisture content. The bulk density and the dry density were calculated from

$$\rho_b = \frac{M_2 - M_1}{V} \tag{3.4}$$

where

M_1 = mass of empty mould

M_2 = mass of mould + soil

V = volume of mould

Also, dry density,

$$pd = \frac{\rho_b}{1+w} \tag{3.5}$$

Where w = moisture content of the soil.

3.2.5 Unconfined compressive strength

Roy (2014) observed that Unconfined Compressive Strength (UCS) is the most common and adaptable method of evaluating strength of stabilized soils. The UCS tests were performed on the soil samples in accordance with BS 1377 part 7 (1990) at Standard Proctor Test (SPT) and West African Standard (WAS) energy levels. In moulding the samples, the soil samples were filled into a properly oiled split mould and collar of height 80mm and diameter 38mm. At Standard Proctor Test, the samples were given 2 blows using a 3.15kg rammer falling through a height of 30cm for the first layer. More samples were added into the mould and given another 2 blows for the second layer and another 2 blows for the final layer after addition more sample. The mould was then untied and excess sample trimmed off with spatula. The moulded samples were then wrapped in polyethene, labeled properly and immersed in wet river sharp sand to be cured for 1, 7, 14, 28 and 60 days.

Before testing the moulded samples were trimmed to a height of 76 mm and moisture content test conducted on the excess soil samples. The mass of the wet sample was also determined to calculate the bulk unit weight. The specimens were, one after another, then placed centrally on the lower platen of the UCS testing machine and a compressive force applied to the specimen. The UCS machine was then switched on and the load reading taken from the proving ring dial gauge at deformation intervals of 0.05, 0.1, 0.2, 0.3, 0.4, etc. continuously until the sample shears. The machine was then unloaded and the sample removed. The procedure was repeated for each sample after 1, 7, 14, 28, and 60 days.

Record was also taken simultaneously of the axial deformation and axial force at regular interval until failure of the samples occurs. UCS of the samples were calculated at the point, on the stress–strain curve, at which failure occurred using the following equation:

$$\text{compressive strength} = \frac{\text{failure load}}{\text{Cross}-\text{sectional area of specimen}} \tag{3.6}$$

The results obtained from different percentage proportions of the stabilizers as well period of curing are shown in Appendix

Cross-sectional area of specimen were determined from

$$A_f = \frac{A_o}{1-\mathcal{E}} \tag{3.7}$$

where:

A_f is area of specimen at shearing load,

$\mathcal{E}$ is strain,

A_o was computed from:

$$A_o = \pi r^2 \tag{3.8}$$

where:

r is initial radius of the specimen

Strain (£) was computed from

$$\mathcal{E} = \frac{\Delta l}{l_0} \tag{3.9}$$

ΔL =

change in length deformation of the (specimen)

Lo= is initial length of the specimen

Figure 3.3: UCS Machine

3.6 Microstructure Test

Elemental composition and mineralogy of the soil samples, Cement and Zeolite would be was determined using X-ray Fluorescence (XRF) and X-ray Diffraction (XRD) and Scanning Electron Microscopy SEM. The optimum mixtures of the cement-zeolite stabilized clay was subjected to same microstructural test to examine the microstructure and further elucidate the clay interactions and hence the soil cementation and stabilization mechanisms.

3.6.1 Scanning electron microscopy

The Scanning Electron Microscope (SEM) has become one of the most widely used electron microscopes (Susan Swapp, 2017). It examines microscopic structure by scanning the surface of materials, similar to scanning confocal microscopes but with much higher resolution and much greater depth of field (Susan Swapp, 2017). An SEM image is formed by a focused electron beam that scans over the surface area of specimen, it is not formed by instantaneous illumination of a whole field as for a TEM (Susan Swapp, 2017). The most important feature of SEM is the three-dimensional appearance of its images because of its large depth of field (Natasha, e al., 2019). The depth of field can reach the order of tens of micrometers at 103 × magnification and the order of micrometers at 104 × magnification (Natasha, e al., 2019). An SEM is relatively easily operated and maintained, compared to TEM "Transmission Electron Microscopy vs Scanning Electron Microscopy". In addition, SEM system allows chemical information from a specimen to be obtained by using various techniques, including equipping the X-ray energy Dispersive Spectrometer (EDS).

3.6.2 X-ray diffraction technique

X-ray Diffraction (XRD) is a rapid analytical technique primarily used for phase identification of a crystalline material and can provide information on unit cell dimensions (Abdullah, *et al*., 2014). The material to be analyzed will be finely grounded, homogenized, and average bulk composition is determined (Barbara, *et al*., 1999). In this study, XRD was carried out on the black clay, cement, zeolite and on the optimum composition of the stabilized clay sample.

3.6.3 X-ray fluorescence

X-ray Fluorescence (XRF) is a non-destructive analytical technique used to determine the elemental composition of materials (Karl *et al.,* 2020*)*. XRF analyzers determine the chemistry of a sample by measuring the fluorescent (or secondary) X-ray emitted from a sample when it is excited by a primary X-ray source (Karl, *et al.,* 2020*)*. Each of the elements present in a sample produces a set of characteristic fluorescent X-rays ("a fingerprint") that is unique for that specific element, which is why XRF spectroscopy is an excellent technology for qualitative and quantitative analysis of material composition. in this study, XRF will be used to detect the amount of stabilizer, in the form of oxide, in several stabilized clay sample.

CHAPTER FOUR

4.0 RESULTS AND DISCUSSION

4.1 Index Properties of the Tropical Black Clay

The results of preliminary tests conducted on the natural soil, for identification and classification purposes, is presented on Table 4.1. From the table, the soil contained high composition of clay sized particles without gravel.

Table 4.1: Geotechnical Properties of Natural Black Cotton Soil

Property	Quantity
Percentage passing BS No 200 sieve (%)	79.03
Moisture Content (%)	35.06
Liquid limit (%)	58
Plastic limit (%)	26.6
Plasticity Index (%)	31.4
Specific Gravity	2.43
AASHTO Classification	A-7-6
USCS	CH
Maximum Dry Density (g/cm³) Standard Proctor	1.692
Optimum Moisture Content (%) Standard Proctor	18.0
Colour	Greyish black
Dominant clay mineral	Montmorillonite

According to the American Association of State Highway and Transportation Officials (AASHTO), the natural soil falls under A-7-6 soils, which is termed silty-clay/clayey materials (more than 35% passing sieve No. 200) and are generally rated fair to poor for any engineering application. Based on Unified Soil Classification System (USCS), the soil is classified under clay of high plasticity (CH). The soil, in its natural state, is not suitable for use for road construction unless stabilized to improve its strength and durability. The low value of MDD and high value of OMC recorded is also an indication of poor soil for road construction. Based on these properties, the natural soil falls below the standard recommended for most civil engineering construction works especially highway construction (Osinubi and Medubi, 1997) and therefore needs stabilization.

4.2 Detectable Oxide Composition of Materials used

The oxide composition, determined using X-ray Fluorescence analysis, of materials used in the study are presented:

Table 4.2: Oxide Composition of Natural Clay, Cement and Zeolite

Oxide	Clay (%)	Cement (%)	Zeolite (%)
Calcium oxide (CaO)	7.81	64.44	0.84
Silicon dioxide (SiO_2)	1.57	0.44	69.12
Magnesium oxide (MgO)	2.13	4.57	0.65
Potassium oxide (K_2O)	1.68	0.56	1.09
Sodium oxide (Na_2O)	58.81	21.60	0.73
Aluminum oxide (Al_2O_3)	15.86	4.13	10.79
Ferric oxide (Fe_2O_3)	0.99	1.06	4.2
Sulfur trioxide (SO_3)	1.16	0.11	0.04
LOI	8.99	0.76	4.5-7.0

The oxide composition of Zeolite composed of predominantly Silica oxide (69.12%), Alumina Oxide (10.79%) and Iron Oxide (4.2%), sum up to a value of <70% oxide composition which is required for pozzolanic reactivity and satisfy the criteria for using a material as pozzolana. The Clay composed majorly of Sodium oxide (58.81%), Aluminum oxide (15.86%) and calcium oxide (7.81%). The Cement used majorly composed of calcium oxide (64.44%), Sodium oxide (21.60%) and magnesium oxide (4.57%).

4.3 Unconfined Compressive Strength

Unconfined Compressive Strength (UCS) is regarded as the most common and adaptable method of evaluating the strength of stabilized soils (Alhassan and Alhaji, 2007). According to Singh and Singh (1991), it is the main test recommended for the determination of the required amount of additive to be used in stabilization of soils. The variation of UCS with increase in zeolite from 0 to 15% (at 3% addition) at specific cement contents and for 1, 7, 14, 28 and 60-days curing periods were studied and the results for the five curing periods are presented in Figures 4.1, 4.2, 4.3, 4.4 and 4.5 respectively.

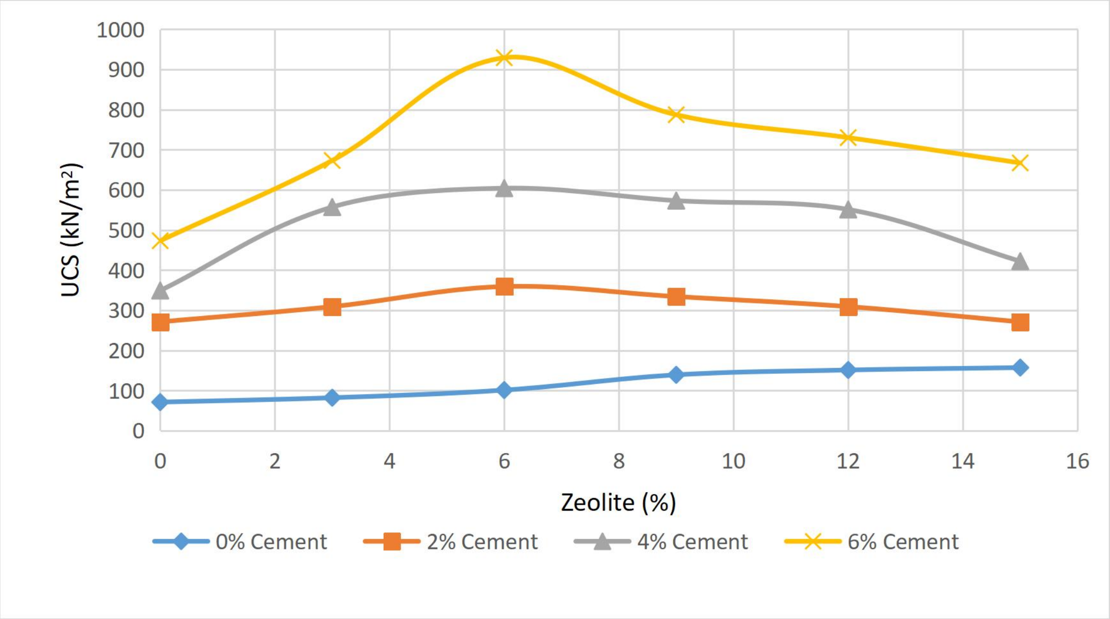


Figure 4.1: Variation of unconfined compressive strength with various dosages of both cement and zeolite after 1 day curing period

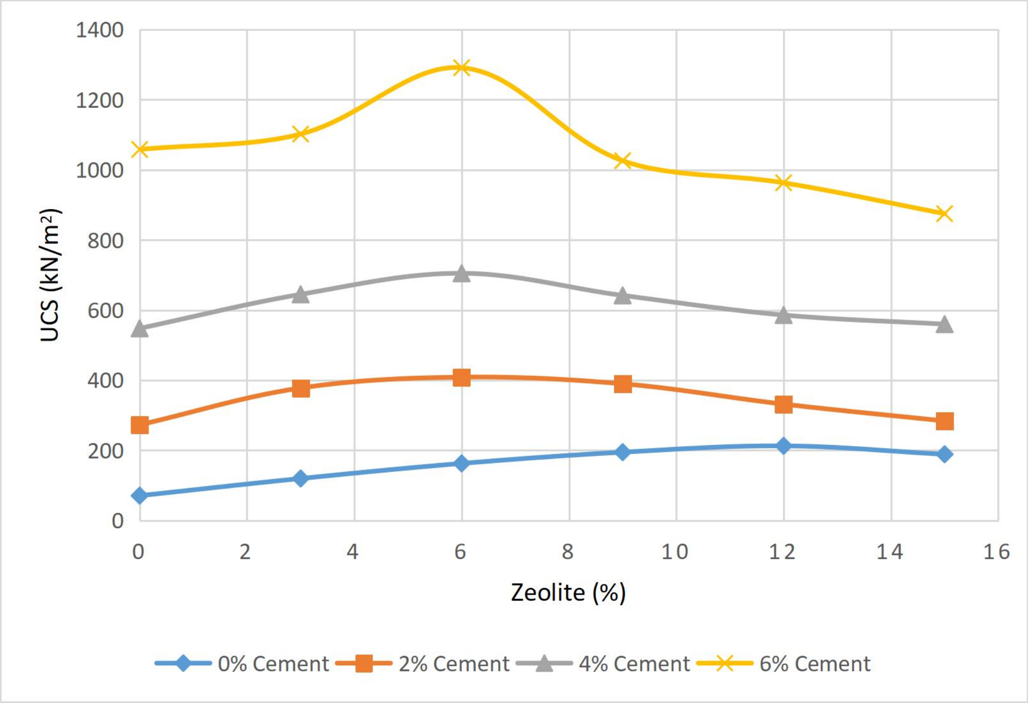


Figure 4.2: Variation of unconfined compressive strength with various dosages of both cement and zeolite after 7 days curing period

