ANGLIA RUSKIN UNIVERSITY

THE EFFECT OF LIGHT CRUDE OIL CONTAMINATION ON THE GEOTECHNICAL PROPERTIES OF KAOLINITE CLAY SOIL

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Dedication

To Dr (Mrs) Chidinma Ota, Chukwudumaga Brian Ota and Chizara Helen Ota
For the Love and Joy they have brought to my life

And

To Elder (Chief) Nnechi Ejitu Ota and Elder (Mrs) Helen Nnechi Ota
For their Love and Care towards me.
Crude oil is of economic importance to any nation. However its poor management and disposal method has resulted in oil spillage and ground contamination. Most emphasis on crude oil contamination on the environment has been on surface and ground water pollution, as well as plants and animals with little or no attention paid to the engineering properties of the contaminated soil. This research is thus aimed at investigating the effect of light crude oil contamination on the geotechnical properties of kaolinite clay soil. This research is a laboratory based experiment in which the contaminated soil was prepared by adding different percentages of light crude oil (2%, 5%, 8%, 10%, 15% and 20%) measured by weight of the dry soil sample and mixed until a uniform mixture was obtained. The British Standard Test Methods for Civil Engineering purposes BS 1377: 1990 (EUROCODE 7: EN 1997: 2-5) was adopted with few modifications. The calculations were based on the fact that the pore space in the contaminated soil is occupied by water and crude oil so water content was calculated from the fluid content and the rate of evaporation of crude oil during oven drying was also considered.

The classification results showed that crude oil contamination caused an increase in linear shrinkage, liquid limit, plastic limit and plasticity index between 0% to 20% contaminations. The compaction result showed that there was an increase in maximum dry density while the optimum moisture content decreased between 0% and 15% of crude oil contamination. The result showed that the soil could not compact at 20% contamination and above. Also, the coefficient of permeability increased with increase in the percentage of crude oil contamination while the coefficient of consolidation value (Cv) increases with increase in the percentage of contamination. There was a decrease in the cohesion value and the frictional angle due to the introduction of the crude oil into the soil. Although crude oil altered the geotechnical properties of the kaolinite clay soil and reduced its strength, the soil can still be used for geotechnical purposed after remediation.

**Key words:** Light crude oil, Clay soil, Contamination, Geotechnical properties.
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Abbreviations

API gravity - American Petroleum Institute gravity
ASTM- American Society of Testing and Material
BS - British Standard
C - Carbon
CBR – California Bearing ratio
CL – Clay
COSHHA - Control of Substances Hazardous to Health
DNAPLs - Dense Non-Aqueous Phase Liquids
H - Hydrogen
LL – Liquid Limit
LNAPLs - Light Non-Aqueous Phase Liquids
LPG - Liquid Petroleum Gas
MDD - Maximum Dry Density
Mo – Weight of Oil
Ms – Weight of Dry soil
Mw – Weight of Water - Weight of water
NAPLs - Non-Aqueous Phase Liquids
NOSDRA - Nigerian National Oil Spill Detection and Response Agency
OMC - Optimum Moisture Content
PAHs - Polynuclear Aromatic Hydrocarbons
PL – Plastic Limit
SEM - Scanning Electron Micrograph
SM - Silty Sand
SP - Poor Graded Sand

SPSS - Statistical Package for the Social Sciences

UNDP - United Nations Development Program

Wf - Fluid Content

Wo - Oil Content

Ww - Water Content

XRD - X-ray Diffraction

XRF - X-ray Fluorescence
Notations

$\Delta H$ – Settlement

$C$ - Cohesion (kN/m$^2$)

$Cu$ - Apparent cohesion (kN/m$^2$) (Shear strength of a soil when subjected to zero confining pressure)

$Cv$ – coefficient of Consolidation

$e_0$ - initial void ratio

$Gs$ – Specific Gravity

$k$ - Coefficient of Permeability (m/s)

$K$ - Hydraulic Conductivity (m/s)

$MDD$- Maximum Dry Density (Mg/m$^3$)

$Mv$ – Coefficient of Volume Compressibility

$\phi$ – Frictional Angle ($^0$)

$OMC$- Optimum Moisture Content (%)

$P$- Pressure (KN/m$^2$)

$s$ - Degree of Saturation

$Va$ - Air void

$W$ - Moisture Content (%)

$\varepsilon$ - Strain (%)

$\rho$ –Bulk Density (Mg/m$^3$)

$\rho_D$. Dry Density

$\rho_s$ - Particle density

$\sigma$- Compressive Stress

$\sigma_1$ – Major Principal Stress
σ₂ Intermediate Principal Stress

σ₃ – Minor Principal Stress

σ₋₁ – Major Effective Principal Stress

σ₋₂ Intermediate Effective Principal Stress

σ₋₃ – Minor Effective Principal Stress

(σ₋₁ – σ₋₃) – Deviator Stress
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CHAPTER 1: INTRODUCTION

1.1. Oil spills and ground contamination

Crude oil is of significant economic importance to the development of any nation in terms of energy generation, fuel, employment and as a source of raw materials for petrochemical industries. However, the exploration, exploitation and production of this crude oil may cause oil spills which contaminate the soil. The crude oil contamination may result from leakage from storage tanks, spills and leaks during transportation, poor waste collection and disposal facilities. Most of the crude oil spillages are accidental, although there are some known cases where crude oil was spilled purposely as in the Gulf war in 1991, where it was reported that between August 1990 and February 1991 about 1.1 billion (1,100,000,000) litres of crude oil were deliberately spilled into the Arabian Gulf, Persian Gulf and in the Kuwait desert, making this the largest oil spill in history (Khamehchiyan et al., 2006, Al-Sanad et al., 1995). Seven hundred kilometres of Kuwait and Saudi Arabian coastline were severely polluted as was an area of approximately 49 square kilometres in the Kuwait desert. However, vast majority of the crude oil spills are a consequence of aging facilities and human errors by the oil companies. Well known examples include the July 2001 Shell pipeline explosion in the Port Harcourt area of Nigeria which caused an 18-day spillage thereby putting environment and life within the community in danger (Turner, 2001). According to Turner (2001), oil extraction in the Niger Delta region of Nigeria has caused severe environmental degradation owing to a legacy of oil spills, lax environmental regulations and government complicity during military regimes that once governed the country. The 2010 explosion of BP deep water horizon drilling rig in the Gulf of Mexico
that spilled about 91 million litres of oil affecting about 110km of Louisiana coastline is another example of crude oil spill that was caused by negligence by the operating oil companies (Browner, 2010).

1.1.1 Crude Oil production in low latitude climate (tropical climate and the dry climate)

Low-latitude Climates (tropical climates and dry climates) - are controlled by equatorial and tropical air masses. The low latitude climate includes the tropical rainforest, tropical savanna climate and the desert climate i.e. arid and semi-arid climate (figure 1.1).

Tropical climates are hot all year-round because they are near the equator. These climates are generally in or near the Inter-tropical Convergence Zone (ITCZ), where winds from the northern and southern hemispheres converge and hot, humid air rises, causing large amounts of precipitation to fall. According to Juo and Franzluebbers (2004) one-third of the soils of the world are in the tropics and these support nearly half of the world population and yet more is known about the soil resources in the temperate regions than is known about the tropical zone. In 2008 statistics about 40 percent of the world’s human population lived within the tropical zone and by 2060, 60% of the human population will be in the tropics, owing to high birth rates and migration (Pidwirny, 2008).
Soils in the tropics are classified into four groups according to the dominant clay mineralogy: kaolinitic soils, oxidic soils, allophanic soils and smectite soils. Kaolinitic soils are deeply weathered with a sand, loamy sand, or sandy loam topsoil and clayey subsoil dominated by kaolinite. Oxidic soils are strongly weathered red and yellowish, fine-textured soils that typically have low bulk density and large amounts of stable microaggregates. The allophanic soils are dark-coloured and young soils derived from volcanic ash with low bulk density, high water retention and contain predominantly allophanes, imogolite, halloysite.
and amorphous aluminium in the clay fraction. The last group are the smectite soils which are loamy to clayey alluvial soils containing moderate to large amounts of smectite (Juo and Franzluebbers, 2004).

As shown in figure 1.2, large quantity of the crude oil production and reserves in the world is within the low latitude climates (tropical and dry climates) and there is the possibility of crude oil contamination of the tropical soils.

Figure 1.2 World crude oil production and reserves (USGS: Mineral commodity summary 2011)
1.1.2 Crude oil in Niger Delta of Nigeria

Nigeria including the Niger Delta region is within the tropical zone of the world and is the 6th largest exporter of crude oil (Bonny light) in the world and the largest in Africa (USGS, 2011). Oil spill in Nigeria (especially in the Niger Delta) has been a regular occurrence and the resultant degradation of the surrounding environment has been a source of concern to the communities living in the region and the multinational companies operating there (Nwilo and Badejo 2006). Ekundayo and Obuekwe (2004) examined the effect of crude oil spill on soil physio-chemical properties at a spill site in the Niger Delta area of Nigeria and they observed that about 36,567 litres of crude oil was spilled at the site which penetrated a depth of 8.4 metres with total hydrocarbon content of about 10.04ppm to 23.6 ppm. Many of the cases of crude oil contamination in Niger Delta are caused by sabotage and negligence. A UNDP report of 2006 stated that oil spills have devastated the environment of the fertile land of Niger delta of Nigeria and in the last 30 years there has been around 6,817 oil spills resulting in more than 1.3 billion (1,300,000,000) litres of oil been spilled into the creeks and soils of southern Nigeria and some 70% of the oil has not been recovered (Opukri and Ibaba, 2008). According to Nwilo and Badejo (2006), in January 2006, the Nigerian National Oil Spill Detection and Response Agency (NOSDRA) declared that it had located more than 1,150 oil spill sites abandoned by various oil companies within the Niger Delta and little has been done regarding the constant crude oil spill within the Niger Delta area of Nigeria compared to the much published crude oil spill in the Gulf of Mexico in 2010. According to NOSDRA report, the scale of crude oil contamination/ spills in the Niger Delta area dwarfs the Deep water Horizon spill of the Gulf of Mexico by a wide margin but little is
known about it due to the fact that most oil company in Nigeria are rarely scrutinized while the media and the government seems uninterested. Nigeria (Niger Delta in particular) being part of the tropical zone has vast quantity of kaolinitic soil with kaolinite as the dominant clay mineral (Badmus and Olatinsu, 2009 and Ugbe, 2011). A considerable increase in soil utility for engineering works is expected as Nigeria aspires towards improved infrastructural development. Frequent occurrence of road pavement failure and building collapse has made it imperative for understanding closer scrutiny of the geotechnical properties of clay soils within the Niger Delta region. Because clay is predominant in most of the subgrade soils of Nigeria (as in most countries within the tropical zone) and easy to acquire, they have found wide application in engineering construction works (Oyediran and Durojaiye, 2011).

1.1.3 Engineering behaviour of contaminated soil

Presently there is no consenus of opinion or undisputable proof about the effect of crude oil on the engineering behaviour of clay soil. When soil is contaminated by crude oil, it is subjected to a change in its engineering properties. Clay soil which is electro-chemically active is mostly affected by the environment (Rehman et al., 2007). Oil contamination might affect the stability and permeability of compacted clay in landfill sites and oil leaks might cause expansion or contraction of the soil on which pipelines are laid (Rahman et al., 2010b).

Since the advent of engineered waste disposal, most industrial wastes are discharged after treatment into land based containments. However, industrial wastes in contact with soil, or accidental spillages of chemical substances, may
lead to changes in soil properties, resulting in improvement or degradation of the engineering characteristics of soil and sometimes leading to functional or structural failure of structures. Any change in engineering properties or behaviour of soil strata may lead to loss of bearing capacity and an increase in total or differential settlement of foundation systems of the structure (Rehman, et al., 2007). Polluted water may attack foundation structures and may affect the workability as well as durability of concrete when used for mixing concrete (Klein and Sarsby, 2000). Recent case histories of structural damage to industries and residential buildings from chemical contamination of soils serve to emphasize the importance of the modification of engineering properties of soil by chemicals (Rao et al., 2008). Sharma and Reddy (2004) stated that the presence of chemicals may influence the soil properties and behaviour and that detailed understanding of the different phases of the soil is critical when considering re-use of contaminated soil.

Much attention has been paid to the effect of crude oil spills on the environment in terms of pollution of surface and ground water, flora, fauna, air pollution and fire hazards. But little research has been done on the nature of soil-crude oil interaction and the effects of crude oil on the geotechnical properties of soil. Most of the Built Environment and infrastructure, such as houses, roads and bridges are built on soil. Soil-waste interactions could alter the effective grain size of soil particles, liquid limit, plastic limit, shrinkage limit, specific gravity, hydraulic conductivity (permeability), compaction characteristics, strength properties and consolidation. Modification of soil properties can then cause or exacerbate various geotechnical problems such as; landslides, ground subsidence, settlement, erosion, progressive failure, underground structural stability, foundation
durability and corrosion (Shin et al., 1997; Sunil et al., 2009 and Rahman et al., 2010b). Sunil et al., 2009 stated that excessive acidity or alkalinity can be detrimental to the soil and that the sulphate content of the soil increases with increases in leachate concentration. This sulphuric attack from the sulphate content may affect buried concrete leading to foundation problems. Also, Simons et al. (2002) stated that acidic groundwater can attacks the concrete used in foundation.

1.1.4 Remediation of crude oil contaminated land

When crude oil is spilled on soils, the extent of the contamination would depend on the properties of the soil (Fine et al., 1997). Crude oil contaminated land would need major task of remediation and /or reclamation for it to be suitable for any purpose in any respects. Remediation may involve soil washing methods, vacuum extraction and separation by centrifuge and screen systems, absorption or biological methods. In all cases, the clean-up works will require knowledge of the geotechnical properties of the soil type. Very few studies exist that addresses the geotechnical properties of contaminated soils. Very few have also addressed the specific problem soil types in the tropics, where most of the largest oil reserves are concentrated.

Based on these introductory paragraphs, it has been noted that;

- Crude oil production is an important resource in the world’s economy.
- Crude oil production cause spills which may be due to negligence, poor waste management, sabotage or war.
- International organisations e.g. UNDP and government agencies has acknowledged that there is huge environmental pollution due to crude oil
contamination within the crude oil producing areas e.g. Niger Delta region of Nigeria.

- Bulk of the crude oil production is within the low latitude climate (tropical zone and dry zone) that has vast quantity of kaolinite clay soil.
- More emphasis has been on the effect of crude oil contamination on the surface and groundwater quality, air pollution, fire hazard and plant and animal of the affected area.
- Less emphasis has been on the effect of crude oil contamination on the soil structure of the contaminated land.
- There is the need to examine the effect of crude oil contamination on the engineering behaviour of tropical soil especially kaolinite soil to ascertain its suitability and usability as a construction material and the ability of crude oil contaminated kaolinite soil to support engineering structures.

1.2 Aims and Objectives

The overall aim of this research project is to investigate the influence of light crude oil contamination on the geotechnical properties of kaolinite clay. Kaolinite clay is chosen as the clay soil of interest based on the fact it is the dominant clay mineral in the soil type of the low latitude climate (tropics and dry climate). This research is undertaken within an engineering context and is primarily a laboratory experimental investigation. This primarily due to the difficulty in accessing a crude oil contaminated site within the UK and the legality of brining in crude oil contaminated soil sample from the tropical zone into the UK.

The specific objectives are to;
• Review existing literature on the effect of crude oil contamination on the engineering properties of kaolinite clay soil.

• Establish methods and testing protocols to quantify the effect of crude oil penetration on kaolinite clay soil.

• Conduct laboratory tests to measure the effects of light crude oil on the index properties (liquid limit, plastic limit, plasticity index and linear shrinkage), compaction, shear strength, permeability and consolidation properties of a typical kaolinite clay soil.

• To compare the geotechnical result of the light crude oil contaminated kaolinite clay soil with that of the uncontaminated kaolinite clay soil.

• To determine the usability of a light crude oil contaminated kaolinite soil as a construction material based on the effect of light crude oil contamination on its geotechnical properties.

1.3 Scope and limitation of the Research

This study is focused on the effect of light crude oil contamination on the geotechnical properties of kaolinite clay. The geotechnical properties of interest in this research are liquid limit, plastic limit, plasticity index, linear shrinkage, compaction, permeability, shear strength and consolidation. This research scope is based on the fact that kaolinite clay soil is the dominant clay type in the low latitude climate (tropical and dry climate). Bulk of the world’s crude oil reserve is found within the low latitude climate zone. Within this geographical location i.e. low latitude climate there are countries that do not have proper regulations regarding how to handle crude oil spills, e.g. in Nigeria, implementation of environmental agency regulation is poor and as such there is little or no
remediation of crude oil contaminated land. Furthermore, clay soil is used in many construction projects, so there is the need to investigate the effect the crude oil contamination will have on the geotechnical properties of the dominant clay soil within this climatic zone.

The samples were mixed by hand in the laboratory and the range of crude oil contamination was between 0% and 20%. The reason for mixing the contaminated soil in the laboratory rather than conducting the geotechnical tests on contaminated soil samples from a polluted oil field was due to the legality issues stated in Section 1.2 of bringing contaminated soil from a tropical country into United Kingdom. The British Standard Test Method for Civil Engineering purposes BS 1377:1990 (EUROCODE 7: EN 1997: 2 - 5) was adopted and used in the geotechnical soil tests.

The limitations encountered in this research are the accessibility of light crude oil contaminated site, lack of some geotechnical equipments in the university laboratory used for the laboratory experiment and lack of some specialist trained operators for some tests to avoid errors especially on the chemical analysis test. This research does not cover the effect of crude oil contamination on other types of clay soil e.g. montmorillonite or illite, and did not use other test methods e.g. the American Society for Testing and Material (ASTM) method in testing for the geotechnical parameters. The limitations noted here do not, however, undermine the validity of the research undertaken and its main findings. It should be remembered that scientific research is a never-ending quest aimed at the understanding of some phenomenon which requires continuous measurement and examination.
1.4 Structure of the thesis

The thesis is divided into 8 chapters as shown in figure 1.3. Chapter 1 deals with the introduction to the research topic focusing on the importance of crude oil, causes and sources of crude oil contamination of land, crude oil production in low latitude climates and the tropical zones of the world. It also discusses the soil type within the tropical zone with an example of crude oil contamination of land within the Niger Delta region of Nigeria. The chapter also identifies the aim and objectives of the research and the scope and limitation of the research.

Chapter two presents a selective review of publish information on this subject. Chapter 3 further presents the basic physics and chemistry of clay mineralogies especially kaolinite. It also contains the basic properties and the nature of hydrocarbons. This chapter examined the clay structure and how fluids are accommodated in the clay soil.

From the published material it was possible to determine the methodology to be used for the research and the methodology is divided into chapters 4 and 5. Chapter 4 deals with the materials used for the research, why the materials were selected, the method of sample preparation and why the samples were prepared in that particular way. It also explains the reasons for selecting the different percentages of contamination used and the possible evaporation of crude oil during the test. It also deals with the different geotechnical properties that are to be tested and why these were chosen. Chapter 5 deals with the equipment and test procedures for the geotechnical test and the reason for choosing each test procedure.

The results of the test that were conducted using the test procedures in chapter 5 are presented, analysed and discussed in chapter 6. It also explains how to
determine the water content and fluid content of the contaminated soil samples and why there were repetitions of some tests. Chapter 6 also explained the use of statistical method and their reliability in the analysis of the results.

Chapter 7 summarises the findings and conclusions from the research and examined if the research work meets the goal set in Chapter 1. It outlines the contribution to knowledge of the research and examines the implication of the findings on the soil structures and the possible use of light crude oil contaminated soil in construction. And Chapter 8 deals with suggestions and recommendation for future work.
Figure 1.3 Structure of the thesis
CHAPTER 2: LITERATURE REVIEW 1: SOIL – CHEMICAL INTERACTION

2.1 Introduction

This chapter reviews published materials (literatures) on how chemicals interact with soil. It includes the composition of soil, how land is contaminated and then it looked at crude oil pollution of ground. It also reviewed literatures on how organic and inorganic chemicals interact with clay soil.

2.2 Soil Composition

The geotechnical use of soil is mainly on the mechanical behaviour of the soil and soil is considered to be a three phased material (liquid, solid and gas). Contamination of soil may cause the composition of these phases to change (Sharma and Reddy, 2004) as the application of chemicals may change the original geotechnical properties of soils (Park et al. 2006). There has been cases of geotechnical failures such as tilting of storage tanks and ground soil heave below industrial structures due to changes in soil properties (Sivapullaiah and Manju, 2005).

Soil includes all earth material (organic and inorganic) occurring in the zone overlying the rock crust and the nature of each individual particle in soil is derived from the minerals it contains which are affected by the original rock from which the particle was eroded. The behaviour of man-made structures depends upon the properties of the soil materials on which they rest. However, soil is not a cemented or solid material i.e. unlike rock the individual particles are not sufficiently bonded together. According to Simons et al. (2002) soil comprises of a skeleton of soil grains in frictional contact with each other. It is a
natural aggregate of mineral grains with or without organic constituents that can be separated by gentle mechanical means (Murthy, 2002). The behaviour and properties such as permeability, compressibility, shrinkage, swelling and shear strength of soils depend on the interaction of the three phases of soil (Sharma and Reddy, 2004). In all soil, the solid phase is the framework and enclosed within the framework is the pore space which is shared by the liquid and gaseous phases (Figure 2.1). The flow of pore water can be restricted by the size of the pore and the degree of saturation thus giving rise to low permeability ($k$) particularly in clay (Simons et al., 2002). The presence of chemicals other than water in the pore space may influence the soil properties and behaviour (Sharma and Reddy, 2004). In geotechnical engineering, soil solid phase is classified according to particle sizes as boulders, gravels, sand, silt, clay and colloids. According to Sharma and Reddy (2004), silicate minerals are the most common minerals found in soils and clay soil is one of the silicates. The behaviour of clay soil is strongly influenced by the physiochemical interactions between clay particles and pore fluid chemistry and the properties of these soils are sensitive to changes in the electrolyte concentration of the pore fluid (Barbour and Yang, 1993). Furthermore the geotechnical properties of clay soils are dominated by the mineralogical composition, the physiochemical interaction between clay particles, inter-particle forces, pore fluid chemistry and soil structure. Many reactions and interactions between the soil and organic matter are possible with consequential effects on plasticity, shrinkage, compressibility, hydraulic conductivity and strength (Mitchell and Soga, 2005). Organic matter interacts with clay by adsorption onto the particle surfaces through ionic exchange and attraction of large organic molecules to clay surface by Van der Waals forces and
intercalation i.e. the entry of organic molecules between silicate layers, (Mitchell and Soga, 2005).

Figure 2.1 Soil Mass (from Sharma and Reddy, 2004)
2.3. Land contamination

Many sites comprising naturally occurring soils are been contaminated or polluted by careless or intentional introduction of chemical substances due to industrial activities (Barnes, 200). Land is considered to be contaminated if it contains elevated concentrations of potentially harmful substances, the contaminants may be present in solid, liquid and gaseous forms (Sharma and Reddy 2004) and changes between these states may occur with time or due to external influences (Rudland and Jackson 2004). According to Sarsby (2000), current and historic industrial and manufacturing activities, the abandonment of land, build-up of toxic chemicals and heavy metals in soils can have serious adverse effect on the soil quality. Extreme cases land contamination can cause the virtual sterilisation of the soil making it devoid of any environmental function. Contamination can, at its worst, affect the soil to such a degree that it can destroy the soil’s ability to perform its required functions. However many of these sites can be brought back into use after remedial treatment (Filauro et. al, 1998).

Contaminated soil may be identified by soil surface cracking patterns, colours, odour and volume change in addition to the relevant analytical chemistry (Fang et al., 2007). However, crude oil-related contaminants that are hazardous to the environment are hard to detect and their effects are long term and contaminated soils may be expensive to clean up completely (Korzeniowska, 1998). So mathematical modelling of contaminant fate and transport has become an integral part of risk management strategies both as a diagnostic and as a predictive tool (Gorder, 1997).
Direct determination of the distribution of ground pollution is often made using trial pits which allows for in-situ observation and description of the various materials in the ground. However, trial pits only provide limited ‘snapshot’ of the ground on which the contaminated zones can be easily observed. Geophysical methods may be used to provide an overview of the presence and extent of contamination and to identify the pathway and obstruction. Even though geophysical techniques are good, they have limitations for example, ground-penetrating radar cannot be used if there is an inorganic contamination in the ground (Nathanail et al., 2002).

2.4. Oil pollution of ground

A critical environmental impact of the petroleum industry is the spillage of oil and related products that may cause severe soil contamination. Oil contamination on the water is relatively easy to collect and remedy, however, when soils are contaminated with oil the process is much longer and there may be significant environmental and engineering effects. For this reason, it is very important to investigate the properties of oil-contaminated soils (Jia, 2011). According to Tuncan and Pamukcu, (1992) once hydrocarbons come into contact with the soil, they alter its physical and chemical properties. The degree of alteration depends on the soil type, the specific composition of the hydrocarbon spilled and the quantity spilled. Once a spill or a leakage occurs, the hydrocarbon liquid, under gravity moves down to the groundwater, partially saturating the soil in its pathway. Upon reaching the groundwater table, this liquid may spread horizontally by migration within the capillary zone, thereby further saturating the soil (Tuncan and Pamukcu, 1992). Clay particles are
chemically active soil particles and their behaviour is always affected by the environment to variable degree depending on the clay particles mineralogy. Generally, hydrocarbon is more viscous than water therefore it relatively moves slower in the groundwater. Some hydrocarbons might be trapped and clogged, reducing pore volume and may led to a reduction in hydraulic conductivity of contaminated soils (Khamehchiyan et al. 2007).

Motor oil is a hydrocarbon product which is very polluting and contains polynuclear aromatic hydrocarbons (PAHs) and high level of heavy metals. It may also enter the ground after being placed in a landfill site (Vazquez-Duhalt, 1987). The contamination of soil by light hydrocarbons is one of the most frequently encountered environmental problems. Leakage from underground storage tanks at refuelling stations is the most common reason but other sources of light hydrocarbon contamination are refineries and production facilities (Aikman et al., 2002). Once diesel fuel has been spilt over the ground it can percolate into the subsurface and ultimately come to exist in three different states i.e. aqueous, gaseous and non-aqueous phase liquids (NAPLs). Yu, et al. (2007) and Rehman et al. (2007) observed that some NAPLs will dissolve in water and others will volatilize and become isolated in the soil pores in the form of thin liquid films on soil particles or colloidal material including natural organic matter. According to Sharma and Reddy (2004), NAPLs exist in the soils in dissolved, adsorbed, gaseous, and free NAPLs phases. In unsaturated soils all the four phases exist (Figure 2.2a), but in saturated soils only the dissolve, adsorbed and free NAPL phases exist (Figure 2.2b).
NAPLs are classified into dense non-aqueous phase liquids (DNAPLs) with densities greater than water and light non-aqueous phase liquids (LNAPLs) with densities less than water. LNAPLs are mostly petroleum products, i.e. gasoline, kerosene and diesel, while DNAPLs are primarily chlorinated solvents, polycyclic aromatic hydrocarbon (PAHs) resulting from industrial activities. The characteristics of NAPLs that affect their behaviour in soil are density, viscosity,
solubility, vapour pressure, volatility, and capillary pressure (Sharma and Reddy, 2004). Hydrocarbon contamination may lead to fire on the ground with consequential effect on the erodability of soil and on water infiltration. In addition, aggregation of fine particles and fusing of minerals may lead to decrease in soil-organic matter aggregate stability. Fire-induced or fire-enhanced soil water repellence has often been cited as the major cause of post-fire enhanced runoff and erosion (Shakesby et al., 2007). Hydrocarbon contamination can also affect the physiochemical characteristic of a soil. Osuji and Ezebuiro (2006) observed that a mangrove floor contaminated by hydrocarbons had a higher acidic value, moisture content and lower electric conductivity.

The permeability of a soil is significantly affected by the density and viscosity of the fluid fillings its voids. According to Sharma and Reddy (2004), there is an increase in intrinsic permeability (k) when there is an increase in density and decrease in viscosity. Also, capillary pressure increases with decrease in soil pore size. In addition there is a decrease in initial moisture content and increase in interfacial tension.

Although ground remediation via biodegradation of petroleum hydrocarbons may be successfully conducted under controlled conditions, the bioremediation of large volumes of hydrocarbon contaminated soils presents significant technical challenges, particularly when contamination occurs in soils of high clay content (Danielle et al., 2006).

Land contamination is detrimental to the building and structure standing on it. Any change in the engineering properties and behaviour of the soil strata may lead to the loss of bearing capacity and differential settlement of the foundation system of the structure (Rehman et al., 2007). According to Rahman et al. (2010)
the spillage of oil into the ground do not just affect the ecosystem but also the safety of the civil engineering structures and the cleaning up of the hydrocarbon contaminated soil is a complicated job due to the high cost and limitations in the disposing the excavated soil. Again, lack of proper management of disused oil and illegal dumping of other hydrocarbon components in many developing countries have contributed to the problem in tackling the environmental issue. Oil contamination can adversely affect the soil microbes and plant as well as contaminate ground water resources for drinking or agricultural purposes. This is because hydrocarbon may either remain close to the source of leak or migrate within the groundwater system or absorbed on the soil grains as an immobile residual fluid. Also the properties of soils and migration path changes in composition and properties of migration substances control the rate of migration (Rahman et al., 2010b).

2.5. Interaction between soils and chemicals

The contamination of land by chemicals depends on a number of factors such as the permeability of the soil and adsorption properties of the soil. The extent of contamination depends on the chemical composition of the contaminant and the properties of the soil (Fine et al., 1997).

To date the majority of investigations into the interaction between engineering soils and chemicals have been related to containment of refuse in landfill sites (Gettinby, 1999; Sarsby, 2001). This is because of the widespread use of landfill as a means of waste disposal. This process produces a polluted water (leachate) containing a large spectrum of chemicals (Table 2.1) which could potentially affect the geotechnical properties of clay layers within liners which are used to
contain waste. Any increase in permeability or loss of plasticity could severely affect the ability of clay layers to act as a barrier. However the pore fluid chemistry may significantly alter the chemical compositions of clays by means of exchangeable cations which govern the engineering properties of clays in most cases (Yukselen-Aksoy, 2011).

Concentration profiles of contaminant mixture components in a mineral layer of a composite landfill liner have indicated that the various mineral layers have a selective sorption capacity (Kalbe et al., 2001). This was reported as corresponding to the different parameters of organic compounds (especially solution in water), properties of the mineral layer (in particular mineral phase composition, particle size distribution and moisture content) and the thickness.

Table 2.1 Leachate compositions

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Total Range (mg/l)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminium</td>
<td>0.27-2.7</td>
</tr>
<tr>
<td>Ammonia nitrogen</td>
<td>0-3000</td>
</tr>
<tr>
<td>Alkalinity</td>
<td>0-20850</td>
</tr>
<tr>
<td>Arsenic</td>
<td>0.021-0.13</td>
</tr>
<tr>
<td>BOD</td>
<td>2.0-57700</td>
</tr>
<tr>
<td>Boron</td>
<td>4.2-7.4</td>
</tr>
<tr>
<td>Cobalt</td>
<td>0.01-95</td>
</tr>
<tr>
<td>Cadmium</td>
<td>0.001-17.0</td>
</tr>
<tr>
<td>Calcium</td>
<td>5-7200</td>
</tr>
<tr>
<td>COD</td>
<td>0-89250</td>
</tr>
<tr>
<td>Chloride</td>
<td>4.7-4816</td>
</tr>
<tr>
<td>Parameter</td>
<td>Range</td>
</tr>
<tr>
<td>---------------</td>
<td>----------------</td>
</tr>
<tr>
<td>Chromium</td>
<td>0.002-18</td>
</tr>
<tr>
<td>Conductivity</td>
<td>400-50000 (µS/cm)</td>
</tr>
<tr>
<td>Copper</td>
<td>0-9.9</td>
</tr>
<tr>
<td>Fluoride</td>
<td>0.27</td>
</tr>
<tr>
<td>Iron</td>
<td>0.09-2500</td>
</tr>
<tr>
<td>Lead</td>
<td>0-12.3</td>
</tr>
<tr>
<td>Magnesium</td>
<td>13.3-15600</td>
</tr>
<tr>
<td>Manganese</td>
<td>0.06-1400</td>
</tr>
<tr>
<td>Mercury</td>
<td>0.002-19.5</td>
</tr>
<tr>
<td>Nickel</td>
<td>0.002-79</td>
</tr>
<tr>
<td>Nitrate</td>
<td>0-1300</td>
</tr>
<tr>
<td>Nitrite</td>
<td>0-25</td>
</tr>
<tr>
<td>pH</td>
<td>3.7-9.1(µS/cm)</td>
</tr>
<tr>
<td>Phosphorous</td>
<td>0-154</td>
</tr>
<tr>
<td>Potassium</td>
<td>2.8-3770</td>
</tr>
<tr>
<td>Silica</td>
<td>12-34</td>
</tr>
<tr>
<td>Sodium</td>
<td>0-7700</td>
</tr>
<tr>
<td>Strontium</td>
<td>0.94-72</td>
</tr>
<tr>
<td>Sulphate</td>
<td>1-2000</td>
</tr>
<tr>
<td>Sulphide</td>
<td>0-30</td>
</tr>
<tr>
<td>Suspended solid</td>
<td>0-700</td>
</tr>
<tr>
<td>TOC</td>
<td>0-28500</td>
</tr>
<tr>
<td>Total dissolved solid</td>
<td>0-44900</td>
</tr>
<tr>
<td>Zinc</td>
<td>0-1000</td>
</tr>
</tbody>
</table>

(Sarsby, 2001, Sharma and Reddy, 2004, Aziz et al., 2010)
2.5.1. Inorganic Chemicals

Mesri and Olsen (1971) looked at the mechanisms that controlled the permeability of clay and they stated that while the coefficient of permeability was lowest for water that is a polar fluid and has a high dielectric constant, it is smaller for polar fluid of low dielectric constant and largest for non-polar fluids. They also stated that the coefficient of permeability is generally lower when the absorbed cations are monovalent rather than divalent. In addition, they observed that a reduction in electrolyte concentration to reduce the coefficient of permeability but the effect of the electrolyte concentration decreases as the valency of the cations increases. This was found to be smaller in sequence from smectite to illite to kaolinite.

Barbour and Yang (1993) reviewed literatures on the influence of clay-brine interaction on the geotechnical properties of clayey soils and observed that the presence of brine will cause the plasticity of soil to decrease. Also there was a decrease in liquid limit and plasticity index with an increase in pore fluid concentration. Subsequently, Ho and Pufahl (1997) conducted laboratory tests to investigate the effect of brine contamination on the properties of fine-grained soil. They found out that the liquid limit decreased (from 75% to 58%) with increase in the brine concentration (0ml of NaCl to 4ml of NaCl) and the brine also caused a decrease in hydraulic conductivity (from $5 \times 10^{-10} \text{ m}^2/\text{s}$ to $1 \times 10^{-10} \text{ m}^2/\text{s}$) of the soil sample when a voids ratio of 0.8 was exceeded (with 4ml NaCl solution). Brine contamination changed the clay soil from non-dispersive to dispersive thus the clay particles had a minimum electrochemical attraction and failed to adhere closely to or bond with other soil particles. Hence the clay soil could erode easily in the presence of flowing water as individual clay platelets
are split off and carried away. Ho and Pufahl (1997) stated that inter-particle bonding forces were reduced as water molecules were drawn into the soil samples and that swelling resulted from this reduction in the inter-particle bonding forces made the soil prone to erosion.

Rajasekaran and Rao (2002) worked on the permeability characteristics of lime treated marine clay and observed an increase in permeability value up to a maximum value of 15 – 18 times that of untreated soil. However the compression index of the natural marine clay soil decreased from 0.85 to 0.49 and the pre-consolidation pressure increased from 36kN/m$^2$ to 70kN/m$^2$. Also the shear strength characteristic using falling cone method showed that the shear strength increased from 1.2kN/m$^2$ to 9.7kN/m$^2$. The authors stated that this increase may be due to higher absorption of calcium ions by the soil particles as a result of cation exchange phenomena. Again they stated that the addition of lime induces aggregation and cementation effects on soil particles that result in a considerable reduction of compression index and increase in preconsolidation pressure.

Sivapullaiah and Manju (2005) investigated the geotechnical properties of a low plasticity soil (wL= 38%) using NaOH solution. They reported that the liquid limit of the test soil was increased with increasing NaOH concentration. They observed that due to formation of new swelling compounds, dispersion and deflocculation of clay, the geotechnical properties (especially, hydraulic conductivity) of clay were significantly changed. They added that the increase in consistency limits could be attributed to dispersion of the clay particles when clay was permeated with salt solutions and that salt solutions might cause the clay to
form new swelling compounds and these new compounds might have increased the liquid limit of CL clay (Sivapullaiah and Manju 2005).

Ouhadi and Goodarzi (2006) noted that when Alum (Aluminium Sulphate, \( \text{Al}_2(\text{SO}_4)_{3.18\text{H}_2\text{O}} \)) is added to a cohesive soil it controls the dispersivity of soil due to the pH and ionic exchange effects that increase the plasticity index of the soil. Also there was an increase in the soil compressibility, a decrease in permeability (from \( 1 \times 10^{-9} \text{m/s} \) to \( 1 \times 10^{-11} \text{m/s} \)) with increase in the quantity of Alum. This was attributed to the decrease of void ratio and increase in density mainly due to the replacement of the sodium ions by aluminium ions which provide a reduction in repulsive force.

Park et al. (2006) studied the effect of surfactant and electrolyte solution on the properties of soil and they concluded that NaPO\(_3\) caused a decrease in the hydraulic conductivity of kaolinite and it caused the maximum dry density to increase from 18.9kN/m\(^3\) with water to 19.7kN/m\(^3\) with NaPO\(_3\), the optimum moisture content to decrease from 11.6% with water to 9.0% with NaPO\(_3\). They stated that NaPO\(_3\) decreases the surface tension so that the solution could move freely through the pores in the soil. Also Park et al. (2006) also indicated the addition of the electrolyte solutions (Triton X-100 and biosurfactant) caused an increase in electrolyte concentration, which decreased the double layer thickness. The large increase in inter-particle attraction made possible by the reduction of the diffuse double layer was responsible for the flocculation of the clay mixture on mechanical remolding. This effect resulted in increased strength of kaolinite mixtures.

Similarly Sunil et al. (2006) worked on the effect of pH on the geotechnical properties of laterite. They observed that an increase in the pH of leachate, i.e.
from acidic towards alkaline, had a strong influence on the chemical characteristics of lateritic soil and could cause a change in the Atterberg limits of the soil. This they attributed to the leaching of metallic cations which could take place under acidic conditions. Also they observed that leachate caused the maximum dry density to decrease by about 20% (from 16.87 kN/m$^3$ to 13.93 kN/m$^3$) and the optimum moisture content to increase from about 22% to about 26%. Nayak et al. (2007) also worked on the hydraulic conductivity and compaction characteristics of leachate–contaminated lateritic soil with acidic pH and found that the maximum dry density decreased but only by about 3% (from 15.47 kN/m$^3$ to 14.98 kN/m$^3$). However, the optimum moisture content increased significantly (from 19.52% to 25.01%) when the percentage leachate contamination was increased from 5% to 20%. On the other hand, Alainachi and Alobaidy (2010) found that the addition of salt water to soil prior to compaction caused an increase in the maximum dry density of between 4% and 8% of the original value, i.e. from (17.4 – 18.6) kN/m$^3$ to (18.0 – 20.1) kN/m$^3$ with the optimum moisture content decreasing by about 10% of its original value, i.e. from (11 – 15) % to (10 – 13) %. Permeability doubled (approximately from 2.69 x$10^{-7}$ m/s to 5.86 x$10^{-7}$ m/s) and voids ratio increased from 1.02 to 1.10 when the percentage contamination was increased from 5% to 20% (Nayak et al., 2007).

Singh and Prasad (2007) studied the effect of chemicals on compacted clay (bentonite) liners and they observed that heavy compaction test results indicate that addition of Acetic acid caused reduction in both OMC and MDD whereas addition of Aluminium hydroxide causes increase in OMC. Also, reduction in MDD and both Acetic acid and Aluminium hydroxide caused the cohesion ($C_u$) decreased by about 50% whereas the angle of internal friction ($\phi_u$) remained...
unchanged. Also these chemicals caused a decrease in the hydraulic conductivity of the tested soil.

Rao et al. (2008) mixed clay soil with spent dye effluent and observed an increase in liquid limit from 58% to 80% after adding the dye to the soil. However, the liquid limit value returned to its original value after the contaminated soil had been left for 15 days. They stated that the addition of dye to the soil would result in changes in the pore fluid chemistry leading to physio-chemical interactions between the individual particles. Rao et al. (2008) also observed an increase in the value of the Coefficient of Consolidation from 4597.95 m²/yr to 15515.71 m²/yr (for a pressure of 80kN/m²) when the dye was added to the soil.

During a laboratory-based investigation of the shear strength characteristics of leachate-contaminated lateritic soil, Sunil et al. (2009) observed an increase in liquid limit from 50% to 58% as the percentage of acidic leachate contamination increased from 0 to 20%. They also observed a slight increase in cohesion (from 18kN/m² to 20kN/m²) and a decrease in frictional angle 30° to 26° with increase in contamination. Sunil et al. (2009) reported that the pH of lateritic soil increased from 4.31 to about 6.9 with increase in the concentration of leachate and that at the same time the cation exchange capacity doubled approximately, increasing from an initial value of 6.23meq/100g to 12.2meq/100g. Addition of the leachate caused a major increase in the electrical conductivity of the soil, i.e. from 25μ/cm to 320μ/cm.

Arasan (2010) reviewed effect of chemicals on geotechnical properties of clay liners and concluded that chemicals significantly affect the geotechnical properties of clay and clay liners. However, there no consensus regarding the
effect of chemicals on the geotechnical properties of clay and clay liners in that the behaviour of the low plasticity clays (CL and kaolinite clay) is different from the high plasticity clay (CH and bentonite clay). He stated that the liquid limit and swelling decreases with increasing chemical concentration for high plasticity clay but the liquid limit and swelling increases with increasing chemical concentration for low plasticity clay. Also the hydraulic conductivity increases with increasing chemical concentration for high plasticity clay but the hydraulic conductivity decreases with increasing chemical concentration for low plasticity clay.

The effect of chemicals on the geotechnical properties may be explained by Diffuse Double Layer (DDL). If the chemical solutions tended to reduce the thickness of the DDL and flocculate the clay particles, it results to reduction of liquid limit, swelling and increasing of hydraulic conductivity of high plasticity clays. However, if the chemical solutions tended to increase the thickness of the DDL and disperse the clay particles, it results to increase in liquid limit, increase in swelling and reduction of hydraulic conductivity of low plasticity clays (Arasan 2010).

Some researchers have looked at the effect that particles (as opposed to chemicals) have on the properties of soil and concluded that there was consequential alteration of the properties to the soil. For instance, Akbulut and Saglamer (2004) looked at the effect of addition of silica fume and fly ash on the hydraulic conductivity of granular soil and found that both caused a decrease in the permeability of the soil. Yarbasi (2007) found that the addition of silica fume and fly ash to a soil caused the maximum dry density of the soil to decrease whilst its optimum moisture content increased. Generally the additions produced an increase in the compressive strength.
Lin et al. (2006) worked with ground fine sewage sludge particles (passing No 200 sieve) that had been incinerated in a furnace at 800°C. The sludge ash and hydrated lime were mixed in the ratio of 4:1, respectively and they observed that there was a decrease in the plasticity index value of clay soil when mixed with a sewage sludge ash and ash-hydrated lime mixture. They also found that there was improvement on the CBR value, triaxial shear strength and unconfined compression strength.

Muhardi et al. (2010) conducted a laboratory tests on the engineering characteristics of coal ash and they found that the optimum moisture content for fly ash and bottom ash was 19.8% and 21.5% respectively with maximum dry density of 15.0 kN/m³ and 12.9 kN/m³ respectively, i.e. significantly lower than the dry density of sandy soil (typically from 17 –20 kN/m³).

2.5.2. Organic Chemicals

Micheal and Lins (1954) stated that clay can be much more permeable to organic liquids than pure water and they reported that extremely large values of hydraulic conductivity about $1 \times 10^{-4}$ m/s were observed when liquid hydrocarbons dominated the fluid phase of clays. Such a value of hydraulic conductivity value for the contaminated clay was more characteristic of fine sand than clay.

Acar and Ghosh (1986) studied the role of soil activity in hydraulic conductivity of compacted soils permeated with acetone. They observed that hydraulic conductivity of the soil decreased with increase in soil activity due to the permeation with acetone. This decrease in hydraulic conductivity was believed to be due to a decrease in the volume of the soil, which reduced the size of the pores and controlled the structural stability of the compacted soil with higher soil
activity values. On the other hand, when Bowders et al. (1986) mixed soil with methanol to study the effect of methanol addition on Atterberg limits and permeability of a clay liner, they found that the permeability of a clay liner mixed with methanol was higher than that of clay mixed with water only. They also observed that the change in liquid limit, plastic limit and permeability was negligible but when the aqueous solution contain more than 80% methanol there was an increase of about 28% in liquid limit and about 32% in plastic limit.

Chan et al. (1986) also observed an increase in plastic and liquid limits in proportion to the concentration of the organic liquids when clay samples were soaked overnight in liquid phenol solutions before being subjected to geotechnical tests (using the ASTM methods). There was also an increase in both optimum dry density and optimum moisture content with increase in concentration of the organic liquids.

Mitchell and Jaber (1990) looked at the factors controlling the long term properties of clay liners and identified the most important factors controlling the influence of organic chemicals on hydraulic conductivity of clay as; (1) the exposure of the clay to pure organic or a dilute solution, (2) polarity of the organic chemicals, (3) the dielectric constant of the organic chemicals and (4) the water solubility. However they stated that in practice, the exposure of clay barriers to water-insoluble, pure or concentrated organics is likely only in the case of spills, leakage from tanks containing organic dense non-aqueous phase liquids or where there is accumulation at low spots above liners.

Evgin and Das (1992) performed a series of triaxial tests on contaminated and uncontaminated clean sands. The results showed that the oil saturated samples drastically reduced the friction angle for loose and dense samples. On the other
hand, it apparently increased the volumetric strain. They concluded that settlement of footing would increase as a result of oil contamination.

Meegoda and Rajapakse (1994) applied various concentrations of organic chemicals (propanol, glycerol, acetone and hexane) as permeants to clay under short–term and long-term exposure condition. They reported that when chemicals were allowed to permeate for an extended time period, there would be a gradual replacement of the pore fluids of the clay that do not contribute to fluid flow by the permeating chemicals. This gradual replacement of the pore fluid molecules leads to a modification of the intermolecular forces. Due to long term chemical contamination of soils, the soil structure may change with associated change in the mechanical properties of the soil.

Al-Sanad et al. (1995) undertook laboratory experiments in which sand was mixed with crude oil to determine the geotechnical properties of sands contaminated by oil as a result of exploded oil wells and destruction of oil storage tanks in Kuwait at the end of the Gulf war. The oil contamination applied was up to 6% by weight. In their plot they observed a slight increase in the maximum dry density from 18.63kN/m³ to 19.12kN/m³ for a 2% addition of crude oil, but addition of further oil caused the maximum dry density to decrease i.e. to 19.03kN/m³ at 4% and 17.95kN/m³ at 6% contamination. However the optimum moisture content showed a decrease with increase in oil contamination i.e. 12% at 0% contamination and 2% at 6% contamination. The effect on the compaction characteristics (maximum dry density and optimum moisture content) were stated as reflecting the lubricating effect caused by the presence of oil, which facilitates compaction and reduces the amount of water needed to reach maximum density. In addition their results showed a reduction in the
coefficient of permeability of about 20% from $1.72 \times 10^{-5}$ m/sec to $1.38 \times 10^{-5}$ m/sec, for clean soil when contaminated with 6% (by weight) of crude oil. The two foregoing observation are complimentary because the oil has higher viscosity than water so it would make it more difficult for the pore fluid to flow. At the same time this higher viscosity (and surface tension) would promote retention of oil between soil particles. The CBR initially increased from 25% to 32% with an increase in oil contamination from 0 to 2% but greater contamination caused CBR to fall to 31% (at 4% addition) and to 4% (at 6% contamination). The authors attributed this change in CBR value to the presence of excess oil and low maximum density associated with the mix which would also account for the increase in compressive index from 0.03 (clean soil) to 0.07 (at 6% contamination). A marginal decrease in the frictional angle from $32^0$ (at 0% contamination) to $30^0$ (at 6% contamination) was reported but this apparent change may have been due to incorrect measurement of true pore pressure between particles.

Shin et al. (1997) also conducted laboratory tests on crude oil-contaminated quartz sand as they were concerned with the stability of slopes and the bearing capacity of foundations of coastal structures. Like Al-Sanad et al. (1995) they found a decrease of about 25% in the friction angle ($43^0$ to $30^0$), a decrease in the ultimate bearing capacity of shallow foundation ($350$ kN/m$^2$ to $50$ kN/m$^2$) and a decrease in permeability ($6.8 \times 10^{-5}$ m/sec to $1 \times 10^{-5}$ m/sec) when the oil content was increased by 4%. However, they plotted a compaction curve using both moisture content (water as the only lubricant) and oil content (oil as the lubricant) and found that the test with crude oil as lubricant has maximum dry density of $16.63$ kN/m$^3$ at oil content of 9.4% while the test with water as the
lubricant has maximum dry density of 16.21kN/m³ at moisture content of 19.1%. They concluded that oil contamination of sand gives higher maximum dry unit weight when the crude oil was used as the lubricant, this result was the opposite of the findings of Al-Sanad et al. (1995).

Aiban (1998) examined the effects of temperature on contaminated soil strength, porosity and compaction with samples collected from east Saudi Arabia. The compressibility and deformation of oil-contaminated sand increased as the temperature was increased above room temperature. The shear strength was found to be independent of testing temperature when samples compacted to their maximum dry densities. Shin and Das (2001) studied the load capacity for oil partially saturated sand at oil content ranged between 0% and 6%. The results indicated that the load capacity dropped with the increase of oil content.

Shah et al. (2003) studied the Stabilization of fuel oil contaminated soil samples from a petrochemical complex near Vadodara City in Gujarat State, India and they found that the soils exhibited major changes in their geotechnical parameters including a 4% decrease in maximum dry density, 66% decreases in cohesion, 23% decrease in angle of internal friction and 35% decrease in unconfined compressive strength (UCS) and 11% increase in liquid limit.

Khamehchyan et al. (2006) and Khamehchyan et al. (2007) conducted laboratory experiments involving crude oil and coastal soils (clayey and sandy) in Iran. The soils were classified as silty sand (SM), poor graded sand (SP) and clay (CL), and samples were mixed with 0%, 4%, 8%, 12%, and 16% (measured by weight of the dry soil) of crude oil. Their test methods were based on American Society of Testing and Material (ASTM) standards. The authors, Khamehchyan et al. (2006) and Khamehchyan et al. (2007) noted a decrease in maximum dry density
with increase in oil content from 18.54kN/m$^3$ to 17.45kN/m$^3$ and from 18.83kN/m$^3$ to 17.45kN/m$^3$ for the clay and silty sand respectively as contamination went from 0% to 16%. The reduction in dry density was reported as being faster in silty sand and clay samples due to the lubricating effect of oil which facilitated compaction and reduced the amount of water needed to reach maximum density whereas for poorly graded sand the reduction in the dry density was slower because in this case the pore spaces were larger so that oil could move through the soil in the same way as water. The optimum moisture content (i.e. fluid content – oil content) decreased from about 16% to 4% for clay, from 15% to 4% for silty sand and from 13% to 3% for poorly graded sand as the oil contamination increased from 0 to 16%. From direct shear tests they reported a reduction in cohesion and an increase in frictional angle (from about 26$^0$ to 35$^0$) for the clay as the oil content was increased. On the other hand the frictional angle decreased from 34$^0$ to 24$^0$ in silty sand and also in the sand samples as reported previously by Al-Sanad et al. (1995) and Shin et al. (1997). 

Also, uniaxial compression tests on clay showed a decrease in compressive strength from 400kN/m$^2$ for plain soil to about 100kN/m$^2$ at 16% oil content. Constant head permeability tests indicated that in all cases, the coefficient of permeability decreased with increase in oil content from $7 \times 10^{-7}$ to $4 \times 10^{-8}$ m/s for clay, from $1 \times 10^{-5}$ to $2 \times 10^{-7}$ m/s for silty sand and from $5 \times 10^{-3}$ to $5 \times 10^{-5}$ m/s for poor graded sand as contamination increase from 0% to 16%. Furthermore the effect of oil content on permeability decreased with increase in soil porosity.

Korzeniowska-Rejmer (2007) conducted a laboratory experiment on sand, silt and clay soils and he stated that oil contamination causes significant soil
settlement because chemical substances in high concentrations which fill the pores of soil can affect the mineral composition. He observed that there was an alteration in the grain size distribution curve in soil subject to persistent effect of oil-related contamination, there was a decrease in sand fraction and a significant increase in silt content as a result of disintegration of sand particle. There were also effects relating to the flocculation of clay particles. The changes in the grain size distribution have important effect on the value of internal friction angle, cohesion and modulus compressibility and these affect the stability of foundation and structure placed directly on oil-contaminated subsoil.

Mashalah et al. (2007) carried out an extensive laboratory testing program to determine the effects of crude oil contamination on some of the geotechnical properties of clayey and sandy soils such as SM (silty sand), SP (poorly graded sand) and CL (lean clay) sampled from the coastal soils from the southern coastal plain of Iran in the Persian Gulf. The contaminated samples were prepared by mixing the soils with crude oil at 2%, 4%, 8%, 12%, and 16% measured by dry weight of the soil. The results indicated a decrease in strength, permeability, maximum dry density, optimum water content and Atterberg limits.

Rehman et al. (2007) conducted laboratory experiments wherein they mixed clay soil with crude oil to make the soil fully saturated with crude oil at its natural dry unit weight of 12kN/m³ and the oil and soil mix were air-dried for one week before a comprehensive laboratory test were conducted on the conditioned contaminated clay. They observed that the addition of oil increased the liquid limit (172% to 185%), plastic limit (48% to 50%) and the plasticity index (123 to 136). The shrinkage limit also increased from 26% to 28%. When the soil was
subjected to standard Proctor compaction the maximum dry density increased from 11.7kN/m$^3$ (uncontaminated soil) to 15.6kN/m$^3$ (contaminated soil) and the optimum fluid content (water and oil) decreased from 43% to 8.5%. Compression index was reported to increase from 0.342 to 0.932, which was attributed to open structure of the contaminated soil due to the presence of oil, and the percentage of swelling increased from 28.5% to 29%. Also they conducted a swelling pressure test and the result showed a decrease from 680kN/m$^3$ to 200kN/m$^3$ for uncontaminated soil and contaminated soil respectively.

Rahman et al. (2010a) mixed the soil samples with engine oil (up to 16% by dry weight of the soil) and tested the mixture using essentially British Standard Test procedures. Two type of soil were used i.e. granitic soil consisting of 64% sand, 34% silt and 2% clay and metasedimentary soil consisting of 34% gravel, 37% sand, 27% silt and 2% clay. In all cases, the liquid limit decreased with increasing oil content (from 62% to 55% for the metasedimentary soil and 43% to 38% for the granitic soil) and the plastic limit also decreased (from 41% to 36% for metasedimentary soil and from 24% to 19% for granitic soil) as the oil content increased from 0% to 16%. The compaction result of the granitic soil sample showed that at 0% oil content. The maximum dry density was 14.71kN/m$^3$ and the optimum moisture content was 17.2%. But as the oil content increased from 4% to 8%, the maximum dry density decreased from 14.42kN/m$^3$ to 14.22kN/m$^3$ while the optimum moisture content increased from 19.4 to 22.5%. Between 12% and 16% oil content, the maximum dry density decreased from 13.63kN/m$^3$ to 13.44kN/m$^3$ but there was no significant change in the optimum moisture content. But the compaction result for the metasedimentary
soil shows that the maximum dry density increased from 15.50kN/m$^3$ to 18.63kN/m$^3$ and the optimum moisture content decreased from 21.9% to 8.5% between 0% and 16% increase in oil content. The compaction characteristics of the granitic soil are in line with finding of Khamenehchyan et al. (2006) and Khamenehchyan et al. (2007) whereas the behaviour of the metasedimentary soil was supported by the findings of Rehman et al. (2007). Again, Khamenehchyan et al. (2006) and Khamenehchyan et al. (2007) conducted an unconsolidated undrained triaxial compression test on both types of soil. Their result shows that the shear strength value $C_u$ decreased from 27kN/m$^2$ to 8kN/m$^2$ and 28kN/m$^2$ to 6kN/m$^2$ for metasedimentary soils and granitic soils respectively as the oil content increased from 0% to 16%.

In a subsequent study Rahman et al. (2010b) mixed oil with basaltic residual soil. The classification of the Basaltic residual soil sample showed that it was made up of sandy loam and silty loam. The classification result based on oil content (i.e. water content – weight of water) shows that the liquid limit decreased from 66% to 40%, the plastic limit decreased from 42% to 25.5% and the plasticity index decreased from 31 to 11.8 as the oil content increased from 0% to 16%. Also with increase in oil content, the compaction result showed that maximum dry density decreased from 16.38kN/m$^3$ to 14.71kN/m$^3$, optimum moisture content decreased from 23.5% to 16.5% for the basaltic residual soil. Also, the falling head permeability test result showed the coefficient of permeability decreased from $3.7 \times 10^{-7}$m/s to $0.25 \times 10^{-7}$m/s. Again the strength characteristics $C_u$ showed a decrease in value with increase in oil content, it decreased from 125kN/m$^2$ to 38kN/m$^2$. 
Olgun and Yildiz (2010) studied the effect of organic fluid on the geotechnical behaviour of a highly plastic clayey soil by using different organic fluid (methanol, ethanol, isopropyl alcohol and acetic acid) at different organic/ water ratio as the permeating fluid, the laboratory test procedure was according to the American society of testing and material (ASTM) standard. Their result showed that organic fluid caused the liquid limit and plasticity index to decrease e.g. the liquid limit and plastic limit in distilled water was 106% and 69.8% but it decreased in 80% organic fluid/ 20% water to 45% and 4.2% in acetic acid. The hydraulic conductivity (k) value increases with increase in the percentage of organic fluid/ water ratio e.g. at 0% organic fluid/ 100% water the hydraulic conductivity was $2.12 \times 10^{-12}$ m/s, but at the ratio of 20% organic fluid/80% water, it was $4.84 \times 10^{-12}$ m/s for Methanol, $3.88 \times 10^{-12}$ m/s for Ethanol, $4.02 \times 10^{-12}$ m/s for Isopropyl alcohol and $6.7 \times 10^{-12}$ for Acetic acid, The shear strength parameters showed an increase in cohesion and friction angle with increase in organic fluid, e.g. at 0% organic fluid/100% water ratio, cohesion was 25.5kN/m², and frictional angle was $3.97^0$, but at 20% organic fluid/80% water ratio cohesion (c) increased to 27kN/m² and frictional angle (Ø) increased to $4.93^0$ for methanol. The reported effect of liquid contaminants on geotechnical properties are summarised in tables 2.2 and 2.3.

2.6. Summary

The trend as observed from the reviewed literature (Table 2.2) shows that increase in the concentration of inorganic chemicals to soil cause a decrease in the liquid limit and the plasticity index, a decrease in dry density and an increase in optimum moisture content. Also increase in the concentration of inorganic
chemicals caused an increase in permeability, a decrease in compression index, a decrease in frictional angle and an increase in cohesion.

From Table 2.3, it has been noted that crude oil caused the liquid limit, plastic limit and plasticity index values to decrease in sand and sandy soils whereas it caused the same parameters to increase in clay soil. Also crude oil caused an increase in the strength parameter of clay and clayey soils but a decrease in that of sand and sandy soils. The table also showed that crude oil caused a decrease in the compaction and permeability value of sandy soils and an increase in the compressive index of sandy soil.

To date, little research has been done on the influence of crude oil contamination on the geotechnical properties of clay soils and the accommodation of hydrocarbon within the internal structure of clay soil and the consequential effects on geotechnical characteristics. Most of the research on the crude oil contamination has been focused on the contamination of surface and ground water and its effect on aquatic life (Bu-Olayan et al., 1998). The few that are on the effect of crude oil contamination on the geotechnical properties of soil focused on sand and on silty sand. As can be seen from Table 2.3, there is lack of knowledge on the effect of crude oil contamination on the compaction characteristics, consolidation, shear strength characteristic and permeability of clay soil. Also, this research will look at the effect of crude oil at different percentage of contamination on the geotechnical properties of clay soil.
Table 2.2 Summary of the effect of inorganic contaminant on the geotechnical properties of soil

<table>
<thead>
<tr>
<th>Geotechnical property</th>
<th>Contaminant</th>
<th>Effect</th>
<th>Author</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Alum (Aluminium Sulphate ( \text{Al}_2(\text{SO}<em>4)</em>{3.18\text{H}_2\text{O}}) ))</td>
<td>Increase</td>
<td>Ouhadi and Goodarzi (2006)</td>
</tr>
<tr>
<td></td>
<td>leachate</td>
<td>Increase</td>
<td>Sunil et al (2009)</td>
</tr>
<tr>
<td>Shear Strength</td>
<td>Brine</td>
<td>Increase</td>
<td>Barbour and Yang (1993).</td>
</tr>
<tr>
<td></td>
<td>Alum (Aluminium Sulphate ( \text{Al}_2(\text{SO}<em>4)</em>{3.18\text{H}_2\text{O}}) ))</td>
<td>Increase</td>
<td>Ouhadi and Goodarzi (2006).</td>
</tr>
<tr>
<td>Cohesion</td>
<td>Leachate</td>
<td>Increase</td>
<td>Sunil et al (2009)</td>
</tr>
<tr>
<td>Frictional Angle</td>
<td>Leachate</td>
<td>Decrease</td>
<td>Sunil et al (2009)</td>
</tr>
<tr>
<td>CBR</td>
<td>Salt water</td>
<td>Increase</td>
<td>Alainachichi and Aloibaidy (2010)</td>
</tr>
<tr>
<td>Maximum Dry density</td>
<td>Leachate</td>
<td>Decrease</td>
<td>Nayak et al (2007)</td>
</tr>
<tr>
<td></td>
<td>Salt water</td>
<td>Increase</td>
<td>Alainachichi and Aloibaidy (2010)</td>
</tr>
<tr>
<td>Optimum</td>
<td>Leachate</td>
<td>Increase</td>
<td>Nayak et al</td>
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<tr>
<td>Property</td>
<td>Material</td>
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<td>Moisture content</td>
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<td>Compressibility Index</td>
<td>Lime</td>
<td>Decrease</td>
<td>Rajasekaran and Rao (2002)</td>
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<td></td>
<td>Alum (Aluminium Sulphate [Al₂(SO₄)₃.18H₂O])</td>
<td>Increase</td>
<td>Ouhadi and Goodarzi (2006).</td>
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<tr>
<td>Permeability</td>
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<td>Decrease</td>
<td>Ho and Pufahl (1997).</td>
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<td></td>
<td>Alum (Aluminium Sulphate [Al₂(SO₄)₃.18H₂O])</td>
<td>Increase</td>
<td>Ouhadi and Goodarzi (2006).</td>
</tr>
<tr>
<td>Contaminant</td>
<td>Geotechnical Property</td>
<td>Soil Type</td>
<td>Effect</td>
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<tr>
<td>Crude Oil</td>
<td>Liquid Limit, Plastic Limit, Plasticity Index</td>
<td>Granitic and Metasedimentary Soil</td>
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<td></td>
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<td>Silty and Loamy Sand</td>
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<td>Clay</td>
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<td></td>
<td></td>
<td>Clay</td>
<td>Increase</td>
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<tr>
<td>Property</td>
<td>Material</td>
<td>Change</td>
<td>Reference</td>
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<td></td>
<td>Clayey and Sandy Soil</td>
<td>Decrease</td>
<td>Khamehchyan et al (2006 and 2007)</td>
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<tr>
<td>Frictional Angle</td>
<td>Clay</td>
<td>Increase</td>
<td>Olgun and Yildiz (2010)</td>
</tr>
<tr>
<td>Cohesion</td>
<td>Clay</td>
<td>Increase</td>
<td>Olgun and Yildiz (2010)</td>
</tr>
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<td>Increase</td>
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<tr>
<td>Maximum Dry Density</td>
<td>Clay</td>
<td>Increase</td>
<td>Chan et al (1986)</td>
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</tbody>
</table>
2.7 State of the art- Existing knowledge and contribution to research

It has been stated that the behaviour of man-made structures depends upon the properties of the soil materials on which they rest while silicate minerals are the most common minerals found in soils of which clay soil is one of the silicates. However the behaviour of clay soil is strongly influenced by the physiochemical interactions between clay particles and pore fluid chemistry as well as the properties of these soils are sensitive to changes in the electrolyte concentration of the pore fluid. The presence of chemicals other than water in the pore space may influence the soil properties and behaviour.

From the literature review, there has been a lot of researches on the effect of chemical contamination on the geotechnical properties of soil but the amount of researches on the effect of crude oil contamination on the engineering properties of soil is small compared to that on the effect of crude oil contamination of soil on agricultural plant or on groundwater and surface water. Also, of the few researches that deal with effect crude oil contamination on the geotechnical properties of soil, there is so little that deals on clay soils majority has been on sand. Furthermore, the few researches on the effect of crude oil contamination on the geotechnical properties of clay soil have been mostly on montmorillonite clay while there has been no proper research on geotechnical properties of crude oil contaminated kaolinite clay soil. Also a considerable amount of information relating to crude oil as a contaminant has been published for soils formed in cold and temperate climates. However, there is only limited data on soils formed in tropical and dry (arid and semi-arid) climatic regions and kaolinite is the dominant clay soil in the tropical and dry climates where the vast amount the worlds crude oil are found.
This research will look at the effect of light crude oil contamination on the geotechnical properties (liquid limits, plastic limits, plasticity index, compaction characteristics, coefficient of permeability, cohesion and frictional angle, coefficient of consolidation and coefficient of compressibility) of kaolinite and will bridge this gap where there seems to be lack of knowledge. Therefore it is vital to establish the response of contaminated kaolinite clay soil in terms of engineering behaviour and comparison will be made between contaminated and uncontaminated soils.
CHAPTER 3: LITERATURE REVIEW 2: CRUDE OIL AND CLAY CHEMISTRY

3.1 Introduction

The contamination of soil by chemical may affect its behaviour and properties, therefore understanding the composition of the contaminating chemical and composition of the soil are important to the understanding of the geotechnical properties of contaminated soil. This chapter deals with hydrocarbon, its different forms and the crude oil chemistry. It also deals with the clay soil, its structure, mineral composition and interaction between chemicals and clay soil.

3.2 Hydrocarbons

Natural gas and petroleum are hydrocarbons formed by gradual decomposition of marine life and other biological materials and are found in porous rock formations in the upper strata of some areas of the earth’s crust (Routh et al 1971). Petroleum is a fossil fuel derived from ancient organic matters such as zooplankton and algae due to vast quantities of their remains settling at sea or lake bottoms, mixing with sediments and being buried. As the depth of the deposits on the sea or lake bed increased then intense heat and pressure built up in the lower region. These processes caused the organic matter to change first into a waxy material (kerogen) and with more heat into liquid and gaseous hydrocarbons via a process known as catagenesis.

That is to say that petroleum is a complex mixture of hydrocarbons that occur in the Earth. It may be in liquid, gaseous, or solid forms. The term crude oil is often restricted to the liquid form but in a technical term it also includes natural gas and the viscous or solid form known as bitumen (North, 1985 and Bates and
The liquid and gaseous phases of petroleum constitute the most important of the primary fossil fuels. The non-hydrocarbon component commonly found in crude oil and natural gases are sulphur, nitrogen and oxygen and their compounds, also found in crude oil are organo-compounds of certain heavy metals principally vanadium and nickel (North, 1985). Petroleum is used mostly by volume for producing fuel oil and gasoline (petrol), both of which are important primary source of energy. Due to its high energy content, transportability and relative abundance, petroleum has become the world's most important source of energy. Petroleum is also the raw material for many chemical products, including pharmaceutical products, solvents, fertilizers, pesticides, and plastics.

Petroleum is separated into its hydrocarbon fractions by distillation whereby the fractions are separated using their different distilling temperatures. The products of this fractional distillation are compressed natural gas, liquefied natural gas, petrol (gasoline), kerosene, diesel, lubricating oil and a residual fraction.

Hydrocarbons are often referred as Non- Aqueous Phase Liquids (NAPLs) in that even though they exist in liquid state, they do not mix with water. Differences in the physical and chemical properties of water and NAPLs result in the formation of a physical interface between the liquids that prevents the two fluids from mixing. NAPLs are classified as light non-dense aqueous phase liquid (LNAPLs) if they have densities less than that of water and dense non-aqueous phase liquid (DNAPLs) if they have densities greater than water. NAPLs can travel long distances either vertically or horizontally under gravitational influence (Sharma and Reddy, 2004, Rehman et al, 2010b).
3.2.1. Hydrocarbon structure

Hydrocarbons are a class of organic chemical compounds composed only of carbon (C) and hydrogen (H) elements. Hydrocarbons are divided into different subgroups based on covalent bonding (formed as a result of the sharing of one or more pairs of bonding electrons) between the carbon atoms in the compounds, as indicated in Figure 3.1.

![Diagram of hydrocarbon structure]

- = carbon electron, x = hydrogen electron

Figure 3.1 Examples of Covalent bonding of Hydrocarbon (Routh et al 1971)
The carbon atoms join together to form the framework of the compound with the hydrogen atoms attached to them in many possible configurations (some examples are shown in Figure 3.2).

The physical property of individual hydrocarbons depends in large measure on their structure and the types of chemical bonds that link together the atoms of their constituent molecules. For example, natural crude rubber is a hydrocarbon polymer, a chainlike molecule consisting of many units linked together and benzene is a ringed hydrocarbon with double bond (Figure 3.13).

Figure 3.2 Different configurations of hydrogen and carbon atoms (King et al., 1977)
Hydrocarbons are classified as either aliphatic or aromatic on the basis of their properties. Aliphatic hydrocarbons are derived by chemical degradation of fats or oils and aromatic hydrocarbons constitute a group of related substances obtained by chemical degradation of certain pleasant-smelling plant extracts (North, 1985). The aliphatic and aromatic compounds are distinguished on the basis of structure (as illustrated in Figures 3.3 and 3.4 respectively).

![Ethane](image1)

![Isobutane](image2)

Figure 3.3 Examples of Aliphatic hydrocarbon structure

![Benzene](image3)

![Toluene](image4)

![Naphthalene](image5)

Figure 3.4 Examples of Aromatic hydrocarbon structures (King et al. 1977)
Aliphatic hydrocarbons are divided into three main groups of alkanes, alkenes, and alkynes according to the types of bonds they contain. Alkanes have only single bonds, alkenes contain a carbon-carbon double bond, and alkynes contain a carbon-carbon triple bond. Alkanes are described as saturated hydrocarbons, while alkenes, alkynes, and aromatic hydrocarbons are said to be unsaturated. Aromatic hydrocarbon compounds contain a benzene ring as a structural unit, e.g. benzene, toluene, naphthalene.

3.2.2. Aliphatic hydrocarbons

An aliphatic compound is a hydrocarbon compound containing carbon and hydrogen joined together in straight chains, branched chains or non-aromatic rings and it includes alkanes, alkenes and alkynes.

The most abundant sources of alkanes are natural gas and petroleum deposits. Natural gas contains approximately 60–80% methane, 5–9% ethane, 3–18% propane, and 2–14% higher hydrocarbons (KCPC, 2003). Smaller quantities of alkanes can also be found in a variety of natural materials. Apart from the alkanes that are readily available from petroleum, alkanes are also synthesized in the laboratory and in the industry by the hydrogenation of alkenes (Bates and Jackson, 1987).

Alkanes have relatively low boiling points compared with polar molecules, i.e. a molecule that has a slightly positive charge at one end and a slightly negative charge at the other (for instance water) of comparable molecular weight. The boiling points of alkanes increase with increasing number of carbons because the intermolecular attractive forces, which individually are weak, become cumulatively more significant as the number of atoms and electron in the
molecule increases (King et al., 1977). Alkanes burns in air to produce carbondioxide (CO₂) and water (H₂O) and release heat.

The lower alkenes are produced commercially by cracking and dehydrogenation (removal of molecular hydrogen) of the hydrocarbons present in natural gas and petroleum. Higher alkenes and cycloalkenes are normally prepared by reactions in which a double bond is introduced by elimination. Alkenes react with a much richer variety of compounds than alkanes.

The physical properties of alkenes and alkynes are generally similar to those of alkanes or cycloalkanes with equal numbers of carbon atoms. Alkynes have higher boiling point than alkanes or alkenes. Because the electric field of an alkyne with its increased number of weakly held electrons, it is more easily distorted and it produced a stronger attractive force between molecules.

3.2.3. Aromatic hydrocarbons

Aromatic hydrocarbons are significantly more stable and are classified as either arenes, (which contain a benzene ring as a structural unit) or non-benzenoid aromatic hydrocarbons, which possess special stability but lack a benzene ring as a structural unit. All aromatic hydrocarbons are either liquids or solids at room temperature and they are insoluble in water.

Distillation of coal tar (a condensate from the roasting of coal) gives a number of hydrocarbon fractions; benzene, toluene, and other low-molecular-weight aromatic compounds (given off at low temperatures). The higher-boiling fractions are sources of aromatic compounds of higher molecular weight. Petroleum has now replaced coal as the principal source of aromatic hydrocarbons. The stability of the benzene ring enables certain processes known
generally as catalytic reforming to be undertaken in which alkanes are converted to aromatic hydrocarbon by a combination of isomerisation and dehydrogenation events as illustrated in equation 3.1.

\[
\text{Catalytic Reforming} \\
\text{C}_6\text{H}_{12} \xrightarrow{3.1} \text{C}_6\text{H}_6 + 3\text{H}_2 + \text{Heat} \\
\text{Cyclohexane} \quad \text{Benzene} \quad \text{Hydrogen}
\]

The aromatic hydrocarbons formed by catalytic reforming are used to boost the octane rating of gasoline and as starting materials for the synthesis of a variety of plastics, fibres, dyes, agricultural chemicals, and drugs.

Lubricating oils are produced from the fractional distillation of petroleum at the highest temperature (usually over 300°C) and consist of hydrocarbon with 20 or more carbon atom which can be separated into oils of different viscosity by fractional distillation.

3.3. Crude oil

Crude oil is a fossil fuel which is found trapped in underground rock from which it is obtained by drilling. It is a mixture of hydrocarbons which vary in state from almost solid to gas (Table 3.1) and can be found ranging from highly flammable light liquids to highly viscous and heavy tar-like materials (King et al, 1977, Ainley et al, 1985). Crude oil may be light or viscous because of their relatively molecular weights (Table 3.2).
Table 3.1 Composition of crude oil (KCPC, 2003)

<table>
<thead>
<tr>
<th>Formula</th>
<th>Name</th>
<th>Boiling point(°C)</th>
<th>Normal state at Room Temp (+20°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CH4</td>
<td>Methane</td>
<td>-161</td>
<td>gas</td>
</tr>
<tr>
<td>CH3CH3</td>
<td>Ethane</td>
<td>-89</td>
<td></td>
</tr>
<tr>
<td>CH3CH2CH3</td>
<td>Propane</td>
<td>-42</td>
<td></td>
</tr>
<tr>
<td>CH3CH2CH2CH3</td>
<td>Butane</td>
<td>-0.5</td>
<td></td>
</tr>
<tr>
<td>CH3CH2CH2CH2CH3</td>
<td>Pentane</td>
<td>+36</td>
<td>Liquid</td>
</tr>
<tr>
<td>CH3(CH2)6CH3</td>
<td>Octane</td>
<td>+125</td>
<td></td>
</tr>
</tbody>
</table>

However, crude oil does not consist simply of straight chain hydrocarbons, but is a mixture of linear, branched, cyclic and aromatic carbon compounds, and may contain sulphur, oxygen, and nitrogen in addition to carbon and hydrogen.

Table 3.2 Molecular size of crude oil fractions (KCPC, 2003)

<table>
<thead>
<tr>
<th>Fraction</th>
<th>No Of carbon</th>
<th>Boiling Point (°C)</th>
<th>% of crude oil fraction</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gases</td>
<td>1 – 4</td>
<td>&lt;0</td>
<td>2</td>
</tr>
<tr>
<td>Light Naphtha</td>
<td>5 – 7</td>
<td>27-93</td>
<td>34</td>
</tr>
<tr>
<td>Heavy Naphtha</td>
<td>6 – 10</td>
<td>93 -177</td>
<td>34</td>
</tr>
<tr>
<td>Kerosene</td>
<td>10 – 15</td>
<td>177 – 293</td>
<td>11</td>
</tr>
<tr>
<td>Light Gas Oil</td>
<td>13 – 18</td>
<td>204 – 343</td>
<td>21</td>
</tr>
<tr>
<td>Heavy Gas Oil</td>
<td>16 - 40</td>
<td>315 - 565</td>
<td>31</td>
</tr>
</tbody>
</table>

The composition of crude oil varies greatly from one source to another. The chemical composition of crude oil varies between regions and even within the same geologic formation, so that no two batches of crude oil are chemically identical (KCPC, 2003).

The petroleum industry often characterizes crude oils according to their geographical source e.g. Alaska North Slope Crude. Oils from different geographical areas have their own unique properties; they can vary in consistency from a light volatile fluid to a semi-solid. Classification of crude oil types by
geographical source is generally not a useful classification scheme for response personnel, because general toxicity, physical state, and changes that occur with time and weathering are not primary considerations (NOAA, 2007) rather crude oil is classified based on its effect on the environment into;

**Class A: Light, Volatile Oils.** These oils are highly fluid, often clear, spread rapidly on solid or water surfaces, have a strong odour, a high evaporation rate, and are usually flammable. They penetrate porous surfaces such as dirt and sand, and may be persistent in such a matrix. They do not tend to adhere to surfaces and flushing with water generally removes them. Class A oils may be highly toxic to humans, fish and other biota. Most refined products and many of the highest quality light crudes can be included in this class.