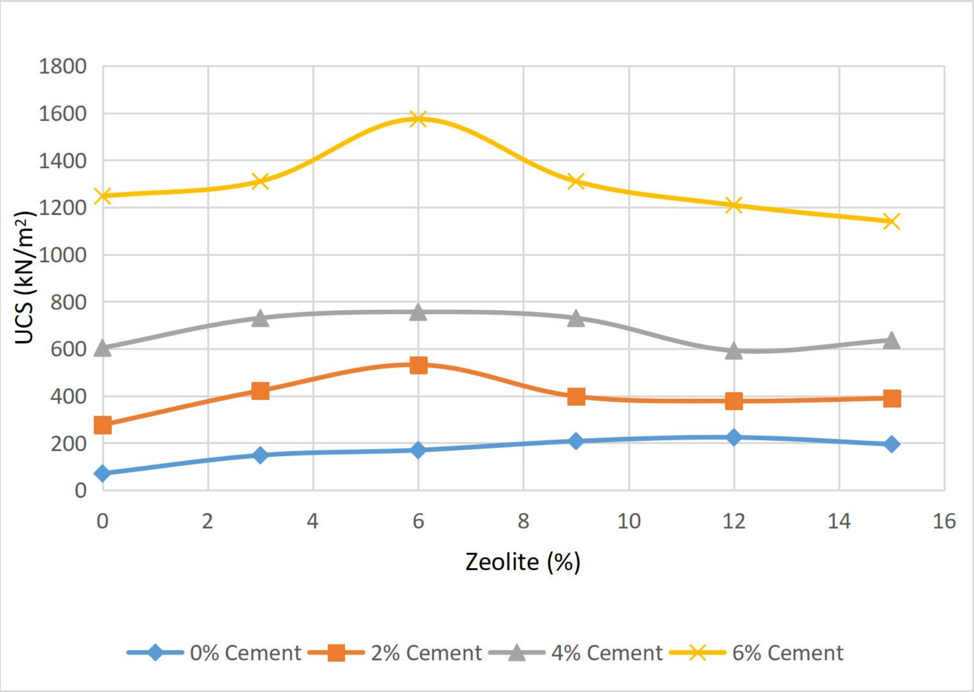


Figure 4.3: Variation of unconfined compressive strength with various dosages of both cement and zeolite after 14 days curing period

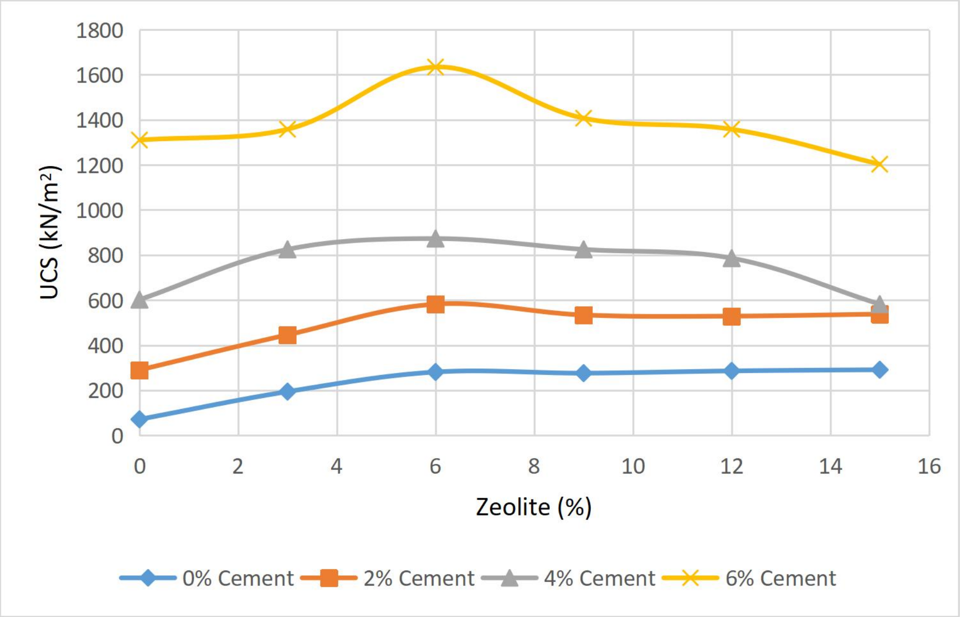


Figure 4.4: Variation of unconfined compressive strength with various dosages of both cement and zeolite after 28 days curing period

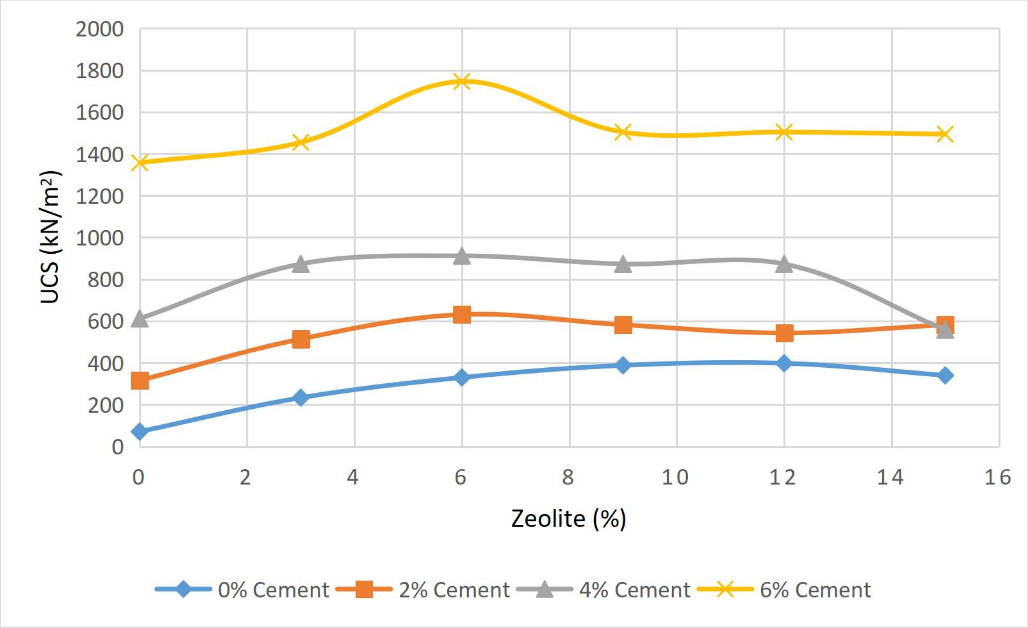


Figure 4.5: Variation of unconfined compressive strength with various dosages of both cement and zeolite after 60 days curing period

From the results from Figure 4.1-4.5, it was observed that there was a tremendous improvement in the UCS value of the soil with addition of cement and zeolite, when compared with the relatively low UCS value of 71 kN/m^2 for the soil at natural state as depicted in Figures 4.1-4.5.

Apart from the increase in UCS recorded with increase in cement content, there was also increase in UCS with increase in zeolite content at specific cement contents, up to the peak UCS values at 6% zeolite, after which the values dropped. This trend was observed at all the curing periods considered (Figures 4.6 to 4.9). Also, observation and comparison of the figures revealed that as the curing period increases, UCS of the treated soil increased. At 1 day curing period, UCS of the soil increased from 71 kN/m^2 for the natural soil to a maximum value of 929 kN/m^2 at 6 and 4% cement and zeolite respectively, representing 1309% increase in strength. At 7 days curing period, UCS of the soil rose to a maximum value of 1291 kN/m^2 at 6 and 4% cement and zeolite respectively, representing 1818% increase in strength, while at 14 days curing period, UCS of the soil increased to a maximum value of 1574 kN/m^2 at 6 and 4% cement and zeolite respectively, representing 2217% increase in strength. At 28 days curing period, UCS of the soil increased to a maximum value of 1634 kN/m^2 at 6 and 4% cement and zeolite respectively, representing 2301% increase in strength, while at 60 days curing period, UCS of the soil increased to a maximum value of 1746 kN/m^2 at 6 and 4% cement and zeolite respectively, representing 2459% increase in strength. This tremendous increase in UCS of the treated soil, with increase in curing period is attributed to the progress of hydration reaction of cement and the pozzolanic reaction of the zeolite.

Addition of zeolite resulted in additional increase in the UCS. This increase in UCS with introduction of zeolite, resulted from the pozollanic reaction between the lime (CaOH) liberated from the hydration reaction of the cement and the pozollanic zeolite to form secondary cementitious materials in the soil matrix (Osinubi and Medubi, 1997). This is primarily due to the formation of various compounds such as calcium silicate hydrates (C-S-H) and calcium aluminate hydrates (C-A-H) and micro fabric changes, which are responsible for the strength development (Negi *et al*., 2013), that peaks at 6% zeolite.

Since cement hydration, with subsequent liberation of Ca(OH) is time dependent, effect of pozzolanic reaction of the zeolite will also be time dependent as observed in the results. The drop in strength after 6% zeolite content may be attributed to excess amounts of SiO_2, Fe_2O_3 and Al_2O_3, from zeolite in the presence of less liberated by-products of cement hydration reaction (lime). This excessive SiO_2, Fe_2O_3 and Al_2O_3 (from zeolite), with less available Ca(OH) within the matrices of the mixtures, created barrier that hinders bonding of the soil particles by the formed cementitious materials, resulting to decrease in strength. This is expected because of the cementitious calcium silicate hydrate (C-S-H) formed on addition of water to the mixture of clay, cement and zeolite. The optimal mixture that gave the highest UCS value was observed at 6% zeolite, which is just sufficient to react with the lime (in cement) and the calcium hydroxide liberated from cement hydration.

From the results, it is observed that, at 6% zeolite content, it is possible to achieve UCS values above the 1710 kN/m^2 recommended by TRRL (1977) and 1750 kN/m^2 reported by Khanna and Justo (2011), which is regarded as criteria for design cement content, considered adequate for soil-cement composite for road base.

4.4 Effect of Zeolite on Stiffness of the Cement Stabilized Clay Soil

The stress-strain behavior of the soil treated with specific cement and zeolite contents are presented in Figures 4.6 to 4.9. Figure 4.6 shows the stress-strain relationship for samples containing 0% cement with varying zeolite content. It revealed a gradually prolonged failure with higher strain at failure of between of 2% and above. At 2% cement content (Figure 4.7), the strain at failure was observed to have reduced to between 1.1 – 1.5% with those at 0 and 3% zeolite content having higher strain values of 1.5% and 6, 9, 12 and 15% zeolite content having low values of 1.1% strain. These trends probably resulted from the hydration or ion exchange reaction of cement that liberated calcium hydroxide as by-product. This reaction causes flocculation of clay minerals to larger particles thus reducing the strain at failure. The trend is similar for 4% cement addition with strain at failure occurring at 0.8%.

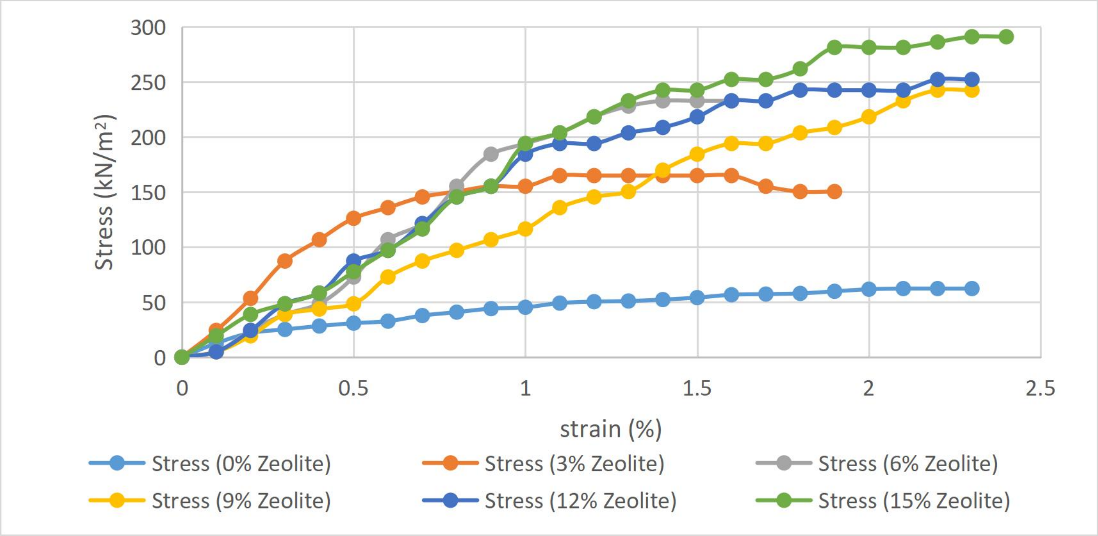


Figure 4.6: Stress-Strain relationship graph for 0% Cement with varied composition of zeolite

This trend differs for 6% cement content, where the strain at failure extended marginally to above 1.25%. This may be attributed to the higher amount of cement and pozzolanic zeolite whose reaction is probably persisting, resulting to delay in the agglomeration of clay particles result to brittleness in the mixture.