**Class B: Non-Sticky Oils.** These oils have a waxy or oily feel. Class B oils are less toxic and adhere more firmly to surfaces than Class A oils, although they can be removed from surfaces by vigorous flushing. As temperatures rise, their tendency to penetrate porous substrates increases and they can be persistent. Evaporation of volatiles may lead to a Class C or D residue. Medium to heavy paraffin-based oils fall into this class.

**Class C: Heavy, Sticky Oils.** Class C oils are characteristically viscous, sticky or tarry and are brown or black in colour. Flushing with water will not readily remove this material from surfaces but the oil does not readily penetrate porous surfaces. The density of Class C oils may be near that of water and they often sink. Weathering or evaporation of volatiles may produce solid or tarry Class D oil. Toxicity is low but wildlife can be smothered or drowned when contaminated. This class includes residual fuel oils and medium to heavy crudes.
**Class D: Non fluid Oils.** Class D oils are relatively non-toxic, they do not penetrate porous substrates and are usually black or dark brown in colour. When heated, class D oils may melt and coat surfaces which become very difficult to clean. Residual oils, heavy crude oils, some high paraffin oils, and some weathered oils fall into this class.

However, weather conditions and water temperature greatly influence the behaviour of crude oil and refined petroleum products in the environment. For example, as volatiles evaporate from a Class B oil, may become a Class C oil. If a significant temperature drop occurs (e.g. at night), a Class C oil may solidify and resemble a Class D oil and upon warming, the Class D oil may revert back to Class C (NOAA, 2007).

Also crude oils are categorized according to the molecular weight distribution of their constituents, and distinctions are made between light, medium, and heavy crude oil. Physical properties that distinguish heavy crudes from lighter ones include higher viscosity as well as heavier molecular composition, heavy oil has over 60 carbon atoms and hence a high boiling point and molecular weight.

Although low molecular weight hydrocarbons suitable for use as fuels are present in crude oil, it could be separated by distillation. Most of the hydrocarbons present in most crude oil are of high molecular weight and are broken down into smaller hydrocarbons through cracking (the process whereby complex organic molecules such as heavy hydrocarbons are broken down into simpler molecules by breaking the carbon-carbon bonds). Equation 3.2 and figure 3.5 shows more clearly what happens to the various atoms and bonds during the cracking of hydrocarbon.
Two processes, namely cracking and fractionation (basic refining process for separating crude petroleum into intermediate fractions of specified boiling point ranges) are used to transform amorphous black crude into useful materials. There are two forms of cracking i.e. thermal cracking and catalytic cracking, which involve different chemical reactions and give different distributions of products. In thermal cracking the hydrocarbons are heated to 750-900 °C in the absence of oxygen so they will tend to break up into smaller fragments. Thermal cracking breaks the carbon-carbon bonds so that each carbon atom ends up with a single
electron, i.e. free radicals are formed (Figure 3.6) and these can react in numerous ways.

![Thermal cracking diagram](image)

Figure 3.6 Thermal cracking of hydrocarbon (Clark, 2003)

In catalytic cracking the hydrocarbons are heated to a comparatively low temperature of 500-600 °C in the presence of a catalyst (figure 3.7). This causes the hydrocarbons to break up and form positively charged fragments and because of the relatively low temperature. They can recombine as well as break up. Modern cracking uses zeolites as the catalyst. Zeolites are complex aluminosilicates carrying a negative charge, which can remove hydrogen from an alkane together with the two electrons which bind it to the carbon. This leaves the carbon atom with a positive charge. It also produces high proportions of branched alkanes and aromatic hydrocarbons like benzene.
During the fractionation of crude oil, the fractionation column (Figure 3.8) is hot at the bottom and cool at the top and the crude oil is separated into fractions according to weight and boiling point. The lightest fractions, including petrol and liquid petroleum gas (LPG) vaporise and rise to the top of the tower, kerosene (aviation fuel) and diesel oil, stay in the middle of the tower, heavier liquids separate lower down and the heaviest fractions with the highest boiling points settle at the very bottom (Table 3.2).
Figure 3.8 Fractional Distillation (Ophardt, 1998)
3.4 Clay soils

Clay is a fine grained soil that shrinks and swells according to its water content. Clay minerals are typically formed over long periods of time by the gradual chemical weathering of rocks usually silicate-bearing rocks by low concentrations of carbonic acid and other diluted solvents. In addition to the weathering process, some clay minerals are formed by hydrothermal activity. Clay deposits may be formed as residual deposits in soil, but thick deposits are usually formed as the result of a secondary sedimentary deposition process after they have been eroded and transported from their original location of formation. Clay deposits are typically associated with very low energy depositional environments such as large lakes and marine basins. Primary clays are located at the site of formation while secondary clay deposits have been moved by erosion and water from their primary location.

The origin of clays is very closely related to the geological environment in which they are formed, temperature and chemistry are the primary factors which determine the type of clay which are formed (Mitchell and Soga, 2005). Clay minerals can originate from weathering of shale, slate, sandstone and limestone and also from the devitrification of volcanic ash that was deposited under marine conditions during geologic times. A subtropical climate influences the development of soils formed from the devitrification of volcanic ash and accelerates the desiccation and weathering processes of the source rock. Clay minerals have the capacity to change volume by adsorbing water molecules or other polar ions into their structure (swelling properties). Although all clays attract water to their surface (adsorption) some clay bring water into their structure (absorption). Sensitive clays have a higher proportion of water among
their small particles and during drought periods, there may be a severe loss of water and soil shrinkage occurs on the sensitive clay.

Swelling potential of a clay deposit may be due to local geology (rock type, age and the soil particle arrangement) and engineering factors (placement moisture content, Atterberg limits and dry density), while environmental factors (confining pressure, type and degree of wetting and water chemistry) control the amount of swelling (Sabtan, 2005). Furthermore, the magnitude of microscopic swelling, permeability and compressibility of clay depends on mineralogy, valence of cations, cation exchange capacity and structure at the atomic scale through clay layer orientation and texture at the aggregate scale through grain and pore distribution (Dananaj et al., 2005). According to Barbour and Yang (1993), soil grain can be described as elementary particles inside the soil aggregate (micro fabric) and the arrangement of the soil aggregate and the total porosity is distributed through a system of micro-pores and macro-pores. The hydraulic and mechanical performances of the natural soils are then controlled by the interactions between the aggregates. The compressibility of the soil structure is tied to the rearrangement of the soil aggregate and also to the shear strength at the inter-aggregate contact. The inter-aggregate pores control the hydraulic conductivity of natural clay.

3.4.1 The Structure of clay

According to Jeong et al. (2004) clay minerals are formed by chemical weathering, hydrothermal alteration and diagenesis. Chemical weathering is the breakdown of rocks which due to the chemical reaction between the minerals in the rocks and substances in the environment, such as water, oxygen, and weakly
acidic rainwater, hydrothermal alteration is the change of pre-existing rocks or minerals caused by the activity of hot solutions, such as fluids accompanying or heated by magma, diagenesis is the alteration of sediment at low pressure and temperature that can result in change to the rock’s original mineralogy and texture. Clay mineral may also be formed by physical weathering in continental and marine environments.

Clay particles consist of grains less than 2μm in diameter, and have a crystallographic habit which is sheet-like (Smith, 2006). Clay particles have a large surface area per unit volume and a complex structure compared to granular soils. Clay minerals are essentially hydrous aluminium silicates with magnesium or iron replacing wholly or in part, the aluminium in some minerals. Clay mineral composition is determined by the type and arrangement of the atoms which makes up the crystal structure of the solid matter.

The structural unit of the grain in cohesive soils comprises primarily silica and alumina of ferric oxide with varying amounts of other elements (Fang and Daniels, 2006). Clay can also contain organic matter or water-soluble salt.

Clays are divided into three main groups (kaolinite, montmorillonite and illite) based on their crystalline arrangement and particular engineering properties are assigned to a clay belonging to the same group (Murthy, 2002). In the more common clay minerals (kaolinite, montmorillonite, illite) certain atomic combinations are found to be repetitive (Fang and Daniels, 2006). One such combination is a sheet-like arrangement of silica tetrahedra (Figure 3.10) in which each of the structure consisting of four tightly packed oxygen atoms and a central silicon atom (Figure 3.9). Another basic combination is a sheet-like structure comprised of Aluminium octahedra (Figure 3.12) in which each of the
structure consisting of six oxygen or hydroxyl ions arranged around an Aluminium atom (Figure 3.11). The hexagonal planar network of Silica tetrahedra is known as a ‘Silica sheet’ and the network of octahedral units is termed a ‘Gibbsite sheet’. The crystal structure of many clay particles consists chiefly of alternations of these sheets (Figure 3.13).

In the tetrahedral structure (Figure 3.9), the Silicon ion shares its charge equally between the four oxygen ions, leaving each of the oxygen atoms with one excess negative charged ion. This anion tends to react readily with alkali and alkali earth ions although the $\text{SiO}_4^{4-}$ anion does have another option open to satisfy the charges. It is also possible for an oxygen ion to bond with two Silicon ions and thereby have its charge balanced. Theoretically this could happen by the three face oxygen ions, the two edge oxygen ions or the single corner oxygen ion
bonding with two silicon ions (Figure 3.10). The face sharing or edge sharing would require the two highly electropositive silicon ions to be closer together than is physically feasible making this option impossible. Thus only the sharing of the corner oxygen ion is a viable option (Sharma and Reddy, 2004).

![Figure 3.11 Octahedral arrangement of atom (Home, 1998)](image1)

The aluminium ion is "more comfortable" in an octahedral coordination (Figure 3.11) whereby the ion can be said to share +0.5 of its charge with each of the surrounding oxygen ions, leaving each oxygen ion with a negative 1.5 charge. This excess negative charge on the oxygen ions needs to be balanced and the charge can at least be partially balanced if each oxygen ion is bonded with two aluminium ions (Home, 1998, whitlow 2001). Once again, this could theoretically happen by the three face oxygen ions, the two edge oxygen ions, or
the single corner oxygen ion bonding with two \( \text{Al}^{2+} \) ions (Figure 3.11). In this case aluminium is slightly less electropositive than silicon and is able to approach close enough that corner oxygen ions can be shared. In a matrix of these octahedral units, each oxygen atom will be bonded to two aluminium ions, leaving it with a remaining -1 charge (Sharma and Reddy). When this type of structure is continued in three dimensions the Gibbsite mineral is produced (figure 3.12).

3.4.2. Clay minerals

The alternation of silica and gibbsite structure form the different clay minerals, figure 3.13 shows different alternations of silica and gibbsite sheets and there is a strong bonding (covalent bond) within each sheet (tetrahedral and octahedral formation) and between the sheets themselves. They may also be a relatively weak secondary bond (Hydrogen bond or Van der waal bond) between adjacent layers of two or three sheets (Mitchell and Soga, 2005). This weak bonding between layers permits excellent cleavage of clays and also the adsorption of metallic cations and organic substances on to the clay-mineral surface (Blatt, 1982). The specific arrangement of the sheets gives rise to three main different clay minerals (Kaolinite, Illite and Montmorillonite) with different engineering characteristics as a result of their differing platelet formations.
a = Kaolinite, b = Illite and c = Montmorillonite

Figure 3.13 Clay structure showing the alternation of sheets to give different clay minerals (Home, 1998)

**Kaolinite** is a hydrous aluminum silicate (with the chemical composition of $\text{Al}_2\text{O}_3\cdot2\text{SiO}_2\cdot2\text{H}_2\text{O}$) formed by the chemical weathering of feldspar and decomposition of aluminium silicate rocks. It belongs to the 1:1 clay minerals i.e. one silica sheet (tetrahedral) to one alumina sheet (octahedral), in alternating sequence as shown in Figures 3.13 and 3.14. The strong bond between the sheets (Figure 3.15) is the hydrogen bond (Sharma and Reddy, 2004). Kaolinite is not prone to shrinking or swelling with changes in water content. Among the physical characteristics of kaolinite are; its crystal habits which includes foliated and earthy mass where crystals of any size are rare and usually microscopic, also
the cleavage is perfect in one direction and is basal. It has a hardness of 1.5 – 2 with specific gravity of 2.6 (Mitchell and Soga, 2005).

(a) Silica and Alumina alternating sequence in Kaolinite

(b) 1:1 Sheet arrangement showing hydrogen bonding in kaolinite

Figure 3.14 Kaolinite sheet arrangement (Home, 1998)
Montmorillonite is a 2:1 mineral group i.e. it has two silica sheets (tetrahedral) to one alumina sheet (octahedral), arranged as shown in Figures 3.13 and 3.16, with alternation of the silica tetrahedron and alumina octahedron structures. In montmorillonite, one octahedral layer is sandwich between two tetrahedral layers and there are isomorphous substitutions in both the tetrahedral and octahedral layers. No apparent secondary bonding exists to connect these sheets together (Figure 3.17) and as a result water entering between these sheets causes high swelling to several times the original volume of montmorillonite crystal due to variable distance of the internal adsorptive surface and this makes montmorillonite a useful mineral for several purpose (Sharma and Reddy, 2004). Montmorillonite makes greater use of water and has a greater affinity for water than kaolinite because of the variable distance of the internal adsorptive surface, (Figures 3.13 and 3.16). Consequently, montmorillonite is regarded as expansive clay. However, this volume change can also present a problem as in swelling soils where large seasonal movement is induced in buildings sat on the clay. Montmorillonite is the main constituent in a volcanic ash called bentonite, which
is used in drilling mud, the bentonite gives the water greater viscosity (thickness of flow) which is very important in keeping a drill head cool during drilling.

(a) Alumina and Silica sheet alternating sequence

(b) 2:1 (Silica: Alumina) Sheet arrangement

Figure 3.16 Sheet arrangement in Montmorillonite (Home, 1998)
(a) Tetrahedra and Octahedra sheet arrangement

(b) Bond arrangement

Figure 3.17 Montmorillonite mineral (Home, 1998)
Illites are essentially a group of non-expanding, clay-sized, dioctahedral, micaceous minerals. Structurally the basic unit is a layer composed of two inward-pointing silica tetragonal sheets with a central octahedral sheet (Figures 3.13 and 3.18). It is non-expanding because the silica and gibbsite layers are held together strongly by the interlayer cation (potassium) i.e. the sheets are connected to each other by the K-bond (Figures 3.13 and 3.19). Because the interlayer cations are not easily removed, water will not cause much expansion (swelling) between the layers. This group is the dominant clay mineral in argillaceous rocks, formed by the weathering of silicates (primarily feldspar) through the alteration of other clay minerals and during the degradation of muscovite. Formation of illite is generally favoured by alkaline conditions and by high concentrations of Aluminum and Potassium. The general formula for illite is \((K, H)\text{Al}_2(Si, Al)_4\text{O}_{10}(OH)_2 - x\text{H}_2\text{O}\), where \(x\) represents the variable amount of water that this group could contain. The structure of this group is similar to the montmorillonite group with silicate layers sandwiching a gibbsite-like layer in between, in an s-g-s (Silica-gibbsite-silica) stacking sequence. The comparative weak interlayer forces caused by fewer interlayer cations in illite allow for more variability in the manner of stacking. The variable amounts of water molecules would lie between the s-g-s sandwiches as well as the potassium ions.
Figure 3.18 Bond arrangement in Illite (Home, 1998)

Figure 3.19 Sheet arrangement of Illite mineral (Mitchell and Soga 2005)
3.4.3 Charges in clay

Most clay minerals develop net charges on the clay surface (Mitchell and Soga, 2005), this net charges may be due to broken crystal bonds or isomorphous substitution (i.e. irreversible replacement of one atom by another of similar size in a crystal lattice without disrupting or changing the crystal structure of the mineral).

The main source of charge on clay minerals is from isomorphous substitution which confers permanent charge on the surface of most layered silicates. However ionization of hydroxyl groups on the surface of other soil colloids and organic matter can result in what is describes as pH dependent charges-mainly due to the dependent on the pH of the soil environment. Unlike permanent charges developed by isomorphous substitution, pH-dependent charges are variable and increase with increasing pH, also the presence of surface and broken - edge -OH groups gives the kaolinite clay particles their electronegativity and their capacity to absorb cations.

All clay particles have a finite size and the external or interlayer surfaces are generally negatively charged but at the edge of the mineral they can be positively or negatively charged in that there are some oxygen atoms that do not have their negative charges satisfied by cations in the mineral structure, thus at the edge of the mineral negative charges exist (Figure 3.20). Clay minerals hold an excess of negative charge on the surface. The spaces between mineral particles contain water with positively charges ions (cations) and negatively charged ions (anions). Therefore, the density of cations near the negatively charged mineral surface is high and is reduced as the distance from the surface increased.
With isomorphous substitution internal charges are not satisfied because of ionic substitution of Al$^{3+}$ for Si$^{4+}$ or Mg$^{2+}$ for Al$^{3+}$ (Mitchell and Soga, 2005). The net negative charge makes it possible for the clay soil to interact with other polar ions. The charge density and electric potential vary as a function of distance from clay surface, surface charge density, surface potential, electrolyte concentration and valence, dielectric constant of the fluid and temperature (Barbour and Yang, 1993). Where the cation from isomorphous substitution does not provide enough positive charges for neutralization within the mineral structure the excess negative charges must be neutralized at the mineral surface by adsorbing cations from the environment (pore fluid) and the phenomenon gives rise to cation exchange capacity (Fang and Daniel, 2006).

The behaviour of the soil mass is profoundly influenced by particle-water inter-relationships, the ability of the soil particle to adsorb exchangeable cations and the amount of water present (Murthy, 2002). A clay soil is composed of electrically negative mineral surfaces while water is composed of electric dipoles.
and predominantly positive charge ions (Fang et al., 2007). Hence contaminant flow in a soil is affected by electromagnetic energies and not by mechanical energy alone. However water movement between soil particles in a contaminated soil-water system is influenced by electric-viscous effects and the type and concentrations of the exchangeable ion play an important role. Ho and Pufahl (1997) observed that the presence of Na\(^+\) ions among the pore water molecules generates the response of the negatively charged clay particles to brine and in turn causes changes in the physical properties of the soils.

In clay-organic interaction, there is exchange of a hydrogen ion for another cation on the clay surface, if a hydrogen ion is present it will interact with the organic molecule, often changing its chemical character. Also, small organic molecules can be inserted between the swelling layers of clays in polymer adsorption whereby only the clay surface is involved, not the interlayer position and this is due to the irregular forms of the polymers (Mitchell and Soga, 2005).

All clay minerals have the property of plasticity, i.e. they readily react with water (Mitchell and Soga, 2005). As water moves in and out of the soil system the platelets are continually reacting with the water in hydration and dehydration cycles. This fundamental behaviour allows for manipulation of the chemistry of the clay and therefore it can be engineered by allowing the substitution of ions into the structure in place of the water molecules. By covering the points of contact on the clay molecules that are frequently taken up by water molecules with more stable ions the engineering performance can be greatly enhanced, thus reducing the desire of clays for dissociated water molecules (Mitchell and Soga, 2005).
3.5 Accommodation of fluid in clay

The interaction of water and clay is used to illustrate how fluids are accommodated in clay soils. According to Mitchell and Soga (2005), knowledge of soil water properties is important in understanding and quantifying geotechnical phenomena such as fluid flow through soil. Clay particles because of their small size, unique crystal structure and platy shape have very large specific surface areas and are especially influenced by the unbalanced force fields at the interfaces between soil and water.

Water has a dipolar molecular structure and this makes it possible to have a strong bond with clay soil (Sharma and Reddy, 2004). Water adsorption by clay soil is through the mechanism of hydrogen bonding, hydration of exchangeable cations, attraction by osmosis and charge surface-dipole attraction (Figure 3.21).

Clay particles and water molecules form hydrogen bond since the surface of the soil mineral are composed of either oxygen or hydroxyl in which case the oxygen will be attracted to the positive corner and hydroxyl will be attracted to the negative corner of the water molecule.

Clay minerals have a negative charge on their surfaces which are balanced by cations that are presences in dissolved forms in pore water (Mitchell and Soga, 2005) however these cations are not attached to the surface of the clay minerals which makes them exchangeable. But the cations are electrostatically attracted to the negative charged clay minerals and they (cations) have thermal energy so they tend to move around. The cations repels each other and water molecules try to surround them i.e. hydrate the cations, so the cations do not crowd the surface of the clay rather they form a diffused layer around the minerals. The charged surface and the distributed charge is known as the diffused double layer and the
water within this layer is known as double layer water (Figure 3.22). The double-layer thickness is the distance from the soil surface required to neutralize the net charge on the particle, or the distance over which there is electrical potential. The electrolyte concentration (concentration of cations) decreases exponentially with distance from the surface of the clay (Sharma and Reddy, 2004).

Figure 3.21 Mechanisms of water adsorption by clay surface;
(a) Hydrogen bonding, (b) Ion hydration, (c) Attraction by osmosis, (d) Dipole attraction (Mitchell and Soga, 2005)

According to Mitchell and Soga (2005), the diffuse double layer phenomena have some consequences on the soil interactions, for example, flocculation, dispersion,
swelling, shrinkage and plasticity are fundamental to virtually all aspects of soil behaviour. Exposure of soil mineral to inorganic chemicals can initiate a variety of processes that can cause reactions and alterations of the particles and other constituents of the soil mass for example, acids dissolves carbonates, iron oxides and the alumina octahedral layer of clay mineral, while bases (greater than 7 on the pH scale) can break silicate minerals. Oxidation of sulphides leads to the formation of sulphuric acid which can break down some rock mineral and reduction of sulphate creates an alkaline environment. In contaminated soil the liquid phase may undergo significant change in its composition depending on the chemicals, the adsorbed water, diffuse double-layer water and free water properties may be affected.

Figure 3.22 Diffuse double layer concept showing distribution of ions adjacent to clay surface (Mitchell and Soga, 2005)
3.6 Accommodation of organic chemical in clay

Knowledge of clay - organic chemical interaction is important in geotechnical and geoenvironmental engineering in solving problems such as contaminant migration, containment and the remediation of the contaminated soil. Crude oil has an affinity for clay and does not penetrate deeply into clay soil compared with sandy soil (Holliday and Deuel, 2009). Organic materials interact with clays by (1) adsorption on the particle surfaces e.g. hydrogen bonds (2) ion exchange (3) attraction of large organic molecules to clay surfaces by van der waals forces and (4) intercalation (entry of organic molecules between silicate layers). According to Mitchell and Soga (2005), the adsorption of organic compounds on clay surfaces in aqueous systems depends on the available surface and the ability of the organic molecules to displace water molecules that would otherwise be adsorbed. Cationic organics can exchange for inorganics adsorbed cations but if the organic cation is larger than the cation site then all exchangeable cations cannot be displaced. Also attraction of large organic molecules to clay surfaces by van der Waals forces may contribute to the total amount of organics held. The polarity, polarizability, solubility, size and shape are the important properties of organic molecules that are relative to their interaction with clays.

The most common mechanism that has been observed in adsorption of organic compounds is the ion exchange between the cations held in inner spaces of clay minerals and negatively charged organic molecules. Adsorption of organic compounds to soil is also influenced by molecular properties, including the electronic structure that is determined by the nature of constituent atoms and functional groups, the partitioning coefficient and size and shape of molecules.
3.7 Infiltration of crude oil into soil

According to (Youdeowei, 2012) any material with voids is porous and, if the voids are interconnected then it possesses permeability. Permeability or hydraulic conductivity is also the ability of a soil to conduct or discharge water under a hydraulic gradient. It depends on soil density, degree of saturation, viscosity and particle size. Materials such as clays and silts in natural deposits have large values of porosity (or void ratio) but are nearly impermeable, primarily because of their very small void sizes.

Oil spills on a permeable soil surface undergo three main processes (surface flow, infiltration and evaporation) which control the extent of the spill and the subsequent environmental impacts. The larger the area of pool, for the same volume spilt, the shallower is the zone of soil contaminated.

Crude oil can penetrate deep into the ground when spilt and the infiltration of hydrocarbon into the soil to a large extent are dependent upon the structure and characteristics of the soil, that is, porosity, humidity, organic matter content. In the pore space of crude oil contaminated soil since different phases coexist i.e. soil air phase, water phase and organic phase which seeps to the ground, the spreading of crude oil may be through the processes of advection, diffusion, dispersion, adsorption and biochemical degradation. However, the advective transport process represents the dominant process of infiltration and the air phase can be assumed to be inactive and Darcy’s law is acceptable to define the rate of penetration into the soil (Grimaz, et al., 2007). The principles of advective hydrocarbon transport are gravitational and capillarity. Hydrocarbons move through the soil under a three phase flow condition, displacing air and water.
Infiltration refers to the initial process of fluid (or contaminant) movement into unsaturated zone through the soil surface and the maximum rate at which fluid can move into the soil is called the infiltration capacity or potential infiltration rate. Infiltration of spilled oil is an important mechanism by which hazardous hydrocarbons may pollute the groundwater. Most oils act as non-aqueous phase liquids (NAPLs) and their migration in the vadose zone is influenced by the interaction between air, water and oil which occupies the pore space. The vadose zone which is the area between the surface of the land and the aquifer water table has a moisture content that is less than the saturation point and the pressure is less than atmospheric. Chemicals released at or near the land surface must pass through the vadose zone before reaching the water table. The depth to the water table (which is equivalent to the thickness of the unsaturated zone) is one of the parameters that determine whether or not a pollutant will reach the water table from a surface spill (Youdeowei, 2012).

And the degree of saturation of the soil with water will influence the rate of penetration of the crude oil into the soil. The more (water) saturated the soil, the larger is the surface pool and the smaller the depth of penetration of the oil into the ground. The larger the percentage of oil evaporated from the pool area the more limited is the volume of surface penetration by oil and the smaller is the depth of soil contaminated (Gawdzil, and Zygadlo, 2010).

Groundwater flow or solute (pollutant) transport at a given location depends on the permeability of the subsoil and the potential or hydraulic gradient. According to Grimaz et al. (2007) variations in soil permeability results in a deviation from the gravitationally dominated vertical flow; as the hydrocarbon encounters layers of slightly less permeable materials, or if materials with smaller pores are
encountered, it will tend to flow mostly in the horizontal direction until it encounters a path of less resistance. The distribution of the hydrocarbons in the soil is dependent on the pore size between the soil grains and the pore pressures of the air, water, and hydrocarbons occupying these pore spaces. The permeability of a soil for a hydrocarbon and water differs owing to differences in viscosity.

Vertical migration proceeds until the interface between the unsaturated zone and saturated zone is met, and at that point, the free phase solvent would either spread laterally, continue through the water table vertically as DNAPL, or a combination of both. Also as crude oil moves from a spill site, it contaminates the soil in the vadose zone since its components are largely water insoluble and less dense than water. Hydrocarbon free product tends to reside and spread along the water table boundary (Youdeowei, 2012). Unsaturated or vadose zone often contains greater amounts of organic matter than the saturated zone. Density and solubility are among the primary physical properties affecting the transport of separate phase liquids in the soil and water.

The variable possess by the soil are infiltration capacity and hydraulic conductivity and the variable possess by the crude oil are density, viscosity and interfacial tension. Both the soil and crude oil variables are responsible for the crude oil infiltration and migration in the soil during spill. In the field, the hydraulic properties of the soil permeability and porosity are heterogeneous and isotropic but in this research the hydraulic properties is homogeneous due to the fact that the test was conducted on a soil containing only kaolinite.
CHAPTER 4: METHODOLOGY 1: MATERIALS AND SAMPLE PREPARATION

4.1 Introduction

This chapter highlights the source of the materials (clay and crude oil) and the reasons for choosing the materials used in this research. It also deals with the method of preparing the test samples and how the different percentages of contaminated samples were mixed and prepared. It also deals with the reasons for choosing the different percentages of crude oil contamination used in the experiment. Chapter 4 also defined the different geotechnical properties that were tested in this research, the reasons for conducting the testing on contaminated soil and the significance of the tests to an engineer.

4.2 Materials for the research

4.2.1 Clay soil

The fine grained soil used in the experiments was a grey smooth to medium CLAY soil. The clay was supplied in the form of a wet damp block sealed in a plastic bag and each bag weighed about 50kg. The chemical analysis of the clay soil shows that it contains 63 % SiO$_2$, 23 % Al$_2$O$_3$, 2% K$_2$O, 2% Fe$_2$O$_3$, 1% TiO$_2$ and other minor compounds. The XRF and XRD results (appendix 3) showed that the soil is a kaolinite clay soil.

4.2.2 Crude oil

The hydrocarbon for this research is a crude oil (Forties Buzzard) and the chemical composition of the crude oil shows that it is a Brent light crude oil. It has a brown colour and a pungent smell with a flash point of - 50°C to 100°C.
(PMC). It also has a density of 7.36 – 9.81 kN/m³ at 15⁰C, kinematic viscosity of <7mm²/s at 40⁰C. The boiling point of the crude oil is -10 to 800⁰C with a vapour pressure of 40 – 700 kN/m² (Appendix 2b and 5). The forties buzzard crude oil blend has sulphur level of 0.2% by weight and an API gravity of 44.6⁰. API gravity is the American Petroleum Institute gravity which is a measure of how heavy or light petroleum liquid is compared to water (Bates and Jackson, 1987).

4.2.3 Reasons for choosing Kaolinite clay soil

The reason for choosing kaolinite as clay soil of interest for the research is based on the fact that kaolinite is the dominant clay mineral in the low latitude climate (tropical and dry climate zones) where there is vast crude oil exploration and reserve. As highlighted in chapter 1 there has been less research on the effect of crude oil contamination on tropical soils compared to that on the temperate soils. Again there are few researches on crude oil contamination within this low latitude climate zone and the few researches within this zone have been mostly on sandy soil or silty soil. There has been limit research on the effect of crude oil contamination on kaolinite clay and there is a gap in knowledge regarding the effect of crude oil contamination on the geotechnical properties of kaolinite clay soil. Also a good knowledge of the effect of crude oil contamination on the geotechnical properties of the clay soil will be of benefit on its use in the construction industry. For instance, Nigeria is within the tropical zone and has vast quantity of kaolinitic soil with kaolinite as the dominant clay mineral particularly in the Niger Delta region of Nigeria (Ugbe, 2011). There is large crude oil exploration within the Niger Delta region of Nigeria. A considerable increase in soil utility for engineering works is expected as Nigeria aspires
towards improved infra-structural development. Frequent occurrence of road pavement failure and building collapse has made it imperative for a proper understanding of the geotechnical properties of clay soils within the Niger Delta region as clay is predominant in most of the subgrade soils of Nigeria likewise in most countries within the tropical zone and due to the relative abundance of these soils and ease of acquisition they have found wide application in engineering construction works.

4.2.4 Reasons for choosing Brent crude oil

Wide variety of bench mark crude oil worldwide are considered to be light crude oil and as explained in Section 3.4 of Chapter 3, light crude oil are highly fluid, often clear, spread rapidly on solid or water surfaces. It will be easier for light crude oil (e.g. Brent crude oil or bonny light crude oil) to move into the soil than for heavy crude oil. Contamination of soil by light crude oil is one of the most encountered environmental problems (Aikam, 2002). Most soils are contaminated by light crude oil and it has the ability to infiltrate into the soil. Brent crude oil is a form of light crude oil likewise Bonny light crude oil which is dominant in the Niger Delta region of Nigeria. So the light crude oil was chosen because it is widely produced in the low latitude climate and in the tropics, again light crude is the dominant crude oil type within the Niger Delta area of Nigeria.

4.2.5 Reasons for using wet clay sample

The clay sample used in this research was supplied in wet form (as explained in Section 4.2.1), the reason for the supply of the clay in wet form was to avoid the clay from mixing with other form of soil e.g. sand which may be possible if the
clay sample was to be supplied in dry form. The wet clay sample reduces the risk of clay dust being blown around which if inhaled may be hazardous. Although the clay was supplied in wet form, it was sliced and dried during preparation so as to make it easier for the crude oil to mix with the soil sample.

4.3 Sample Preparation

This section deals with the way in which the soil samples for the tests were prepared and how the crude oil was mixed with soil before the tests.

4.3.1 Preparation of dry clay sample

Although the clay soil was supplied in wet lump block, due to its low permeability it was not possible to mix or inject the crude oil into the wet block therefore there was the need to prepare it in a dry powder form so that the different percentages of the crude oil contamination can be mixed. The pulverized dry powder clay sample was prepared from the wet lump by cutting it into tiny slices using a potato peeler and the sliced pieces were first air dried for 48 hours (Figure 4.1) then later oven dried at 50°C to ensure zero moisture. After drying the sliced clay sample, it was sieved using no 200 sieve (75 µm aperture sieve) and the sieved sample was stored to be used for the experimental test.
4.3.2 Determining the percentages of crude oil contamination

The contaminated soil sample for this research was prepared by mixing the dry soil sample with the light crude oil in the amount of 2%, 5%, 8%, 10%, 15% and 20% measured by weight of the dry soil sample to represent the different levels of crude oil contamination. The level of contamination in this research was defined as the percentage weight of crude oil with respect to dry weight of soil. These percentages of contamination represents points close to source of the contamination to point away from the source of the contamination since the concentration of contamination decreases as the horizontal distance increases, i.e. 20% is the point close to the source of contamination and 2% is the point away
from the source of the contamination while 0% represents clean uncontaminated soil.

**Reasons for choosing small increments**

The reason for choosing these different percentages of contamination is to examine if there are changes in the geotechnical properties as the concentration of the contamination varies. The selected percentages of contamination is of small increments i.e. 2%, 5%, 8%, 10%, 15% and 20% and these small increments were selected so as to examine how small a contamination could be to cause a change in the geotechnical properties of a soil. In a situation where the amount of crude oil contamination is up to 100%, it will draw attention and criticism which will result in the remediation of the contaminated soil but in a situation where the contamination is within this small increment, it can be easily ignored or overlooked.

**Reasons for not choosing higher percentage of contamination**

Higher percentages of contamination e.g. 25%, 50%, 75% and 100% were not selected (even though there may be points with these percentages of contamination especially within hours of the contamination) due to the fact that the mixed soil samples will be too wet for classification tests. The peak value in the compaction tests on the soil samples with more than 20% crude oil cannot be reached, it will be on the wet side of the compaction curve without increasing water. Excess crude oil will drain out of the sample during tests e.g. oedometer, triaxial and permeability tests thereby making it difficult for the tests to be conducted.

Apart from the above reasons for limiting the percentages of the light crude oil contamination to 20%, Shroff (1997) stated that the maximum percentage of
crude oil present in disturbed and undisturbed contaminated soil samples is within 10% after the crude oil contamination of soil, and this was also used as a justification to limit the percentage of contamination for this research to 20%.

So the main reason for selecting the small increment (2% to 20% crude oil contamination) for this research is based on the fact that in most cases crude oil contamination in undisturbed sample will be within the range of 20% contamination and this range can easily be ignored by the authorities since more attention will be paid to higher percentages of contamination. In a situation where crude oil might remain after remediation, it will be within this range. So there is the need to examine the effect of crude oil contamination on soil within a small increment.

4.3.3 Preparation and mixing of contaminated sample

The contaminated soil sample for each geotechnical test in this research was prepared by measuring out seven different portions of the dry soil sample. Six portions of the dry soil samples were mixed with different percentages of crude oil (2, 5, 8, 10, 15 and 20%) measured by dry weight of the soil sample (Figure 4.2). The remaining one portion was left uncontaminated (i.e. 0% contamination) which will act as control upon which the effect of the light crude oil contamination on the kaolinite clay soil will be compared. The mixing of the crude oil with the dry soil sample was done by hand and the mixed samples were then stored in a cellophane bag for 72 hours, this was to allow for proper mixing so as to attain a homogenous mixture. The cellophane bag also prevented the evaporation of the crude oil within the incubation period.
4.3.4 Adsorption of crude oil into the clay soil

According to Rehman et al. (2007) once crude oil spills, it move down through the soil due to gravity and saturate the soil in its part and it may spread horizontally by migrating within the capillary zone. The properties of the soil and the migration substances control the rate of migration (Rahman et al. 2010b). The spreading of a liquid spill depends on its physical characteristics (viscosity, density, and surface tension) and on its surface properties. According to Grimaz et al (2007), the migration of crude oil in the vadose zone is influenced by the interaction between the three immiscible fluids (i.e. air, water and oil). However, the advective transport process (the contaminant movement by flowing water in response to a hydraulic gradient) is the dominant process of infiltration and the air phase can be assumed to be inactive. Darcy’s law is also acceptable to define the
rate of penetration into the soil while crude oil viscosity and density remain constant. So the crude oil infiltrates the soil sample during the mixing and sample preparation stage (as describe in Section 4.3.3).

However the adsorption of organic compounds into the inner surfaces of the clay minerals is attributed to the bonding mechanism and the intercalation can be accompanied by electron or proton transfer resulting in charged layers. So as organic molecules are incorporated into the inter-layer spacing, strong covalent bonds and weak interactions occur between molecules and the host lattice. Since the intercalation process itself can produce charged layers in clay minerals. Changes in charge distribution on clay surfaces can even further increase the affinity of organic molecules to the clay surfaces thereby increasing the rate of adsorption of organic compounds into the external and internal surfaces. Also the intercalation of organic compounds may result in the enlargement or shrinkage of the interlayer spaces between the clay minerals. For example, polar organic compounds can penetrate into spaces between kaolin-like layers expanding the crystal from 0.72 nm to 1.00-1.47 nm (Yariv and Lapides, 2002). It is also possible for organic compounds to partially exclude water molecules from the interlayer spaces resulting in the reduction of thickness of the interlayer spaces. The interlayer thickness is also influenced by the orientation of adsorbed organic molecules (Satyanarayana and Wypych, 2004). Furthermore, larger concentrations of organic compounds lead to the larger concentrations of adsorbed compounds and the larger spaces between minerals. Such changes in the thickness of interlayer spaces may affect the soil properties, including the swelling behaviour and strength of soil (Yariv and Lapides, 2002).
4.3.5 Test for possible crude oil evaporation

Water content has been widely applied in geotechnical calculations relating to soil properties such as liquid and plastic limits, unconfined compressive strength and unit weight of soil. However the water content of contaminated soil cannot be calculated simply based on the weight loss of the sample during drying. Rather the calculation should be based on the difference in physical properties of pore fluid constituents. Organic fluids exhibiting boiling points higher than the boiling point of water will not evaporate easily at the temperature of the boiling point of water. On the other hand, organic compounds exhibiting boiling points lower than boiling point of water will evaporate more easily at the boiling point of water. So the evaporation rate of organic compounds from soil depends on the vapour pressure of the liquid and its boiling point.

Crude oil evaporation rate does not require consideration of wind velocity, turbulence level, surface area, thickness or size scale, the only factors important for evaporation are chemical and physical properties of the crude oil, time and temperature (Fingas, 2004). In crude oil contaminated soil, the evaporation of oil from the soils and the percentage of the remained oil solid material are extremely dependent on size and composition of soil particles. The evaporation of the oil also depends on the thermal properties of soil and oil. Evaporation increase with decreasing soil particle size and decreases with increasing oil content under the same environmental condition such as temperature and time (Khamehchiyan, 2007).

Since crude oil evaporate with temperature and time, it important to determine the rate of evaporation or the percentage of the crude oil that will evaporate and the percentage that will remain as oil residual if the contaminated soil samples are
place in the oven when determining the moisture content of the contaminated samples.

To test for this possible crude oil evaporation, six different soil samples (40g each) were measured out and the different percentage of contamination (2%, 5%, 8%, 10%, 15% and 20%) measured by dry weight of the soil sample were mixed with the different soil samples to form the contaminated soil samples. Then the contaminated soil samples were weighed in a container and placed in the oven at 105°C for 24 hours. At the end of the 24 hour period, the contaminated soil samples together with the containers were weighed again. The result of the test is shown in table 4.1 and figure 4.2

**Table 4.1 showing rate of crude oil evaporation**

<table>
<thead>
<tr>
<th>% Cont.</th>
<th>A</th>
<th>M1</th>
<th>M2</th>
<th>M3</th>
<th>M2 - M3</th>
<th>E</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>0.8</td>
<td>46.28</td>
<td>87.08</td>
<td>86.48</td>
<td>0.6</td>
<td>75</td>
</tr>
<tr>
<td>5</td>
<td>2</td>
<td>46.24</td>
<td>88.24</td>
<td>87.04</td>
<td>1.2</td>
<td>60</td>
</tr>
<tr>
<td>8</td>
<td>3.2</td>
<td>46.32</td>
<td>89.52</td>
<td>87.92</td>
<td>1.6</td>
<td>50</td>
</tr>
<tr>
<td>10</td>
<td>4</td>
<td>46.18</td>
<td>90.18</td>
<td>88.58</td>
<td>1.6</td>
<td>40</td>
</tr>
<tr>
<td>15</td>
<td>6</td>
<td>46.31</td>
<td>92.31</td>
<td>90.51</td>
<td>1.8</td>
<td>30</td>
</tr>
<tr>
<td>20</td>
<td>8</td>
<td>46.22</td>
<td>94.22</td>
<td>92.22</td>
<td>2</td>
<td>25</td>
</tr>
</tbody>
</table>

% Cont. = Percentage of crude oil contamination

A = Amount of crude oil added per 40g of soil sample measured by the weight of the dry soil

M1 = Mass of tin

M2 = Mass of tin + soil + crude oil before drying

M3 = Mass of tin + soil + crude oil after drying

M2 – M3 = Quantity of crude oil that evaporated
E = Percentage of evaporated crude oil (i.e. \((M2 - M3)/A\) * 100)

i.e. \(E = \left(\frac{M2 - M3}{A}\right) \times 100\) .......................................................... Equation 4.1

A graph of \(E\) was plotted against percentage of contamination to determine the rate of evaporation.

Figure 4.3 Graph of percentage of evaporation against percentage of contamination

Figure 4.3 shows that the percentage of crude oil evaporation decreases with increase in percentage of contamination under the same environmental condition.
i.e. under the same time and temperature. According to Khamehchiyan (2007), the cause of change in crude oil evaporation in the samples is due to thermal conductivity.

Due to the fact that crude oil will evaporate in the oven during test, the moisture content of all crude oil contaminated soil samples was calculated based on equation 4.2.

$$w\% = (1 - mn) \frac{W_t}{W_d} - (1 + n) \quad \text{equation 4.2}$$

where;

$W_t =$ weight of wet contaminated soil

$W_d =$ weight of dry contaminated soil

$m =$ crude oil residual after drying

$n =$ oil content before drying

An example illustrating the use of equation 4.2 in calculating the moisture content of crude oil contaminated soil is shown in Appendix 6.

4.3.6 Experimental Precautions

The experimental precaution explains the precaution that were taken during the laboratory tests, the hazards associated with the test materials and equipments used in the experiment, the risks and the steps taken to eliminate the risks.

4.3.6.1 Hazards associated with clay soil

The clay soil may contain free silica and if allowed to dry out during the test experiment, it may liberate dust containing respirable silica and this may pose danger to health by prolonged exposure. The health effects that can result from clay particle or dust are transient irritation of the eyes, abrasion of the skin, and
gastrointestinal irritation through ingestion. Inhalation of clay dust may cause severe irritation of nasal tissues, the throat and respiratory tract and could lead to lung damage. This hazard was eliminated by wearing face mask and hand gloves during the test especially during the sample preparation stage.

4.3.6.2 Hazards associated with crude oil

The crude oil may contain aliphatic, naphthenic and aromatic hydrocarbons and it may also contain gases, sulphur and nitrogen compounds. Crude oil may cause cancer and it is classified as a category 2 carcinogen. The toxic components of crude oil may enter the body by different routes; by breathing the vapour, by absorption through the skin or eyes, by swallowing or accidental ingestion. It also contains benzene and prolonged exposure to benzene can cause anaemia and other blood related diseases including leukaemia. It may contain significant quantity of polycyclic aromatic hydrocarbons some of which have been shown to induce skin cancer. Also crude oil is flammable and vapour containing hydrogen sulphide may accumulate during storage or transportation of crude oil. Heavy hydrocarbon gases and vapours can displace air from a confined environment and lead to suffocation. This risks were eliminated by wearing face mask and hand gloves. The mixing of the crude oil with the soil samples were conducted in a fume cupboard.

4.3.6.3 Storage and handling precautions

Due to the fact that crude oil is flammable, mixing of the crude oil with soil was done in a fume cupboard. The laboratory was well ventilated and any spillage was thoroughly cleaned immediately. The crude oil was stored in a container with air-
tight lid and it was stored with the soil samples in a secure store room. All experimental apparatus and the laboratory were cleaned at the end of each day during the period of laboratory experiment. During any experiment care was taken to adhere to appropriate United Kingdom’s legal provisions such as the Health and Safety at Work Act (1974), the Control of Substances Hazardous to Health Regulations (COSHH 2002), the Personal Protective Equipment at Work Regulations (1992) and the Manual Handling Operations Regulations (1992). Although crude oil may be biodegradable, spillage may penetrate the soil and cause ground water contamination, so the contaminated soil samples and crude oil were disposed of via an authorised licensed waste disposal site.

Laboratory coats were worn throughout the experimental test period. Although gloves were worn during the tests, hands were also washed after making contact with contaminated samples. And to avoid inhalation of clay dust and crude oil vapour, face mask was worn during experiments.

4.4 Geotechnical properties of soil

Soils are naturally occurring materials which are regarded as engineering materials and their physical characteristics can be determined by experiment and the application of the methods of analysis enables these properties to be used to predict their likely behaviour under working conditions (Head, 2006).

Among the advantages of measuring soil geotechnical properties by means of laboratory testing include having full control of the test conditions (including boundary conditions), it permits a greater degree of accuracy of measurements, changes in conditions can be simulated to represent the condition during and after construction and soil parameters can be derived within a reasonable time scale.
The understanding of geotechnical properties of soil has been of benefit to the engineer in terms of reduction of uncertainties in the analysis of foundations and earthwork, erection of structures and the use of soil as construction materials (e.g. earth dams and embankments).

In this research the geotechnical properties that were tested are index properties or consistency limits, compaction characteristics, permeability characteristics, consolidation characteristics and shear strength characteristics.

**4.4.1 Index properties (consistency limits)**

The index properties of soil relates to the amount of water in the soil (moisture/water content) and the way it can influence the soil behaviour. The consistency of fine soils varies drastically according to the amount of water present; when completely dry the soil may be hard (solid) while at high water contents it may be almost a slurry/liquid. The measurement of moisture content is use in classifying cohesive soil and assessing their engineering properties. A classification of the behaviour of soil into solid, semi-solid, plastic and liquid may be made on the basis of its moisture content, the transition between these states is gradual rather than abrupt (Sarsby, 2000). These consistency limits includes liquid limit, plastic limit, plasticity index and linear shrinkage. These limits which relate a change from solid state to fluid state or to semisolid state with change in water content are also known as Atterberg limits.

**4.4.1.1 Importance of soil index properties and its significance to an engineer**

Soil index properties are used extensively by engineers to discriminate between the different kinds of soil within a broad category, e.g. clay will exhibit a wide
range of engineering properties depending upon its composition. The Atterberg limits can be used to distinguish between silt and clay and it can distinguish between different types of silts and clays.

Classification tests to determine index properties will provide the engineer with valuable information on the soils characteristics when the results are compared against empirical data relative to the index properties determined. The index properties tests are used widely in the preliminary stages of building any structure to ensure that the soil will have the correct amount of shear strength and not too much change in volume as it expands and shrinks with different moisture contents.

4.4.1.2 Reason for index properties test on contaminated soil

Soil contamination may attack foundation of structures such as footings, caissons, piles and sheet piles. If the polluted water or contaminated soil is used for mixing concrete, it will affect the workability and durability of the concrete. In embankment construction, the moisture-unit weight relationship of soil will also be affected. A number of polar organic molecules have been observed to form interlayer complexes with clay and to cause expansion of the clay lattice (Sharma and Reddy, 2004). Crude oil contamination may cause significant changes in the Atterberg limits of the contaminated soil and Atterberg limits are particularly useful indices often used directly in soil specifications for controlling soils used in engineered fills. Test to determine the index properties of crude oil contaminated soil will be significant in the use of the crude oil contaminated soil for any geotechnical/ engineering purposes.
4.4.2 Compaction

Compaction is the process of reducing the void ratio of the soil through the removal of air by applying mechanical energy. Compaction improves soil properties by increasing the soil density and shear strength and decreasing compressibility. Compaction depends on the dry density, water content, compaction effort and type of soil (Sharma and Reddy, 2004). Many civil engineering projects require the use of soils as 'fill' materials and when soil is placed as an engineering fill, it is normally compacted to a dense state so as to obtain satisfactory engineering properties. Compaction on site is usually affected by mechanical means such as rolling, ramming or vibrating. Control of the degree of compaction is necessary to achieve a satisfactory result at a reasonable cost. Laboratory compaction tests provide the basis for control procedures used on site.

4.4.2.1 Importance of soil compaction test and its significance to an engineer

The importance of compaction is to ensure stability of structure and to minimise settlement as compaction is extensively employed in the construction of embankments and increasing the strength of roads and runways. Compaction reduces the void ratio and increases the shear strength making it more difficult for water to flow through soil. This is important if the soil is being used to retain water such as would be required for an earth dam. Compaction enables an engineer to understand the factors affecting the acceptability of fill material. It also enables an engineer to understand the factors affecting the field compaction of soils and to be able to interpret the results of the laboratory compaction tests. Laboratory compaction test provide the maximum dry density and optimum moisture content of soil for a given compaction energy. Laboratory test also
provide information on the suitable range of moisture contents for the placement in the field and are used to provide control of field compaction. However in most cases with certain soils the desire to hold volume change to a minimum may be more important than just an increase in shearing resistance.

4.4.2.2 Reason for compaction test on contaminated soil

Soil used in the construction industry requires preliminary testing in most localities in order to detect contamination, determine its suitability for construction, and select the proper type of foundation. In engineered fill, the soil is selected, placed and compacted to an appropriate specification with the objective of achieving a particular engineering performance which is adequate for the fill. Testing for compaction on contaminated soil will give an idea of the possible use of the contaminated soil as an engineered fill or its suitability for construction and the type of foundation to be adopted in the area with the contaminated soil. Compaction tests are critical in determining if contaminated clay soils are compatible with building, what kind of equipment should be used to compact the soil, and how much compaction is required before constructing the foundation. Soil compaction tests may also be performed to monitor problem areas for released contaminants and to keep an eye on environmental remediation programs. If land does require environmental clean-up, a soil test will be performed at the end of the clean-up to confirm that the soil has been properly handled and the land is safe to use.
4.4.3 Shear Strength

All the structures and buildings found in or on the land impose loads on the soil which support the foundations of such structure. The load impose on the soil may cause shear failure which occurs when the shear stress set up in the soil mass exceed the maximum shear resistance (shear strength) which the soil can offer. The shear strength of the soil is to guard against disastrous failure. So the shear strength of a soil is its resistance to shearing stresses, it is a measure of the soil resistance to deformation by continuous displacement of its individual soil particles. Shear strength in soils depends primarily on interactions between particles. Shear failure occurs when the stresses between the particles are such that they slide or roll past each other. Soil derives its shear strength from two sources: cohesion between particles (stress independent component) and frictional resistance between particles (stress dependent component). Cohesion is the cementation between sand grains or the electrostatic attraction between clay particles (Smith, 2006).

4.4.3.1 Importance of soil shear strength test and its significance to an engineer

The safety of any geotechnical structure is dependent on the strength of the soil, if the soil fails, the structure that is founded on it can collapse. Understanding shear strength is the basis to analyse soil stability problems like lateral pressure on earth retaining structures, slope stability and bearing capacity.

The knowledge of shear strength is very important to an engineer in that in the design of foundations the evaluation of bearing capacity is dependent on the shear strength and it is used in the design of embankments for dams, roads, pavements, excavations, levees etc. The analysis of the stability of a slope is done using shear
strength and it is used in the design of earth retaining structures like retaining walls, sheet pile, coffer dams, and other underground structures.

4.4.3.2 Reason for shear strength test on contaminated soil

Shear strength as a geotechnical property of soil is used in the assessment of engineering problem so the reason for shear strength test on the contaminated soil is to observe the difference in the shear strength of the soil due to the presence of the crude oil contaminant. The information obtained from the shear strength test on the crude oil contaminated soil tested in the laboratory will help the geotechnical engineer to design the structure that will be built on a crude oil contaminated land. The test will also help in the remediation of the contaminated land.

4.4.4 Permeability

Permeability of a soil is a measure of the soils capacity to allow the flow of a fluid through it (Head and Epps, 2011) or the ease with which fluid can pass through a soil, i.e. it is the property of a porous media that permits the transmission of fluid through it.

Soils are permeable (i.e. water may flow through them) because they consist not only of solid particles, but a network of interconnected pores. The degree to which soils are permeable depends on a number of factors, such as soil type, grain size distribution, water content, degree of compaction and stress history. The ability of soil to transmit water is characterized by the coefficient of permeability (or hydraulic conductivity).
4.4.4.1 Importance of soil permeability test and its significance to an engineer

Knowledge of the permeability characteristics of a soil is required for many construction projects in which drainage is an important feature. Permeability is a major parameter used in the design and assessment of land fill sites and the investigation of contaminated ground, the design of earth dams and sheet pile walls, and in assessing the potential for lowering groundwater levels.

The permeability of the soil either on natural ground or on contaminated soil is a critical parameter for design and construction purposes as well as for numerical modelling applications.

In environmental site characterization and geotechnical design, the importance of permeability to an engineer is based the fact that:

- The rate of flow to wells from an aquifer is dependent on permeability.
- The migration of contaminant seepage through a saturated or unsaturated soil profile is dependent on permeability.
- The design of earth dams is very much based upon the permeability of the soils used.
- Permeability influences the rate of settlement of a saturated soil under load.
- The performance of landfill or tailings impoundment liners is based upon their permeability.
- The stability of slopes and retaining structures can be greatly affected by the permeability of the soils involved.
- Filters to prevent piping and erosion are designed based upon their permeability.
4.4.4.2 Reason for permeability test on contaminated soil

Permeability test on contaminated soil is important since knowledge of the permeability properties of soil is necessary in estimating the quantity of underground seepage. Solving problems involving pumping of seepage water from construction excavation, stability analyses of earth structures and earth retaining walls subjected to seepage forces. Permeability test on crude oil contaminated soil is also needed when determining the rate at which the groundwater is contaminated and in ground water remediation.

4.4.5 Consolidation

Consolidation is the process whereby soils particles are packed more closely together over a period of time under the application of continue pressure and it involves the drainage of water from the pore spaces between the solid particles (Head and Epps, 2011). It can be said that consolidation is the gradual reduction in volume of a fully saturated soil of low permeability due to the drainage of some of the pore water over time. Whereas consolidation is the rate of volume change with time to produce an amount of settlement required, compressibility is the amount of volume changes in soil when subjected to pressure giving the amount of settlement.

4.4.5.1 Importance of soil consolidation test and its significance to an engineer

To an engineer the limitation of settlements to within tolerable limits is sometimes of greater significance in foundation design than the limitation imposed by bearing capacity requirements derived from shear strength.
Estimating the rate of settlement and the time within which the settlement will be completed is an important factor for the engineer in foundation design.

4.4.5.2 Reason for consolidation test on contaminated soil

Consolidation test on crude oil contaminated clay soil is important since it will provide information on the rate and amount of settlement that the soil will undergo with time as clays and other compressible soils can be subject to long-term consolidation under the loads imposed by foundations and above ground structures and settlement may occur even if the applied pressure is within the safe bearing capacity of the soil. So among the reasons for consolidation test on contaminated soil is to know the difference in the rate and amount of settlement compare with that of uncontaminated soil.

4.4.6 Reason for limiting the research to these geotechnical properties

Soil properties that may be affected by crude oil contamination includes chemical and geotechnical properties. The chemical properties include pH value, sulphate content, organic content, carbonate content, chloride content and total dissolve solid, also cation exchange capacity and anion exchange capacity. Although these chemical tests may be of interest for civil engineering purposes, there may be a big source of error on the quantitative chemical analysis of soil due to the selection of the test sample and chemical test should be perform by a special trained chemist (Head, 2006). The chemical property of soil is not of significant important when considering foundation design. The chemical test and analysis of the crude oil contaminated soil is beyond the scope of this research, so the chemical properties of the soil are not tested in this research so as to avoid errors.
This research is limited to the geotechnical properties of soil since they will be of significant important to an engineer in the design of geotechnical structure, foundation and geo-environmental studies (e.g. design of earth barrier in waste containment system), also the geotechnical properties of soil is a useful information when considering the effective use of the contaminated soil and its remediation.

The geotechnical properties of soil considered in this research are index properties, compaction properties, permeability properties, shear strength properties and consolidation properties. However there are some geotechnical properties that were not test for this research. The geotechnical properties and the reason there were not tested are;

- **Particle Size Distribution** – The particle size analysis is use to group the soil particle into separate ranges of sizes and determines the relative proportion by dry mass of each size range. Particle size analysis is performed mostly on soil containing both coarse and fine particle. This test (PSD) was not conducted in this research because the test soil comprises of only clay soil.

- **Erodibility test** – this is used for the identification of soil susceptible to being eroded. However it is the presence of sodium and the relationship of the concentration of sodium cations to other metallic cation which is the prime factor responsible for clay being dispersive (Head and Epps, 2011). This test was not conducted in this research due to the fact that the test requires a specialist chemical testing laboratory and this was not available as at the time conducting the geotechnical test.

- **California Bearing Ratio** – this is an empirical penetration test use to estimate the bearing value of highway sub-base and subgrade. The CRB value enables
the determination of a suitable thickness of sub-base construction to withstand the anticipated traffic conditions over the design life span of the pavement. According to Head and Epps (2011), CBR value is a dimensionless number and is not related to fundamental soil properties governing shear strength or compressibility. The application of CBR value is restricted to pavements construction and not used in the estimation of bearing capacity of ground for foundation (Head and Epps, 2011). The CBR test was not conducted in this research based on the fact that the CBR equipment was not available in the laboratory used to conduct the geotechnical tests.

4.5 Summary

As explained in this chapter the materials used in this research are kaolinite clay soil which is the dominant clay in the low latitude climate which includes the tropical zone of the world and the Brent light crude oil which spread rapidly on soil. The geotechnical properties tested are index properties, compaction, shear strength, permeability and consolidation. These properties are importance to an engineer in that the information obtained from the tests will be useful in the design of any geotechnical structure such as foundations, pavement, dam etc., and conducting these tests on contaminated soil will show if there are significant differences on the result when compared with that of the uncontaminated soil. The geotechnical tests will also be useful on how to effectively use the contaminated soil and on the remediation of the contaminated soil.
CHAPTER 5: METHODOLOGY 2: SPECIFIC TESTS, EQUIPMENT AND TEST PROCEDURES

5.1 Introduction

Knowledge of geotechnical properties of soil is important in determining the use and application of the soil and the information from geotechnical soil test is used by geotechnical engineers when designing structures, foundations, buildings, bridges, tunnels and dams. This chapter presents the different kinds of geotechnical tests conducted on clean uncontaminated soil and crude oil contaminated kaolinite clay soil, the reason for choosing each type of geotechnical test, the equipment and the detailed test procedure used in conducting the each of the different geotechnical test, the important test parameters for each geotechnical test and why compacted samples were used for permeability, consolidation and triaxial tests.

The methods used for the experiments in this research are those specified in the British Standard (BS1377: 1990), for testing soils used for Civil engineering purposes. At the time of commencing the research reported herein, laboratory tests on soils in the United Kingdom were generally carried out in accordance with the British standard and this was the reason for adopting this standard. Soil tests for civil engineering purposes can also be conducted using the American Society for Testing and Materials (ASTM) which is similar to the BS standard but is often different in details. The ASTM standard is mostly used in United States of America. Other countries have their own sets of standards some of which are based on British or US practice. The establishment of EUROCODES (Euro Code 7: EN 1997: 2-5) to harmonize European standards occurred towards the end of the research test period and these codes have not been considered in this work.
The tests conducted in accordance with British Standard BS 1377:1990 were:


5.2 Index properties tests

Index properties refers to those properties of a soil that indicate the type and condition of the soil and it provide a relationship to structural properties such as strength, compressibility, permeability, swelling potential, etc.

Index properties allows for the use of empirical correlations for some other engineering properties. The index properties tested in this research are liquid limit, plastic limit, plasticity index and linear shrinkage.

The principal objective of any soil classification system is to predict the engineering properties and behaviour of the soil based on few simple laboratory or field tests. Laboratory or field test results are used to identify the soil and put it into a group that has soils with similar engineering characteristics (Craig, 2004, Sarsby, 2000). Hence the classification tests indicate the general type of soil and the engineering category to which it belongs.

5.2.1 Liquid limit

This is the water content at which a soil changes from plastic to liquid behavior. It is the minimum water content at which the soil is assumed to flow under its own weight (Sarsby, 2000). The importance of the liquid limit test is to classify soils
because different soils have varying liquid limits. Also to find the plasticity index of a soil there is the need to know the liquid limit and the plastic limit.

5.2.1.1 Kinds of liquid limit test

The most commonly methods for determining liquid limit test are the casagrande method and the cone penetration method.

**Casagrande method** – In this method soil is placed into a circular metal brass cup and a groove of about 2mm wide is made down its center to separate it into two halves. The cup is then lifted and allowed to drop onto a hard rubber base. The number of such blows to cause the two soil halves to come together over a distance of 13mm is recorded and soil sample is taken to determine the moisture content of the soil. The test is repeated and a graph of moisture content versus number of blows is plotted. The flow of the two halves towards each other is related to the moisture content of the soil and the liquid limit is defined as the moisture content when the condition is achieved after 25 blows. This Liquid Limit test is defined by ASTM standard test method D 4318 (Barnes, 2000).

**Cone Penetration method** – This method is based on the measurement of penetration into a soil of a standardized cone of specified mass. The moisture content at the point of each penetration is measured. As the moisture content is increased by small amount, the penetration increases. The test is repeated with the same soil but with further addition of distilled water and a plot of cone penetration versus moisture content is obtained. The liquid limit of the soil is taken as the moisture content at the penetration of 20mm.
5.2.1.2 Research test method and the reason for selecting the method

The cone penetrometer method was used for the liquid limit test in this research and the reason for choosing this method is based on the fact that it is easier to perform and it is less dependent on the operator when determining the liquid limit. The cone penetrometer method gives more reproducible results compared to the Casagrande method. The casagrande method is sensitive to operator error and requires judgment concerning the closing of the gap and the results are affected by the hardness of the rubber base on which the cup is dropped.

5.2.1.3 Liquid limit test procedure

The liquid limit test apparatus using the cone penetration method comprises of a penetrometer which has a sharp-pointed stainless steel cone with a smooth polished surface which is 35mm long (Figure 5.1). The point of the cone has an angle of 30° and the mass of the cone (together with the sliding shaft on which it is mounted) is 80± 0.1 g (Figure 5.2). The cone penetrometer apparatus include a clamp which prevents the cone from falling and a control button which allows its free fall. The apparatus also include flat glass plate (about 500mm square with 10mm thickness) which acts as a surface for mixing samples, a brass cup (55mm diameter and 40mm depth) where the soil to be tested is contained. Additional standard laboratory equipment include timer, weighing scale, wash bottle and distilled water, metal straight edge, palette knives/ spatulas and moisture content tins (Figure 5.1).
Figure 5.1 Liquid limit apparatus.
Test on the uncontaminated soil

To determine the liquid limit of uncontaminated soil, a piece of clay weighing about 300g was cut from the clay as supplied and was sliced into thin sheets and mixed with distilled water on a glass plate until the soil turned into a stiff paste. This paste was pressed into a brass cup using a spatula until the cup was full and the top of the soil was trimmed level using a straight edge. Care was taken to avoid trapping air within the clay soil in the cup. The cup was placed on the platform of the cone penetrometer apparatus and the plunger was lowered so that
the tip of the cone just touched the surface of the soil. The initial reading of the vertical deflection dial gauge was taken and then the cone was allowed to fall freely (by pressing the release button) for 5 seconds. After this time the final reading of the vertical dial gauge was taken and the cone was removed from the clay. Two samples of clay were taken from the place where the cone penetrated the soil and their moisture contents were determined. The brass cup was emptied and cleaned and a repeat measurement of cone penetration was conducted at this moisture state. Two moisture contents were also determined from this second test. After the second test, all of the soil both in the brass cup and on the glass plate were mixed with more distilled water so that a uniform but softer mix was obtained which was then tested using as on the first test. The process was repeated until at least five different penetration values ranging from about 12mm to about 26mm were obtained.

The moisture content of the soil from each of the penetration readings was calculated using the moisture content formula i.e.

\[
\text{Moisture content} = \left( \frac{\text{loss of moisture}}{\text{Dry mass}} \right) \times 100 \]

**Equation 5.1**

Where \( m_1 \) = mass of container, \( m_2 \) = mass of container and wet soil, \( m_3 \) = mass of container and dry soil, then the moisture content (\( w \)) will be;

\[
 w = \left( \frac{m_2 - m_3}{m_3 - m_1} \right) \times 100 
\]

**Equation 5.2**

A graph of cone penetration (as the ordinate) against moisture content (as the abscissa) was plotted and the line of best fit through the data point was subsequently drawn. The value of moisture content which gives a cone penetration of 20mm was defined as the liquid limits. The value of the moisture content was calculated to the nearest 0.1% and normally it is reported to the
nearest whole percentage value, but for this research the moisture content was reported to 0.1%.

**Test on the contaminated soil**

To test for the liquid limit of the contaminated soil, about 300g of dry soil sample (prepared as described in Section 4.3.1) was measured out and was mixed with the required percentage of crude oil measured by weight of the dry soil. The soil sample mixed with crude oil was stored in an air tight cellophane bag for 72 hours this was to allow the soil and the crude oil to mix properly. After the 72 hour period, the liquid limit test was conducted using the same procedure as the uncontaminated soil. The liquid limit value was determined by plotting cone penetration against moisture content. This procedure was used to test for the liquid limit of all the contaminated samples i.e. 2%, 5%, 8%, 10%, 15% and 20%.

5.2.2 Plastic limit

This is the moisture content at which a soil passes from the plastic state to the solid state and becomes too dry to be in a plastic condition i.e. it is the water content of the soil below which it ceases to be plastic. It is the moisture content where the soil sample begins to crumble when rolled into threads of 3mm in diameter. A soil is considered non-plastic if a thread cannot be rolled out down to 3mm at any moisture.

5.2.2.1 Kinds of plastic limit test

The test method commonly used to determine the plastic limit of soil is that which involve rolling the soil into a ball which is then rolled out between the hand and a
glass to form a thread (BS 1377: part 2: 1990: 5.3 and ASTM D 4318, 15) however according to Smith (2006), the cone penetrometer can also be used to determine the plastic limit of a soil in the same way as liquid limit where a penetration of 2mm gives the plastic limit.

5.2.2.2 Research test method and the reason for selecting the method

The British Standard BS 1377: part 2: 1990: 5.3 was the plastic limit test method adopted for this research. Although this method may be dependent on individual operator's finger, hand pressure and judgment concerning the achievement of the crumbling condition at required diameter, it was selected because it is the most popular method used in determining the plastic limit of soil.

5.2.2.3 Plastic limit test procedure

The plastic limit test apparatus comprised of a glass plate for rolling a clay ball sample into threads, a 3mm diameter metal rod for checking the thickness of the rolled threads and moisture content tins.

Test on the uncontaminated soil

To test for the plastic limit of uncontaminated soil, a stiff soil paste was prepared in the same manner as in the liquid limit test (Section 5.2.1.3 for uncontaminated soil). The soil was spread over a glass plate and was gathered up and moulded into a ball by rolling it between the fingers and the palm. The ball was divided into two portions and each portion was further sub-divided into four equal parts. One part was then placed on the glass plate and rolled out manually using a back-and-forth movement to produce a uniform thread about 3mm diameter. If the
thread did not crumble as it was rolled the soil was reformed into a ball and hand-rolled again to dry it. The process was repeated until a 3mm thread was produced which did crumble when it was rolled. The crumbs were collected and placed in a moisture content container. Each of the remaining three parts of the soil portion was subjected to the same process and the average moisture content of the soil crumbs was determined. The process was repeated for the second soil portion. The value of the plastic limit was reported to the nearest whole percentage.

**Test on the contaminated soil**

The plastic limit test for the contaminated soil was prepared in the same way as that for the liquid limit test for the contaminated soil Section 5.2.1.3 and the test procedure was the same as that of the plastic limit for uncontaminated soil.

**5.2.3 Plasticity Index**

The plasticity index is the range of water content within which a soil is plastic and it is the numerical difference between the liquid limit and the plastic limit. It indicates the size of the range over which the material acts as a plastic i.e. capable of being deformed under stress, but maintaining its form when unstressed.

**5.2.3.1 Kinds of plasticity index test**

The method of determining the plasticity index depends on the method used in determining the liquid limit and the plastic limit since the plasticity index is the difference between the liquid limit and the plastic limit.
5.2.3.2 Research test method and the reason for selecting the method

The method that involve calculating for the difference between the liquid limit and the plastic limit was adopted for this research i.e. the BS 1377:1990:5.3 was the method used in calculating the plasticity index for this research.

5.2.3.3 Plasticity index test procedure

Test on the uncontaminated soil

The plasticity index of a soil is the numerical difference between the liquid limit and the plastic limit, and it is a dimensionless number (i.e. PI = LL-PL). Both the liquid and plastic limits are based on moisture contents. Hence the plasticity index for the uncontaminated soil was determined based on liquid limit and plastic limit of the uncontaminated soil in Sections 5.2.1 and 5.2.2.

Test on contaminated soil

The Plasticity index of the contaminated soil was calculated in the same way as in uncontaminated soil using the liquid limit and plastic limit of the contaminated soil in Sections 5.2.1 and 5.2.2.

5.2.4 Linear Shrinkage

Linear shrinkage is the change in the length of a bar of soil sample when dried and is expressed as a percentage of the initial length. Linear shrinkage is the decrease in length of a soil sample when oven-dried, starting with a moisture content of the sample at the liquid limit. In addition to indicating the amount of shrinkage, the linear shrinkage test can provide an approximate estimate of the
plasticity index for soils in which the liquid and plastic limits are difficult to determine (Head, 2006).

5.2.4.1 Kinds of linear shrinkage test

The test method for the linear shrinkage is the BS 1377: part 2: 1990: 6.5. This method is similar to that described in the ASTM standard. The shrinkage limit test was not conducted in this research due to the fact that the equipment required to conduct the test was not readily available as at the time of tests.

5.2.4.2 Research test method and the reason for selecting the method

The BS 1377: part 2: 1990: 6.5 test method was adopted for this research and the reason for using this method is based on the fact that it is simple and easy to conduct.

5.2.4.3 Linear shrinkage test procedure

The linear shrinkage test apparatus comprises of a half-cylinder brass metal mould (about 140mm long and 25mm diameter) that was filled with soil. Additional equipment includes a flat glass plate and palette knives for mixing the soil sample, petroleum jelly to rub on the brass mould to reduce friction and vernier callipers for measuring the length and width of the soil sample (Figure 5.3).
Figure 5.3 Shrinkage limit apparatus

**Test on the uncontaminated soil**

The soil for this test was prepared in the same way as for the liquid limit tests for uncontaminated soil (Section 5.2.1) and the sample was taken at a moisture content approximating to the liquid limit. A thin smear of grease was applied to the inner surface of the semi-cylindrical shrinkage mould to prevent soil sticking to the mould and then the soil sample was pressed into the mould until the mould was slightly overfilled (the soil sample was pressed into the mould in
such a way as to avoid trapping air). The surplus soil was scraped off to level with the top edge of the mould using a straight edge and then the fill mould was left exposed to air dry slowly for a day. Later the soil sample in the mould was oven dried at 60°C on the second day before being dried at 105°C on the third day. After drying at 105°C the sample was taken out of the oven and was allowed to cool then the length of the bar of soil sample was measured using the vernier callipers.

The linear shrinkage value was calculated as a percentage of the original length of the specimen from the formula; 

\[ Ls = \left[ 1 - \left( \frac{LD}{LO} \right) \right] \times 100 \]

Equation 5.3

Where \( L_s = \) Linear Shrinkage, \( L_O = \) Original length of the mould, \( L_D = \) Length of dry soil sample. The linear shrinkage is reported to the nearest whole number.

**Test on the contaminated soil**

The soil for the linear shrinkage test on the contaminated sample was prepared as in the liquid limit test for contaminated sample (Section 5.2.1) and the measurement of the linear shrinkage for the contaminated soil was calculated in the same way as in the linear shrinkage of the uncontaminated soil discussed above.

**5.3 Compaction Test**

Compaction increases the bulk density of soil by driving out air. For a given amount of compactive effort, the density obtained depends on the moisture content (see Section 4.4.2). The main objective of laboratory compaction test is the classification and selection of soils use in fills and embankments and for
providing a specification for earth works. The laboratory tests may be used to define a suitable moisture content range for field compaction (Sarsby, 2000), i.e. the laboratory compaction tests provide the basis for control procedure used on site. Compaction is employed in the construction of road bases, runways, earth dams, embankments and reinforced earth walls and in some cases, compaction may be applied in the preparation of a level surface for building construction. Compaction increases the shear strength of the soil and reduces the voids ratio making it more difficult for fluid to flow through the soil. This behavior would be very important if the soil was being used to construct banks to contain oil spills or if oil contaminated soil was being used in the restoration of a disaster zone e.g. post-earthquake. The essence of the test is to see if contamination of soil by crude oil will have any effect on the compaction characteristics of the soil.

5.3.1 Kinds of compaction test

The British Standard and ASTM both have similar method for the compaction test however the different methods of laboratory compaction tests are;

- Light compaction (the proctor method) – this method uses a 2.5kg rammer that falls through a height of 300mm in a metal mould of 105mm in diameter and 1 litre in volume with 27 blows. The sample in the mould is compacted in three layers. The method represents the BS 1377: 1990: part 4: 3.3 and it is similar to ASTM D 698.

- Heavy compaction – this method uses a 4.5kg rammer and the test procedure is the same as that of light compaction method except that the rammer falls through a height of 450mm and the soil is compacted in
five layers. This method represents the BS 1377: 1990: part 4:3.5 and it is similar to ASTM D 1577. Both the light compaction and the heavy compaction can be done manually by lifting and dropping the rammer or can be done using automatic mechanical machine.

- Vibrating hammer – this method is more appropriate for granular soil rather than cohesive soil and the soil is compacted in a CBR mould of 152 mm in diameter and 2305 cm$^3$ in volume and the soil sample is compacted in three layers using a vibrating machine operating at a frequency between 25 and 45 Hz for a period of 60 seconds. The method represents the BS 1377: 1990: part 4:3.7.

- The California department of transportation developed a test (California Test 216) which measures the maximum wet density, and controls the compactive effort based on the weight (not the volume) of the test sample. The primary advantage of this test is that maximum density test result is fast because evaporation of the compacted sample is not necessary.

5.3.2 Research test method and the reason for selecting the method

Laboratory compaction tests are intended to model the field process and to indicate the most suitable moisture content for compaction (the ‘optimum moisture content’) at which the maximum dry density will be achieved for a particular soil.

In this research the standard light compaction test (2.5 kg rammer method) BS 1377: 1990 part 4:3.3 was used in conducting the compaction test. The reason for choosing this method is because it is suitable for soils containing particles no
larger than 20mm. Although the heavy compaction method (4.5kg rammer method) is also suitable for soil particle no larger than 20mm, it is often specified where higher levels of compaction are necessary in a structure, e.g. an airfield sub-base material. However, excessive compaction poses a risk of fracturing granular soils resulting in the reduction of soil strength parameters. The light compaction method was also used because it is easy and simple, the equipment and apparatus were easily available during the laboratory work of this research.

5.3.3 Compaction test procedure

The standard compaction test apparatus comprised of a cylindrical metal mould of about 105mm in diameter and approximately 115.5mm in height or approximately one litre in volume (Figure 5.4) and a steel rammer weighing 2.5 kg with a 50mm diameter head fastened to a rod which was contained inside a steel tube about 350mm long (Figure 5.5). The rammer slides free in the steel tube. Other equipment include a metal tray for mixing the soil sample, a scale balance with accuracy of 1g for weighing the samples, measuring cylinder, distilled water, an extruder, steel straight edge and a scoop trowel, palette knife, and moisture content tins (Figure 5.6). At the beginning of the experiment, the 2.5kg rammer was checked to make sure that it falls freely through the correct height of drop i.e. 300mm. Although the experiment can be done using the automatic mechanical compaction machine, for this research the experiments for the compaction were done manually.
The compaction apparatus set up involves fitting the cylindrical metal mould with the detachable baseplate and removable extension collar (Figures 5.4 and 5.6).

Figure 5.4 Description of one-litre compaction mould (Head, 2006)
Figure 5.5 Description of 2.5kg compaction rammer (Head, 2006)
Figure 5.6 Compaction apparatus

**Test on the uncontaminated soil**

Approximately 6 kg dry soil sample was measured out from the prepared dry soil sample as described in Section 4.3.1 and placed in a metal tray. Distilled water was added to the dry clay to bring it to a moisture content of 8% and the soil was mixed thoroughly until the colour was uniform. This starting moisture content was chosen because it was on the ‘dry side’ of the optimum water content.
The cylindrical mould, base plate and extension collar were assembled, weighed and placed on a solid floor then sufficient loose soil sample was added to the mould to ensure that after compaction the mould would be about one-third filled.

The soil was then compacted by applying 27 blows of a 2.5kg rammer dropping through a height of 300mm (Figure 5.7).

![Figure 5.7 Raising and dropping of compaction rammer](image)

The rammer was dropped in such a way that it moved progressively around the edge of the mould between successive blows so that the blows were uniformly distributed over the whole area (Figure 5.8).
After the compaction of the first layer, approximately the same amount of soil as for the first layer was added to the mould and the compaction was repeated. A third layer was placed and compacted in the same way so that the compacted soil protruded slightly above the mould into the extension collar (about 6mm or less above the level of the mould body). The extension collar was removed and excess soil was removed to leave the soil level with the top of the mould using the steel straight edge (Figure 5.9). The compacted soil together with the mould was weighed and the value of the weight recorded. After weighing the filled mould, the compacted soil was forced out from the mould using an extruder and soil sample was taken from each of the three compacted layers for moisture content determination.
Figure 5.9 Compacted soil sample

The compacted soil was then broken in small pieces and mixed with the remainder of the prepared soil in the metal tray. Additional water was added to the soil to raise its moisture content (by about 3%) and the soil was thoroughly mixed before the next compaction test was undertaken. This compaction process was repeated for about 12 different water contents to define the soil compaction curve from the ‘dry side’ to the fully saturated state. The data was processed by calculating bulk density, water content and then dry density.