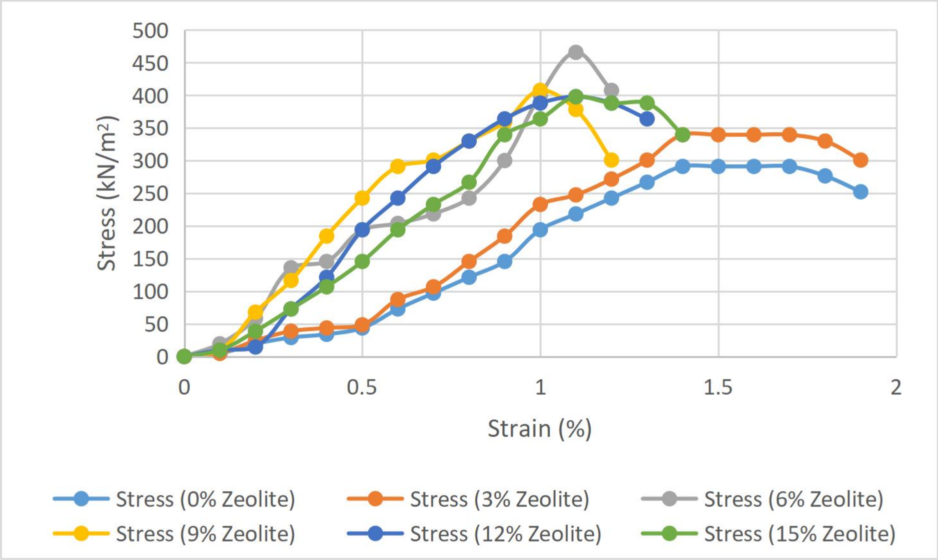


Figure 4.7: Stress-Strain relationship graph for 2% Cement with varied composition of zeolite

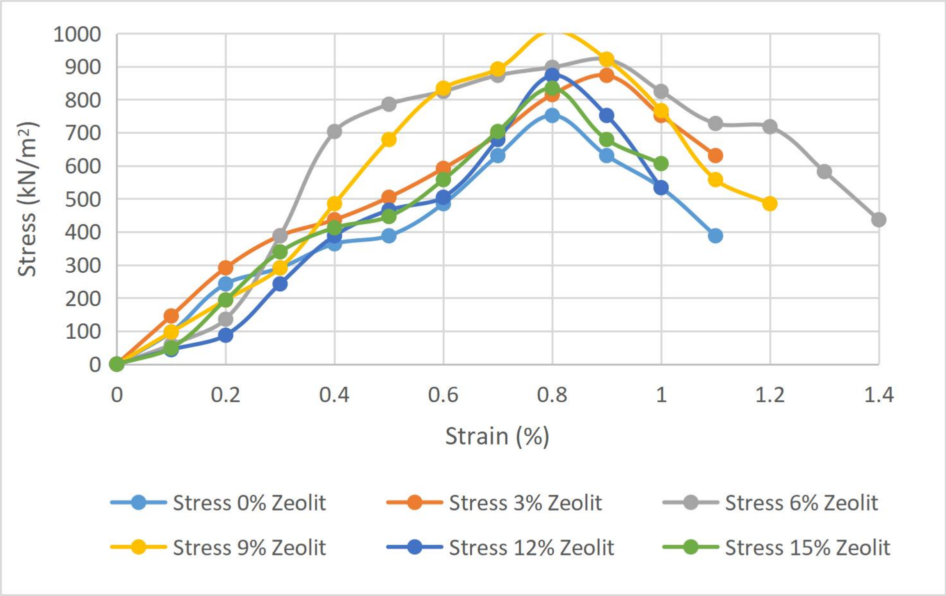


Figure 4.8: Stress-Strain relationship graph for 4% Cement with varied composition of zeolite

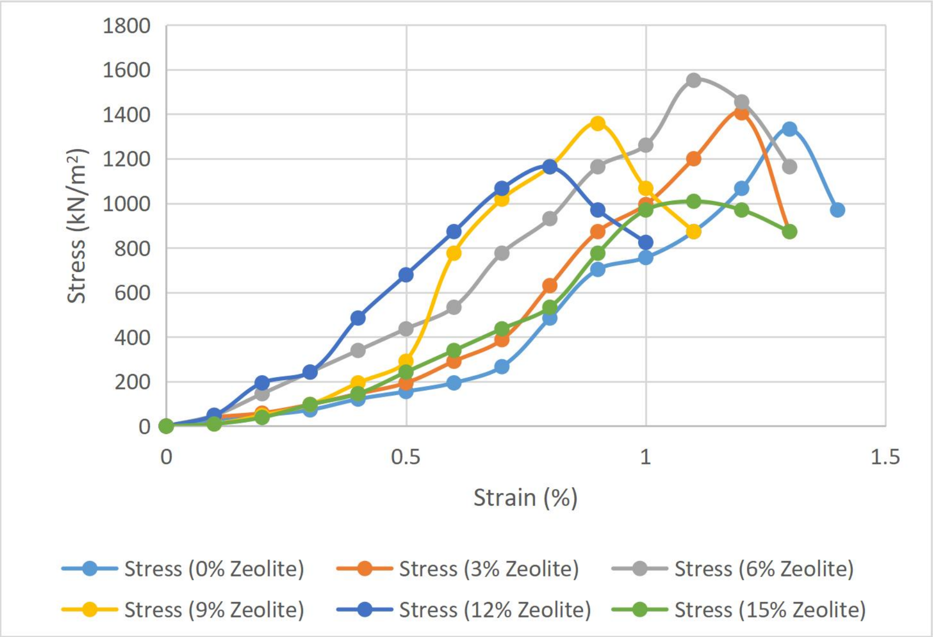


Figure 4.9: Stress-strain relationship graph for 6% cement with varied composition of zeolite

The modulus of elasticity for specimen with 6% zeolite is observed to increase from 211MPa for 0% cement content through 414MPa for 2% cement, to 2850MPa at 4% cement content and finally 2,857MPa at 6% cement content.

4.5 Microstructural Analysis

4.5.1 X- ray diffraction (XRD) analysis

The XRD result for the untreated clay, clay treated with 6% cement, clay treated with 6% zeolite, and Clay treated with 6% cement and 6% zeolite are shown in the Figures 4.11 to 4.13.

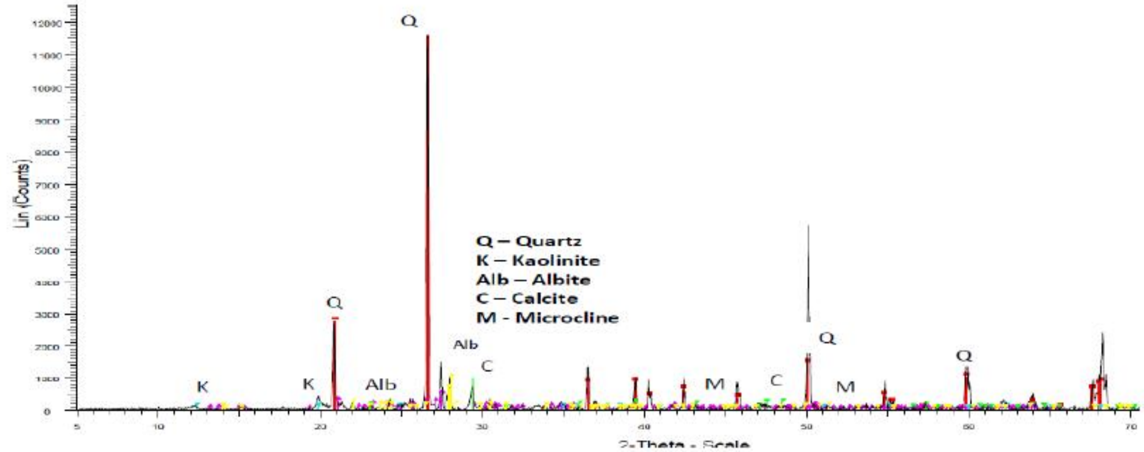


Figure 4.10: X- Ray Diffraction (XRD) of untreated clay

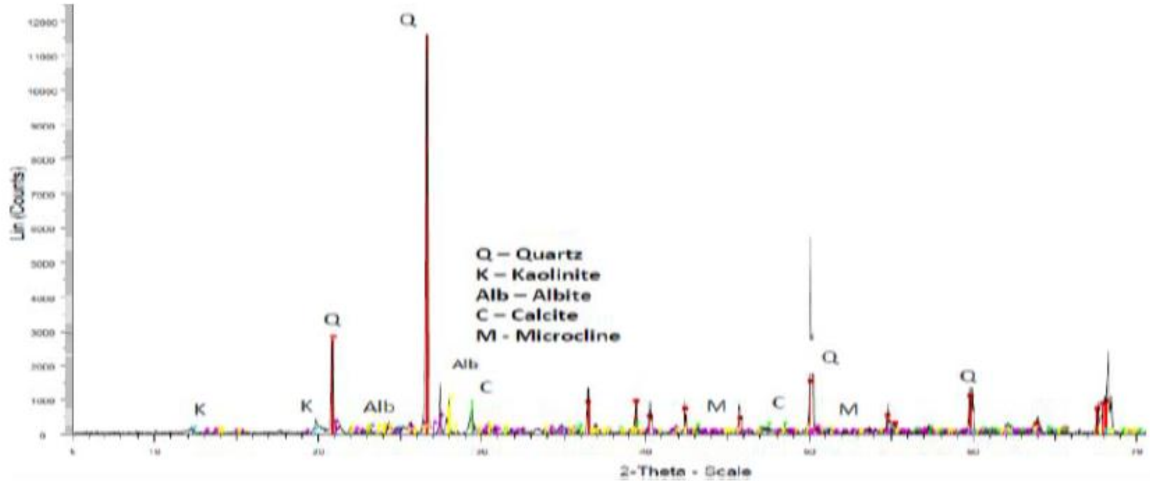


Figure 4.11: X- Ray Diffraction (XRD) of Clay treated with 6% cement

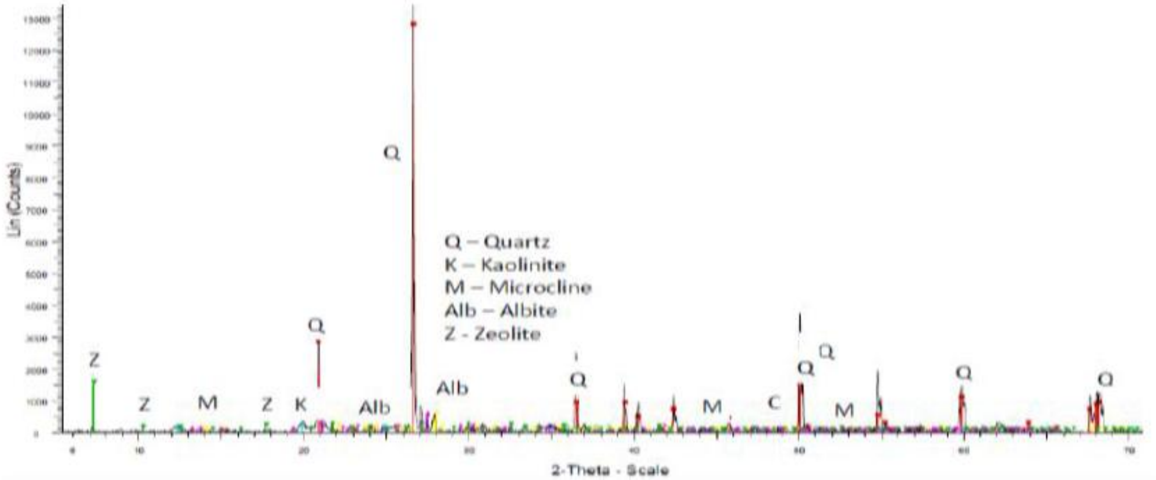


Figure 4.12: X- Ray Diffraction (XRD) of Clay treated with 6% cement

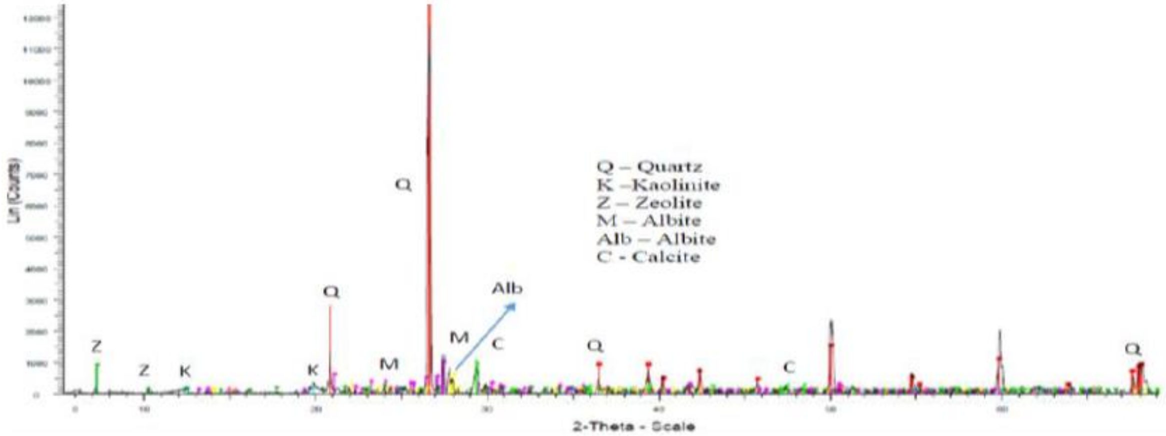


Figure 4.13: X- Ray Diffraction (XRD) of Clay treated with 6% cement

From the figures, the untreated clay consists predominantly of albite, montmorillonite, anorthoclase, quartz, ankerite, calcium silicide and orthoclase as showed in Figure 4.11. When 6% cement was added to the soil, the montmorillonite mineral was disintegrated by the cement, to form kaolinite mineral which is less reactive. The predominant minerals present includes; quarts (SiO_2), kaolinite, Albite and a formation of microcline. Orthoclase was recorded as microcline as presented in Figure 4.12. This process must have resulted from the cation exchange between the lime contained in the cement and ions around the montmorillonite minerals. Appearance of calcite among the resulting minerals resulted from the constituent of cement.

Addition of zeolite to the soil gave similar reaction to that of cement, except that zeolite mineral was recorded among the resultant minerals as shown in Figure 4.13. Albite mineral was observed to be a hard mineral which cannot easily be disintegrated into other minerals. Sodium ions in the zeolite must have contributed to the disintegration of the montmorillonite minerals due to exchange in ions. Addition of both 6% cement and 6% zeolite introduced both zeolite and calcite in the resultant minerals. This combined ionic exchange will result to agglomeration of clay particles to larger particles, which will increase workability and strength gain of the clay soil (Ali *et al.*, 2017).

4.5.2 Energy dispersive spectroscopy (EDS) and Scanning electron microscopy

The EDS allows the amount of the main elements in a sample to be quantified, SEM is a test process that scans a sample with an electron beam to produce a magnified image for analysis, this method is useful in microanalysis. The morphology of the untreated clay specimen and clay mixed with cement, zeolite and combination of cement and zeolite is presented in Figures 4.14 to 4.21. The natural clay, shown in Figure 4.14 revealed whitish flaky clay particles.