The bulk density of each compacted sample was calculated using the formula;

$$\rho = \frac{m_2 - m_1}{1000} \text{ (Mg/m}^3\text{)}$$

Equation 5.4
where; \( m_1 \) = mass of mould, \( m_2 \) = mass of soil and mould, (in this particular experiment the volume was 1000, so the 1000 in the equation is the volume).

The moisture content (w %) of tested soil sample (the specimen) was calculated using the formula;

\[
w\% = \frac{m_2 - m_3}{m_3 - m_1} \times 100
\]

where; \( m_1 \) = mass of container, \( m_2 \) = mass of container and wet soil, \( m_3 \) = mass container and dry soil and \( w \) = moisture content.

Then the dry density was calculated from;

\[
\rho_D = \left[ \frac{100}{100 + w} \right] \rho \text{ Mg/m}^3
\]

where; \( \rho_D \) = dry density, \( \rho \) = bulk density, \( w \) = moisture content

A graph of dry density was plotted against the moisture content to obtain the compaction curve and the theoretical 0%, 5% and 10% air voids plot were calculated and plotted together with the graph (Head, 2006).

From the curve, the maximum dry density was read off and the corresponding moisture content was taken as optimum moisture content. The maximum dry density result was reported to the nearest 0.01Mg/m\(^3\) and the optimum moisture content to two significant figures. The analysis test result will be presented in chapter 6 section 6.5.

**Test on the contaminated soil**

The compaction test for crude oil contaminated soil was done by first measuring out 6kg of dry soil sample (from the dry sample prepare in Section 4.3.1) as in the case of uncontaminated soil, the dry soil was then mixed with the required percentage of crude oil measured by weight of the soil (i.e. 2%, 5%, 8%, 10%,
15% and 20%) and then the mixed soil was stored in an air tight cellophane bag for a 72 hour period to allow for proper and thorough mixing. At the end of the incubation period, the compaction test was conducted in the same way as that of uncontaminated soil.

Also a graph of dry density was plotted against the moisture content to obtain the compaction curve and the theoretical 0%, 5%, 10% air voids plot were calculated and plotted together with the graph. From the graph the optimum moisture content was determined.

5.3.4 Test parameters

The main parameters in compaction test are;

- **Maximum dry density (MDD or \( \rho_{dmax} \))** – This is the dry density obtained using a specific amount of compaction at the optimum moisture content of a soil. This is the peak of the dry density in the curve of dry density against water content.

- **Optimum moisture content (OMC)** – This is the moisture content of a soil at which a specific amount of compaction will produce the maximum dry density. It is the moisture content value at the curve of dry density against the moisture content at which a maximum value of dry density is obtained. At low water content value, most soils tend to be stiff and difficult to compact. At high water content, the dry density decreases with increasing water content because the water occupies an increasing proportion of the void spaces i.e. an increasing proportion of the soil volume is occupied by water.
5.4 Shear Strength Test

Shear Strength is defined as the maximum resistance of the soil to shearing stress under any given conditions. Shear strength is soil’s ability to resist sliding along internal surfaces within the soil mass, so shear strength in soil is the resistance to movement between particles due to physical bonds from particle interlocking, atoms sharing electrons at surface contact points and chemical bonds (cementation) such as crystallized calcium carbonate. Shear strength is one of the most important engineering properties of a soil, because it is required whenever a structure is dependent on the soil’s shearing resistance (Section 4.4.3). Shear strength is needed for engineering situations such as determining the stability of slopes or cuts, finding the bearing capacity for foundations, and calculating the pressure exerted by a soil on a retaining wall.

Factor that influences the shear strength are:

- **Soil composition**: the soil composition includes mineralogy, grain size and grain size distribution, shape of particles, pore fluid type and ions on the soil grain.
- **Initial state**: the initial state can be describe by terms such as: loose, dense, overconsolidated, normally consolidated, stiff, soft, etc.
- **Structure**: refers to the arrangement of particles within the soil mass; the manner in which the particles are packed or distributed. Features such as layers, voids, pockets, cementation, etc, are part of the structure.

The shear strength of soil is described by the equation

\[ \tau = c + \sigma \tan \varphi \]  

Equation 5.7

where \( \tau \) = shear strength, \( c \) = cohesion and \( \varphi \) = angle of internal friction
5.4.1 Kinds of shear strength test

The shear strength value measured in the laboratory is dependent upon the conditions imposed during the test and in some case upon the duration of the test (Head and Epps, 2011) and laboratory tests commonly used are;

• **DIRECT SHEAR TEST**- In this test, a thin soil sample is placed in a shear box consisting of two parallel blocks. The lower block is fixed while the upper block is moved parallel to the fixed one in a horizontal direction. The soil fails by shearing along a plane assumed to be horizontal. This test is relatively easy to perform. It can be performed on soils of low permeability in a short period of time as compared to the triaxial test. However, the stress, strain, and drainage conditions during shear are not as accurately understood or controlled as in the triaxial test. Direct shear test is quick and inexpensive but its shortcoming is that it fails the soil on a designated plane which may not be the weakest plane.

• **UNCONFINED COMPRESSION TEST**- In this test the specimen is not placed in the cell and the specimen is open to air with the minor principle stress ($\sigma_3$) at 0. A cylindrical sample is loaded in the compression and failure occurs along diagonal planes where the greatest ratio of shear stress to shear strength occurs. Very soft material may not show diagonal planes of failure but generally is assumed to have failed when the axial strain has reached a value of 20%. The unconfined compression test is performed only on cohesive soil samples. The cohesion ($c$) is taken as one-half the unconfined compressive strength.

• **TRIAXIAL COMPRESSION TEST or CONFINED COMPRESSION TEST** – The triaxial tests is designed to mimic as closely as possible the
actual field or “in situ” conditions of the soil and the tests are run by saturating the soil, applying the confining stress (called \( \sigma_3 \)) and then applying the vertical stress (sometimes called the deviator stress) until failure occurs.

In triaxial test, a cylindrical sample is confined in a membrane and lateral pressure is applied, pore water drainage is controlled through tubing connected to porous discs at the ends of the sample. The triaxial test permits testing under a variety of loading and drainage conditions and also allows measurement of pore water pressure.

There are three main types of triaxial tests and the type of triaxial test that is performed depends on the drainage conditions of the soils to be tested. The types of triaxial tests are;

**Unconsolidated-Undrained (UU) or Quick Undrained Test (QU) -** In this test the specimen is saturated and confining stress (\( \sigma_3 \)) is applied without drainage or consolidation (i.e. drains are closed on the entire duration of the test). In this test method, the compressive strength of a soil is determined in terms of the total stress, therefore, the resulting strength depends on the pressure developed in the pore fluid during loading. Fluid flow is not permitted from or into the soil specimen as the load is applied, therefore the resulting pore pressure and hence strength differs from that developed in the case where drainage can occur.

Normal stress is increased until failure occurs without allowing drainage or consolidation. This test can be run quicker since no consolidation or drainage is needed. The test can be run with varying values of \( \sigma_3 \) to create a Mohrs circle and to obtain a plot showing \( c \) and \( \varphi \). The test is applicable in most practical situations e.g. foundations. The shear strength of soil as determined
in UU tests corresponds to total stress, and is applicable only to situations
where little consolidation or drainage can occur during shearing.

If the test specimens are 100 % saturated, consolidation cannot occur when
the confining pressure is applied nor during the shear portion of the test since
drainage is not permitted. Therefore, if several specimens of the same
material are tested, and if they are all at approximately the same water
content and void ratio when they are tested, they will have approximately the
same undrained shear strength. The Mohr failure envelope will usually be a
horizontal straight line over the entire range of confining stresses applied to
the specimens if the specimens are fully saturated.

If the test specimens are partially saturated or compacted specimens, where
the degree of saturation is less than 100 %, consolidation may occur when the
confining pressure is applied and during shear, even though drainage is not
permitted. Therefore, if several partially saturated specimens of the same
material are tested at different confining stresses, they will not have the same
undrained shear strength. Thus, the Mohr failure envelope for unconsolidated
undrained triaxial tests on partially saturated soils is usually curved line.

The unconsolidated undrained triaxial strength is applicable to fast soil
failures or to situations where the loads are assumed to take place so rapidly
that there is insufficient time for the induced pore-water pressure to dissipate
and for consolidation to occur during the loading period (that is, drainage
does not occur) e.g. tillage and rapid construction of a large embankment.

Also the UU test is the standard test for bearing capacity of foundation which
is a short term case, since after initial loading, the soil will consolidate and
gain strength.
Consolidated-Undrained (CU) or Consolidated Slow Quick Test - The specimen in this test is saturated and confining stress ($\sigma_3$) is applied which squeezes the sample causing volume decrease. Once full consolidation is achieved, drain lines are closed (no drainage for the rest of the test), and normal stress is applied until failure occurs. Normal stress can be applied faster since no drainage is necessary (pore pressure ($u$) is not equal to 0). In the CU test, complete consolidation of the test specimen is permitted under the confining pressure, but no drainage is permitted during shear. The specimen is completely saturated before application of the deviator stress. Full saturation is achieved by back pressure and pore water pressure is measured during the CU test, thus permitting determination of the effective stress parameters $c'$ and $\varphi'$. But in the absence of pore pressure measurements CU tests can provide only total stress values $c$ and $\varphi$. The analysis carried out in terms of total stress obtained from undrained test can be used to investigate the initial stability of the foundation of a structure or embankment on saturated clay. Alternatively, the analysis can also be used to determine the initial stability of open cut or sheet piled excavation made in clay and the stability against bottom heave of a deep excavation in clay. Moreover, stability of impervious rolled field can be investigated through the test. The CU test can be run with varying values of $\sigma_3$ to create a Mohrs circle and to obtain a plot showing $c$ and $\varphi$. This test is applicable in situations where failure may occur suddenly such as a rapid drawdown in a dam or levee. In the CU test the effective stress analysis is applied. The CU test may apply to a building which has consolidated as drainage has taken place and the
building fails e.g. the failure of footings or foundations with suddenly applied load.

**Consolidated-Drained (CD) Test** - The specimen in this test is saturated and confining stress ($\sigma_3$) is applied which squeezes the sample causing volume decrease. The drain lines are kept open and once full consolidation is achieved, normal stress is applied until failure occurs with drain lines still open. Normal stress is applied very slowly allowing full drainage and full consolidation of sample during test ($u = 0$). The test can be run with varying values of $\sigma_3$ to create a Mohr circle and to obtain a plot showing $c$ and $\varphi$. In the CD test, complete consolidation of the test specimen is permitted under the confining pressure and drainage is permitted during shear. The rate of strain is controlled to prevent the build-up of pore pressure in the specimen. A minimum of three tests are required for $c'$ and $\varphi'$ determination. CD tests are generally performed on well-draining soils. For slow draining soils, several weeks may be required to perform a CD test. In the CD test, the total and effective stress is the same since $u$ is maintained at 0 by allowing drainage. This means that the soil is tested in effective stress conditions. This test is applicable in conditions where the soil will fail under a long term constant load where the soil is allowed to drain (e.g. long term slope stability). For excavated or natural slopes that are exposed for long periods of time, it is necessary to use the drained strength because the unloading produced by erosion or excavation eventually reduces the effective stress on the soil and thereby the strength. This test is used for long term values of shear strength e.g. if a motorway cutting is being envisaged.
5.4.2 Research test method and the reason for selecting the method

The test method used in this research is the **unconsolidated undrained triaxial** (UU) test specified by British Standard BS 1377: 1990 Part 7: clause 8. Triaxial test is the most common method used to determine soil shear strength and it is the most reliable method to model the stress-strain state of the ground than direct shear test. In triaxial test method, the specimens are subjected to uniform stresses and strains. In triaxial test, the complete stress-strain behaviour can be investigated. There is also the possibility to allow the soil to be sheared to failure in its natural weakest plane and the shear strength parameters obtained is more accurate than those obtained from shear box test. The unconsolidated undrained triaxial test method was chosen because the test was conducted on compacted soil rather than undisturbed sample.

5.4.3 Unconsolidated undrained triaxial test procedure

The shear strength of the soil samples for this research was determined using the **unconsolidated undrained triaxial** test method. The triaxial apparatus comprised of a cylindrical soil sample (50mm in diameter by 105mm in high) within a cell that was surrounded by water which can be pressurised. The soil was contained within an impermeable membrane and was sealed at its top and bottom surfaces by a loading cap and base pedestal respectively. The movement of water in and out of the sample was controlled. Because the soil sample is a compacted sample, the saturation is less than 100%. The water surrounding the sample was pressurised by a motorised oil-water constant pressure system capable of maintaining a cell pressure up to 500kN/m² at a constant level. Compression of the sample was measured by a dial gauge with a 25mm travel.
capable of reading to 0.01mm (Figure 5.10). The vertical (deviator) load was measured using a proving ring with a capacity of 4kN. The triaxial cell was sat in a load frame of 10kN capacity with a vertical platen speed of 1mm/min.

![Figure 5.10 Triaxial apparatus arrangement.](image)

**Test on the uncontaminated soil**

The soil sample for the triaxial test on the uncontaminated soil was measured out from the dry sample prepared as described in Section 4.3.1 and was compacted in a CBR mould using standard compaction techniques (but with 62
blows instead of 27 blows) as described in Section 5.3.3 for the uncontaminated soil and the soil was compacted at 15% moisture content. Consideration was not given to the degree of saturation on the samples used for the triaxial tests because it was a compacted sample rather than an undisturbed sample and the degree of saturation in a compacted sample is less than 100%.

The filled compacted CBR mould was mounted on an extruder and a guide holding three 50mm diameter sampling tubes was fixed above the CBR mould so that the sharpened ends of the sampling tubes were in contact with soil. The soil in the mould was then extruded and pushed into the sampling tubes until they were about two-thirds full. Samples were taken from the remaining soil in the mould for moisture content determination. The soil in each sampling tube was pushed out and into a split former using another extruder (a smaller extruder) and the soil protruding from the spilt former was trimmed to form a cylindrical soil sample with smooth ends.

The height (about 105mm) and diameter (about 50mm) of each soil sample was measured and its weight was recorded. The soil sample was placed on the pedestal of triaxial cell base. As the soil sample was placed on the pedestal of the triaxial base, a loading cap was placed on top of the soil sample and then an impermeable cylindrical membrane was fitted around the soil sample covering the lower pedestal and upper loading cap (Figure 5.10). O-rings were then placed around the membrane to seal it on the loading cap and the base pedestal.
The triaxial specimen was now surrounded by the cell body and care was taken to avoid the load piston from pushing the specimen when cell body was lowered over the specimen. Once the cell body was in position, tie rods were used to clamp it to the cell base (Figure 5.11). The piston in the cell body was then gently pushed down until it made contact with a hemispherical dome in the loading cap. The whole cell (the cell body together with the soil sample) was raised, using the loading ram so that the piston was just touching the load ring. The cell was then filled with water and the water was pressurised using a motorized oil-water pressure system. The pressure control knob was turned whilst the reading on a pressure gauge was observed until the gauge reached its desired pressure. Finally the compression rate for the sample i.e. 1 mm/min was
set and cell base was raised until there was a slight movement in the proving ring. After recording the initial deflection dial gauge, the apparatus was ready for a compression triaxial test.

The compression test began by putting the constant strain rate control in motion and readings the proving ring and deflection dial gauge readings at a regular time intervals using a stop clock. The test was continued until failure occurred (Figure 5.12), i.e. when there were three consecutive proving ring readings that showed a non-increasing value (i.e. constant or decreasing value). After the failure had occurred, the apparatus was dismantled and soil sample was taken to determine the final moisture content of the tested sample. Sample for the moisture content was taken from the area close to the failure zone for moisture content test.

Figure 5.12 Soil sample showing failure zone
This same test procedure described above was applied to the two remaining 50mm samples but at different cell pressures (e.g. 200kN/m$^2$ and 400kN/m$^2$). A graph of compressive stress against strain was plotted for the all the three specimens on one graph and the maximum value of the stress and the corresponding strain was read off and recorded for each of the three specimens. Also another graph was plotted for shear strength (i.e. $\sigma_1 - \sigma_3)/2$ against principal stress to give a straight line plot which will represent the Mohr circle and from this straight line plot, the frictional angle and the cohesion intercept were determined.

The shear strength parameters are frictional angle which is reported to the nearest $\frac{1}{2}^\circ$ and cohesion which is reported to nearest 1kN/m$^2$. The analysis and result of the shear strength test will be discussed in chapter 6 section 6.6.

**Test on the contaminated soil**

The crude oil contaminated soil sample for the triaxial test was prepared following the procedure used in the compaction of contaminated soil sample in Section 5.3.3 and the soil was compacted in CBR mould as described above for the uncontaminated. The triaxial test procedure and analysis of result for the contaminated soil was the same as that of the uncontaminated soil.

**5.4.4 Shear strength test parameters**

Shear strength parameters consists of two components: cohesive (c) and frictional ($\varphi$) which are measures of shear strength of soil and the higher these values, higher the shear strength of the soil.
• **Frictional Angle or the angle of internal friction** (φ) - The friction angle is a shear strength parameter of soils, its definition is derived from the Mohr-Coulomb failure criterion and it is used to describe the frictional shear resistance of soils together with the normal effective stress. It is the measure of the shear strength of soils due to friction. In the stress plane of shear stress against effective normal stress plot, the soil frictional angle is the angle of inclination with respect to the horizontal axis of the Mohr-Coulomb shear resistance line. Frictional angle or Angle of internal friction for a given soil is the angle on the graph (Mohr's Circle) of the shear stress and normal effective stresses at which shear failure occurs. It can be determined in the laboratory by the direct shear test or the triaxial stress test.

• **Cohesion** (c) - The cohesion is a term used in describing the shear strength of soils, its definition is mainly derived from the Mohr-Coulomb failure criterion and it is used to describe the non-frictional part of the shear resistance which is independent of the normal stress. In the stress plane of shear stress against effective normal stress, the soil cohesion is the intercept on the shear axis of the Mohr-Coulomb shear resistance line. Cohesion is the shear strength or the force that binds together molecules or like particles in the structure of a soil and cohesion in soil is sometimes caused by cementing material.

The information from shear strength parameters such as the frictional angle and the cohesion that are obtained from triaxial test can be used to check the safety and predict the behaviours of long-term stability of slopes, earth fills and earth retaining structures.
5.5 Permeability

Permeability is a measure of how easily a fluid (water) can flow through a porous medium (soil) i.e. soil permeability is the property of the soil to transmit water and it is commonly measured in terms of the rate of water flow through the soil in a given period of time. The size of the soil pores is of great importance with regard to the rate of infiltration (movement of water into the soil) and to the rate of percolation (movement of water through the soil). Pore size and the number of pores closely relate to soil texture and structure and also influence soil permeability.

Permeability coefficient is used to compute the quantity and rate of water flow through soils in drainage and seepage analysis. Laboratory tests are appropriate for undisturbed samples of fine-grained materials and compacted materials in dams, filters, or drainage structures.

5.5.1 Kinds of permeability test

Constant head permeability test – In this method, water is allow to flow through a column of soil under the application of pressure difference which remains constant (constant head permeameter) until the soil is saturated. The amount of water passing through the soil in a known time (i.e. flow rate) is measured and is used in calculating the coefficient of permeability for the soil sample. The constant head method is suitable in measuring the coefficient of permeability for sands and gravels containing little or no silt (i.e. soil with high permeability) so that the quantity of water flow through the soil is measured in reasonably short time (Head and Epps, 2011).
**Falling head permeability test** – The falling head permeability test involves the flow of water through a relatively short soil sample connected to a standpipe which provides the water head and also allows measuring the volume of water passing through the sample. Before starting the flow measurements, the soil sample is saturated and the standpipes are filled with de-aired water to a given level. The period of time it takes the water to flow from the stand pipe is measured and it used in calculating the coefficient of permeability. This test is suitable for soils with low permeability.

**Triaxial cell permeameter** – This test uses the drained triaxial test apparatus with drain connected to the top and bottom of the specimen and the sample is subjected to all round pressure with no deviator stress, a constant water head is applied using the drainage connections across the sample and a resultant steady-state flow rate is measured which is used in calculating permeability.

**Consolidation cell permeameter** – This method uses the consolidation test apparatus (Casagrande oedometer and the Rowe cell) and can be used to measure the coefficient of permeability both in horizontal and vertical directions. The apparatus can be used to test natural soils containing fabric that can significantly affect the permeability of soil and it can also be used to test compacted material (Sarsby, 2000). In this test a constant head test is conducted by applying a head to bottom drain and allowing water to exit from the top drain.
5.5.2 Research test method and the reason for selecting the method

The **falling head permeability** test method was adopted for this research and the reason for choosing this method is based on the fact that the soil sample is a compacted fine grained soil and the constant head method would not be appropriate for fine grained test. Also the falling head test method was choosing because it has the main advantage over the conventional constant-head test on its ability to determine permeability properties of the soil test material at different levels of hydraulic gradients in a single test and the test setup is simpler. The consolidation cell permeameter was not considered as a test method for this research based on the fact that the values obtained using the consolidation cell permeameter will depends greatly on the relative magnitude value of small quantities and each can vary significantly according to the structure of the soil (Sarsby, 2000).

5.5.3 Permeability test procedure

Permeability test for this research was conducted using the falling head permeability test method. The permeability apparatus comprised of a cylindrical cell body (100mm in diameter and 130mm long), a perforated base plate with straining rods and wing nuts that support the cell body and a top cap that has an inlet for tube and an air release valve (Figure 5.13). The cell body was clamped between the base plate and top cap using the straining rods and the assembled permeameter was sat in a water-filled tank with an overflow. A vertical glass standpipe was used to both apply a water head and measure the flow of water through a sample (Figure 5.14).
Figure 5.13 Sketch of Falling head permeability mould (Head and Epps, 2010).
Test on the uncontaminated soil

The soil samples for permeability tests were measured out from the dry sample prepared as described in Section 4.3.1 and was compacted in a standard compaction mould using standard compaction techniques as described in Section 5.3.3. The sample for the uncontaminated test was compacted at 15% moisture content using the 2.5kg rammer as this was the optimum moisture content for the uncontaminated soil and was considered to be the approximate degree of saturation. After the compaction, the compacted filled mould was covered with cling film and sealed in an air tight bag for 24hours to allow the
soil to attain a uniform moisture condition throughout before installing it in the permeameter apparatus.

The first stage in the setting up of the permeameter was to dig out some soil sample from the mould so that the depth of soil was reduced by half. This was done to reduce the time taken to achieve steady-state permeation by the water to an acceptable length of time and to provide a space above the soil for retention of water and to accommodate soil swelling. Some of the soil that was removed was used for moisture content determination and the length of soil remaining in the mould was measured. The mould was then placed on the perforated base, with a filter paper between the soil and the base, also another filter paper was placed on top of the soil sample. The space above of the compacted soil was filled with steel wool to hold the top filter paper in place and prevent excessive swelling of the soil and ‘slurrying’ of the upper surface (Figure 5.15).

A sealing ring was placed on top of the mould (this was to ensure that there was no water leakage at the top cap) and the permeameter cell cap was clamped onto the mould using tie rods attached to the perforated base (Figure 5.16). The assembled cell was then sat in an immersion tank filled with water.
The void in the mould above the compacted soil was filled by running water from the reservoir into the mould until water seeped out from the top air release valve which was then closed (Figure 5.15). During the permeability test, water was run from the reservoir into the falling-head capillary tube (the height of the water in the capillary tube was recorded) then the reservoir was closed off and the water head in the capillary tube was applied to the soil. During the test the level of water in the capillary tube was recorded at approximately 30 minutes interval. The permeability tests were conducted over a period of four days depending on how long it took the water in the capillary tube to drain out. Four
different permeability tests were conducted on the uncontaminated soil sample. This was done so as to attain a consistent permeability value for the uncontaminated soil.

Figure 5.16 Permeability test arrangement

However, the average cross-sectional area of the manometer tube was determined by filling capillary tube with water and then allowing the water to
run out in stages. At each stage the weight of water and the fall in the water level in the tube was measured. The weight of water at each stage was used to calculate the volume of water flowing out i.e.

\[ \text{Volume} = \frac{\text{Weight}}{\text{Density}} \]

Equation 5.8

A graph of the volume of water discharged against fall in the water level was plotted and the average area of the tube was the slope of the graph.

The data for the permeability test was analysed by plotting a graph of \( \frac{h_0}{h} \) (where \( h_0 = \) applied head at time zero and \( h = \) applied head) against elapse time for the whole testing period. The applied head corresponded to the reading on a vertical scale placed at the side of the glass tube plus the height from the bottom of the ruler above the permeameter (i.e. datum height) as shown in figures 5.15 and 5.16.

The slope of the graph is used in calculating the coefficient of permeability for the soil using the standard formula for a falling head permeameter, i.e.

\[ k = 2.3 \left( \frac{a}{A} \right) \times L \times S \]

Equation 5.9

where:

\( a \) = cross sectional area of the manometer (capillary) tube
\( A \) = cross sectional area of the soil sample in the mould
\( L \) = length of the soil sample in the mould
\( S \) = slope of the graph

The derivation of equation 5.9 will be explained in further in Section 6.7 and the result is reported to two significant figures. The analysis of the test result from the permeability test will be presented in chapter 6 section 6.7.
Test on the contaminated soil

The soil samples for the permeability tests on the crude oil contaminated soils were compacted following the same method used for the compaction of crude oil contaminated soil samples in Section 5.3.3 and the soil sample after compacting in the mould was wrapped in a cling film and sealed in an air tight cellophane bag for 24 hour to allow the contaminated soil to attain a uniform wet condition. At the end of the 24 hour period, the permeability test procedure and calculation was the same as that of the uncontaminated soil.

5.5.4 Permeability test parameters

Coefficient of permeability (k) – This is an important parameter of interest in the permeability test and it is the amount of water flowing through the soil i.e. the mean discharge velocity of flow of water in the soil under the action of a unit hydraulic gradient and it is expressed in meter per second.

5.6 Consolidation

This test involves the gradual reduction of the volume of a fully saturated soil sample with time as water is squeezed out of the pore spaces under an induced or excess pore water pressure.

5.6.1 Kinds of consolidation test

The methods of determining the consolidation of soil in the laboratory are;

Casagrande oedometer test – In this kind of test the soil sample is encased in a steel cutting ring and porous discs saturated with air free water are placed on top
and below the sample. The drainage of the specimen takes place in the vertical direction when vertical stress is applied i.e. consolidation is one-dimensional.

**Rowe consolidation cell** – The apparatus used in this method prevents lateral strain by confining the specimen in a bronze cast ring and provides vertical stress through a rubber membrane (the rubber ‘jack’). It is restrained at the top and bottom by thick metal plates bolted to the bronze ring. The apparatus should only be used where it is acknowledge that the conventional casagrande oedometer is likely to give unreliable and uneconomical data and for testing large samples (Sarsby, 2000).

### 5.6.2 Research test method and reason for selecting the method

The method used in this research is the casagrande oedometer test method and the reason for choosing the method is based on the fact that it was the only reliable consolidation apparatus in the university laboratory as at the time of the laboratory experiment.

### 5.6.3 Consolidation test procedure

The apparatus used for the consolidation test was a standard casgrande oedometer apparatus comprising a cell body, sample confinement ring (about 75mm internal diameter and 20mm high), free draining porous discs and a loading cap (Figure 5.17). The equipment also included a loading frame, weighing scale, moisture content apparatus, dial gauge (accuracy of 0.001mm), stop clock and calibrated masses (0.5, 1, 2, 5, 10 kg weights).
Figure 5.17 Consolidation apparatus

Figure 5.18 Consolidation Oedometer assembled cell
The oedometer cell was assembled by putting the lower porous disc on the base of the cell body, the consolidation ring containing the soil sample was placed on top of the lower porous disc and the upper porous disc was placed on the top of the consolidation ring. The retaining ring was then placed around the consolidation ring in the cell body so that it was secured and was tightened by screw nuts. The loading cap was then placed on top of the upper porous disc and the consolidation cell (Figure 5.18).

**Test on uncontaminated soil**

The soil samples for oedometer consolidation tests were measured out from the prepared dry sample as described in Section 4.3.1 and it was compacted in a standard compaction mould using standard compaction techniques as described in Section 5.3.3. The uncontaminated soil sample was compacted at 15% moisture content using the 2.5kg rammer as this was the optimum moisture content for the uncontaminated soil.

The confining ring was first weighed and it then was pressed into the compacted soil so that the soil filled the ring and protruded above the top. Excess soil was then removed and the trimmings were used for moisture content determination. A fully saturated porous disc was placed on the bottom of the oedometer cell and the filled confining ring was placed on top of a filter paper disc sat on the porous disc. Another filter paper disc and saturated porous disc were placed on top of the soil in the confining ring and the confining ring was fixed in place using clamping screw and the nuts (Figure 5.18). Then a loading cap was placed on top of the upper porous disc and the cell was filled with water, the oedometer cell
was sat on the supporting frame as shown in figure 5.19 and was ready for the test.

The loading beam was set in a horizontal position with the loading yoke resting gently on top of the oedometer loading cap and then the yoke, beam and weight hanger were balanced so that they applied no load to the soil sample. A vertical deflection dial gauge was set up with its traveller resting on the loading yoke (Figure 5.18), this dial gauge measured settlement and the requisite weights were placed on the weight hanger (the loading beam was supported at this stage). The test was commenced by removing the support beneath the load beam (Figure 5.19) and a stop clock was started at the same time. The deflection dial gauge was read at various time intervals over a 24 hour period. At the end of the 24 hour period the final dial gauge reading was recorded and the beam support was put in place again whilst further load was added to the load hanger. The consolidation test procedure was then repeated. Tests were conducted for vertical consolidation pressures of 50kN/m$^2$, 100kN/m$^2$, 200kN/m$^2$, and 400kN/m$^2$. When the loading sequence was completed i.e. 24 hours after a vertical stress of 400kN/m$^2$ has been applied, then the unloading stage of the experiment was started. Unloading was done in stages i.e. from 400kN/m$^2$ to 200kN/m$^2$ to 100kN/m$^2$. Readings of the vertical deflection dial gauge were taken at the same time intervals as for the increasing load part of the test.

At the end of the experiment, the consolidation cell was drained of water and the cell was dismantled. The consolidation ring and the soil sample were weighed and were placed in an oven to dry. At the end of the oven drying, the soil sample together with the consolidation was re-weighed. These data were used to calculate the final moisture content, bulk density and hence voids ratio.
The result was analysed using two different methods; by (1) plotting graphs of compression gauge reading (settlement) against log of time method and (2) plotting compression gauge reading against square-root of time method. The graphs were used to determine the values of the coefficient of consolidation. The amounts of settlement occurring during a loading stage and over the whole consolidation cycle were used to calculate the compressibility parameters for the clay. The analysis of the data obtained during the test will be presented in chapter 6 Section 6.8.
Test on contaminated soil

The contaminated soil for the consolidation test was prepared by measuring out dry soil sample from the prepared dry soil as described in Section 4.3.1 and about 6kg dry soil sample was mixed with the required percentage of crude oil measured by weight as was done for the compaction test of the contaminated soil samples in Section 5.3.3, the mixed samples were compacted using 2.5kg rammer following standard compaction procedure as described Section 5.3.3. The consolidation test procedure and result analysis of the contaminated soil was conducted in the same way as the uncontaminated soil.

5.6.4 Consolidation parameter

The consolidation parameters of interest in this research test are;

- **Coefficient of consolidation** – which indicate the rate of compression and hence the time period over which consolidation settlement will take place.

- **Compression index** – this is the slope of linear portion of the e-log p curve and it is dimensionless.

- **Coefficient of volume compressibility** – this is a measure of the amount by which the soil will compress when loaded and allowed to consolidate. It indicates the compressibility per thickness of the soil.

5.7 Reason for using compacted sample in permeability, triaxial and consolidation tests

It is sometime necessary to carry out shear strength, compressibility and permeability tests on recompacted soil (Head and Epps, 2011), the samples in this research were compacted so as to test at a dry density of 15% moisture
content which was the optimum moisture content of the uncontaminated soil and also to allow for consistence by using the light rammer (2.5kg rammer) method which was used in the compaction test. Also compaction into a standard compaction mould is better than compacting cohesive soil directly into a small tube or a ring (Head and Epps, 2011). Again the reason for using compacted soil for permeability, shear strength and consolidation tests is based on the fact that compaction is used to densify soils during placement to minimize post-construction consolidation and to improve strength characteristics. Structural and supporting capabilities also are evaluated by appropriate tests on samples of compacted soil.

5.8 Summary of research test procedure

The geotechnical tests conducted in this research are the index properties tests which includes cone penetrometer liquid limit test, plastic limit test, plasticity index and linear shrinkage. The compaction test was conducted using the light (2.5kg rammer) method, the shear strength of the soil was determined using the unconsolidated undrained triaxial test method, the permeability of the soil was determined using the falling head permeability test method and the consolidation test was conducted using the casagrande oedometer test method.
CHAPTER 6: ANALYSIS OF TEST RESULTS AND DISCUSSION

6.1 Introduction

This chapter presents the results of the specific geotechnical tests conducted in chapter 5 which highlights the differences in the results on the contaminated soil samples and the uncontaminated soil samples and the differences between the different percentages of contamination. This chapter also discusses the significance of the results and it also looks at the determination of water content from fluid content (i.e. fluid content = water content + oil content) which was used in the calculations especially in index properties tests and compaction test. The reason for the repetition of some tests and the use of some statistical method in the analysis of classification and compaction results for the uncontaminated soil sample is also discussed in this chapter.

6.2 Determination of water content and fluid content

The water content of a soil is normally calculated using the formula:

\[ w(\%) = \left( \frac{W_w}{W_s} \right) \times 100 \] .................................Equation 6.1

where;

\( w = \) water content (\%), \( W_w = \) weight of water and \( W_s = \) weight of dry soil.

The above moisture content formula is applicable where the pore fluid is water alone but when there is contamination and the pore fluid is no longer water alone the formula may not be used.

In the case of crude oil contaminated soil, Khamehchiyan et al. (2007) stated that although crude oil may evaporate under room temperature, some part will remain in the soil and that crude oil evaporation is dependent on the physical and chemical properties of the crude oil, environmental temperature and humidity.
Again Khamehchiyan et al. (2007) stated that the evaporation of crude oil from soils and the percentage of oil which remain depends on the size and composition of the soil particle, thermal properties of the soil and oil. They (Khamehchiyan et al. 2007) stated that evaporation of crude oil increases with decrease in soil particle size and that evaporation decreases with increase in the oil content under the same environmental condition. The authors further stated that heat transfer in clayey soils is slower than in sandy soils so oil in clayey soils absorbs more heat and oil evaporation is also more compared to sandy soil.

In this research since the soil is contaminated by light crude oil and the pore space is occupied by crude oil and water, there is the need to determine the water content from the fluid content.

However due to the fact that the light crude oil will evaporate in the oven during testing, moisture content of all crude oil contaminated soil samples was calculated based on equation 4.2 in Section 4.3.5, i.e.

\[
W\% = (1 - mn) \frac{W_t}{W_d} - (1 + n) \text{equation 6.2}
\]

where;

\( w \) = moisture content

\( W_t \) = weight of wet contaminated soil

\( W_d \) = weight of dry contaminated soil

\( m \) = crude oil residual after drying

\( n \) = oil content before drying

The formula in equation 6.2 was used to calculate of the moisture content of soil samples for the different percentages of contamination i.e. 2%, 5%, 8%, 10%, 15% and 20%.
In this research due to the light crude oil contamination there was the need to calculate the fluid content alongside the water content, so fluid content was calculated from the formula:

Fluid content \( (W_f) \) = water content + oil content

\[ W_f = W_w - W_o \] ........................................................................Equation 6.3

\[ W_f = \left( \frac{M_w + M_o}{M_s} \right) \] ........................................................................Equation 6.4

But \( W_w = \frac{M_w}{M_s} \)

And \( W_o = \frac{M_o}{M_s} \)

Therefore \( W_o = W_f - W_w \) ........................................................................Equation 6.5

where; \( W_w \) = water content, \( W_f \) = fluid content, \( W_o \) = oil content, \( M_o \) = weight of oil, \( M_s \) = weight of dry soil and \( M_w \) = weight of water.

Equation 6.4 was used in calculating the fluid content for the tests that were conducted for the different percentage of contamination by weight i.e. 2%, 5%, 8%, 10%, 15% and 20%.

Applying equation 6.1 will give the fluid content of the contaminated soil but applying equation 6.2 will give the water content of the contaminated soil sample.

6.3 Repetition of tests and use of statistics in the analysis of results

No matter what procedure a researcher employs in the collection of data, it should be assessed, in order to ensure that it is valid and reliable. Validity refers to the ability of the instrument to measure what it is designed to measure and reliability refers to the repeatability or consistency of what is measured by the research instrument and what it has been designed to measure. In other words, a reliable
instrument is one that gives the same results when used on different occasions. In this research, test especially on the uncontaminated soil samples were repeated (conducted several/ different times) to make sure that the apparatus and equipment used in the experiments are working properly without errors and are capable of repeating the tests. Also the tests were conducted several times so as to have reliability and consistency of results of the uncontaminated soil since any change in the geotechnical properties of the soil due to the crude oil contamination will be compared with that of uncontaminated soil i.e. tests on the uncontaminated soil sample for any of the geotechnical tests will act as a control test.

Due to the repetition of test and the need to have a consistent value for the uncontaminated soil sample, there were a lot of scattered points when data from the several tests conducted on the same uncontaminated soil sample was used in a plot. To obtain a straight line or a line of best fit form the scattered plot, statistical method (linear regression) was used in the data analysis of the result. The regression procedure fits the best possible straight line to an array of data points and if no single line can be drawn such that all the points fall on it, the line that minimizes the sum of squared deviations from each data point to the line is used (Wonnacott and Wonnacott 1990). The slope of the fitted line is equal to the correlation between $y$ and $x$ corrected by the ratio of standard deviations of these variables. The intercept of the fitted line is such that it passes through the center of mass $(x, y)$ of the data points. Regression attempt to minimize the distance measured vertically between the observation point and the model line (or curve). Any straight line in two-dimensional space can be represented by this equation:

$$y = mx + c;$$
where \( y \) is the variable on the vertical axis, \( x \) is the variable on the horizontal axis, \( c \) is the \( y \)-value where the line crosses the vertical axis (often called the intercept), and \( m \) is the amount of change in \( y \) corresponding to a one-unit increase in \( x \) (often called the slope).

### 6.3.1 Strength, limitation and confidence level of the statistical method

Regression analysis is the statistical technique that identifies the relationship between two or more quantitative variables. Linear Regression estimates the coefficients of the linear equation, involving one or more independent variables, which best predict the value of the dependent variable.

Regression analysis tries to find the line (and linear equation) that best fits data points. The coefficient of determination \( R^2 \) expresses how good the regression is and the higher the value of \( R^2 \), the better the regression e.g. \( R^2 = 1 \) implies a perfect fit and \( R^2 = 0 \) implies no fit (Mohr 1995). The confidence of the regression method used in this research is based on the fact the \( R^2 \) for the curves are closer to 1 than 0.

The strength of the regression analysis used in determining the line of best fit in this research is based on the fact that Regression analysis provides an opportunity to specify the outcome of a straight line concerning the nature of effects, as well as the explanatory factors and when it is successfully executed (with a statistically valid adjustment), regression analysis can produce a quantitative estimate of net effects. However the limitation of this method (regression analysis) used in the determination of line of best fit lied on the fact that the technique is demanding because it requires several scattered point and conducting many tests (repeated tests) so as to get the required data and implementing the data collection can be
time-consuming. Another limitation of the regression analysis is likely to reach the conclusion that there is a strong link between two variables, whereas the influence of other variables may not have been estimated.

Also statistical mean is used in the calculating the average of the data especially in the plastic limit tests for the uncontaminated soil sample. The statistical mean is a measure which takes into account every item of the data.

The strength of the statistical mean value lies on the fact it uses all the data and takes into account end values but its limitation is based on the fact that it can easily be distorted by large values.

The standard deviation which is a measure of the spread of the data is used in determining the confidence level of the statistical method. In most cases (and in a normal distribution), about 95% of the subjects will have a value within the standard deviations of the mean. Also the confidence of this statistical mean is based on the standard error of the mean which is a measure of how certain we are about the value of this mean. Unlike the standard deviation, the value of the standard error depends on the size of the study. It is small when there is a large amount of data, and big when there is a tiny study. The standard error is used to generate the confidence interval (Wonnacott and Wonnacott 1990).

6.4 Index properties result

The results discussed in this section are the test results for the uncontaminated and contaminated soil samples for liquid limit test, plastic limit test, plasticity index test and the linear shrinkage test that were conducted in chapter 5 Section 5.2.
6.4.1 Liquid limit test result

The liquid limit value was determined from a graph of penetration (mm) against moisture content value obtained during the laboratory test (Section 5.2.1) using cone penetration test method and the moisture content value at 20mm penetration on the graph gave the liquid limit value.

6.4.1.1 Uncontaminated soil sample

The liquid limit value was determined from the moisture content value corresponding to the cone penetration of 20mm. A typical liquid limit test result for the uncontaminated soil sample is shown in Figure 6.1 with a liquid limit value of 37.7%.

Figure 6.1 Liquid limit graph
However because the liquid limit test for the uncontaminated soil acts as a control test upon which the judgement on the effect of the different percentage of light crude oil contamination on the kaolinite clay soil is based, there was the need to have a valid and reliable result for the liquid limit of the uncontaminated soil. So the liquid limit test was conducted 7 different times on the uncontaminated soil sample to observe if there was any variation in the value. Figure 6.2 shows the individual liquid limits of all the 7 different tests on the uncontaminated soil sample plotted on one graph using one common axis.

![Individual LL Plot](image.png)

Figure 6.2 Individual liquid limit plots on the uncontaminated soil sample
The variation in the liquid limit value of the 7 different tests on the uncontaminated soil sample is shown in Table 6.1 and the liquid limit values ranges between 37.7% and 38%. Also Figure 6.3 shows this variation in the value of the liquid limit presented in bar chart.

Table 6.1 Liquid limit values for uncontaminated soil sample

<table>
<thead>
<tr>
<th>Test</th>
<th>LL</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>37.7</td>
</tr>
<tr>
<td>2</td>
<td>37.8</td>
</tr>
<tr>
<td>3</td>
<td>37.8</td>
</tr>
<tr>
<td>4</td>
<td>37.9</td>
</tr>
<tr>
<td>5</td>
<td>37.8</td>
</tr>
<tr>
<td>6</td>
<td>37.9</td>
</tr>
<tr>
<td>7</td>
<td>38.0</td>
</tr>
</tbody>
</table>

Figure 6.3 Chart of liquid limit values for the uncontaminated soil
From figure 6.2 it is readily apparent that there is a significant degree of scatter for data which relate to one material which has been processed and is assumed to be very uniform, so all the data points i.e. penetration and moisture content for the 7 different tests were plotted on one graph (Figure 6.4) and using SPSS software to determine line of best fit, it was possible to obtain a straight line and the liquid limit value was therefore obtained since it is evident from Figure 6.2 that there is a large scatter to the data which do not fit one single unique line.

Figure 6.4 Liquid limit plot using SPSS to determine ‘line of best fit’
The linear regression used in determining the line of best fit for the scattered plot in Figure 6.4 shows the dependency of penetration on water content and the reliability of the plot is 0.8448. As discussed in Section 6.3 the confidence of the plot shows that it is a good fit since the coefficient of regression $R^2$ is high and towards 1.

From the SPSS statistical plot, the equation of straight line; $y = mx + c$ is represented by

$$y = 0.940x - 15.29$$

......Equation 6.6

$R^2 = 0.8448$

where $y = $ Penetration and $X = $ water content.

Therefore; penetration (mm) = 0.9405 x water content (%) − 15.29

$R^2 = $ Reliability of the result which show the confidence in the result.

From Figure 6.4, the liquid limit value is 37.5% and is within the range of the liquid limit value for all the tests on the uncontaminated results (Table 6.1). Fojtova et al. (2009) used the linear regression method to determine line of best fit when they compared the compatibility of two test methods for liquid limits and they came out with equation $y = 1.003x + 2.439$ and $R^2 = 0.978$, their equation is similar to equation 6.6 in that it is an equation of a straight line.

6.4.1.2 Contaminated soil samples

The water content of the contaminated soil samples which was calculated using equation 6.2 was used in plotting the liquid limit graphs for the contaminated soil samples and Table 6.2 shows the liquid limit results for the contaminated soil samples. Also the fluid content for the contaminated soil was calculated using equation 6.4. The fluid content value for the contaminated soil samples was
compared with that of the water content and this highlights the importance of determining the water content rather than the fluid content for the contaminated soil samples.

Table 6.2 Liquid limit values for contaminated soil samples

<table>
<thead>
<tr>
<th>Contamination (%)</th>
<th>Water content LL (%)</th>
<th>Fluid content LL (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.0</td>
<td>41.2</td>
<td>41.2</td>
</tr>
<tr>
<td>5.0</td>
<td>44.2</td>
<td>44.4</td>
</tr>
<tr>
<td>8.0</td>
<td>45.5</td>
<td>46.0</td>
</tr>
<tr>
<td>10.0</td>
<td>48.5</td>
<td>49.0</td>
</tr>
<tr>
<td>15.0</td>
<td>49.9</td>
<td>50.0</td>
</tr>
<tr>
<td>20.0</td>
<td>50.5</td>
<td>51.2</td>
</tr>
</tbody>
</table>

From Table 6.2 the liquid limit results of the contaminated soil samples for the different percentages of contamination when calculated using the water content formula (equation 6.2) ranges from 41.2% to 50.5% whereas when calculated using fluid content formula (equation 6.4), it ranges from 41.2 and 51.2%.

Figures 6.5 and 6.6 shows the variation on the graphs when fluid content was used and when water content was used for one particular crude oil contaminated soil sample (e.g. 8% contamination).
Figure 6.5 Liquid limit graph using fluid content
Figure 6.6 Liquid limit graph using water content

As can be seen from Table 6.2 and Figures 6.5 and 6.6, there was a slight change in the liquid limit values of the contaminated soil samples for the different percentages of contamination when calculations were based on fluid content formula and when it was based on water content formula.
6.4.1.3 Discussion of liquid limit result

The behaviour of clay soil can be related to the interacting factors of the local geology, engineering properties of the soil and the environment of deposition. From Table 6.2 there were slight differences in the values obtained when liquid limit values were calculated using fluid content formula and when they were calculated using water content formula. The result shows an increase in the liquid limit value with increase in the percentage of contamination (Figure 6.7).

![Liquid limits against contamination](image)

Figure 6.7 Graph of liquid limit against percentage of contamination
This increase in liquid limit value is due to exchangeable or bond cations on the surface or interlayer of clay particle due to the contamination. The extra cohesion provided to the clay soil by the light crude oil also contributed to the increase in the liquid limit of the contaminated clay soil. Crude oil has higher viscosity than water, this high viscosity and surface tension would promote the retention of oil between the soil particles.

6.4.2 Plastic limit test result

The plastic limit results presented in this section is obtained from the plastic limit tests conducted in chapter 5 as described in Section 5.2.2 for the uncontaminated and contaminated soil samples.

6.4.2.1 Uncontaminated soil

The plastic limit value for the uncontaminated soil sample was determined during each of the seven liquid limit tests (as described in Section 6.4.1.1) and to determine the plastic limit of the sample, the crumbled thread at the crumbling stage of the plastic limit test was gathered into a moisture content container. The moisture content of the soil sample was calculated following the standard moisture content test procedure (Section 5.2.2) and using equation 6.1. The results of the plastic limit tests for the uncontaminated soil samples is shown in Table 6.3 and the chart in Figure 6.8 shows the variation in the plastic limit values for the seven different tests conducted on the uncontaminated soil.
Table 6.3 Plastic limit result for uncontaminated soil sample

<table>
<thead>
<tr>
<th>Test</th>
<th>PL</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>18.2</td>
</tr>
<tr>
<td>2</td>
<td>17.9</td>
</tr>
<tr>
<td>3</td>
<td>18.1</td>
</tr>
<tr>
<td>4</td>
<td>18.0</td>
</tr>
<tr>
<td>5</td>
<td>17.8</td>
</tr>
<tr>
<td>6</td>
<td>17.9</td>
</tr>
<tr>
<td>7</td>
<td>18.1</td>
</tr>
</tbody>
</table>

Figure 6.8 Chart of plastic limit value for the uncontaminated soil
From Table 6.3 the plastic limit value for the uncontaminated soil ranges from 17.8% to 18.2%. Since the plastic limit value for the uncontaminated soil sample is a control test to determine the effect of the light crude oil contamination on the kaolinite soil sample, there was the need to have a consistence in the value, so in this particular test the statistical mean average for the seven different tests was calculated (Table 6.4). The average plastic limit for the uncontaminated soil was found to be 17.8% with a standard deviation of 0.8.

Table 6.4 Plastic limit for uncontaminated soil sample using SPSS

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean</td>
<td>17.8</td>
</tr>
<tr>
<td>Standard deviation</td>
<td>0.8</td>
</tr>
<tr>
<td>Upper limit</td>
<td>18.6</td>
</tr>
<tr>
<td>Lower limit</td>
<td>17.0</td>
</tr>
</tbody>
</table>

6.4.2.2 Contaminated soil

The plastic limit test for the contaminated soil was prepared as described in Section 5.2 and the test was conducted for the different percentages of contamination using the moisture content formula described in equation 6.2. Again the plastic limit was also calculated using the fluid content formula described in equation 6.4. The result of the plastic limit test for the different percentages of contamination is presented in Table 6.5.
Table 6.5 Plastic limit result for contaminated soil

<table>
<thead>
<tr>
<th>Contamination (%)</th>
<th>Water Content PL (%)</th>
<th>Fluid Content PL (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.0</td>
<td>18.6</td>
<td>18.7</td>
</tr>
<tr>
<td>5.0</td>
<td>20.1</td>
<td>20.4</td>
</tr>
<tr>
<td>8.0</td>
<td>21.4</td>
<td>21.7</td>
</tr>
<tr>
<td>10.0</td>
<td>23.1</td>
<td>23.7</td>
</tr>
<tr>
<td>15.0</td>
<td>21.8</td>
<td>23.8</td>
</tr>
<tr>
<td>20.0</td>
<td>20.6</td>
<td>22.8</td>
</tr>
</tbody>
</table>

From Table 6.5, the plastic limit for the contaminated soil is slightly higher when calculated using fluid content formula than when calculated using water content formula and these values are higher than that of uncontaminated soil. Table 6.5 shows that the plastic limit values of the contaminated soil increases with increase in the percentage of contamination.

6.4.2.3 Discussion of plastic limit result

The plastic limit results shows an increase in the plastic limit values with increase in the percentage of contamination up to 10% crude oil contamination after which there was a slight decrease in the plastic limit value (Figure 6.9).
This increase in the plastic limit value shows that the addition of crude oil to the soil resulted in a change in the pore fluid chemistry which leads to a physio-chemical interaction between the individual soil particles or particle groups. The addition of the crude oil into the soil may have caused the replacement of cations. This increase in plastic limit value for the contaminated soil is due to the fact that since the crude oil in the soil’s pore space reduced the amount of water in the soil.
and increases the soil cohesion. But as the percentage of crude oil increased above 10%, the soil sample appear aggregated hence the drop in plastic limit value.

### 6.4.3 Plasticity Index

The plasticity index (PI) is a measure of the plasticity of a soil i.e. the plasticity index is the size of the range of water contents where the soil exhibits plastic properties. The PI is the numerical difference between the liquid limit and the plastic limit (PI = LL - PL). The value of the plasticity index is reported to nearest whole number. The results presented in this section are obtained from the plasticity index tests conducted in Section 5.2.3 for the contaminated and uncontaminated soil.

#### 6.4.3.1 Uncontaminated soil

The plasticity index for the uncontaminated soil was conducted as described in Section 5.2.3.3 on the seven different soil samples used in the liquid limit and plastic limit tests and the result of the plasticity index for the uncontaminated soil is presented in Table 6.6

#### Table 6.6 Plasticity index result for the uncontaminated soil

<table>
<thead>
<tr>
<th>Test</th>
<th>PI</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>19.5</td>
</tr>
<tr>
<td>2</td>
<td>19.9</td>
</tr>
<tr>
<td>3</td>
<td>19.7</td>
</tr>
<tr>
<td>4</td>
<td>19.9</td>
</tr>
<tr>
<td>5</td>
<td>20.0</td>
</tr>
<tr>
<td>6</td>
<td>20.0</td>
</tr>
<tr>
<td>7</td>
<td>19.9</td>
</tr>
</tbody>
</table>
The plasticity index of the uncontaminated soil sample ranges from 19.5 to 20 and the variation in the values of the plasticity index result for the uncontaminated soil sample is presented in a bar chart (Figure 6.10).

![Figure 6.10 Chart of plasticity index test for the uncontaminated soil](image)

The plasticity index values for the uncontaminated soil are plotted in the plasticity chart (Figure 6.11). The plasticity chart shows that the soil sample has intermediate or medium plasticity. Plasticity chart separates fine-grained soil into two basic groups (clay or silt) by mean of the empirical A-line (Sarsby 2000). The
A-line has the equation $Ip = 0.73(LL - 20)$ over most of its length and divides the clay from the silt. The U-line which has equation $Ip = 0.9(LL - 8)$ is the upper limit of the combinations of plasticity index and liquid limit found on any soil.

Figure 6.11 Plasticity chart for uncontaminated soil

where; L = Low, I = Intermediate or Medium, H = High, V = Very High, E = Extremely High Plasticity, C = Clay and M = Silt.
6.4.3.2 Contaminated soil

The plasticity index for the different percentages of crude oil contamination was prepared as described in Section 5.2.3.3 and the plasticity index was determined by subtracting the values of the plastic limit from that of the liquid limit. The moisture content for both liquid limit and plastic limit was calculated using the water content formula as described in equation 6.2.

The result of the plasticity index presented in Table 6.7 shows the plasticity index values for the contaminated soil are higher than that of the uncontaminated soil. The plasticity index for the contaminated soil increases with increase in the percentage of crude oil contamination. The result also shows that the plasticity index calculated using the water formula is slightly higher that calculated using the fluid content formula.

Table 6.7 Plasticity index result for contaminated soil samples

<table>
<thead>
<tr>
<th>Contamination (%)</th>
<th>Water content</th>
<th>Fluid content</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.0</td>
<td>22.6</td>
<td>22.5</td>
</tr>
<tr>
<td>5.0</td>
<td>24.1</td>
<td>24.0</td>
</tr>
<tr>
<td>8.0</td>
<td>24.1</td>
<td>24.2</td>
</tr>
<tr>
<td>10.0</td>
<td>25.4</td>
<td>25.3</td>
</tr>
<tr>
<td>15.0</td>
<td>28.1</td>
<td>26.2</td>
</tr>
<tr>
<td>20.0</td>
<td>29.9</td>
<td>28.4</td>
</tr>
</tbody>
</table>

The plasticity chart for the contaminated soil samples shows that the contaminated soil samples falls within medium or intermediate plasticity although
the plasticity of the soil sample tends to move high on the plasticity chart as the percentage of crude oil contamination increases (Figure 6.12).

Figure 6.12 Plasticity chart for contaminated soil

6.4.3.3 Discussion of plasticity index result

The swelling properties of clay follow the same trend as their plasticity index properties. Hence a successful method to identify soil volume change relates swelling to plasticity index. The plasticity chart show that the contaminated clay falls within ‘medium clay’ as that of the uncontaminated clay soil (Figure 6.12).
The plasticity index values increases with increase in the percentage of light crude oil contamination (Figure 6.13), this increase in value is due to the extra cohesion imparted to the clay particle by the crude oil.

Figure 6.13 Graph of Plasticity Index against percentage of crude oil contamination
6.4.4 Linear shrinkage test

Linear shrinkage measures the percentage decrease in dimension of a fine fraction of a soil when it is dried after having been moulded in a wet condition approximately at its liquid limit. This section presents the results of the linear shrinkage test conducted in Section 5.2.4 for the contaminated and uncontaminated soils.

6.4.4.1 Uncontaminated soil

In this research four tests were conducted on the uncontaminated soil sample so as to attain a consistent value since the uncontaminated soil sample will act as a control test on the effect of the light crude oil contamination on the linear shrinkage of the kaolinite clay soil. The result of the linear shrinkage test conducted on the uncontaminated soil shows that the value ranges between 9.50% and 9.64% (Table 6.8). Using the statistical mean average, the linear shrinkage for the uncontaminated soil was found to be 9.57.

Table 6.8 Linear shrinkage result for the uncontaminated soil

<table>
<thead>
<tr>
<th>Test</th>
<th>Shrinkage (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>9.58</td>
</tr>
<tr>
<td>2</td>
<td>9.64</td>
</tr>
<tr>
<td>3</td>
<td>9.57</td>
</tr>
<tr>
<td>4</td>
<td>9.50</td>
</tr>
</tbody>
</table>
6.4.4.2 Contaminated soil

The linear shrinkage for the crude oil contaminated soil was prepared and tested as described in Section 5.2.4. The values of the contaminated soil (Table 6.9) shows an increase in the value with increase in the percentage of contamination up to 10% percentage contamination after which there was a slight decrease in value and these values are higher than that of the uncontaminated soil.

Table 6.9 Linear shrinkage result for contaminated soil

<table>
<thead>
<tr>
<th>Contamination (%)</th>
<th>Shrinkage (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.0</td>
<td>10.0</td>
</tr>
<tr>
<td>5.0</td>
<td>11.0</td>
</tr>
<tr>
<td>8.0</td>
<td>12.0</td>
</tr>
<tr>
<td>10.0</td>
<td>13.0</td>
</tr>
<tr>
<td>15.0</td>
<td>12.0</td>
</tr>
<tr>
<td>20.0</td>
<td>10.0</td>
</tr>
</tbody>
</table>

6.4.4.3 Discussion of linear shrinkage test result

The addition of crude oil to the soil cause a change in the pore fluid chemistry of the soil which resulted in an initial increase in the linear shrinkage value up to 10% contamination but after the 10% contamination there was a decrease on the linear shrinkage value (Figure 6.14).

The initial increase in linear shrinkage value is due to that fact that the pore space is occupied by water and crude oil. when the percentage of crude oil contamination was increased, the ratio of crude oil to that of water in the pore space also increased, so the rate of evaporation of the crude oil during drying also increased but above 10% percentage contamination, even though the rate of crude oil evaporation increased there was more crude oil in pore space than
water and the soil sample could not reach plastic limit. The trend in the shrinkage limit value is similar to that of the plastic limit value.

![Figure 6.14 Graph of linear shrinkage against percentage of contamination](image)

**6.4.5 Summary of index properties test results**

The index properties results shows an increase in value with increase in the percentage of crude oil contamination except for plastic limit and linear shrinkage where there was a decrease in value after 10% contamination (Figure 6.15).
The reason for this increase in the index properties values is based on the fact that the presence of hydrocarbon (light crude oil) which is non-polarised liquid acts like adsorbed water (i.e. with electrostatic effect and possible chemical interaction) and caused the reduction in thickness of water film around the clay minerals. Water is a binding agent between clay minerals and its orientation around the clay mineral provides the plasticity characteristics but this will not happen if clay minerals are surrounded by hydrocarbon.
From the index properties result it can be summarised that;

- The plasticity of both contaminated and uncontaminated soils falls within that of the intermediate (medium) clay on the plasticity chart.

- The liquid limit, plasticity limit, plasticity index and linear shrinkage values of the contaminated soil are higher than that of the uncontaminated soil. This increase in index properties values is associated with the bonding cation in the clay minerals.

- The index properties values increased with increase in the percentage of crude oil contamination except for plastic limit and linear shrinkage where there was a decrease in value after 10% contamination.

- Hydrocarbon is a non-polarised liquid and act like adsorbed water and its presence in the contaminated soil caused a reduction in the thickness of the water film around the clay minerals thus the increase in the index properties values.

- The index properties of the crude oil contaminated clay soil used in this research are still within the range of clay soils described by other authors (Comer and Firat, 2010 and Hazzan 2010) as construction clay material i.e. the crude oil contamination up to the level determined in this research did not make the kaolinite clay soil to have the characteristics of other kinds of soils such as sand.

6.5 Compaction Test Result

This section presents the results of the compaction tests conducted in chapter 5 Section 5.3 for the contaminated and uncontaminated soils. Laboratory compaction test on soils provides the basis for the control procedures used on the
site since compaction is a process that reduces the voids ratio of a soil through removal of air by applying mechanical force which brings about an increase in dry density of the soil. During compaction porosity, permeability and water infiltration capacity of the soil are reduced while strength is increased but the soil structures are partly destroyed (Smith, 2006).

To analysis the data for the compaction test, the bulk density of each compacted soil was calculated using the formula in equation 5.4 i.e.

$$\rho = \left( \frac{(m_2 - m_1)}{V} \right) \text{ (Mg/m}^3\text{)} \text{ ........................................ Equation 6.7}$$

where \( m_1 = \text{mass of mould, } m_2 = \text{mass of soil + mould and } V = \text{volume of the mould} \)

The soil moisture content in compaction test is normally calculated using equation 5.5 i.e.

$$w\% = \left[ \frac{m_2 - m_3}{m_3 - m_1} \right] \times 100 \text{ .......................................................... Equation 6.8}$$

but when the soil sample is contaminated with crude oil, equation 6.2 is then used to calculate the moisture content of the soil sample i.e.

$$w\% = (1 - mn) \frac{W_t}{W_d} - (1 + n)$$

The dry density of the soil is calculated using the equation 5.6 i.e.

$$\rho_D = \left( \frac{100}{100 + w} \right) \rho \text{ (Mg/m)}^3 \text{ ........................................ Equation 6.9}$$

where \( \rho_D = \text{dry density, } \rho = \text{bulk density and } w = \text{moisture content} \)

Using equation 6.9 and the moisture content calculated using equation 6.2, a compaction curve which is a graph of dry density against moisture content is
plotted together with 0%, 5%, 10% air voids. The air void is calculated using equation 6.10:

\[
\rho_d = \left( \frac{1 - \left( \frac{V_a}{100} \right)}{\left( \frac{1}{\rho_s} + \frac{w}{100} \right)} \right)
\]

...Equation 6.10

where; \( \rho_d \) = dry density for air void, \( \rho_s \) = particle density, \( V_a \) = air void, and \( w \) = moisture content.

From the graph of the dry density against moisture content, the maximum dry density and its corresponding optimum moisture content was read off and recorded. An example of compaction graph showing maximum dry density (MDD) and optimum moisture content (OMC) is shown in Figure 6.16.

![Figure 6.16 Compaction graph](image-url)

Figure 6.16 Compaction graph
6.5.1 Uncontaminated soil

The preparation and calculation of the compaction test for the uncontaminated soil sample was conducted as described in Section 5.3. In this research six different tests were conducted on the uncontaminated soil sample so as to attain a consistent value for the uncontaminated soil sample. The reason for these six different tests is based on the fact that the result of the uncontaminated soil will act as a control test to assess the effect of the light crude oil contamination on the compaction characteristics of the kaolinite clay soil.

Compaction graph for each of the six different compaction tests on the uncontaminated soil was plotted, i.e. dry density against moisture content (an example is shown in Figure 6.16).

A graph that combined all the six different tests on the uncontaminated soil was plotted on one single graph (Figure 6.17) and it shows that the optimum moisture content of the uncontaminated soil from the different tests ranges between 13% and 15%, whilst the maximum dry density ranges between 1.77Mg/ m³ and 1.82Mg/ m³ as shown in Table 6.10.
Figure 6.17 Compaction graph of individual test on uncontaminated soil

Table 6.10 Optimum moisture content and maximum dry density for the uncontaminated soil

<table>
<thead>
<tr>
<th>Test</th>
<th>OMC(%)</th>
<th>MDD(Mg/m³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>13.0</td>
<td>1.77</td>
</tr>
<tr>
<td>2</td>
<td>14.9</td>
<td>1.82</td>
</tr>
<tr>
<td>3</td>
<td>15.0</td>
<td>1.77</td>
</tr>
<tr>
<td>4</td>
<td>14.8</td>
<td>1.79</td>
</tr>
<tr>
<td>5</td>
<td>14.5</td>
<td>1.78</td>
</tr>
<tr>
<td>6</td>
<td>13.0</td>
<td>1.81</td>
</tr>
</tbody>
</table>
Due to this variation in the optimum moisture content and maximum dry density values of these tests conducted on the same soil sample and because the test on the uncontaminated soil will act as a control test to determine the effect of crude oil contamination on the compaction characteristics of the kaolinite soil, all the tests results were again plotted in on one graph and SPSS software was used to obtain a single compaction curve (Figure 6.18).

![Compaction graph using SPSS](image)

Figure 6.18 Compaction graph using SPSS
The optimum moisture content of 14% and maximum dry density of 1.79 Mg/m$^3$ for the uncontaminated soil was obtained from the graph (Figure 6.18), which is within the range for those from Figure 6.17, also the optimum moisture content is above the 5% air void line.

6.5.2 Contaminated soil

The preparation and compaction test for the contaminated soil samples was conducted as described in Section 5.3. The moisture content calculation used in the plot for the compaction curve was based on equation 6.2. An example of compaction graph of a contaminated soil sample where the moisture content was calculated using equation 6.2 is shown in Figure 6.19. The results of the compaction tests for the contaminated soil samples are shown Table 6.11. The contaminated soil could not compact after 15% contamination and as such the optimum moisture content and the maximum dry density could not be obtained for the 20% compaction, this indicates that there was too much oil present in the soil to reach effective compaction.
Figure 6.19 Example of compaction curve for crude oil contaminated soil sample

Table 6.11 Compaction result for the contaminated soil samples

<table>
<thead>
<tr>
<th>% Contamination</th>
<th>OMC (%)</th>
<th>MDD (Mg/m³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>10.0</td>
<td>1.82</td>
</tr>
<tr>
<td>5</td>
<td>6.5</td>
<td>1.82</td>
</tr>
<tr>
<td>8</td>
<td>6.3</td>
<td>1.83</td>
</tr>
<tr>
<td>10</td>
<td>6.0</td>
<td>1.84</td>
</tr>
<tr>
<td>15</td>
<td>6.0</td>
<td>1.69</td>
</tr>
<tr>
<td>20</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
6.5.3. Discussion and Summary of Compaction Test Result

The shapes of the compaction curve changes with increase in percentage of oil contamination as shown in Figure 6.20. This change in compaction curve is typical of compaction curve of crude oil contaminated soil as reported by other authors (Khamehchiyan et al., 2007 and Rahman et al., 2010).

Figure 6.20 compaction curves for the different percentage of contamination
The compaction results show a decrease in the optimum moisture content with increase in the percentage of crude oil contamination up till 10% contamination (Table 6.12 and Figure 6.21), however the maximum dry density increases as the percentage of crude oil contamination increases up till 10% contamination after which there was a drop in the value (Table 6.12 and Figure 6.22). This trend in the maximum dry density values i.e. increase in maximum dry density value with increase in percentage of crude oil contamination has also been reported by other authors (Meegoda et al., 1998, Shin, 1997 and Al-sanad, 1995). But after 10% crude oil contamination, there was too much oil in the soil for it to reach an effective compaction and at 20% contamination the contaminated soil could no longer compact.

Table 6.12 Compaction Result

<table>
<thead>
<tr>
<th>% Contamination</th>
<th>OMC (%)</th>
<th>MDD (Mg/m³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>14.0</td>
<td>1.77</td>
</tr>
<tr>
<td>2</td>
<td>10.0</td>
<td>1.82</td>
</tr>
<tr>
<td>5</td>
<td>6.5</td>
<td>1.82</td>
</tr>
<tr>
<td>8</td>
<td>6.3</td>
<td>1.83</td>
</tr>
<tr>
<td>10</td>
<td>6.0</td>
<td>1.84</td>
</tr>
<tr>
<td>15</td>
<td>6.0</td>
<td>1.69</td>
</tr>
<tr>
<td>20</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The decrease in optimum moisture content with increase in percentage of crude oil contamination reflects the lubricating effect caused by the presence of crude oil, which facilitated compaction and reduced the amount of water needed to reach maximum density. The changes in compaction value is also due to the fact that the crude oil has partially occupied the inter-particle spaces and the
presence of the crude oil has changed the soil to a state of looser material (i.e. appears aggregated) than in the uncontaminated soil. It therefore means that when the crude oil was added to the soil, it made its way to the void space and shared the void space with water. This action reduced the amount of water that the void space was supposed to accommodate thus lowering the optimum moisture content. As stated in Chapter 3, when the crude oil was introduced into the clay soil it covered the points of contact on the clay molecules that were frequently taken up by water molecules with more stable ions thereby affecting the engineering performance and reducing the desire of clay for dissociating water molecules. So crude oil contamination will increase the time it will take for clay soil to compact or the soil will require a more compactive effort to achieve its desired compaction. It will be difficult for the soil to compact above 20% percentage light crude oil contamination.
Figure 6.21 Graph showing the effect of light crude oil contamination on optimum moisture content
Figure 6.22 Graph showing the effect of crude oil contamination on maximum dry density
6.6 Shear Strength Test Result

This section presents the result of the unconsolidated undrained triaxial test described in Section 5.4.3 for the contaminated and uncontaminated soil samples. The result was analysed from a plot of stress against strain relationship. The stress in this case is the compressive stress. The strain was calculated using the formula;

\[ \varepsilon = \left( \frac{x}{L_0} \right) \times 100\% \]  

Equation 6.11

where; \( x \) = the amount of compression at any stage during the test measured in (mm), \( L_0 \) = Initial length of the soil specimen measured in (mm).

The compressive stress was calculated using the formula

\( \text{Compressive Stress } \sigma = \left( \frac{CR(100 - \varepsilon) \times R}{Ao} \right) \times 1000 \)  

Equation 6.12

where; \( CR \) = Calibration of the loading ring, \( Ao \) = Initial cross sectional area of the soil sample, \( \varepsilon \) (\%) = Strain and \( R \) = loading dial gauge reading.

Also the Compressive stress = \( (\sigma_1 - \sigma_3) \) = Deviator stress (D) between the observed load ring reading \( R \) and the initial load ring reading \( R_0 \).

The initial cross-section Area of the soil sample was calculated from formula

\( Ao = \pi d^2 / 4 \)  

Equation 6.13

where; \( d \) = diameter of the soil specimen

6.6.1 Uncontaminated soil

The triaxial test for the uncontaminated soil was tested as described in Section 5.4.3 and a graph of compressive stress was plotted against strain for each of the three specimens as shown in Figure 6.23.
The curves for all the three soil specimens were then plotted on one graph (Figure 6.24). The peak compressive stress and the corresponding strain from the curves for all the three specimens were then read off and recorded (Table 6.13).
Figure 6.24 Graph of compressive stress against strain for the different soil specimen
Table 6.13 Showing Compressive stress and Strain

<table>
<thead>
<tr>
<th></th>
<th>( D = \sigma_1 - \sigma_3 )</th>
<th>( \sigma_3 )</th>
<th>( \sigma_1 )</th>
<th>Strain ( \varepsilon ) (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>350</td>
<td>100</td>
<td>450</td>
<td>14</td>
</tr>
<tr>
<td>B</td>
<td>380</td>
<td>200</td>
<td>580</td>
<td>14.6</td>
</tr>
<tr>
<td>C</td>
<td>432</td>
<td>400</td>
<td>832</td>
<td>16</td>
</tr>
</tbody>
</table>

The value of the major principle stress at failure was calculated from the formula

\[
\sigma_1 = (\sigma_1 - \sigma_3) + \sigma_3 \text{......................................................... Equation 6.14}
\]

\[
\sigma_1 = D + \sigma_3 \text{......................................................... Equation 6.15}
\]

where \( D = \) deviator stress and \( \sigma_3 = \) cell pressure

The undrained shear strength (\( C_u \)) for each of the tested soil specimens was calculated using the formula

\[
C_u = \frac{1}{2}(\sigma_1 - \sigma_3) \text{......................................................... Equation 6.16}
\]

But \( \sigma_1 - \sigma_3 = \) Deviator stress (\( D \))

So \( C_u = \frac{1}{2}(\sigma_1 - \sigma_3) \text{......................................................... Equation 6.17} \)

\[
C_u = \frac{1}{2}D \text{......................................................... Equation 6.18}
\]

The Mohr circle at the point of failure for the soil specimens was derived from a plot of shear strength (equation 6.18) against stress at failure (equation 6.15) and the graph produced a straight line for each of the soil specimen used in the triaxial test (Figure 6.25). A line was drawn through the peak of soil specimen lines and from the intercept of this line on the y-axis (shear strength). It was possible to determine the cohesion of the soil sample and also from the slope of the straight line it was possible to determine the frictional angle (Figure 6.25).
In undrained triaxial test, it is assumed that frictional angle ($\varphi$) equal to zero, meaning that the soil is purely cohesive material and has no friction. This perception is incorrect in that the apparent undrained cohesion results principally from the frictional resistance within the soil and also the frictional angle is not
zero due to the fact that the specimens are partially saturated i.e. the degree of saturation is less than 100% since the specimens were compacted. So the Mohr failure envelope for unconsolidated undrained triaxial tests on partially saturated soils is usually a curve and as such frictional angle (\( \phi \)) is not equal to zero. Based on the above statement the frictional angle was determined rather than assuming it to be zero. The effective stress was not determined for this experiment due to the fact the pore pressure was not measured during the test.

Also during the test, the moisture content, bulk density and dry density of each soil specimen was determined using equations 5.5 and 5.6. The result of the triaxial test for the uncontaminated soil sample shows that the cohesion is 146kN/m\(^2\) and the frictional is 8.1°.

### 6.6.2 Contaminated Soil

The preparation and the uncondolidated undrained triaxial test for the contaminated soil samples were done as described in Section 5.4.3 and the data analysis was done in the same way as in that of uncontaminated soil. However the moisture content for the contaminated soil samples was calculated using equation 6.2. The result shows a decrease in cohesion and frictional angle values with increase in percentage of crude oil contamination (Table 6.14).

### 6.6.3. Discussion and summary of shear strength test result

The shear strength of a soil is an essential property of that soil since it controls the bearing capacity of the soil and the stability of the foundation system of any engineering structure on the soil. The shear strength of a clay soil is influenced by a number of factors such as clay mineral, water content, chemistry of the pore
fluid, stress history, effective stress, stress path and soil structure (Barbour and Yang 1992), liquid limit has an influence on the development of strength, in that the higher the values of liquid limit the lower the values of strength and its durability (Stavridakis 2005).

The triaxial test result showed a decrease in the shear strength parameters (frictional angle ($\phi$) and cohesion ($c$)) of the kaolinite soil with increase in the percentage of the light crude oil contamination (Table 6.14). Some authors (Rahman et al. 2010, Khamehchiyan et al. 2007, Shin et al. 1997) observed a similar trend on the shear strength parameter of hydrocarbon contaminated soil. This decrease in shear strength characteristics is due the presence of the light crude oil which has a higher viscosity than water. The crude oil coats the soil and leaves a blanket surrounding the soil particles and by increasing the percentage of crude oil contamination, the chances of inter-particle slippage will also increase resulting in decrease in the shear strength of the soil.

Table 6.14 Cohesion and Frictional Angle Result

<table>
<thead>
<tr>
<th>% Contamination</th>
<th>C (kN/m$^2$)</th>
<th>$\phi^\circ$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>146</td>
<td>8.1</td>
</tr>
<tr>
<td>2</td>
<td>123</td>
<td>7.6</td>
</tr>
<tr>
<td>5</td>
<td>121</td>
<td>6.3</td>
</tr>
<tr>
<td>8</td>
<td>112</td>
<td>5.9</td>
</tr>
<tr>
<td>10</td>
<td>111</td>
<td>4.4</td>
</tr>
<tr>
<td>15</td>
<td>110</td>
<td>3.7</td>
</tr>
<tr>
<td>20</td>
<td>96</td>
<td>3.9</td>
</tr>
</tbody>
</table>
Figure 6.26 Graph of cohesion against percentage of contamination
Figures 6.26 and 6.27 shows a decrease in the cohesion and frictional angle values of the kaolinite clay soil as the percentage of the light crude oil was increased and because the introduction of crude oil into the soil sample made the soil to be slippery. It caused the frictional angle to decrease with increase in the percentage of crude oil contamination. The formation of large sized particle due to the crude oil contamination and decrease in the specific surface area also resulted in less cohesion which caused a decrease in the cohesion value.

Figure 6.27 Graph of frictional Angle against Percentage contamination
Increase in the percentage of crude oil contamination caused a reduced inter-particle friction due the lubrication effect provided by the crude oil which leads to failure within a shorter period of time. This reduction of the angle of internal friction is due to the inter-grain lubrication of the soil particles by the crude oil. So the reduction in the shear strength characteristics of the contaminated clay is due to the physiochemical effects caused by the reduction in dielectric constant and mechanical interaction caused by high pore fluid viscosity.

Crude oil contamination enhances soil degradation which destroys the inter-molecular forces between soil grains and consequently results in lowering of values of cohesion. This lowering of the cohesion value due to the crude oil contamination may enhance soil erosion.

6.7 Permeability Test Result

This section presents the result of permeability tests in which the preparation and test procedure was as described in Section 5.5.3. The falling head permeability test method was used for the experiment in this research and it involved the flow of water through a relatively short soil sample connected to a standpipe which provided the water head and also allowed measuring the volume of water passing through the soil sample.

During the experiment, the area of the capillary tube used was determined and this was calculated from the volume (mm$^3$) of water within a given height of the capillary (manometer) tube (mm) as described in Section 5.5.3. The average cross-sectional area of the manometer tube was determined by filling it with water and then allowing the water to run out in stages. At each stage the weight of water that passed out from the manometer tube and the fall in the water level in the tube
(i.e. the fall in height of the water inside the manometer tube) was measured. The weight of water at each stage was used to calculate the volume of water flowing out using equation 5.8. i.e.

\[
Volume = \frac{Weight}{Density}
\]

A graph of volume of water discharged against fall in water level (height) was plotted and the slope of the graph gave the area of the capillary tube (Figure 6.28). The calculation for the area of the tube was repeated five times until a consistent value was obtained.