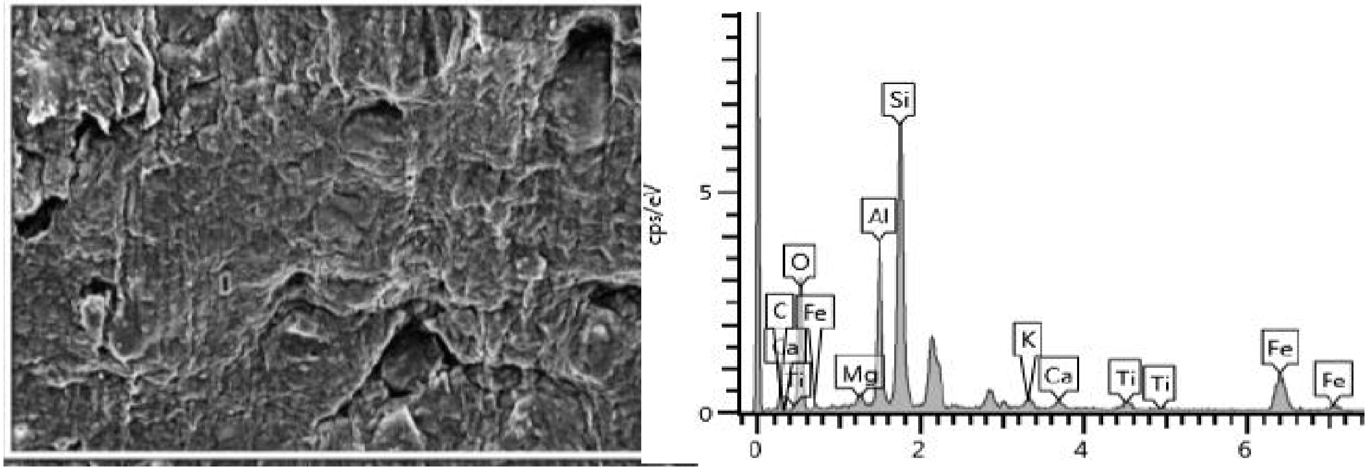


Figure 4.14: SEM for Natural Clay

Figure 4.15: EDS Result for Natural Clay

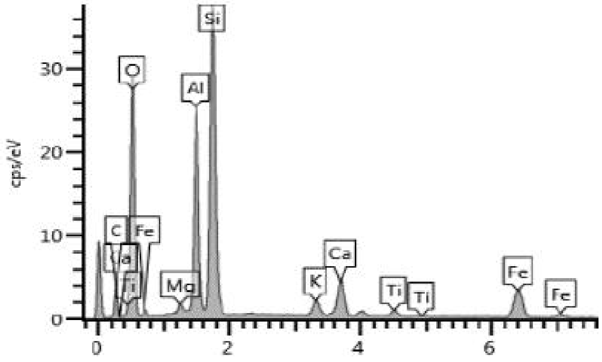
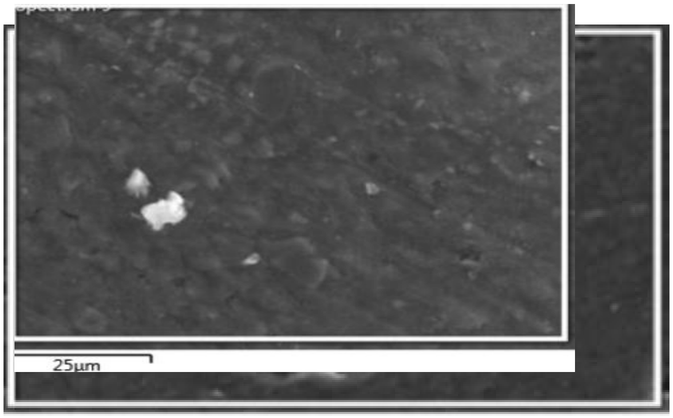


Figure 4.16: SEM for Clay Mixed with Cement

Figure 4.17: EDS Result for Clay Mixed with Cement



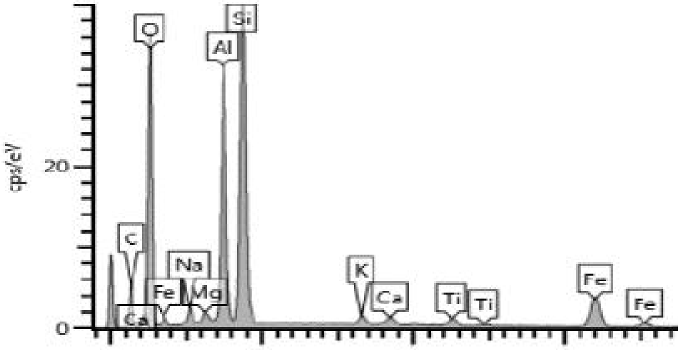
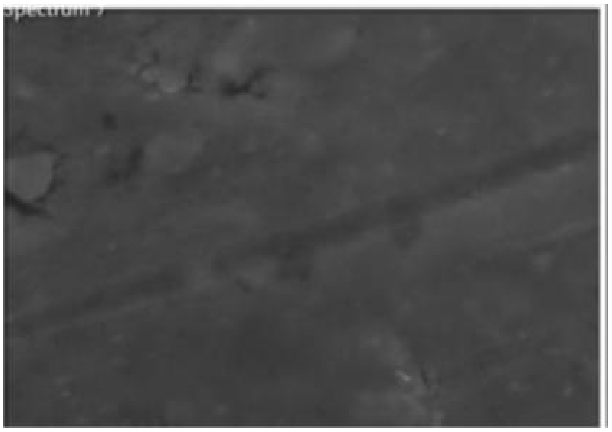


Figure 4.18: SEM for Clay Mixed with Zeolite

Figure 4.19: EDS Result for Clay Mixed with Zeolite



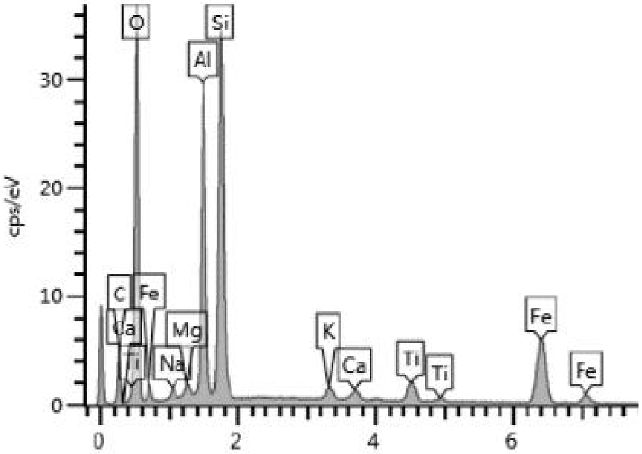


Figure 4.20: SEM for Clay Mixed with cement and zeolite zeolite

Figure 4.21: EDS Result for Clay Mixed with cement and

The EDS recorded silica to alumina ratio of 1.784 which is close to 2, an indication of presence of the montmorillonite minerals. Addition of 6% cement to the clay presented in Figure 4.16, showed relatively compact, non-porous structure with whitish spots suspected to be calcium hydroxide, a byproduct of cement hydration. The silica-alumina ratio of this mixture was 1.558 which is an indication of the start of the process of disintegration of montmorillonite mineral.

Addition of zeolite to the clay revealed similar compact structure, but relatively loose with occasional particulate pores. The silica-alumina ratio recorded for the mixture was 1.381. Addition of 6% cement and 6% zeolite to the clay showed a very compact and smooth structure with very few cracks. The silica-alumina ratio of 1.216 was recorded. This ratio is the minimal from the four mixtures. This justifies the higher UCS values recorded at this composition.

CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

From the study, the following conclusions are drawn:

The soil was classified under CH and A-7-6, according to Unified Soil Classification System (USCS) and American Association for Highway and Transportation Officials (AASHTO) respectively.

Unconfined compressive strength was observed to increase as cement content increase, while at constant cement content, UCS increased to maximum values at 6% zeolite content, after which the strength decreased. This indicates that 6% zeolite content is the optimal.

Clay of high plasticity can be treated with 6% zeolite and less cement content, to satisfy the 1710 kN/m^2 recommended by TRRL (1977) and 1750 kN/m^2 reported by Khanna and Justo (2011), as criteria for design cement content, considered adequate for soil-cement composite as base course material for highway pavements with light to medium traffic.

Microstructurally, the untreated clay consists predominantly of quartz, anorthoclase, orthoclase, ankerite, montmorillonite, silicide and albite. Addition of 6% cement and 6% zeolite to the clay caused ion exchange with the montmorillonite clay mineral thus, converting the mineral to less active kaolinite mineral with introduction of calcite mineral from cement and zeolite mineral from zeolite.

The silica-alumina ratio recorded from EDS results revealed value of 1.784 for untreated clay which reduces to 1.558 for clay mixed with cement. The values further reduced to

1.381 for clay mixed with 6% zeolite and finally to 1.216 for clay mixed with both 6% cement and 6% zeolite.

The SEM result of the specimen of clay mixed with both 6% cement and 6% zeolite showed smooth but compact structure, which justifies the higher UCS value recorded.

5.2 Recommendations

From the study, the following recommendations are drawn:

1. Clay of high plasticity can be treated with 6% zeolite and 4% cement content, to satisfy the 1710 kN/m^2 recommended by TRRL (1977) and 1750 kN/m^2 reported by Khanna and Justo (2011), as criteria for design cement content, considered adequate for soil-cement composite as base course material for highway pavements with light to medium traffic.

2. 6% zeolite can be effectively used with cement for treatment of problematic soils.

5.3 Contribution to Knowledge

The study established that treatment of tropical clay with (4%) cement and 6% zeolite resulted to an Unconfined Compressive Strength (UCS) value that met the 1710 or 1750 kN/m^2, considered adequate for soil-cement composite base course material for road pavements of light to medium traffic.

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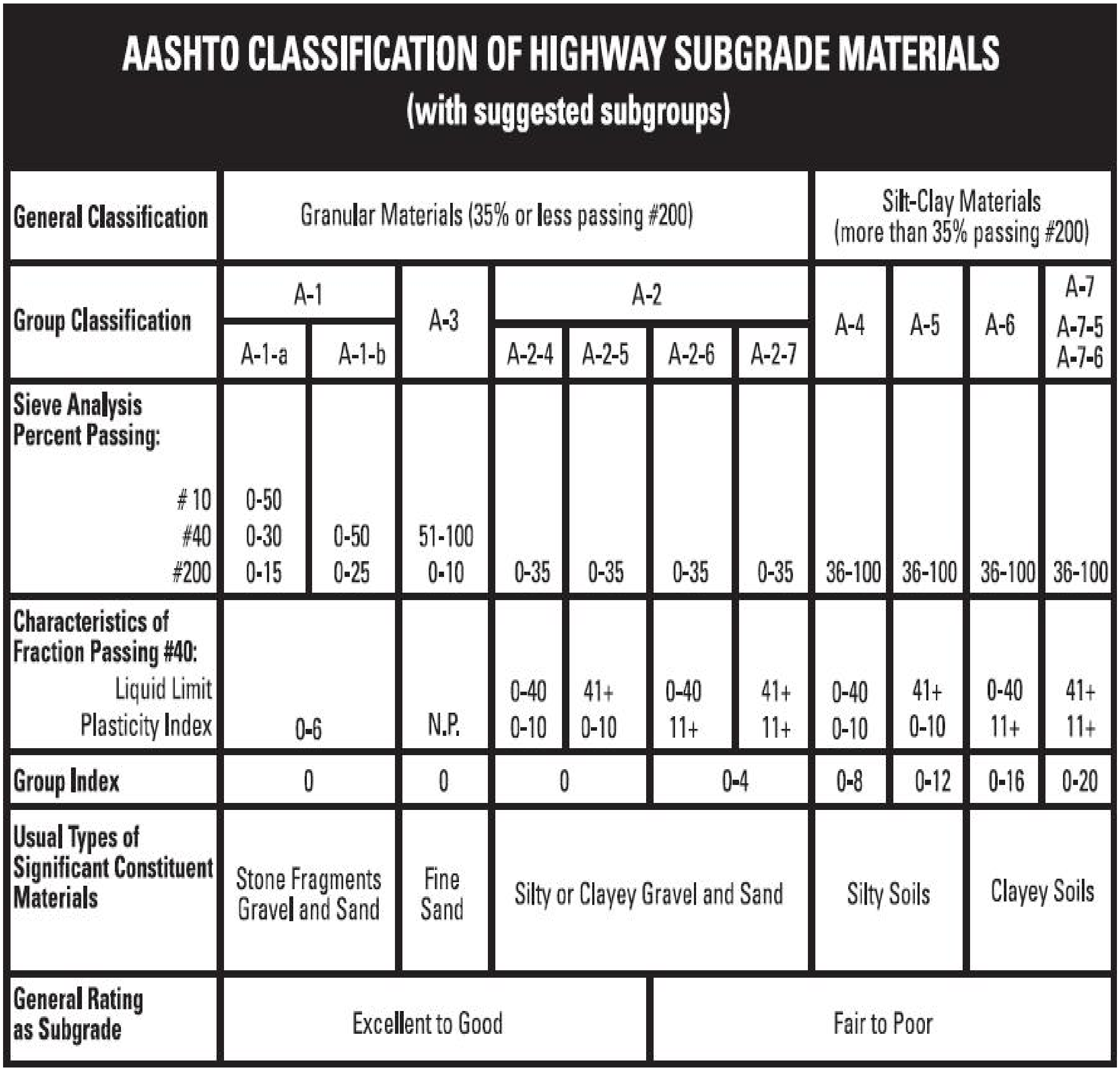
APPENDICES

Appendix A: AASHTO classification table



AASHTO CLASSIFICATION OF HIGHWAY SUBGRADE MATERIALS

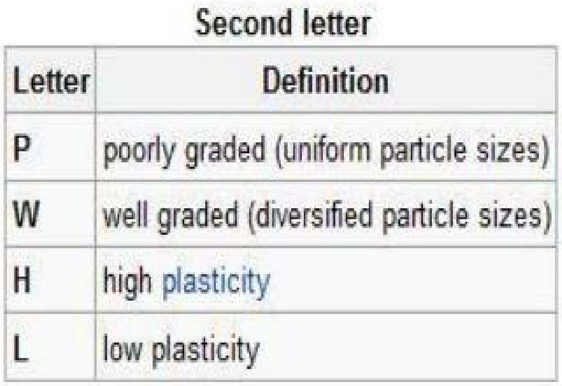
(with suggested subgroups)