Figure 6.28 Graph of volume against height for the area of capillary tube
6.7.1 Uncontaminated soil

The sample preparation and test for the permeability on the uncontaminated soil was done as described in section 5.5.3. During the test, the water level (h) was recorded at a time interval (30 minutes) and a graph of log (h₀/h) in meters was plotted against elapsed time in seconds, where h₀ was the recorded height at time zero.

Figure 6.29 is an example falling head permeability test result conducted on the compacted uncontaminated clay soil used in this research experiment. It is obvious from the graph, that the first run (i.e. day 1) gives a curved relationship and not a straight line as expected from theory. This curved relationship indicates the presence of air in the voids and the soil saturation is less than 100%. In the first run, as the degree of saturation is increased, some air dissolved in the permeating water. So there seem to be high permeability as the air was compressed and permeation leads to steady-state condition. In the second run (i.e. day 2), a more steady permeation was achieved and the curve seem to be more straight for the log (h₀/h) and elapsed time relationship with a longer linear portion. Subsequent runs (i.e. day 3 and day 4) tend to be more of a straight line throughout and all tend towards the coefficient of permeability (k). This achievement of steady-state permeation with the duration of the test is shown clearly in Figure 6.30 when all the data are combined and plotted in one graph.
The falling head permeability test was conducted on the uncontaminated soil specimen for at least four days. A graph of $(h_0/h)$ against elapsed time was plotted for each day until a straight line graph was obtained (Figure 6.29). The data for the four days were then plotted in one graph and the slope of the graph (Figure 6.30) was used to calculate the coefficient of permeability for the soil using equation 6.21 which was obtained through the formula;
\[ K = 2.3 \frac{aL}{AT} \log_{10} \frac{h_0}{h} \] .................................................................6.19

\[ K = 2.3 \frac{a}{A} \frac{L^*}{T} \log_{10} \frac{h_0}{h} \] .................................................................6.20

Since \( \frac{\log_{10} \frac{h_0}{h}}{T} \) = Slope of the graph of \( h_0/h \) against time lapse,

\[ K = 2.3 \left( \frac{a}{A} \right)^* L^* S \] .................................................................6.21

where;

- a = cross sectional area of the manometer (capillary) tube
- \( h_0 \) = recorded water level (height) at time zero
- h = recorded water level (height) at time t
- A = cross sectional area of the soil sample in the mould
- L = length of the soil sample in the mould
- T = elapse time
- S = slope of the graph
Four different tests were conducted on the uncontaminated soil. The reason for conducting the four different tests on the uncontaminated soil was based on the fact that the permeability result for the uncontaminated soil sample will act as a control experiment to know the effect of the light crude oil contamination on permeability characteristics of the kaolinite clay soil. So there was the need to have a reliable value for the uncontaminated soil and the coefficient of permeability (k) for the uncontaminated soil was found to be $5.03 \times 10^{-10}$ m/s.

Figure 6.30 Graph of log (ho/h) against time for four days
6.7.2 Contaminated Soil

The sample preparation and permeability test on the contaminated soil was as described in Section 5.5.3 for the contaminated soil sample. Although there may be concern about the effect of permeating fluid properties on the permeability of soil where the permeating fluid is not water, according to Khamechiyan et al. (2006) hydrocarbon have very low solubility in water and do not change the properties of distilled water at short time during the permeability test so the normal falling head permeability test procedure was used to test for the permeability of the contaminated soil and the result was calculated in the same way as for the uncontaminated soil.

There was a slight increase in the coefficient of permeability (k) values with increase in the percentage of contamination (Table 6.15). In all the permeability tests conducted on the light crude oil contaminated soil in this research, the coefficient of permeability (k) value ranged between $1.51.96 \times 10^{-10}$ m/s and $3.05 \times 10^{-10}$ m/s.

6.7.3 Discussion and Summary of permeability test result

The result of the permeability test (Table 6.15 and Figure 6.31) shows a significant decrease in coefficient of permeability (k) value between the uncontaminated soil and contaminated soil i.e. between 0% contamination and 2% contamination. Other authors (Khamehchiyan et al., 2006 and Al-sanad et al., 1995) also observed this trend in the coefficient of permeability value of a crude oil contaminated soil.
The decrease in permeability value between the uncontaminated soil and the contaminated soil sample (i.e. between 0% and 2%) is attributed to the reduction of pore volume due to trapped crude oil within the soil particle since oil occupies some pore space and also due to the fact that pore fluid is no longer water alone but crude oil and water. As stated in chapter 3 the bonding or substitution of ions within the clay structure allows for the manipulation of the clay chemistry by the contaminating fluid. The decrease in the coefficient of permeability value of the contaminated soil compared to that of the clean soil (i.e. 0% contamination) may be associated with the clogging of some inter-particle space with crude oil. However there was an increase in the permeability values of the contaminated soils with increase in the percentage of contamination i.e. between 2% and 20% oil contamination (Table 6.15 and Figure 6.31). The reason for this may be that as the percentage of crude oil contamination increases, the clay soil appears more like fine sand than clay. That is there was an increase in the void and the clay soil particle appeared aggregated. According to Izdebska-Mucha et al. (2011), in non-polluted soil, the soil fabric may be densely packed with dominating face-to-face and edge-to-edge types of contacts and inter-particle pores are more common.

Table 6.15 Permeability for contaminated soil

<table>
<thead>
<tr>
<th>% contamination</th>
<th>k x 10^{-10} (m/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>5.03</td>
</tr>
<tr>
<td>2</td>
<td>1.51</td>
</tr>
<tr>
<td>5</td>
<td>1.76</td>
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<tr>
<td>8</td>
<td>1.97</td>
</tr>
<tr>
<td>10</td>
<td>2.01</td>
</tr>
<tr>
<td>15</td>
<td>2.28</td>
</tr>
<tr>
<td>20</td>
<td>3.05</td>
</tr>
</tbody>
</table>
but in polluted soil the clay matrix is loosely packed with dominating edge-to-face types of contacts and inter-micro-aggregate pores are more frequent. This observation is among the reason for the increase in coefficient of permeability value as the percentage of crude oil contamination was increased.

Figure 6.31 Graph of permeability against percentage of contamination

Increase in the percentage of oil contamination made the clay soil structure to appear aggregated and the aggregated structure increased the effective pore space and this situation increased the permeability of the soil as the percentage of oil
contamination increased. Rahman et al. (2010b) observed that the permeability of sand dominated soil is higher than that of silt or clay dominated soil. So since permeability increases as the percentage of fine decreases, it is expected that the permeability at 2% crude oil contamination will be lower than that at 20% crude oil contamination. So an increase in the percentage of crude oil contamination caused an increase in the coefficient of permeability. But the value of the coefficient of permeability even at 20% contamination was still lower than that of the clean soil. This reduction in the coefficient of permeability values of the crude oil contaminated soil is due to the fact that when crude oil and water are mixed together, a total or complete mixture of oil and water is not easily achievable. There is usually a boundary layer of soil and water mixture present. Crude oil in soil will trap some of the water, consequently lowering the coefficient of permeability of polluted soils.

The permeability of the clay soil both the contaminated and uncontaminated clay soil up to the level tested in this research are still within the typical permeability value of clay soil as shown by Hazen (1930) in Table 6.16 and the UK Environment Agency guideline (2009) requires clay used in capping for land fill site should be within the following limit; Permeability $5 \times 10^{-10} \leq P \leq 5 \times 10^{-9}$, so the contaminated soil samples within the level tested in this research are still within the limit recommended by the environment agency for landfill construction. However the contaminated soil should be stabilized or capped before being used for landfill construction to avoid surface and groundwater contamination.
Table 6.16 Typical Values of coefficient of permeability of soils (after Hazen, 1930)

<table>
<thead>
<tr>
<th>Soil</th>
<th>K (m/sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Clean Gravel</td>
<td>$1 \times 10^{-2} - 1$</td>
</tr>
<tr>
<td>Coarse Sand</td>
<td>$1 \times 10^{-2} - 1 \times 10^{-4}$</td>
</tr>
<tr>
<td>Fine Sand</td>
<td>$1 \times 10^{-4} - 1 \times 10^{-6}$</td>
</tr>
<tr>
<td>Silt</td>
<td>$1 \times 10^{-5} - 1 \times 10^{-7}$</td>
</tr>
<tr>
<td>Clay</td>
<td>Less than $1 \times 10^{-8}$</td>
</tr>
</tbody>
</table>

6.8 Consolidation Test Result

This section presents the analysis and result of the oedometer test procedure described in Section 5.6.3. During the consolidation oedometer test the initial and final moisture content of the soil was calculated following the moisture content calculation procedure described in equation 6.2. The moisture content was then used to calculate the initial void ratio using the equation:

$$e_0 = \left(\frac{w \cdot G_s}{S}\right) \cdot 100$$ ……………………………………………equation 6.22

where; $e_0$ = initial void ratio, $w$ = moisture content, $G_s$ = specific gravity, $S$ = degree of saturation and the void ratio was calculated from the equation;

$$e = e_0 - \Delta e$$

$$\Delta e = \left(\frac{1 - e_0}{H_o}\right) \times \Delta H$$ ………………………………………………..….Equation 6.23

where; $\Delta e$ = change in void ratio, $e_0$ = initial void ration, $\Delta H$ = settlement, and $H_o$ = height of sample.
6.8.1 Uncontaminated soil sample

Based on the oedometer (consolidation test) experiment carried out in the laboratory (as described in Section 5.6.3), graphs of compression gauge reading against log-time and compression gauge reading against square-root time were plotted and these graphs were used to determine the values of the consolidation characteristics for the uncontaminated soil sample. So the consolidation result on the contaminated and uncontaminated soils was analysis using the log of time fitting method and the square root of time fitting method.

Log of time fitting method

In analysing the consolidation result using the log-time method, the 0% mark of the graph (Figure 6.32) was derived from the upper curved end of the graph and two sets of points on the time values with ratio 1:4 (e.g. 0.25 and 1 or 0.5 and 2) were selected to represent ab and cd (Figure 6.32). The vertical distances between ab and cd were measured to represent ae and cf respectively and the distances ae and cf were measured upward to points ag and ch respectively. Then a horizontal line was drawn across points g and h to give the 0% (U= 0) of the graph.

Also the 100% mark of the graph was derived by drawing a line on the straight portion of the curve to produce line JK, also a line was drawn on the tangent of the straight line portion of the end of the curve to produce LM (Figure 6.32). The lines JK and LM meets at a point N and this point N represents the 100% (U = 100) of the graph.

The 50% mark on the consolidation graph was derived using the formula $\frac{1}{2}(U_0- U_{100})$ and the point was marked on the graph represented as OP (Figure 6.32). At
the intersection of the line **OP** (50%) with the curve, the time for 50% was read off from line **OQ**.

![Settlement log-time graph](image)

Figure 6.32 Settlement log-time graph

The coefficient of consolidation using the log of time method was calculated using equation 6.25 which was derived from the formula;

232
\[ Cv = \frac{T_{50}}{t_{50}} h^2 \]  

...Equation 6.22

\[ Cv = 0.197 \frac{h^2}{t_{50}} \]  

...Equation 6.23

\[ Cv = \frac{0.197 \left( \frac{h}{1000} \right)^2}{t_{50}} \times 60 \times 24 \times 365.25 \]  

...Equation 6.24

\[ Cv = \frac{0.026 \bar{H}}{t_{50}} \text{ m}^2/\text{year} \]  

...Equation 6.25

where; \( h \) = the length of maximum drainage path

The height (H) of the specimen = 2h

\( \bar{H} \) = mean specimen height during load increment measured in mm

\( t_{50} \) = the value of \( t_{50} \) on the log time graph measured in minutes

**Square root of time fitting method**

This method involves a plot of dial gauge readings against square root of time and the experimental curve is usually divided into three parts consisting of a short curved portion representing an initial compression due to the compression of air or reorientation of the particles in the sample, a straight line portion which is due to the expulsion of water/fluid from the sample and a second curved portion towards the end of the curve representing any secondary compression. To get the theoretical 0% of the graph, the straight portion of the graph (Figure 6.33) was extended downward and upward to intersect the settlement ordinate at \( R \) forming line \( RS \) and this point \( R \) represents \( U = 0 \) i.e. 0%.

From the graph (Figure 6.33), point \( a \) was chosen on the line \( RS \) and the value of \( ab \) was multiplied by 1.15 to produce \( bc \) then a line was drawn from \( R \) to
intersect point \( c \) and the line was produced to form line \( RT \). The 90\% (\( U = 90 \)) of the graph was marked at the point where line \( RT \) intersect the settlement curve at point \( d \) (Figure 6.33) and the corresponding value of \( \sqrt{t_{90}} \) was read off.

Figure 6.33 Settlement Square – Root time graph
For the square root of time method the coefficient of consolidation was calculated using equation 6.29 which was derived from the formula;

\[ C_v = \frac{T_{90}}{t_{90}} h^2 \]  \hspace{1cm} \text{Equation 6.26}

\[ C_v = 0.848 \frac{h^2}{t_{90}} \]  \hspace{1cm} \text{Equation 6.27}

\[ C_v = \frac{0.848 \left( \frac{h}{1000} \right)^2}{t_{90}} \times 60 \times 24 \times 365.25 \]  \hspace{1cm} \text{Equation 6.28}

\[ C_v = \frac{0.112}{t_{90}} H^2 \text{ m}^2/\text{year} \]  \hspace{1cm} \text{Equation 6.29}

where; \( h \) = the length of maximum drainage path

The height (H) of the specimen = 2h

\( \tilde{H} \) = mean specimen height during load increment

\( t_{90} \) = the value of \( t_{90} \) on the square root time graph measured in minutes

According to Barnes (2000) the square root of time method gives a reasonable straight line so less judgement is required in obtaining the coefficient of consolidation value but the log of time method is used when a straight line portion cannot be easily deduced from the square root of time method but Head and Epps (2011) stated that it is preferable to calculate coefficient of consolidation from the log of time method rather than the square root of time method because the middle of the laboratory settlement curve is the portion which agrees most closely with the theoretical curve. So in this research the coefficient of consolidation was calculated using both the log of time method and the square root of time method and the result from both methods was compared.
The coefficient of volume compressibility (Mv) was calculated using equation 6.30:

\[
Mv = \frac{1000 \left( -\frac{\delta e}{\delta p} \right) m^2 / MN}{1 + e_1} \]

where; \( e_1 \) = the void ratio at the beginning of the load increment
\( \delta e \) = change in void ratio for the increment and \( \delta p \) = change in pressure (kN/m\(^2\)).

The compression index (Cc) was derived from the slope in the linear portion of the curve of void ratio (e) plotted against the log of pressure (i.e. log p) as shown in Figure 6.34.

![Graph of Log P against Void ratio showing slope for Cc](image)

Figure 6.34 Graph of Log P against Void ratio showing slope for Cc
6.8.2 Contaminated Soil

The sample preparation and test for the consolidation characteristics on the contaminated soil was done as described in Section 5.6.3 and the analysis of the data together with the calculation of the consolidation parameters (coefficient of consolidation (Cv), coefficient of volume compressibility (Mv) and compressibility index (Cc)) was done in the same way as that of uncontaminated soil described in Section 6.8.1 but the moisture content for the contaminated soil was calculated using equation 6.2.

6.8.3 Discussion and Summary of Consolidation Result

The results in Tables 6.17a and 6.17b and Figures 6.35a and 6.35b shows a decrease in the Coefficient of Consolidation (Cv) values with increase in pressure. It also shows that the coefficient of consolidation increases with increase in the percentage of contamination as shown in Figures 6.36a and 6.36b and Tables 6.17a and 6.17b.

Table 6.17a Coefficient of Consolidation (Cv) using Log of time fitting method (t_{50})

<table>
<thead>
<tr>
<th>Percentage of contamination (%)</th>
<th>0</th>
<th>2</th>
<th>5</th>
<th>8</th>
<th>10</th>
<th>15</th>
<th>20</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pressure</td>
<td>50</td>
<td>100</td>
<td>200</td>
<td>400</td>
<td>200</td>
<td>100</td>
<td>50</td>
</tr>
<tr>
<td>0</td>
<td>1.3473</td>
<td>1.3166</td>
<td>1.2573</td>
<td>1.1467</td>
<td>1.0951</td>
<td>1.0702</td>
<td>1.0458</td>
</tr>
<tr>
<td>2</td>
<td>1.5434</td>
<td>1.3447</td>
<td>1.2841</td>
<td>1.2263</td>
<td>1.1712</td>
<td>1.093</td>
<td>1.0681</td>
</tr>
<tr>
<td>5</td>
<td>1.6411</td>
<td>1.365</td>
<td>1.3339</td>
<td>1.2739</td>
<td>1.2449</td>
<td>1.1353</td>
<td>1.0842</td>
</tr>
<tr>
<td>8</td>
<td>1.8355</td>
<td>1.3924</td>
<td>1.3607</td>
<td>1.3297</td>
<td>1.2699</td>
<td>1.1851</td>
<td>1.1318</td>
</tr>
<tr>
<td>10</td>
<td>2.1436</td>
<td>1.4492</td>
<td>1.384</td>
<td>1.3525</td>
<td>1.3217</td>
<td>1.2335</td>
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</tr>
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<td>1.6411</td>
<td>1.5315</td>
<td>1.4626</td>
<td>1.4293</td>
<td>1.3339</td>
<td>1.2165</td>
</tr>
</tbody>
</table>
Table 6.17b Coefficient of Consolidation (Cv) using Square root of time fitting method ($t_{90}$)

<table>
<thead>
<tr>
<th>Pressure</th>
<th>0</th>
<th>2</th>
<th>5</th>
<th>8</th>
<th>10</th>
<th>15</th>
<th>20</th>
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<tbody>
<tr>
<td>50</td>
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<td>2.5267</td>
</tr>
<tr>
<td>100</td>
<td>1.2932</td>
<td>1.3382</td>
<td>1.3718</td>
<td>1.3994</td>
<td>1.4498</td>
<td>1.5438</td>
<td>1.6483</td>
</tr>
<tr>
<td>200</td>
<td>1.2505</td>
<td>1.2932</td>
<td>1.3249</td>
<td>1.3516</td>
<td>1.3994</td>
<td>1.4882</td>
<td>1.5285</td>
</tr>
<tr>
<td>400</td>
<td>1.1711</td>
<td>1.2098</td>
<td>1.2804</td>
<td>1.3061</td>
<td>1.3516</td>
<td>1.4355</td>
<td>1.4734</td>
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<tr>
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<td>0.9456</td>
<td>0.9639</td>
<td>1.0761</td>
<td>1.1828</td>
<td>1.2098</td>
<td>1.2381</td>
</tr>
</tbody>
</table>

Figure 6.35a Plot of coefficient of consolidation (using $t_{50}$) against Pressure
Figure 6.35b Plot of coefficient of consolidation (using \( t_{90} \)) against Pressure
Figure 6.36a Coefficient of consolidation against percentage contamination

Figure 6.36b Coefficient of consolidation against percentage contamination
According to Rao et al., (2008) an increase in coefficient of consolidation indicates an increase in coefficient of permeability, so the decrease in coefficient of consolidation with increase in pressure (Figure 6.35) is attributed to the reduction of pore volume due to trapped crude oil. Also since the crude oil occupies the pore space, the permeability will increase with increase in the percentage of crude oil contamination and because of this higher permeability, the time required for consolidation will increase.

Again the decrease in Cv value with increase in pressure may be attributed to the binding action of the contaminated soil due to the interaction of crude oil and clay minerals present in the soil, that is, the binding action of the contaminated soil formed due to physio-chemical interactions of the crude oil and the clay soil.

An increase in the coefficient of consolidation with increase in percentage of contamination (Figure 6.35) reflects the presence of the crude oil which is an organic matter. Another reason for this increase in the values of Cv with higher crude oil contamination could be due to the initial settlement and expulsion of oil from the soil matrix with increasing loadings through the process of consolidation which is time dependent. The possible reason for an increase in coefficient of consolidation may be attributed to change in the structure that may take place due to the interaction between clay minerals and crude oil, this interaction between the crude oil and the clay soil decreases the repulsive force between the clay particles. It also imparted flocculated structure with some binding action that could be responsible for induced pre consolidation pressure and increases coefficient of consolidation. Clay soil have negatively charged mineral surfaces (Fang et al., 2007) and hydrocarbons have positively charged hydrogen atoms on its surface so the binding action results from the fact crude oil is a reactive chemical that forms
a covalent linkage (bond) with the hydroxyl groups of the clay minerals and the covalent bonding may be responsible for the observed binding action.

The decrease in the values of coefficient of consolidation noticed for crude oil contaminated soils is related to the decrease in the values of coefficient of permeability for crude oil contaminated soils. The consolidation process from the crude oil contaminated soils is achieved longer than those from uncontaminated soil. This means that less area will be consolidated for a given year than for the uncontaminated soils. This tends to suggest, however, that the soil is more prone to settlement when polluted than when unpolluted.

On the graph of void ratio ($e$) against log $p$, there was a decrease in void ratio with increase in the percentage of crude oil contamination (Figure 6.36 and Table 6.18), although this decrease was not consistent but the general trend showed a decrease in value, also the void ratio values also decreased with increase in pressure. The increase in void ratio value observed (Figure 6.37) may be due to the initial reduction in the percentage of fine as the crude oil caused the clay soil particle to form sand size particles which created large void spaces between the soil matrix but as the percentage of contamination was increased this void space was filled with more crude oil which caused the reduction in the void ratio value.

Table 6.18 Void Ratio

<table>
<thead>
<tr>
<th>P</th>
<th>log p</th>
<th>0%</th>
<th>2%</th>
<th>5%</th>
<th>8%</th>
<th>10%</th>
<th>15%</th>
<th>20%</th>
</tr>
</thead>
<tbody>
<tr>
<td>50</td>
<td>1.6990</td>
<td>0.5909865</td>
<td>0.539589</td>
<td>0.612461</td>
<td>0.547588</td>
<td>0.483165</td>
<td>0.551114</td>
<td>0.427505</td>
</tr>
<tr>
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<td>2.0000</td>
<td>0.5483212</td>
<td>0.523777</td>
<td>0.579663</td>
<td>0.516948</td>
<td>0.452548</td>
<td>0.526142</td>
<td>0.430308</td>
</tr>
<tr>
<td>200</td>
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<td>0.592977</td>
<td>0.521281</td>
<td>0.462803</td>
<td>0.531482</td>
<td>0.422785</td>
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<td>0.5384876</td>
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<td>0.518836</td>
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<td>0.564765</td>
<td>0.654838</td>
<td>0.590606</td>
<td>0.527456</td>
<td>0.604514</td>
<td>0.505098</td>
</tr>
</tbody>
</table>
Figure 6.37 Graph of void ratio against log pressure

Figure 6.38 and Table 6.19 shows a plot of settlement against pressure, the result shows that there was a decrease in settlement values with increase in pressure. Again the settlement value decreased with increase in the percentage of crude oil contamination.
Table 6.19 Settlement

<table>
<thead>
<tr>
<th>P</th>
<th>log p</th>
<th>0%</th>
<th>2%</th>
<th>5%</th>
<th>8%</th>
<th>10%</th>
<th>15%</th>
<th>20%</th>
</tr>
</thead>
</table>

Figure 6.38 Graph of settlement against pressure
From Figure 6.39 and Table 6.20, there was a decrease in the coefficient of volume compressibility (Mv) with increase in consolidation pressure when the pore fluid was replaced by crude oil but there was an inconsistent change in the Mv value with increase in pressure as the percentage of crude oil contamination increases. The reason for this observed phenomenon is that there was no resistance for the compression of the clay soil as the percentage of crude oil contamination increased.

The probable reason for the initial increase in the coefficient of volume compressibility value (Mv) under varying loading conditions could be due to the rearrangement of the newly bonded soil particles into the macro voids created as the soil was compressed. However, the decrease in the values of Mv with increase in the percentage of crude oil contamination (Figure 6.40) may have been due to the presence of fewer voids as more bonded soil matrix was formed with higher oil content. Hence the contaminated soil experienced little compression, while the excess oil was squeezed out from the sample. It was also observed that the values of Mv decreased under increased pressures.
Table 6.20 Coefficient of Volume Compressibility

<table>
<thead>
<tr>
<th>P</th>
<th>Log P</th>
<th>0%</th>
<th>2%</th>
<th>5%</th>
<th>8%</th>
<th>10%</th>
<th>15%</th>
<th>20%</th>
</tr>
</thead>
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<td>50</td>
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<td>0.173</td>
<td>0.339</td>
<td>0.337</td>
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<tr>
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<td>0.154</td>
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<tr>
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<td>0.041</td>
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<td>0.115</td>
</tr>
</tbody>
</table>

Figure 6.39 Graph of coefficient of volume compressibility against pressure
When the pore space of the soil was occupied by organic fluid rather than water, there was a decrease in the compressibility of the sample and this decrease in volume was accompanied by an apparent increase in pre-consolidation stress. This behaviour is based on the fact that when the pore fluid changes from water to
crude oil, the increase in effective stress and the contraction of the diffused double layer caused the contraction of the soil particles and shrinkage of the sample. Because of the increase in the effective stress between the soil particles, the resistance of the soil structure to compression increased and this resulted in a decrease in compressibility. Also, expansion of the double layers within the soil aggregates will result in swelling and an increase in the sample volume.

The result of the compression index (Figure 6.41 and Table 6.21) shows an increase in the compression index values with increase in percentage of crude oil contamination. This increase in compression index may be due to mechanical factor such as viscosity which enhances the sliding of particles due to lubrication of soil particle and it is also attributed the to open structure of the contaminated soil due to the presence of oil.
Table 6.21 Compression Index

<table>
<thead>
<tr>
<th>Percentage of Contamination (%)</th>
<th>Cc</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0.038</td>
</tr>
<tr>
<td>2</td>
<td>0.056</td>
</tr>
<tr>
<td>5</td>
<td>0.067</td>
</tr>
<tr>
<td>8</td>
<td>0.075</td>
</tr>
<tr>
<td>10</td>
<td>0.08</td>
</tr>
<tr>
<td>15</td>
<td>0.083</td>
</tr>
<tr>
<td>20</td>
<td>0.084</td>
</tr>
</tbody>
</table>

Figure 6.41 Graph of compression index against percentage of contamination
CHAPTER 7 SUMMARY AND CONCLUSION

7.1 Introduction

The main essence of this research was to see how light crude oil contamination affects the geotechnical properties of kaolinite clay soil. This chapter summarizes the findings of the test results discussed in the previous chapters. It also evaluates the contribution to knowledge as well as the extent to which the results meet the research objectives regarding possible use of the crude oil contaminated soil.

7.2 Summary of Results

Most structures such as buildings, bridges, roads etc. rely on the properties of soil for their stability. However “problem soil” has properties that make them unsuitable to be safely and economically used for the construction of civil engineering structures.

Many soils can prove to be problematic in geotechnical engineering projects, because they may expand, collapse, disperse, undergo excessive settlement, have a distinct lack of strength or are soluble. These characteristics may be attributable to their composition, the nature of their pore fluids, their mineralogy or their fabric. When the water content of some soils change, it may cause problem to civil engineering activities that the soil has been designed for. The change in the water content of some of these soils may cause changes to their volume and such volume change can damage structures that are built on the soils.

Water content has been widely applied during many essential calculations related to soil properties such as liquid and plastic limits, unconfined compressive strength and compaction of soil. But the water content of crude oil contaminated soil cannot be calculated simply based on the weight loss of the sample during
drying but based on the difference in physical properties such as the pore fluid constituents.

Organic fluids exhibiting boiling points higher than the boiling point of water will not evaporate easily at the temperature of the boiling point of water. On the other hand, organic compounds exhibiting boiling points lower than the boiling point of water will evaporate more easily at the boiling point of water. Since the oven temperature for drying soil sample during moisture content determination is usually set at the temperature slightly higher than the boiling point of water \( (105^0\text{c}) \), evaporation chart was created to estimate the rate of evaporation of the crude oil during in the moisture content determination. The water content was calculated considering the fact that the soil pore space contains a mixture of water and crude oil i.e. fluid content = water content + oil content.

So this research examined the effect of light crude oil contamination on the geotechnical properties of kaolinite clay soil to see whether the interaction of the oil and clay makes the soil behave like a problem soil or not. The results of the study provided details on the index properties, compaction characteristics, permeability, consolidation and shear strength characteristics of soils contaminated with different percentages of crude oil measured by the dry weight of the soil.

The geotechnical behaviour of clay soils is influenced by the mineralogical composition, physiochemical interactions between clay particles, inter-particle forces, pore fluid chemistry and soil structure. Remediation work or use of crude oil contaminated soil for any possible geotechnical application requires a good knowledge of the geotechnical properties and behaviour of the crude oil contaminated soil.
The results, discussions and findings presented in the preceding chapters of this thesis, provided an eloquent suggestion that the aim and objectives of the research has been achieved.

- Through the literature review it was noted that limited studies has been done regarding the effect of crude oil contamination on kaolinite clay soil which is the dominant soil type in the low latitude climate (tropical zone and dry zone) where the bulk of the world’s crude oil production takes place.

- In order to adequately understand the effect of crude oil contamination on kaolinite clay, the structure of clay soil, clay minerals and structure of hydrocarbon was examined. The type of bonding structure that exists in the clay soil and how the hydrocarbon (crude oil) was accommodated within the clay soil has also been shown.

- The pores space of the clay soil is occupied by air and water and the introduction of crude oil will alter this relationship. So a formula was derived for the actual water content rather than the ‘fluid content’ and all the geotechnical calculations was based on this actual water content.

- Kaolinite clay soil has electrically negative mineral surfaces and the crude oil has positively charged hydrogen atoms on its surface so the binding action between the clay soil and the hydrocarbon resulted from the fact that crude oil is a reactive chemical that forms a covalent linkage (bond) with the hydroxyl groups of the kaolinite clay minerals which resulted in a covalent bonding.

- The British Standard BS1377: 1990 (Euro code 7; EN1997:2-5) was adopted for the geotechnical tests used in this research and the results of the kaolinite
clay soil showed a slight alteration with introduction of the light crude oil when compared to the result of the uncontaminated soil.

From the tests conducted in the laboratory to examine the effect of light crude oil contamination on the geotechnical properties of kaolinite clay soil the following observations were made;

- The index properties results of the crude oil contaminated soil showed an increase in values with increase in percentage of crude oil compared to that of uncontaminated soil. This increase in classification values is due to the fact that hydrocarbon is a non-polarised liquid and its presence in the contaminated soil caused a reduction in the thickness of the water film around the clay minerals. The increase in the index properties values is therefore associated with the bonding cation in the clay minerals.

- The compaction result showed that maximum dry density (MDD) increased from 1.82 to 1.84 Mg/m$^3$ and that the optimum moisture content decreased from 10% at 0% contamination to 6% at 15% contamination. This phenomenon is due to the fact that when the crude oil was added to the soil, it made its way to the void space and shared the void space with water and this action reduced the amount of water that the void space was supposed to accommodate there by lowering the optimum moisture content. However at 20% contamination and above, the soil was too wet for compaction test. The lubricating effect of the crude oil caused the increased maximum dry density and reduced the amount of water needed to reach maximum density.

- Crude oil contamination resulted in the decrease of shear strength parameters (cohesion and frictional angle) of the soil. The cohesion value decreased from 146kN/m$^2$ for the uncontaminated soil to 96kN/m$^2$ at 20% contamination.
Also, the frictional angle decreased from 8.1\(^0\) for the uncontaminated soil to 3.9\(^0\) at 20% for the contaminated soil. This decrease in shear strength values can be attributed to the fact that crude oil has a higher viscosity than water and also to the lubrication and slippage of the soil particle caused by the presence of the crude oil. So the crude oil contaminated soil has lower shear strength compared to uncontaminated soil. A decrease in the strength of any material including soil reduces its engineering use. This reduction in the strength value may increase the risk of soil erosion due the the decrease in cohesion value.

- The coefficient of permeability decreased with the introduction of crude oil to the soil from 5 x 10\(^{-10}\) m/s at 0% contamination to 1.51 x 10\(^{-10}\) m/s at 2% contamination. But there was an increase in permeability value as the percentage of crude oil contamination increased i.e. the coefficient of permeability increased from 1.51 x 10\(^{-10}\) m/s at 2% contamination to 3.05 x 10\(^{-10}\) at 20% contamination m/s. The initial reduction in the coefficient of permeability (k) value between the uncontaminated soil and the contaminated soil is attributed to the reduction in the pore space due to the trapped oil within the soil particle. The slight increase between the contaminated soils i.e. 2% to 20% is attributed to the fact that as the percentage of the crude oil contamination increases, the clay structure appeared aggregated. So the coefficient of permeability increased with increase in the percentage of crude oil contamination. This increase in permeability is of significant concern in geotechnical work as it portends the risk of surface and groundwater contamination and increases in the rate of settlement.
• The coefficient of consolidation value (Cv) increased with increase in the percentage of contamination. This increase in value is attributed to the initial settlement and expulsion of oil from the soil matrix. This can also be attributed to changes in the soil structure caused by the interaction between the clay minerals and the crude oil. The introduction of the crude oil decreased the repulsive force between the clay particles.

• However there was a decrease in the Cv value with increase in pressure and this can be attributed to the reduction of pore volume due to trapped crude oil and the binding action between the crude oil and the clay soil.

• Also there was a decrease in void ratio with increase in percentage of crude oil contamination due to the fact that pore space of the soil was occupied by water and crude oil. Again due to the fact that there was no resistance for the compression of the clay soil as the percentage of crude oil contamination was increased.

• The coefficient of volume compressibility (Mv) decreased with increase in pressure as the percentage of crude oil contamination increased. This decrease in coefficient of volume compressibility with increase in percentage of crude oil contamination was due to the presence of fewer voids and little compression in the crude oil contaminated soil caused by crude oil occupying the void space of the soil matrix.

• Crude oil contamination affected mechanical characteristics such as viscosity to enhance the sliding of particles due to lubrication of soil which in turn increased the compression index of the crude oil contaminated soil.
In clay soil – crude oil contamination system, the clay particles carry a net negative charge and it attracts cations from the environment. Crude oil being an organic compound contains a large amount of hydrogen ions and the number of
monovalent hydrogen cations required is twice the number of divalent cations to neutralize the net negative charge of the clay soil. This leads to a thicker diffused double layer that increased the liquid limit. Also the plastic limit of soil is observed to increase slightly with the increase in crude oil contamination because of the increased diffused double layer thickness. The presence of monovalent cations in the soil pore water system weakens the soil, and crude oil is rich in hydrogen which is a monovalent cation. The hydrogen ion concentration becomes higher in the contaminated soil- water system, this monovalent hydrogen ion weakens the soil and hence unconfined compressive strength may decrease.

The crude oil contamination is responsible for the high plasticity, high shrinkage, high compressibility, low permeability and low strength observed in the contaminated kaolinite soil compared with that of the uncontaminated kaolinite soil (Figure 7.1). The strength of the contaminated soil is reduced due to the high water content and plasticity caused by the crude oil contamination. So crude oil contamination does not just affect the quality of the soil and ground water, it also alters the physical and geotechnical properties of the oil contaminated soil.

The light crude oil contamination up to the level tested in this research did not make the contaminated kaolinite clay to behave like a problem soil because to the geotechnical and highway engineers, a problem soil is one that poses problem to construction. Such problem may be as a result of instability of the soil which makes it unsuitable as a construction material in foundations, buildings, highways, water retaining structures, dams. Problem soil may be soils that are expansive/ swelling and collapsing soils (Ola, 1987).
7.3 Effect on soil structure

The liquid limit of the kaolinite clay was found to be dependent on the pore fluid composition and it increased with increase in the percentage of crude oil contamination. In addition, the shear strength decreased with increase in the percentage of crude oil contamination since unconfined compressive strength has been found to be dependent on the dielectric constant of the pore fluid. This decrease in the strength of crude oil contaminated soils makes the soil susceptible to compaction, deformation and erosion since high-strength soils resist deformation. This may be due to the fact that as water content increases – cohesion decreases, because increasing water content causes greater separation of clay particles (and thus easier slippage) which might enhance erosion of the contaminated soil.

The study of compressibility is important for safe engineering design and for determination of probability of structural settlement. It depends on the consolidation properties of soil, such as, coefficient of compression and compression index, coefficient of permeability (k) etc. It also depends upon many factors like type of soil, void ratio, degree of saturation, soil structure, stress history, nature of pore fluid etc. This study has established that crude oil contamination caused an increase in the compressibility of the soil and has higher coefficient of consolidation than the uncontaminated soil. This suggests that there is a higher risk of settlement of the crude oil contaminated soil.

7.4 Effect on construction

Soil is a vulnerable and essentially non-renewable resource that fulfils a number of functions and services for society which are central to social, economic and
environmental sustainability, whilst also fulfilling the function of serving as the raw material and platform for construction (Defra, 2009).

Strength is an important factor considered during the design of a structure and from this research it has been established that crude oil contamination lowered the strength of kaolinite clay soil, it therefore means that the used of the crude oil contaminated soil might be limited due to the reduction in its strength with increase in crude oil contamination.

It was found that the reduction in strength of the crude oil contaminated soil increased as a proportion of the percentage of contamination. The practical implication of this is that crude oil contaminated kaolinite soil may suffer greater settlement during and after construction, if measures are not taken to stabilise the soil with lime, cement or other binders. This would have further implications on satisfying the agreed timeline for the construction contract and penalties may be incurred if these timelines are exceeded.

Due to the fact that crude oil contamination caused a reduction in strength and increase consolidation values, crude oil passing through or standing in construction sites risks becoming buried, trapped or otherwise integrated into the soil or structure, and contamination of formwork or shuttering into which cement is to be poured may necessitate expert advice from civil engineers before construction can continue. Also cleaning of crude oil construction site requires careful supervision to ensure that the work is undertaken methodically and safely, these problems might delay the progress of the construction work.

Structures that have lightweight and shallow foundation are more suitable for contaminated site than those with deep foundations. Deep foundation may involve the removal of large quantity of soil and after construction involving
deep foundation on contaminated soil. It may also be technically difficult to implement additional remediation measures or a design to reduce contaminant migration.

Due to the increase in the compressibility of the soil with increase in the percentage of crude oil contamination, before buildings or roadways can be constructed on crude oil contaminated soil, the grade level should be raised by adding compacted fill. However, adding significant amounts of compacted fill puts significant loads on the soil which can cause significant settlements.

7.5 Possible use of contaminated soil in construction

Structural developments are considered on sites where the contamination level is low and the possibility of extensive migration of contaminants from such site would most likely be low. However, there should be proper geotechnical investigation for the construction of buildings at sites with history of crude oil contamination into the subsurface environment. Compaction of the soil in areas susceptible to the risk of oil pollution may reduce the contamination of lower soil layers while providing time for clean-up measures to be implemented.

The suitability of petroleum contaminated soils in road construction shows that in construction applications, stabilizing the soil with cement, mixing it with crushed stone aggregate for use in road bases or sub bases as well as using it as a fine aggregate replacement in hot mix asphalt concrete, improves it potential for use in road construction.