General Classification	Granular Materials (35% or less passing #200)							Silt-Clay Materials (more than 35% passing #200)			
Group Classification	A-1		A-3	A-2				A-4	A-5	A-6	A-7
	A-1-a	A-1-b		A-2-4	A-2-5	A-2-6	A-2-7				A-7-5 A-7-6
Sieve Analysis Percent Passing:											
# 10	0-50										
#40	0-30	0-50	51-100								
#200	0-15	0-25	0-10	0-35	0-35	0-35	0-35	36-100	36-100	36-100	36-100
Characteristics of Fraction Passing #40:											
Liquid Limit				0-40	41+	0-40	41+	0-40	41+	0-40	41+
Plasticity Index	0-6		N.P.	0-10	0-10	11+	11+	0-10	0-10	11+	11+
Group Index	0		0	0		0-4		0-8	0-12	0-16	0-20
Usual Types of Significant Constituent Materials	Stone Fragments Gravel and Sand		Fine Sand	Silty or Clayey Gravel and Sand				Silty Soils		Clayey Soils	
General Rating as Subgrade	Excellent to Good					Fair to Poor					

APPENDIX B: UNIFIED Classification Table (Consistency Limit)

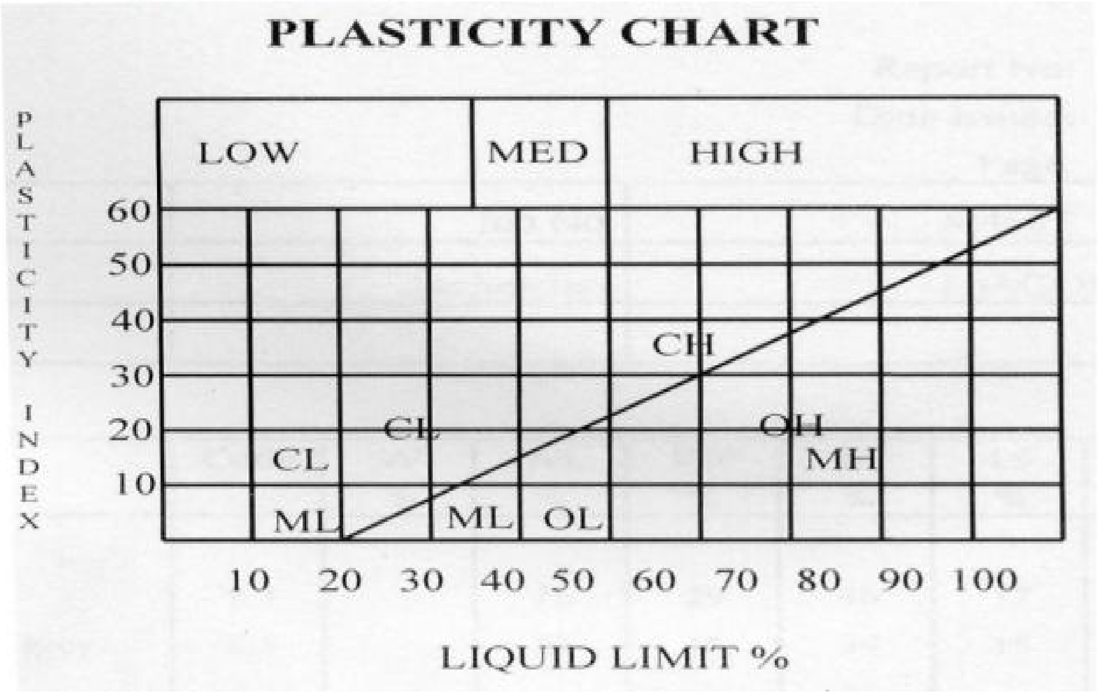
First and/or second letters

Symbol	Definition
G	gravel
S	sand
M	silt
C	clay
O	organic

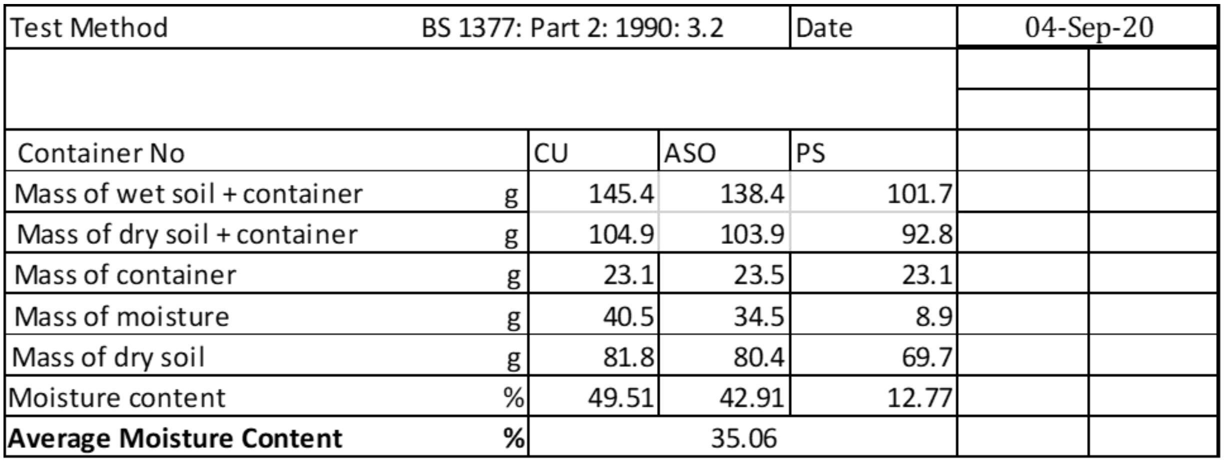


Second letter

Letter	Definition
P	poorly graded (uniform particle sizes)
W	well graded (diversified particle sizes)
H	high plasticity
L	low plasticity

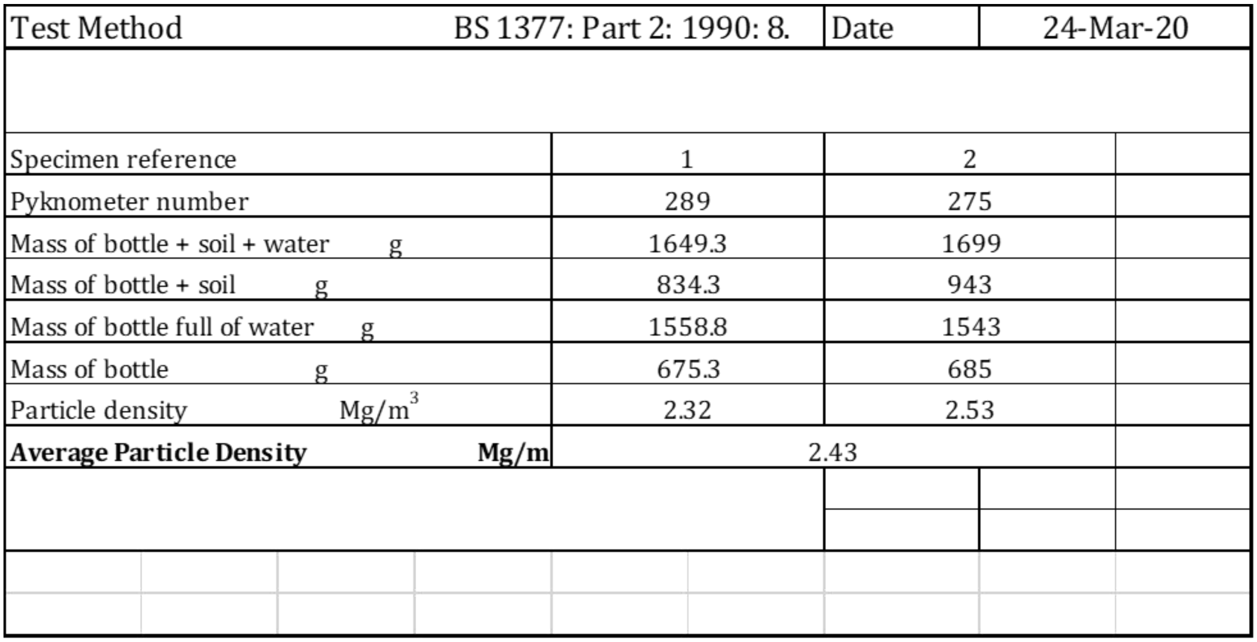


Appendix C: Natural Moisture Content



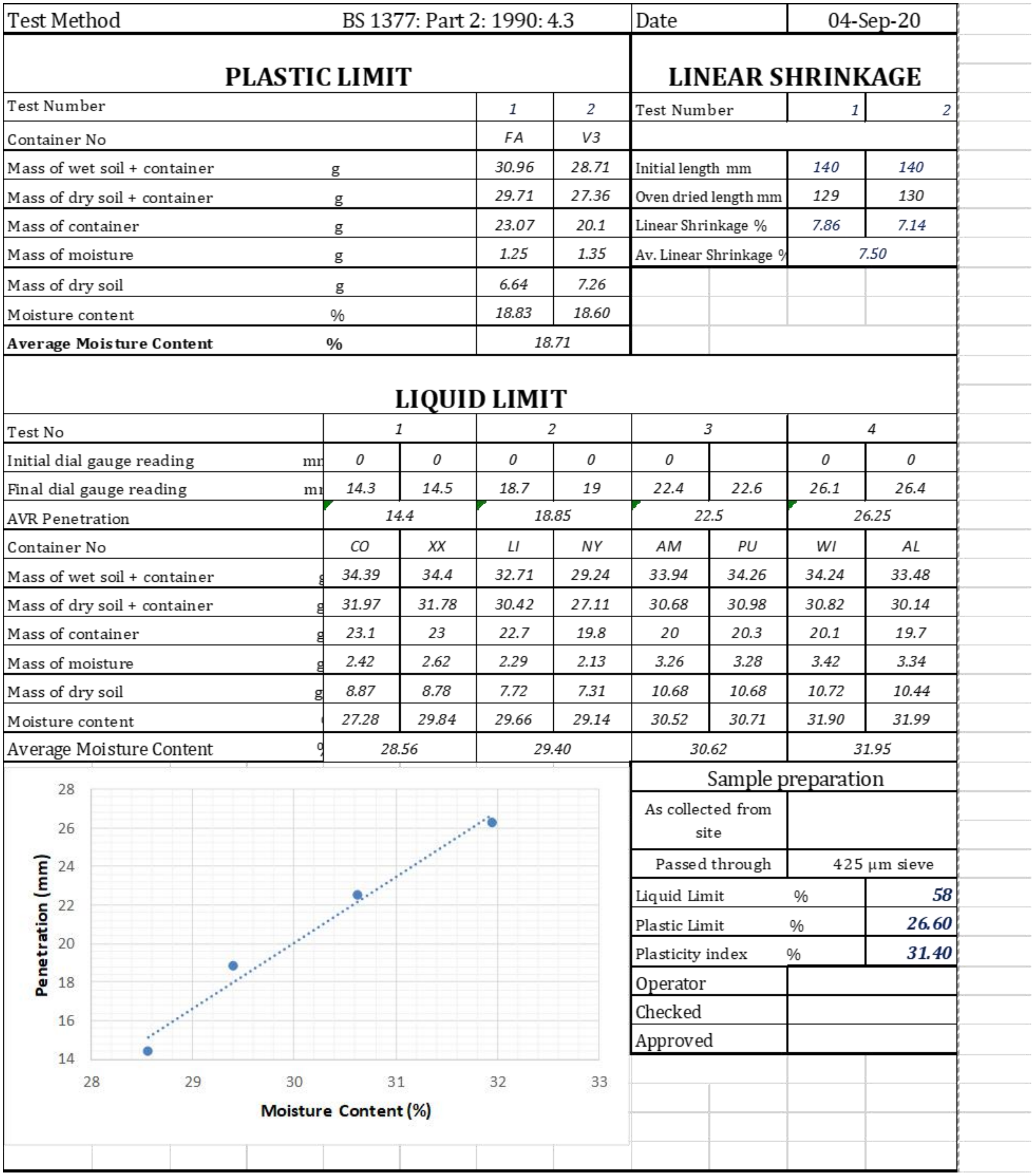
Test Method	BS 1377: Part 2: 1990: 3.2			Date	04-Sep-20	
Container No		CU	ASO	PS		
Mass of wet soil + container	g	145.4	138.4	101.7		
Mass of dry soil + container	g	104.9	103.9	92.8		
Mass of container	g	23.1	23.5	23.1		
Mass of moisture	g	40.5	34.5	8.9		
Mass of dry soil	g	81.8	80.4	69.7		
Moisture content	%	49.51	42.91	12.77		
Average Moisture Content	**%**	35.06				

Appendix D: Specific Gravity Test



Test Method	BS 1377: Part 2: 1990: 8.	Date	24-Mar-20
Specimen reference	1	2	
Pyknometer number	289	275	
Mass of bottle + soil + water g	1649.3	1699	
Mass of bottle + soil g	834.3	943	
Mass of bottle full of water g	1558.8	1543	
Mass of bottle g	675.3	685	
Particle density Mg/m^3	2.32	2.53	
Average Particle Density Mg/m	2.43		

Appendix E: Atterberg limit Test



Test Method	BS 1377: Part 2: 1990: 4.3	Date	04-Sep-20

PLASTIC LIMIT

Test Number		1	2
Container No		FA	V3
Mass of wet soil + container	g	30.96	28.71
Mass of dry soil + container	g	29.71	27.36
Mass of container	g	23.07	20.1
Mass of moisture	g	1.25	1.35
Mass of dry soil	g	6.64	7.26
Moisture content	%	18.83	18.60
Average Moisture Content	**%**	18.71	

LINEAR SHRINKAGE

Test Number	1	2
Initial length mm	140	140
Oven dried length mm	129	130
Linear Shrinkage %	7.86	7.14
Av. Linear Shrinkage %	7.50	

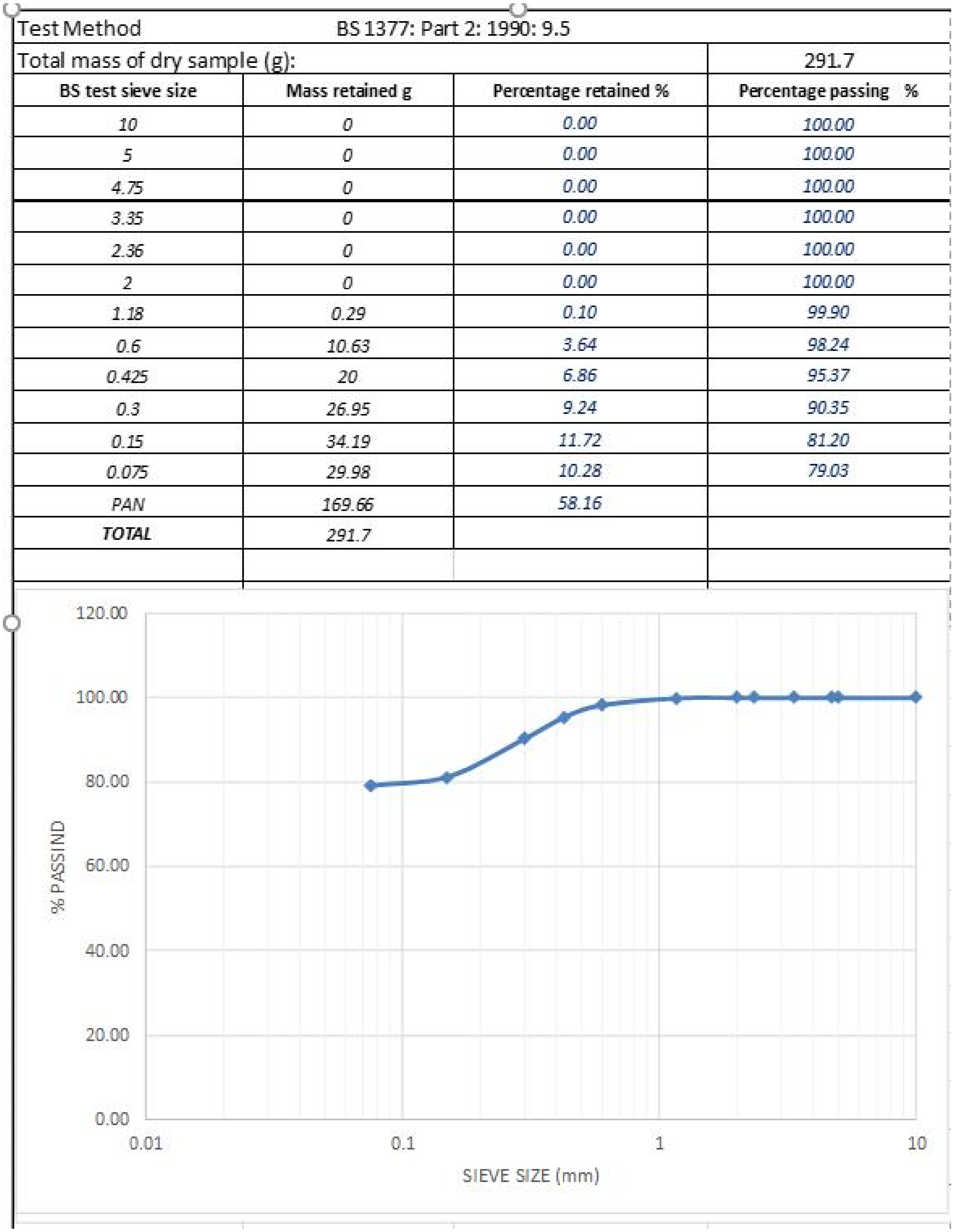
LIQUID LIMIT

Test No		1		2		3		4	
Initial dial gauge reading	mr	0	0	0	0	0		0	0
Final dial gauge reading	mı	14.3	14.5	18.7	19	22.4	22.6	26.1	26.4
AVR Penetration		14.4		18.85		22.5		26.25	
Container No		CO	XX	LI	NY	AM	PU	WI	AL
Mass of wet soil + container	g	34.39	34.4	32.71	29.24	33.94	34.26	34.24	33.48
Mass of dry soil + container	g	31.97	31.78	30.42	27.11	30.68	30.98	30.82	30.14
Mass of container	g	23.1	23	22.7	19.8	20	20.3	20.1	19.7
Mass of moisture	g	2.42	2.62	2.29	2.13	3.26	3.28	3.42	3.34
Mass of dry soil	g	8.87	8.78	7.72	7.31	10.68	10.68	10.72	10.44
Moisture content		27.28	29.84	29.66	29.14	30.52	30.71	31.90	31.99
Average Moisture Content	9	28.56		29.40		30.62		31.95	

Sample preparation

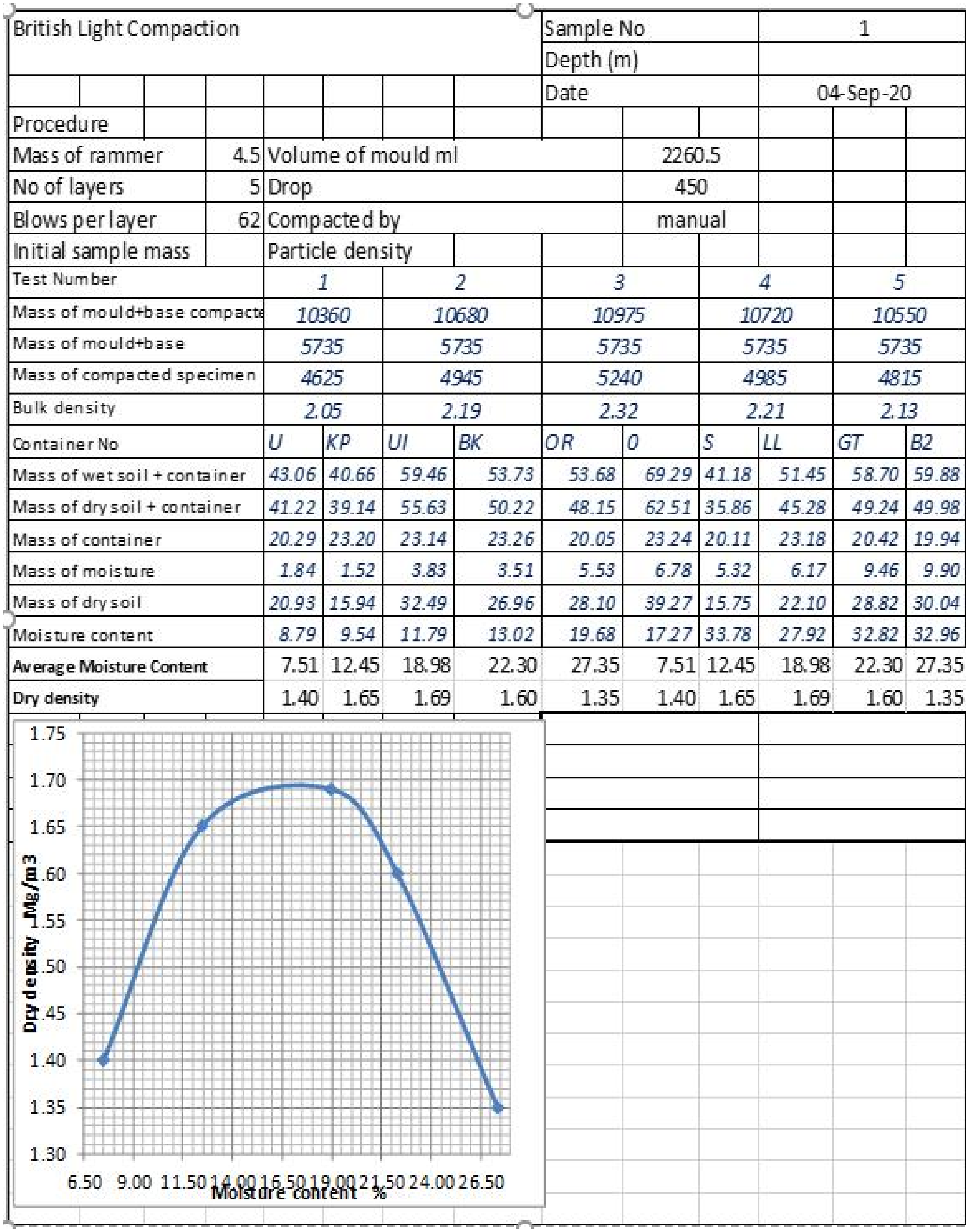
As collected from site		
Passed through	425 µm sieve	
Liquid Limit	%	58
Plastic Limit	%	26.60
Plasticity index	%	31.40
Operator		
Checked		
Approved		

Appendix E: Particle Size Distribution



Test Method	BS 1377: Part 2: 1990: 9.5		
Total mass of dry sample (g):			291.7
BS test sieve size	**Mass retained g**	**Percentage retained %**	**Percentage passing %**
10	0	0.00	100.00
5	0	0.00	100.00
4.75	0	0.00	100.00
3.35	0	0.00	100.00
2.36	0	0.00	100.00
2	0	0.00	100.00
1.18	0.29	0.10	99.90
0.6	10.63	3.64	98.24
0.425	20	6.86	95.37
0.3	26.95	9.24	90.35
0.15	34.19	11.72	81.20
0.075	29.98	10.28	79.03
PAN	169.66	58.16	
TOTAL	291.7		

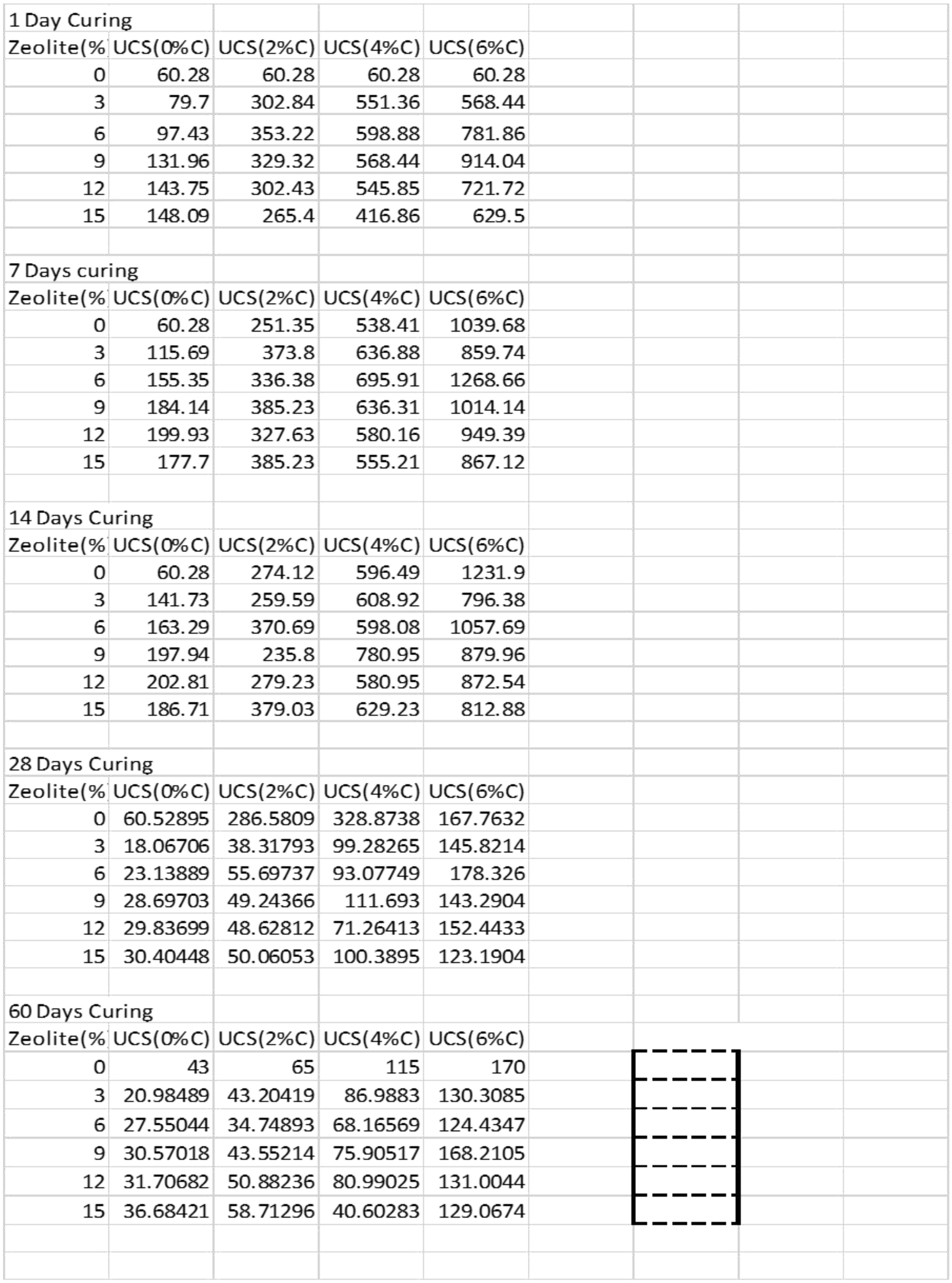
Appendix F: Moisture Density Relationship (Compaction)



British Light Compaction		Sample No	1
		Depth (m)	
		Date	04-Sep-20
Procedure			
Mass of rammer	4.5	Volume of mould ml	2260.5
No of layers	5	Drop	450
Blows per layer	62	Compacted by	manual
Initial sample mass		Particle density	