Although the research has shown that crude oil contamination will alter the geotechnical properties of kaolinite soil used in the experiment, the crude oil contaminated kaolinite soil may be usefully applied in highway and stabilized
soil. In such application there is the need to stabilize or cap the soil to prevent or minimized the release of the contaminants into the ground water or surface water system and environment. Stabilization can be achieved by mixing the soil with cement or lime; mixing it with crushed stone aggregate for use in road bases or sub bases; and using it as a fine aggregate replacement in hot mix asphalt concrete.

7.6 Conclusion
Knowledge of soil composition is useful in the determining the geotechnical properties of the soil. It can be helpful in predicting the future behaviour of the soil. Knowledge of the geotechnical properties of contaminated soils are equally important if infrastructural projects located on crude oil contaminated sites are to be sustained. However, the saturation of soil by crude oil which is characterized by physico-chemical properties that differ from that of water has been found to have a deteriorating effect on the soil’s mechanical and filtration parameters, plasticity, consolidation, compaction and strength properties. This was based on the fact that the calculation of moisture content in crude oil contaminated soil considered the fact that the pore fluid was made up of water and crude oil. Also considered was the evaporation of the crude oil during oven drying.

The variables possessed by the clay soil are hydraulic gradient and infiltration capacity and the variables possessed by the crude oil are viscosity and density. These variables influenced the geotechnical properties of the crude oil contaminated soil. The viscosity of the crude oil enhanced the sliding of the clay soil particle due to lubrication. Also, the crude oil reduced the amount of water
in the pore space, which caused a reduction in the compaction characteristics of the kaolinite soil. The shear strength result showed a decrease in value with the introduction of the crude oil. This reduction in value is due to the fact that crude oil has a higher viscosity than water and also to the lubrication and slippage of the soil particle caused by the presence of the crude oil. The introduction of crude oil into the clay soil reduced the pore space of the soil which affected the infiltration capacity of the kaolinite clay soil.

Crude oil contamination makes the contaminated soil to appear aggregated but the contaminated soil at least to the level investigated in this research (i.e. up to 20% contamination) can be used in engineering and construction works. The contaminated soil can be used in road construction as subgrade material although there is need to stabilize the soil and cap it to avoid surface and ground water contamination. Also, structures within the vicinity of crude oil contaminated soils should be safeguarded to avoid increase in settlement of the foundation footings.

Soils with properties that cannot be safely and economically used for the design and construction of civil engineering structures are termed problem soils. The crude oil contamination did not make the kaolinite clay soil to behave like a problem soil.

Since this research was aimed at the effect of crude oil contamination on the geotechnical properties of kaolinite clay soil which is the dominant clay type in the low latitude climate (tropical climate and dry climate). The Niger Delta of Nigeria is within the low latitude climate. The shear strength of crude oil contaminated soil determined in this research if compared with that of the Niger Delta of Nigeria which has a frictional angle between 2° to 10°. It can be said
that the frictional angle of the crude oil contaminated soil up to the level investigated in this research is within the range of the kaolinite soil in the low latitude climate, even though the introduction of the crude oil reduced the shear strength of the kaolinite soil.

The crude oil contamination caused an increase in the index properties, coefficient of permeability and consolidation characteristics. It also caused a decrease in compaction and strength of the kaolinite clay soil. Crude oil contaminated kaolinite clay can be used as a construction soil. This will help in solving the problem of government regulation on excavation and dredging of construction soil and reduction of high landfill tax for disposal of contaminated soil.

7.7 Contribution to Knowledge

It is a known fact that crude oil spill/ contamination is an environmental problem world-wide in terms of damage to agricultural land, pollution of surface and ground water, damage and death to aquatic and marine lives. However, the findings of this research based on crude oil contamination of clay soil (up to the level investigated in this research suggests the following:

- This research has bridged the gap in knowledge on the geotechnical properties of light crude oil contaminated kaolinite clay. Kaolinite clay soil is the dominant clay soil of the low latitude climate which has the bulk of the world’s crude reserve and production.
- The pore fluid of kaolinite clay soil is sensitive to change. Introduction of crude oil in the pore space of the soil caused a change in the pore fluid chemistry which altered the geotechnical properties of kaolinite clay soil.
This will not however prevent its usage as construction material and does not make the clay soil to behave like a ‘problem soil’.

- Increase in the percentage of crude oil contamination caused kaolinite clay soil to appear aggregated.
- The crude oil contaminations caused an increase in permeability and liquid limit values, a decrease in shear strength values and compaction values. There is also an increase in coefficient of consolidation value with increase in the percentage of crude oil contamination. This trend in the results of the tested geotechnical properties in this research should be of great concern to the geotechnical engineer and the structures to be placed on the contaminated soil as there is the risk of settlement and contamination of surface and groundwater. So there is the need to remediate, stabilize or cap any crude oil contaminated soil before it use for any geotechnical application.
- The research has updated the knowledge and information about the use of light crude oil contaminated kaolinite clay soil with the regards to its geotechnical properties in the construction industry. The knowledge will be of great importance to the civil engineer when designing structures and foundation with regards to strength of the soil and risk of settlement.
- Findings from the research have been presented in conferences and seminar and have been published in conference proceedings. Papers are currently under review with the Canadian Geotechnical Journal, Electronic Journal of Geotechnical Engineering and Journal of Resources, Conservation and Recycling.
CHAPTER 8: RECOMMENDATION FOR FUTURE RESEARCH

8.1 Introduction

Due to the negative effect of crude oil exploration, exploitation, transport and management, large areas of land often become contaminated by crude oil; leading to modification of the soil chemistry which affect the geotechnical properties of clay soil. The research described in the previous chapters, has identified the following areas for further research on the effect of crude oil contamination on the geotechnical properties of clay soil to form the basis of future regulations regarding the handling of crude oil and to deal with crude oil contaminated soils.

8.2 Recommendation for Future Research

- The long term effect of the crude oil contamination on soil properties should be determined and compared with the present study. In the laboratory experiment, the tests were conducted after mixing the soil with crude oil and thus the tests are considered short term tests but in the field, contaminated soil may remain in place for months or even for years, so it is important to consider the aging effect on the soil material for subsequent geotechnical tests.

- Also future work should consider the effect of temperature on crude oil contaminated soil since crude oil exploration, transport and usage are conducted both in tropical and temperate zones of the world and crude oil spillage do happen in all the zones. Also there are different grades of oil crude oil which evaporates at different temperatures, so future research should consider different kinds of crude oil. Again the low latitude climate
zones have different climatic season e.g. rainy and dry seasons. These seasons have varying temperature which might affect the evaporation rate of the crude oil.

- Crude oil contamination migrates both in horizontal and vertical direction, so there should be a further research on the effect of crude oil contamination with regard to distance and direction of contamination.

- In this research, the lubricant or fluids within the soil were crude oil and water, future research should look at a situation where crude oil is the only lubricant, that is, without water.

- Again in this research, during the compaction test and index properties test, the amount of crude oil at any particular percentage of contamination was constant while the amount of water added to the test to reach optimum moisture content was increased, future research should look at a situation where the amount of water should be constant and that of crude oil varies.

- Future research should look on how to view the crude oil interaction with soil in coloured photograph e.g. using scanning electron micrographs (SEM).

- The clay type for this research is kaolinite, future research should examine the effect of crude oil contamination on other type of clay e.g. montmorillonite, illite etc. and also on a mixture of different types of soil.

- In this research, the emphasis was on the effect of crude oil contamination the geotechnical properties of soil, future research should also look at the cation exchange capacity of crude oil contaminated soil and the effect of the crude oil contamination on the chemical properties of the soil.
8.3 Remediation option for crude oil contaminated clay soil

The effectiveness and efficiency of remediation of crude oil contaminated soil depends on the type of crude oil, type of soil, weather condition, the depth of the crude oil penetration and the location of the contaminated site. There are two basic remediation treatment methods: in-situ and ex-situ treatment which may involve thermal treatment, biological treatment, chemical extraction and soil washing techniques.

In situ treatment methods

In this treatment method the contamination in the subsoil is not excavated or scraped, treatment is carried out in the subsoil (in situ) either by biological means such as oil degradation by microorganisms, or chemical-physical processes such as incineration, air sparging, and soil air suction extraction or through combinations of these processes. This process depends on the nature of contaminants.

In situ bioremediation – This method involves the degradation of the crude oil by micro-organisms and may be applied with the help of spreading units if the contamination occurs at the surface. This method also depends on the depth of the crude oil penetration since oxygen is required for the degradation of the crude oil. In situ bioremediation may through;

- Biostimulation – This is the process of adding nutrient, electron acceptor and oxygen to stimulate existing bacteria involve in bioremediation.

- Bioaugmentation - This is the addition of a group of indigenous microbial strains or genetically engineered microbes to treat the contaminated soil. It is
mostly used where native microorganisms do not have the metabolic capability to perform the remediation process.

**Air sparging** – This involves the volatilisation of the soil to extract the crude oil contaminant from the soil by mobilization of the volatile crude oil using hot air. Air sparging also accelerates the growth of aerobic bacteria in the contaminated area by oxygen feeding.

**Slurping** – This involves the extraction of the contaminated crude oil through a well by vacuum. This method is mostly applicable if the crude oil contamination occurs in the boundary area between groundwater-saturated and groundwater-unsaturated soils.

**In situ steam injection** - This is a thermal treatment method that can be applied to remove the volatile organic compound from contaminated soil. It involves injection of steam at high temperature and compressed air into the contaminated soil. The temperature of the injected steam is always higher than the boiling points of the volatile crude oil. The process enhances the conversion of the crude oil to gaseous or volatile phase. The air, vapour and the released hydrocarbon compounds are removed by extraction wells.

**Ex situ Treatment Methods**

In this treatment method the contaminated soil is removed to an off site remediation facility. This method is mostly applied if the amount of contaminated soil is small or if the crude oil contamination occurred at the surface in a location where an in-situ treatment is not possible. In ex-situ treatment method there is greater degree of remediation as compared to in-situ treatment due to the
controllability of many factors (moisture, temperature, salinity, pH). Ex situ treatment method could be through:

**Thermal method** – this method involves steam stripping and combustion.

**Chemical extraction methods** which involves the use of different solvents, where the contaminants are disassociated from the soil, dissolved or suspended in the solvents. The advantage of this method is its applicability for different types of crude oils and soils, but it is more suitable for soil with low clay content.

**Biological methods** – The ex situ bioremediation may be;

Land farming – This is a method in which contaminated soil is spread over a prepared bed along with some fertilizers and occasionally rotated.

Composting - This is a process of piling contaminated soil organic substances such as manure or agricultural wastes.

Soil washing is most applicable for crude oil contaminated coarse soil and not with soil with high clay content.

Bioremediation treatment method may be a good remediation option for crude oil contaminated kaolinite soil.
REFERENCES


272


Ola, S.A. 1987. Laboratory testing and geotechnical characterization of black cotton soil and expansive shales in Nigeria. 9th regional conference for Africa on soil mechanics and foundation engineering 1: Pp 991-995.


<http://www.uoguelph.ca/~terisatu/ogbodospill.pdf> [Accessed 18th March 2010].


BIBLIOGRAPHY


APPENDICES
Appendix 1

Details of Publications and Achievements


Ota, J. O. and Sarsby, R. W. Shear Strength and Drainage characteristics of a clay soil contaminated by crude oil used as a construction material, Electronic Journal of Geotechnical Engineering, In press.


Conference Presentations and Seminars


of Engineering, Built and Environment, University of Wolverhampton, 15th October, 2008.

24 - 04 - 2009

The Head,
Department of Construction and Infrastructure
School of Engineering and Built Environment
University of Wolverhampton, Wolverhampton
United Kingdom.

TESTIMONIAL
I wish to acknowledge that your student, Mr. Jonathan Ota attended the International Conference on Global Food Crisis organized in our University.

He presented a paper titled, Effect of Hydrocarbon Contamination of Land on soil Structure of Agricultural Land which was well received because of its relevance to the hydrocarbon region of Nigeria.

His paper has also been published in our pre-conference Book of proceedings.

I hope that the knowledge gained by Mr Ota during this conference has given him a clearer view of the challenges of agricultural production and possible solutions to the global food crisis.

Prof. Chigozie C. Asiabaka
Dean, School of Agriculture and Agricultural Technology
Chairman, Conference Organising Committee.
16th June 2008

Dear Jonathan Ota,

GREEN5 International Conference
1st to 4th July 2008
Vilnius, Lithuania

We are very pleased that you will be able to attend the GREEN5 conference in Vilnius, Lithuania. The GREEN5 conference runs from Tuesday 1st July to Friday 4th July, inclusive, and as a registered delegate you will be able to attend all technical sessions and social events. You are requested to contribute your knowledge to the discussion on the effects of oil spills on the environment, and on the ground in particular, during the course of the conference.

It gives us great pleasure to invite you to attend GREEN5 and we look forward to seeing you in Vilnius.

Yours sincerely,

(Dr B Jankauskas)
(On behalf of GREEN5 Organising Committee)
APPENDIX: 2

Health and Safety of Test Material

A. Clay Soil

SAFETY DATA SHEET – PREPARED CLAY BODIES

COMPOSITION/INFORMATION ON THE COMPONENTS

Prepared Clay Body is a mixture of natural Minerals and may contain ball clay, china clay, ground quartz, ground cristobalite and fluxes (naturally occurring felspathic material or nepheline syenite). Small amounts of calcined alumina bearing minerals may also be added.

HAZARD IDENTIFICATION

Main Hazards - Prepared clay body contains free silica. If allowed to dry out dust containing respirable silica may be liberated. Danger of serious damage to health by prolonged exposure.

Health effects (Eyes) - Dust will cause transient irritation by abrasion.
Health Effects (Skin) - Dust may cause irritation by abrasion.
Health Effects (Ingestion) - Gastrointestinal irritation.
Health Effects (Inhalation) - Exposure to dust may have the following effects: severe irritation to nose, throat and respiratory tract and possibly lung damage due to respirable Silica.

FIRST AID MEASURES

Eyes - Immediately flush the eye with plenty of water, for at least 10 minutes, holding the eye open. Obtain medical attention if soreness or redness persists.
Skin - Wash skin thoroughly with soap and water. Obtain medical attention if blistering occurs or redness persists.
Ingestion - Wash out mouth with water. Do not induce vomiting. Obtain medical attention.
Inhalation - Remove from exposure, keep warm and rest. If there is difficulty in breathing, give oxygen. Obtain medical attention.
Advice to Physicians - Treat symptomatically.

FIRE FIGHTING MEASURES

Extinguishing Media
Not combustible. Select extinguishing agent appropriate to other materials involved.

Special Hazards of Products
Avoid the formation of dust clouds.

ACCIDENTAL RELEASE MEASURES

Spillages
Avoid creating a dust. Sweep, preferably vacuum up, and collect in suitable containers for recovery or disposal.

Handling and Storage
Store in sealed packaging. Avoid creating dust.

EXPOSURE CONTROLS/PERSONAL PROTECTION

Occupational Exposure Standards - Contains cristalline silica. UK EH40 OES 0.4mg/m3 TWA. As SiO2 dust, respirable.
Engineering Control Measures - Use of the basic principles of Industrial Hygiene will enable this material to be used safely.
Respiratory Protection - Dust respirator if conditions are dusty.
Hand Protection - Only required for those with sensitive skin.
Eye Protection - Provide eyewash.
Body Protection - Normal work wear. Overall or apron.
Protection During Application - In all cases of protective equipment the user must ensure that the equipment complies with the relevant standard. If there is any doubt the user should show this data sheet to the supplier of the equipment to ensure the correct equipment is available.

PHYSICAL AND CHEMICAL PROPERTIES

Physical State - Prepared clay body has a soft and pliable consistency and contains 18-20% water. It may also be sold as a granulate of much lower moisture content (3%) or as a water suspension also known as casting slip.
Colour - Normally brown, grey or white. Vegetable stain (green) may be introduced for identification purposes.
Odour - None.
Solubility in Water - Negligible.
Specific Gravity 2.6
B. Crude Oil

SAFETY DATA SHEET
Crude oil

1. IDENTIFICATION OF THE SUBSTANCE/PREPARATION AND OF THE COMPANY/UNDERTAKING

Identification of substance/preparation
Crude oil
Synonyms

Application
Refinery feedstock

Company Identification
Petroplus Refinery & Marketing Limited (PRML)
Coryton Refinery, The Manorway, Stanford Le Hope, Essex, SS17 9LL

Emergency Telephone Number
+44 (0) 1375 673310

Last Updated
10/06/2007

2. COMPOSITION/INFORMATION ON INGREDIENTS

Chemical Composition
Complex hydrocarbon mixture comprising mainly of aliphatic, naphthenic and aromatic hydrocarbons. It may also contain gases, sulphur and nitrogen compounds.
CAS-No. 8002-06-9
EINECS No.: 232-298-5

Hazardous Components
Polycyclic aromatic hydrocarbons will be present, some of which have been shown by experimental studies to induce skin cancer.

<table>
<thead>
<tr>
<th>Component</th>
<th>CAS No</th>
<th>Exposure Limit</th>
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</thead>
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<tr>
<td>Benzene</td>
<td>71-43-2</td>
<td>1 ppm (8 hour)</td>
</tr>
<tr>
<td>Hydrogen sulphide</td>
<td>7783-06-4</td>
<td>10 ppm (15 min) 14 mg/m3 (15 min) 5 ppm (6 hour) 7 mg/m3 (8 hour)</td>
</tr>
</tbody>
</table>

3. HAZARDS IDENTIFICATION

Extremely flammable.
May cause cancer, classified as a category 2 carcinogen.
Contains Benzene. Prolonged or repeated exposure to benzene can cause anaemia and other blood diseases, including leukaemia.
This material may contain significant quantities of polycyclic aromatic hydrocarbons (PCAs), some of which have been shown by experimental studies to induce skin cancer.
Vapours containing hydrogen sulphide may accumulate during storage or transport and may also be vented during filling of tanks. Hydrogen sulphide has a typical bad egg smell but at high concentrations the sense of smell is rapidly suppressed.


Sheet No: P00001  Material Name: Crude oil
instruments for determining its concentration.
Harmful if swallowed - aspiration hazard (If viscosity<7 cSt at 40°C)
Harmful to aquatic organisms, may cause long-term adverse effects in the aquatic environment.

4. FIRST-AID MEASURES

Eyes
Wash eye thoroughly with copious quantities of water, ensuring eyelids are held open. Obtain medical advice if any pain or redness develops or persist.

Skin
Wash skin thoroughly with soap and water as soon as is reasonably practicable. Remove heavily contaminated clothing and wash underlying skin.
Product can be removed from the skin using warmed medicinal paraffin. Never use gasoline or kerosine to remove product from skin or clothing.
In extreme situations of saturation with this product, drench with water, remove clothing as soon as possible and wash skin with soap and water. Seek medical advice if skin becomes red, swollen or painful.

Ingestion
If contamination of the mouth occurs, wash out thoroughly with water.
Except as a deliberate act, the ingestion of large amounts of product is unlikely. If it should occur, do not induce vomiting; obtain medical advice.

Inhalation
If exposure to vapour, mists or fumes causes drowsiness, headache, blurred vision or irritation of the eyes, nose or throat, remove immediately to fresh air. Keep patient warm and at rest. If any symptoms persist obtain medical advice. Unconsciousness casualties must be placed in the recovery position. Monitor breathing and pulse rate and if breathing has failed, or is deemed inadequate, respiration must be assisted, preferably by the mouth to mouth method. Administer external cardiac massage if necessary. Seek medical attention immediately.

EXPOSURE TO HYDROGEN SULPHIDE:
Casualties suffering ill effects as a result of exposure to hydrogen sulphide should be immediately removed to fresh air and medical assistance obtained without delay.
It is advisable that all who are engaged in operations in which contact with H2S may reasonably be anticipated, should be trained in the techniques of emergency resuscitation and in the care of an unconscious patient.

Medical Advice
Inhalation of hydrogen sulphide may cause central respiratory depression leading to coma and death. It is irritant to the respiratory tract causing chemical pneumonitis and pulmonary oedema. The onset of pulmonary oedema may be delayed for 24 to 48 hours. Treat with oxygen and ventilate as appropriate. Administer broncho-dilators if indicated and consider administration of corticosteroids. Keep casualty under surveillance for 48 hours in case pulmonary oedema develops.
If ingested, do not induce vomiting. Drinking milk or medicinal paraffin may be beneficial. Aspiration of the product is unlikely to occur except as the result of ingestion, followed by vomiting or regurgitation in a partially or totally unconscious individual, when immediate effects are most likely to result from the aspiration of acidic stomach contents. If it should occur, transport casualty immediately to hospital.

5. FIRE-FIGHTING MEASURES

Extinguishing Media
Foam, water fog, dry powder. Do not use water jets.

For major fires call the Fire Service. Ensure an escape path is always available from any fire. There is a danger of flashback if sparks or hot surfaces ignite vapour. FIRES IN CONFINED SPACES SHOULD BE DEALT WITH BY TRAINED PERSONNEL WEARING APPROVED BREATHING APPARATUS. Any spillage should be regarded as a potential fire risk. Combustion Products: Toxic fumes may be evolved on burning or exposure to heat.

6. ACCIDENTAL RELEASE MEASURES

As this product has a very low flash point any spillage or leak is a severe fire and/or explosion hazard. Isolate spillage from all ignition sources including road traffic. Ensure good ventilation. Evacuate all non essential personnel from immediate area. Wear protective clothing. Spilled material may make surface slippery. Recovery of large spillages should be effected by specialist personnel. It is advised that stocks of suitable absorbent material should be held in quantities sufficient to deal with any spillage which may be reasonably anticipated. Large and uncontained spillages should be smothered with foam to reduce the risk of ignition. The foam blanket should be maintained until the area is declared safe. Protect drains from potential spills to minimise contamination. Do not wash product into drainage system. Vapour is heavier than air and may travel to remote sources of ignition (eg. along drainage systems, in basements etc.). If spillage has occurred in a confined space, ensure adequate ventilation and check that a safe, breathable atmosphere is present before entry. In the case of spillage on water, prevent the spread of product by the use of suitable barrier equipment. Recover product from the surface. Protect environmentally sensitive areas and water supplies. Any spillage which results in pollution at sea must be treated in accordance with the guidelines in MARPOL 73/78 Annex 1. In the event of spillages contact the appropriate authorities. If spillage occurs call the Environment Agency Emergency Hotline on 0800 807050 (24 hours a day, 7 days a week).

7. HANDLING AND STORAGE

Storage Conditions

Do not enter storage tanks without breathing apparatus unless the tank has been well ventilated and the tank atmosphere has been shown to contain hydrocarbon vapour concentrations of less than 1% of the lower flammability limit and an oxygen concentration of at least 20% volume. Always have sufficient people standing by outside the tank with appropriate breathing apparatus and equipment to effect a quick rescue. Confined spaces contaminated with hydrogen sulphide must always be considered as constituting potentially life threatening environments. Entry into such spaces must never be undertaken except under extreme emergency when no alternative is possible and then only by trained operators wearing air-supplied breathing apparatus of an approved type and following procedures strictly in accordance with the Statutory Regulations governing such entry.

Handling Precautions

Ensure good ventilation and avoid as far as reasonably practicable the inhalation and contact with vapours, mists or fumes which may be generated during use. If such vapour, mists or fumes are generated, their concentration in the workplace air should be controlled to the lowest reasonably practicable level.

Avoid contact with skin and observe good personal hygiene.

Avoid contact with eyes. If splashing is likely to occur wear a full face visor or chemical goggles as appropriate.

Do not siphon product by mouth.

Whilst using do not eat, drink or smoke.

Fire Prevention

Light hydrocarbon vapours can build up in the headspace of tanks. These can cause flammability/ explosion hazards even at temperatures below the normal flash point (note: flashpoint must not be regarded as a reliable indicator of the potential flammability of vapour in tank headspaces). Tank headspaces should always be regarded as potentially flammable and care should be taken to avoid static electrical discharge and all ignition sources during filling, ullaging and sampling from storage tanks.

When the product is pumped (e.g. during filling, discharge or ullaging) and when sampling, there is a risk of static discharge. Ensure equipment used is properly earthed or bonded to the tank structure.

Explosive air/vapour mixtures may form at ambient temperature.

Product contaminated rags, paper or material used to absorb spillages, represent a fire hazard, and should not be allowed to accumulate. Dispose of safely immediately after use.

Empty containers represent a fire hazard as they may contain flammable product product residues and vapour. Never cut, weld, solder or braze empty containers.

8. EXPOSURE CONTROLS/PERSONAL PROTECTION

Exposure Controls

Personal Protective Equipment

Wear face visor or goggles in circumstances where eye contact can accidentally occur.

If skin contact is likely, wear impervious protective clothing and/or gloves.

Protective clothing should be regularly inspected and maintained; Overalls should be dry-cleaned, laundered and preferably starched after use.

Sheet No:  P00001

Material Name: Crude oil

Respiratory Protective Equipment
If operations are such that the excessive generation of vapour, mist or fume may be anticipated, to which operators may unavoidably be exposed, then suitable approved respiratory equipment should be worn. Note: Approved air-supplied breathing apparatus must be worn where there may be potential for inhalation of hydrogen sulphide gas. The use of respiratory equipment must be strictly in accordance with the manufacturers instructions and any statutory requirements governing its selection and use.

9. PHYSICAL AND CHEMICAL PROPERTIES

Appearance and Odour
Brown liquid
Pungent

Flash Point
-50 to 100 °C (PMC)

Explosive LEL
0.6%

Explosive UEL
8.0%

Properties
Density at 15°C using test method ASTM D1298
750-1000 kg/m³
Kinematic Viscosity at 40°C using test method ASTM D445
≤7 mm²/s - viscous liquid
Boiling point/range using test method ASTM D86
-10 - 800°C
Vapour pressure using test method ASTM D323
40 - 700 kPa

10. STABILITY AND REACTIVITY

Stable at ambient temperatures. Hazardous polymerisation reactions will not occur.

Conditions to Avoid
Sources of ignition.

Materials to Avoid
Avoid contact with strong oxidizing agents.

Hazardous Decomposition Products
Thermal decomposition products will vary with conditions. Incomplete combustion will generate smoke, carbon dioxide and hazardous gases, including carbon monoxide.

11. TOXICOLOGICAL INFORMATION

Eyes
Unlikely to cause more than transient stinging or redness if accidental eye contact occurs.

Skin
Unlikely to cause harm to the skin on brief or occasional contact but prolonged or repeated exposure may lead to dermatitis. As with all such products containing potentially harmful levels of PCBs, prolonged or repeated skin contact may eventually result in dermatitis or more serious irreversible skin disorders including cancer.

Ingestion

Unlikely to cause harm if accidentally swallowed in small doses, though larger quantities may cause nausea and diarrhoea. Lower viscosity crude oils may injure the lungs if aspiration occurs, e.g., during vomiting.

Inhalation
Likely to be irritating to the respiratory tract if high concentrations of mist or vapour are inhaled. May cause nausea, dizziness, headaches and drowsiness if high concentrations of vapour are inhaled. May be toxic by inhalation when hydrogen sulphide is present in the vapour.

ABUSE: Under normal conditions of use the product is not hazardous; however, abuse involving deliberate inhalation of very high concentrations of vapour, even for short periods, can produce unconsciousness and/or result in a sudden fatality.

Carcinogenicity/Chronic Toxicity
Exposure to benzene may result in affects to the hematopoietic system causing blood disorders including anaemia and leukaemia. Benzene is classified by EEC as a category 1 carcinogen - substances known to be carcinogenic to man. IARC assessment: benzene - carcinogenic to humans (Group 1).

12. ECOLOGICAL INFORMATION

Mobility
Spillages may penetrate the soil causing ground water contamination.

Persistence and degradability
This product is inherently biodegradable.

Bioaccumulative potential
There is no evidence to suggest bioaccumulation will occur.

Ecological information
Harmful to aquatic organisms. Spills may form a film on water surfaces causing physical damage to organisms. Oxygen transfer could also be impaired. May cause long-term adverse effects in the aquatic environment.

13. DISPOSAL CONSIDERATIONS
Dispose of via an authorised person/licensed waste disposal contractor in accordance with local regulations.

14. TRANSPORT INFORMATION

**NOTE** Transport regulations depend on flashpoint, initial boiling point and vapour pressure.

Flash point (closed cup) < 23°C, initial boiling point <= to 35°C, vapour pressure at 50°C >110kPa

ADR/RID: Petroleum Crude Oil, UN No. 1267, Flammable liquids, Class 3, Packing Group I, Classification code F1, Hazard Identification No. 33

UN: Petroleum Crude Oil, UN No. 1267, Flammable liquids, Class 3, Packing Group I

IATA/ICAO: Petroleum Crude Oil, UN No. 1267, Flammable liquids, Class 3, Packing Group I

IMO: Petroleum Crude Oil, UN No. 1267, Flammable liquids, Class 3, Packing Group I

Emergency action code: Flammable Liquid, 3WE

Flash point (closed cup) < 23°C, initial boiling point <= to 35°C, vapour pressure at 50°C <175kPa

ADR/RID: Petroleum Crude Oil, UN No. 1267, Flammable liquids, Class 3, Packing Group II, Classification code F1, Hazard Identification No. 33

UN: Petroleum Crude Oil, UN No. 1267, Flammable liquids, Class 3, Packing Group II

IATA/ICAO: Petroleum Crude Oil, UN No. 1267, Flammable liquids, Class 3, Packing Group II

Sheet No: P00001

Material Name: Crude oil

IMO: Petroleum Crude Oil, UN No. 1267, Flammable liquids, Class 3, Packing Group II
Emergency action code: Flammable Liquid, 3WE
Flash point (closed cup) => 23°C but <=61°C
ADR/RID: Petroleum Crude Oil, UN No. 1267, Flammable liquids, Class 3, Packing Group III, Classification code F1,
Hazard Identification No. 30
UN: Petroleum Crude Oil, UN No. 1267, Flammable liquids, Class 3, Packing Group III
IATA/ICAO: Petroleum Crude Oil, UN No. 1267, Flammable liquids, Class 3, Packing Group III
IMO: Petroleum Crude Oil, UN No. 1267, Flammable liquids, Class 3, Packing Group III
Emergency action code: Flammable Liquid, 3W
Flash Point (closed-cup) > 61°C
Not classified as hazardous for transport (ADR, RID, UN, IMO, IATA/ICAO)

15. REGULATORY INFORMATION

EU Category of Danger
Extremely flammable or Highly Flammable or Flammable or unclassified for flammability (dependant on flash point and initial boiling point).
Carcinogenic category 2
Harmful (if viscosity<7cSt at 40°C)
Dangerous for the environment

EU Labelling
The label must carry the following additional information: “EC Label”, Substance name, EINECS No.
Substance Name: Crude Oil, EINECS No. 233-298-5
For non-fuel use only - “Restricted to professional users. Attention - Avoid exposure - obtain special instructions before use.” must be marked on packaging.

Risk Phrases
R12; R45; R65; R52/53

Safety Phrases
S53; S45; S81

<table>
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<tr>
<th>R/S Code</th>
<th>Description</th>
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<tbody>
<tr>
<td>R12</td>
<td>Extremely flammable, or R11 Highly flammable, or R10 Flammable, or no risk for flammability (dependant on flash point and initial boiling point)</td>
</tr>
<tr>
<td>R45</td>
<td>May cause cancer</td>
</tr>
<tr>
<td>R52/53</td>
<td>Harmful to aquatic organisms, may cause long-term adverse effects in the aquatic environment</td>
</tr>
<tr>
<td>R65</td>
<td>Harmful: may cause lung damage if swallowed. (If viscosity&lt;7 cSt at 40°C)</td>
</tr>
<tr>
<td>S45</td>
<td>In case of accident or if you feel unwell, seek medical advice immediately (show the label where possible)</td>
</tr>
</tbody>
</table>

16. OTHER INFORMATION

Sheet revisions
Date Sections
20/12/2002 8,14

Issue date: 20/12/2002
Revision of sheet dated: 03/07/1998

All reasonably practicable steps have been taken to ensure this data sheet and the health, safety and environmental information contained in it is accurate as of the date specified below. No warranty or representation, express or implied is made as to the accuracy or completeness of the data and information in this data sheet.

The data and advice given apply when the product is sold for the stated applications. You should not use the product other than for the stated applications without seeking advice from us.

It is the user's obligation to evaluate and use this product safely and to comply with all applicable laws and regulations. Petroplus shall not be responsible for any damage or injury resulting from use, other than the stated product use of the material, from any failure to adhere to recommendations, or from any hazards inherent in the nature of the material. Purchaser of the product for supply to a third party for use at work, have a duty to take all necessary steps to ensure that any person handling or using the product is provided with the information in this sheet. Employers have a duty to tell employees and others who may be affected of any hazards described in this sheet and of any precautions that should be taken.
## APPENDIX 3
### XRD and XRF Results

#### A. XRD of Clay Soil

**MATCH SCORE TABLE**

<table>
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<tr>
<th>Listed MS file name</th>
<th>OTA01.MS</th>
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<tr>
<td>Original data file name</td>
<td>OTA01.DI</td>
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<tr>
<td>Step size, sample time</td>
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<td>Analysis program number</td>
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<td>Peak angle range</td>
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<tr>
<td>Range in D spacing</td>
<td>1.54060 - 17.6596 Ang</td>
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<tr>
<td>Maximum intensity</td>
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<tr>
<td>Type of search</td>
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<tr>
<td>Knowns</td>
<td>46-1045 6-221 29-1486</td>
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<tr>
<td>Reference data base</td>
<td>1996 PDF APDINO:</td>
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<tr>
<td>Number of Alphal peaks</td>
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<td>Background threshold</td>
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<tr>
<td>Number of ref patterns</td>
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<tr>
<td>Nr of patterns matched</td>
<td>126</td>
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<tr>
<td>To continue give RETURN</td>
<td></td>
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<tr>
<td>Nr of phases identified</td>
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<th>I%</th>
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<td>90</td>
<td>21</td>
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B. XRF of Clay Soil

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APPENDIX: 4

Properties and Source of Clay Soil

TERRACOTTA CLAYS

STANDARD RED (Smooth - Medium)
An extremely popular general purpose clay based on a blend of Etruria Marls. Suitable for throwing, handling & casting. *Highly recommended
Firing: 1080°C - 1160°C (Light red becoming darker as temperature is increased)

STANDARD RED GROGGED (Medium)
The addition of grogg in this body improves strength in throwing and increases resistance to cracking and warping especially on larger pieces. Suitable for throwing and modelling.
Firing: 1080°C - 1160°C (Light red becoming darker as temperature is increased)

TERRACOTTA FINE 120'S (Smooth)
A general blend of Etruria Marls produced to a much finer degree giving a pleasing smooth finish. Suitable for machining and throwing.
Firing: 1080°C - 1160°C (Light red becoming darker as temperature is increased)

V636 (Smooth)
A very well behaved clay resulting from a blend of Etruria Marls and Shropshire Fire Clays. Good plasticity, suitable for throwing and modelling.
Firing: 1020°C - 1160°C (Medium red becoming darker as temperature is increased)

RBC 60s (Medium to Smooth)
This body contains fine graded China Clay and Etruria Marls. Developed to improve casting. Suitable for all types of production or studio work.
Firing: 1040°C - 1160°C (Pale red becoming darker as temperature is increased)

SCULPTURE (Medium to Coarse)
A newly developed clay containing a good degree of medium to coarse grogg. Gives open texture yet retaining a good degree of plasticity. Has good resistance to thermal shock. Suitable for large sculptural work, garden ware, slab work etc.
Firing: 1020°C - 1220°C (Light red becoming darker as temperature is increased)

MOULDING CLAY (Very Coarse)
A traditional coarse, pan milled and pug milled North Yorkshire terracotta clay. Highly plastic, suitable for moulding and sculpture work.
Firing: 1020°C - 1100°C (Mid red becoming darker as temperature is increased)

STONeware CLAYS

V9A (Smooth to Medium)
An excellent general purpose modelling clay, which contains Shropshire & South Staffordshire Fire Clays with a fine graded dust free grogg. Suitable for throwing and hand modelling. *Highly recommended
Firing: 1180°C - 1280°C (Buff to grey)

V9G (Medium)
The addition of grogg gives good resistance to cracking and warping. Behaves well under oxidising and reduction conditions.
Firing: 1180°C - 1280°C (Grey buff - speckled grey)

HT (Smooth to Medium)
This stoneware body is especially recommended for reduction and contains a fine grogg free of dust faction.
Firing: 1120°C - 1280°C - (Buff to grey)

Sam M

100 kg needed
### APPENDIX 5
Properties and Source of Crude Oil

![CORYTON LIMS RESULTS - CRUDE_IMP](image)

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**VESSEL**
- **Crude Name**: Sarpen
- **Sample Name**: Forties Buzzard 1074872
- **Sample Number**: 0.060 PPM

**TECHCOM**
- **Free Text**: Sampled by SGS line sample

**Sampling Procedure**

---

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APPENDIX 6

The calculation of moisture content for crude oil contaminated soil

Due to the fact that crude oil will evaporate in the oven during testing, the moisture content of all crude oil contaminated soil samples was calculated based on equation

\[ w\% = (1 - mn) \frac{W_t}{W_d} - (1 + n) \]

Equation 1

Where \( W_t \) = weight of wet contaminated soil

\( W_d \) = weight of dry contaminated soil

\( m \) = crude oil residual after drying

\( n \) = oil content before drying

Rate of evaporation

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<th>M2</th>
<th>M3</th>
<th>M2 - M3</th>
<th>E</th>
</tr>
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<td>86.48</td>
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<tr>
<td>8</td>
<td>3.2</td>
<td>46.32</td>
<td>89.52</td>
<td>87.92</td>
<td>1.6</td>
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<td>4</td>
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<tr>
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<td>94.22</td>
<td>92.22</td>
<td>2</td>
<td>25</td>
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</tr>
</tbody>
</table>

% Cont. = percentage of crude oil contamination

A = Amount of crude oil added per 40g of soil sample measured by the weight of dry soil

M1 = Mass of tin

M2 = Mass of tin + soil + crude oil before drying

M3 = mass of tin + soil + crude oil after drying

M2 - M3 = Quantity of crude oil that evaporated

E = Percentage of evaporated crude oil
i.e. $E = \left(\frac{M_2 - M_3}{A}\right) \times 100$ ........................................Equation 2

A graph of $E$ was plotted against percentage of contamination to determine the rate of evaporation.

![Evaporation Graph](image)

Figure 1 percentage of evaporation with percentage of contamination

Example:

If for 10% crude oil contamination in 3000g of soil sample equals 300ml of crude oil

i.e. $\frac{10}{100} \times 3000 = 300$ml

Then 50g of soil contaminated by 10% crude oil contamination has 5ml of crude oil

i.e. $\frac{10}{100} \times 50 = 5$ml
And based on the rate of evaporation in the above figure 1, if in 10% contamination, 40% of the crude oil evaporated that means that in 5ml of crude oil 2ml will evaporate i.e. \( \frac{40}{100} \times 5 = 2ml \)

So using equation 1, 

\[
\begin{align*}
\text{w\%} &= (1 - mn) \frac{W_t}{W_d} - (1 + n)
\end{align*}
\]

Where \( n = 5\text{ml} \), \( m = (5 - 2) = 3\text{ml} \)