Test Number	1		2		3		4		5	
Mass of mould+base compacte	10360		10680		10975		10720		10550	
Mass of mould+base	5735		5735		5735		5735		5735	
Mass of compacted specimen	4625		4945		5240		4985		4815	
Bulk density	2.05		2.19		2.32		2.21		2.13	
Container No	U	KP	UI	BK	OR	0	S	LL	GT	B2
Mass of wet soil + container	43.06	40.66	59.46	53.73	53.68	69.29	41.18	51.45	58.70	59.88
Mass of dry soil + container	41.22	39.14	55.63	50.22	48.15	62.51	35.86	45.28	49.24	49.98
Mass of container	20.29	23.20	23.14	23.26	20.05	23.24	20.11	23.18	20.42	19.94
Mass of moisture	1.84	1.52	3.83	3.51	5.53	6.78	5.32	6.17	9.46	9.90
Mass of dry soil	20.93	15.94	32.49	26.96	28.10	39.27	15.75	22.10	28.82	30.04
Moisture content	8.79	9.54	11.79	13.02	19.68	17.27	33.78	27.92	32.82	32.96
Average Moisture Content	7.51	12.45	18.98	22.30	27.35	7.51	12.45	18.98	22.30	27.35
Dry density	1.40	1.65	1.69	1.60	1.35	1.40	1.65	1.69	1.60	1.35

Appendix G: Unconfined Compressive Strength



1 Day Curing

Zeolite(%	UCS(0%C)	UCS(2%C)	UCS(4%C)	UCS(6%C)
0	60.28	60.28	60.28	60.28
3	79.7	302.84	551.36	568.44
6	97.43	353.22	598.88	781.86
9	131.96	329.32	568.44	914.04
12	143.75	302.43	545.85	721.72
15	148.09	265.4	416.86	629.5

7 Days curing

Zeolite(%	UCS(0%C)	UCS(2%C)	UCS(4%C)	UCS(6%C)
0	60.28	251.35	538.41	1039.68
3	115.69	373.8	636.88	859.74
6	155.35	336.38	695.91	1268.66
9	184.14	385.23	636.31	1014.14
12	199.93	327.63	580.16	949.39
15	177.7	385.23	555.21	867.12

14 Days Curing

Zeolite(%	UCS(0%C)	UCS(2%C)	UCS(4%C)	UCS(6%C)
0	60.28	274.12	596.49	1231.9
3	141.73	259.59	608.92	796.38
6	163.29	370.69	598.08	1057.69
9	197.94	235.8	780.95	879.96
12	202.81	279.23	580.95	872.54
15	186.71	379.03	629.23	812.88

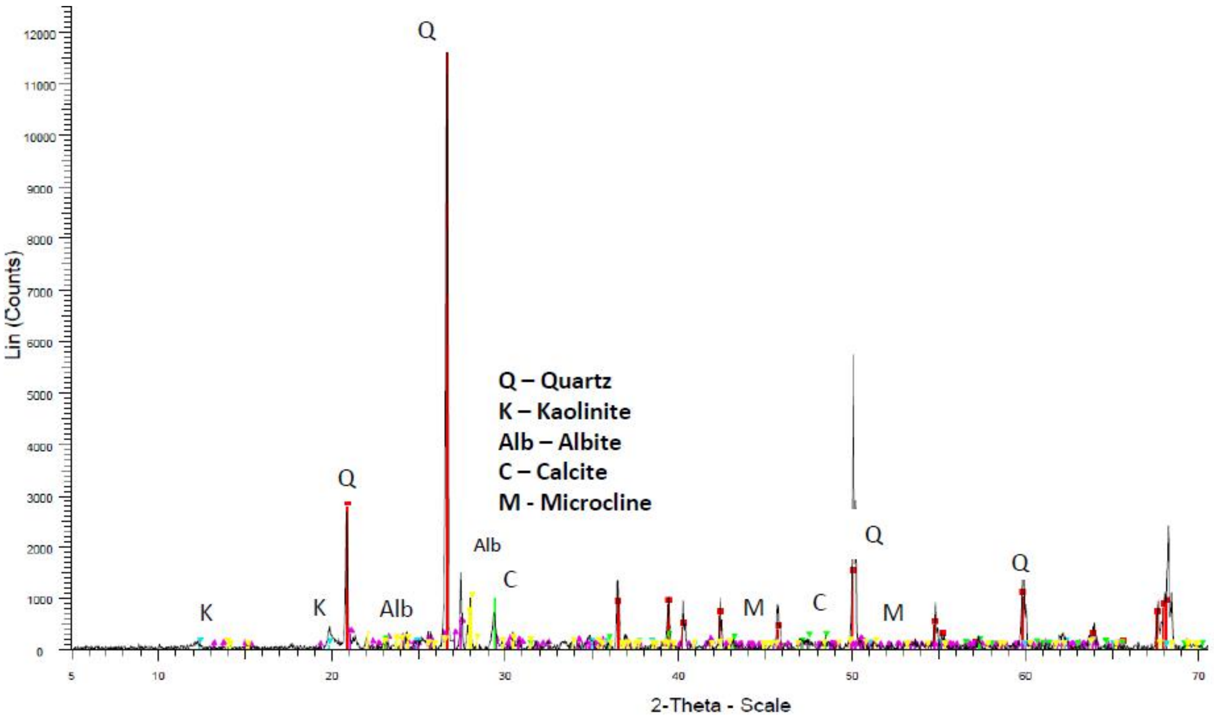
28 Days Curing

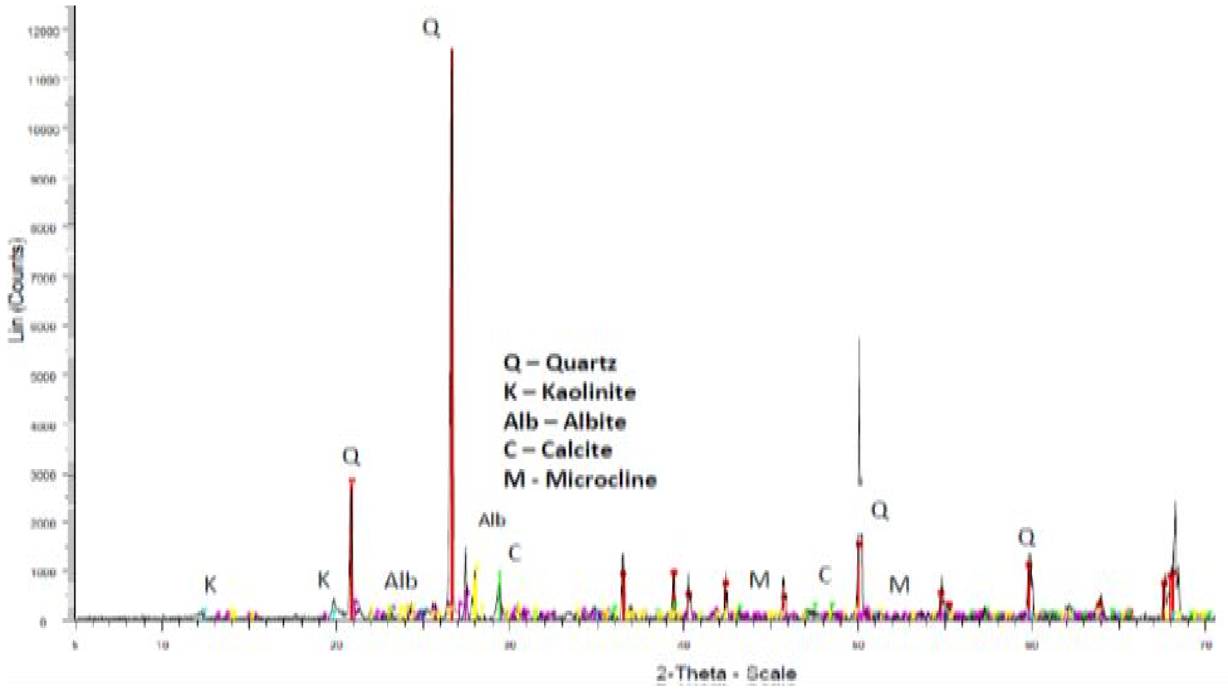
Zeolite(%	UCS(0%C)	UCS(2%C)	UCS(4%C)	UCS(6%C)
0	60.52895	286.5809	328.8738	167.7632
3	18.06706	38.31793	99.28265	145.8214
6	23.13889	55.69737	93.07749	178.326
9	28.69703	49.24366	111.693	143.2904
12	29.83699	48.62812	71.26413	152.4433
15	30.40448	50.06053	100.3895	123.1904

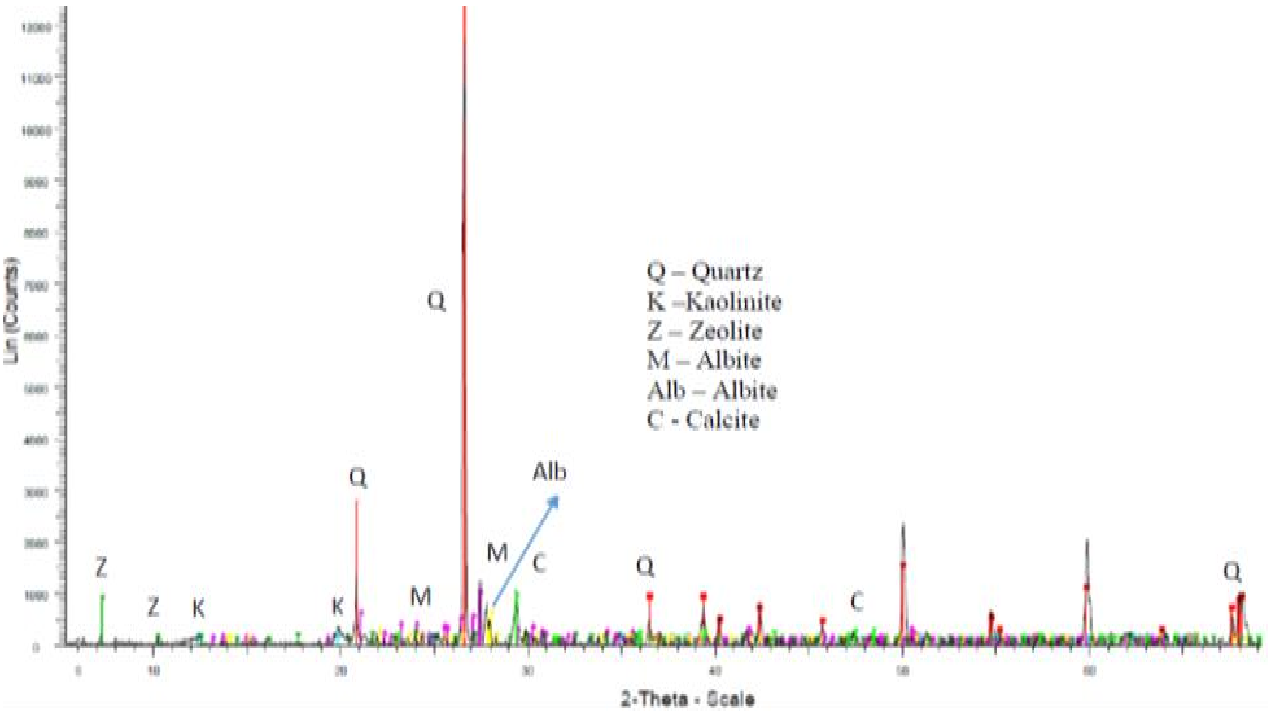
60 Days Curing

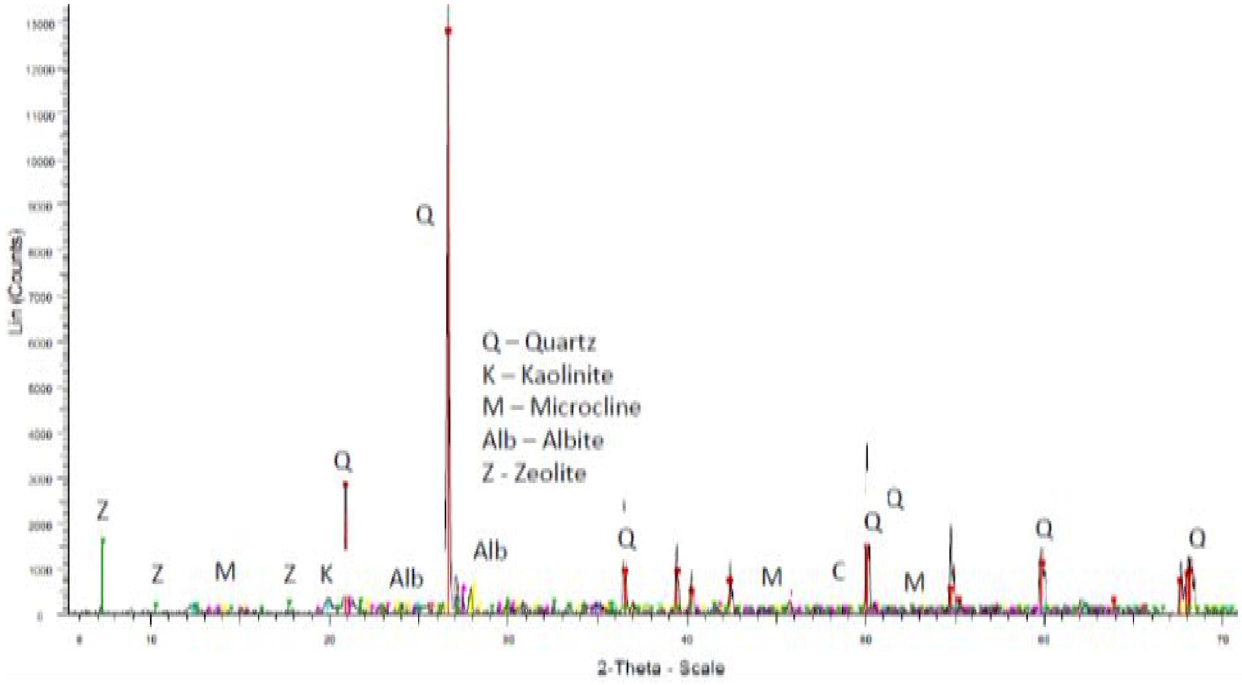
Zeolite(%	UCS(0%C)	UCS(2%C)	UCS(4%C)	UCS(6%C)
0	43	65	115	170
3	20.98489	43.20419	86.9883	130.3085
6	27.55044	34.74893	68.16569	124.4347
9	30.57018	43.55214	75.90517	168.2105
12	31.70682	50.88236	80.99025	131.0044
15	36.68421	58.71296	40.60283	129.0674

Appendix H: X- Ray Diffraction (XRD) Results for Soil Samples

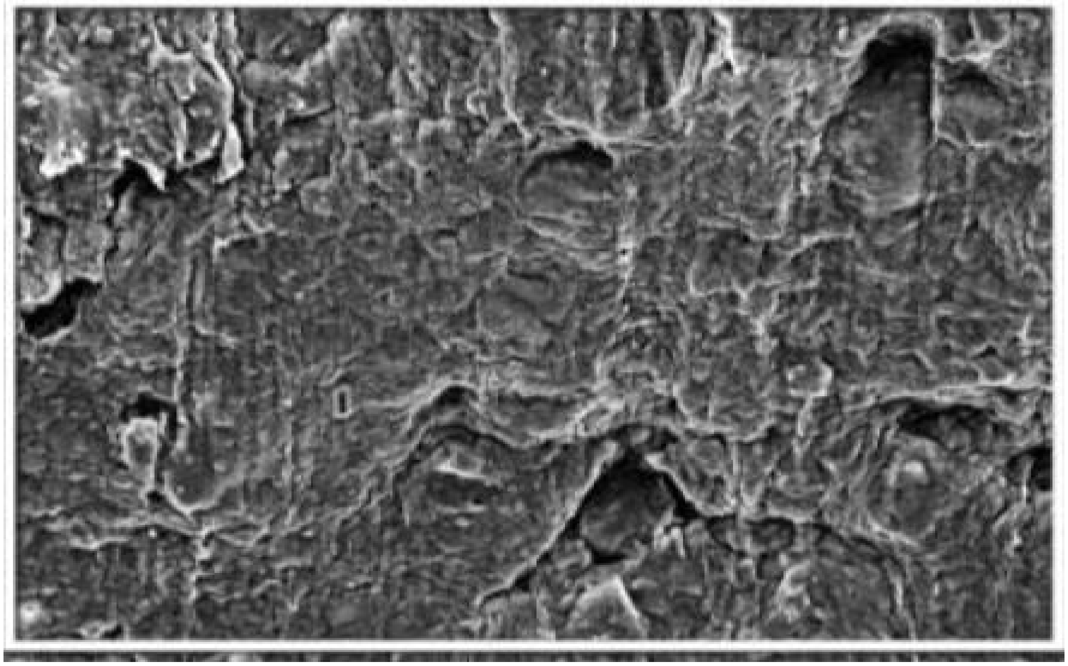




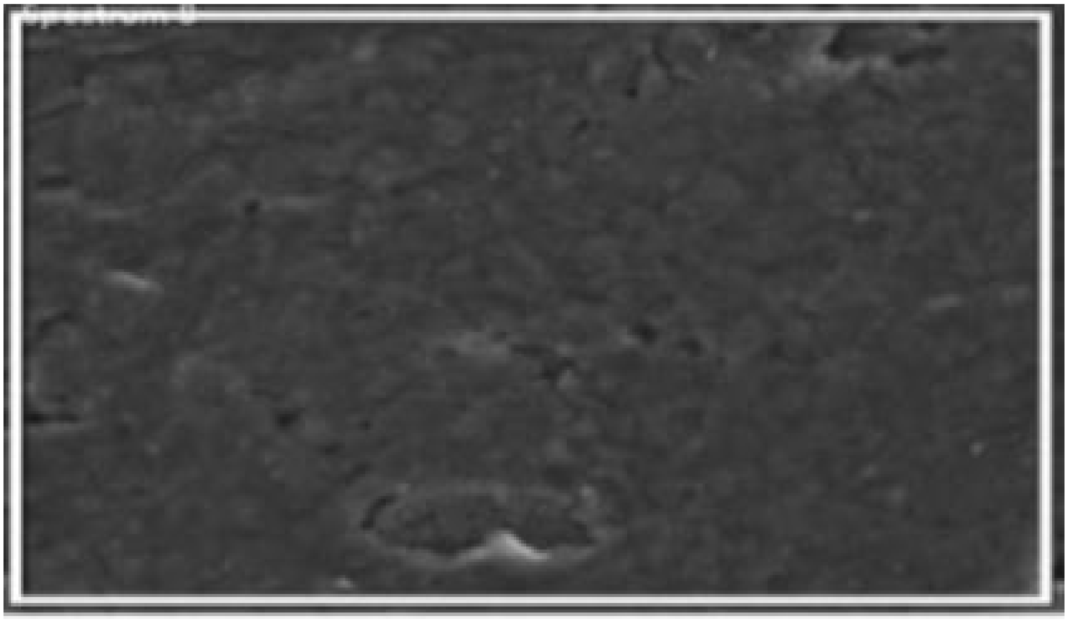




Appendix I: SEM Results for Soil Samples

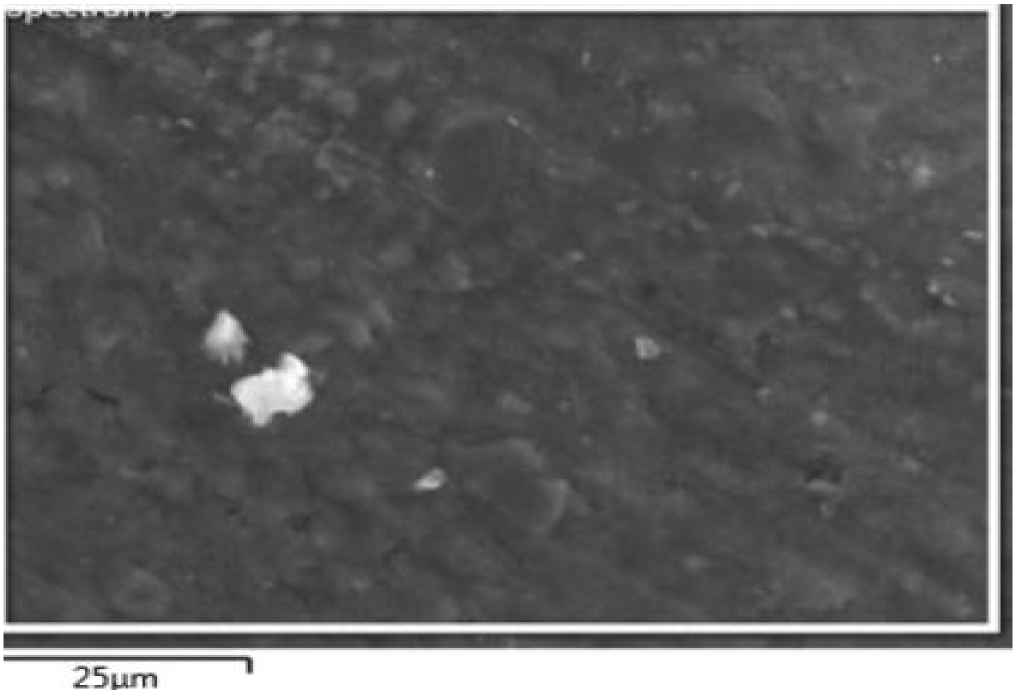


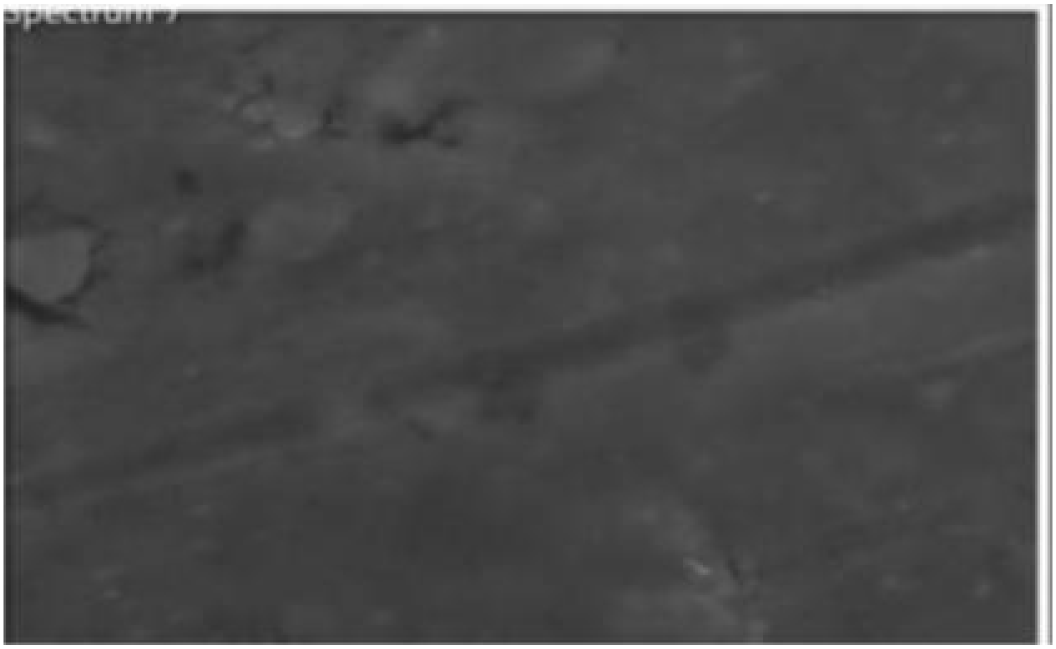
SEM for Natural Clay



SEM for Natural Soil Mixed with Zeolite

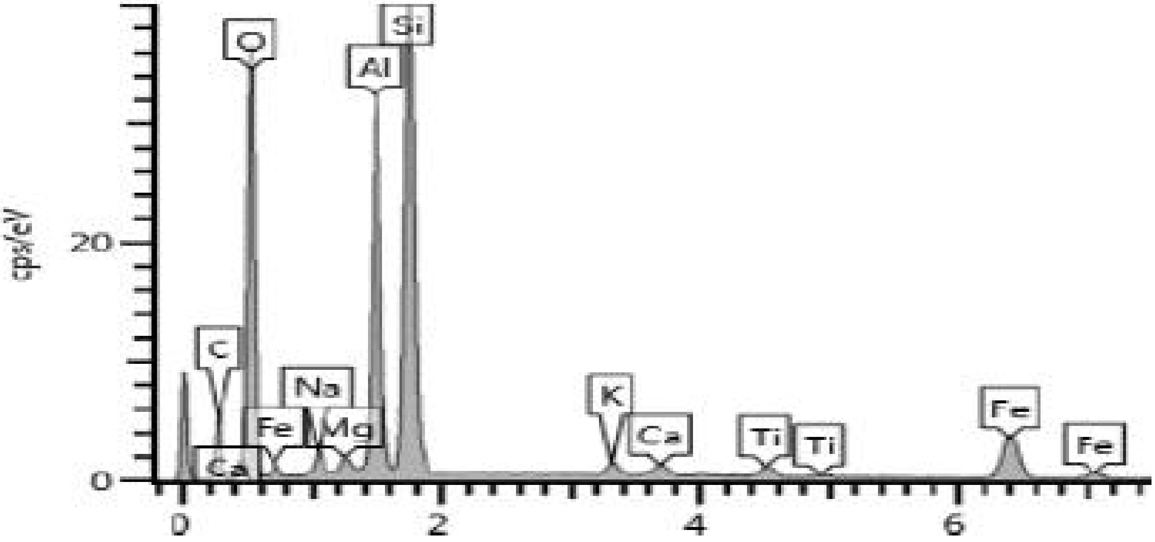
SEM for Natural Soil Mixed with Zeolite and Cement

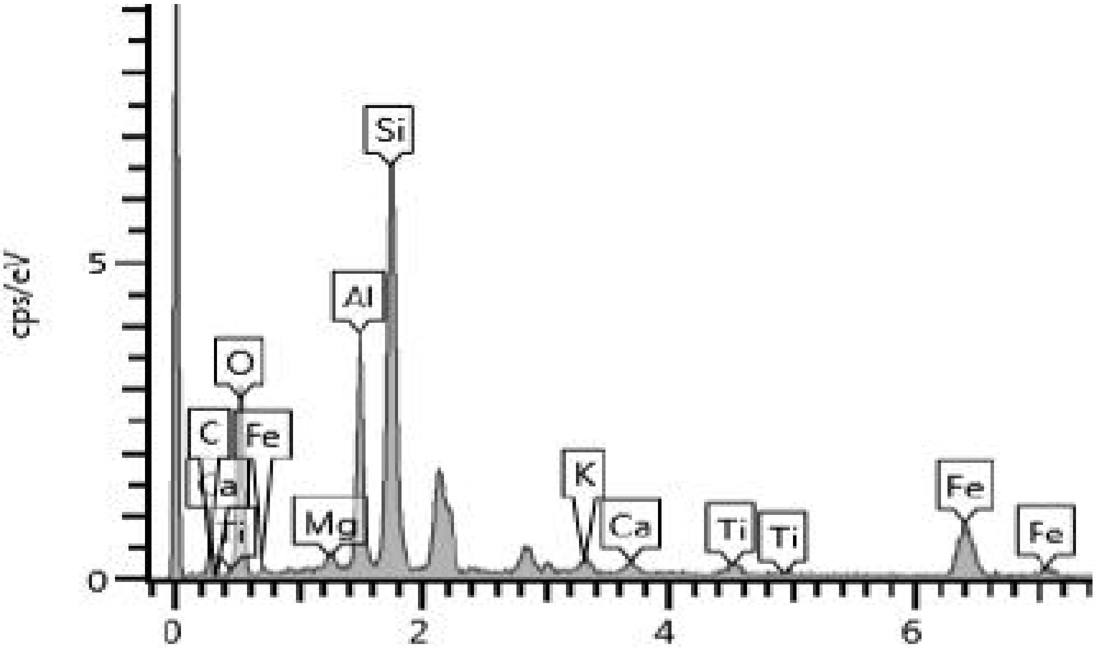




Appendix J: EDS Results

EDS for Natural Soil Mixed with Zeolite





EDS for Natural Soil Mixed with Zeolite and Cement