SOIL CLASSIFICATION AND CONCENTRATION OF SELECTED HEAVY METALS IN SOME SOILS OF THE NIGER DELTA REGION, NIGERIA

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A THESIS SUBMITTED TO THE POSTGRADUATE SCHOOL, FEDERAL UNIVERSITY OF TECHNOLOGY, OWERRI

IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE AWARD OF THE DEGREE OF MASTERS OF SCIENCE (M. Sc.) IN SOIL SCIENCE

JUNE, 2023

CERTIFICATION

This is to certify that this work **"Soil Classification and Concentration of Selected Heavy Metals in some Soils of the Niger Delta Region, Nigeria"** was carried out by I Sule Benjamin Agbene (20124760468) in partial fulfillment for the award of (M.Sc in Soil Survey and Landuse in the Department of Soil Science and Technology) of the Federal University of Technology, Owerri

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DEDICATION

This work is dedicated to the Most Holy Trinity; The source and summit of my life and capabilities, my Mum, Siblings and all lovers of research.

Also in loving memories of my beloved Dad; Hon SULE Nicodemus Yahaya

ACKNOWLEDGEMENTS

To the Almighty God and the Most Blessed Trinity, I owe you all.

How can I pay all my debt on this journey, If not for You Lord.

My untiring project supervisor, Prof Onweremadu E. U, who became a Father and a friend, your sympathy and empathy in my trying moments really gave me a push. I pray you will find available help at your cross roads.

My project co-supervisor, and Head of Department, Dr. Ahukaemere C. M. I know you understand my joy and gratitude better. Your inputs are much appreciated.

Dr. I E Ekpe, for your immense contributions in diverse ways, I remain indebted to you.

My Lecturers, too numerous to mention, you have raised a son; your legacies live.

My Dad, Hon Sule, Nicodemus Yahaya of blessed memories; This was a journey you encouraged me to start and you put in your best, but God's ways are not ours, you could not wait to see me finish. Continue to rest on Daddy.

Mrs Cecilia Sule; my mum and pillar; I lack words to express myself as I write this but Mum, I want you to know that I do appreciate all your struggles and sacrifices. Your prayers were not in vain.

Simon, Pius, Silas and Emma, faithful brothers-turned-friends, you were really a motivation when I was down.

Ocholi Don Michael Akpa, Godwin Ohiri, Reginald Okereke, Vera Aliba, Ogechi, Amara, Nnaemeka, Uche Egbochue, Linda, Patience, Joy, Yemi and Lilian. I love you all

My numerous Uncles, Aunties, Nephews and nieces; I appreciate you all.

Everyone who took out time to make this work a success. God will reward you.

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ABSTRACT

Some physicochemical characteristics and heavy metal levels in selected spilled and unspilled soils of the Niger Delta region, Nigeria were analysed. A total of three locations were sampled and two profile pits (spilled and unspilled) were dug at each location making a total of six (6) profile pits. Samples were collected from genetic horizons starting from the lowest horizon in each pedon. Conventional analytical methods were employed for the determination of these physico-chemical properties and heavy metals. Data gotten from the laboratory analysis were subjected to simple correlation, coefficient of variability and t-test. Results showed that the consistence of the soils was very friable(Egbem-spilled and unspilled, Oguta unspilled), friable(Oguta spilled and Ugwunagbo spilled) and loose in the epipedons and friable in all subsurface horizon, while the soil structure ranged from very weak fine granular to medium subangular blocky in spilled pedons whereas unspilled pedons were predominantly very fine single grained. There were generally fine and medium roots in the studied soils. The soils were basically Sandy Loam (SL) and Sandy Clay Loam (SCL). The value of sand ranged from 740 - 810 g/Kg. Sand and Silt fractions decreased down the pedons. Low clay characterized the epipedons. The soils were well drained and all soils were deep (> 180cm). Bulk density increased with depth for all pedons and ranged from 1.17 – 1.56 gcm^{-3}. Spilled pedons had higher bulk densities (1.37 gcm^{-3}, 1.37 gcm^{-3} and 1.40 gcm^{-3}) than the unspilled pedons (1.33 gcm^{-3}, 1.30 gcm^{-3} and 1.33 gcm^{-3}).Slight variations (CV < 15%) were recorded for bulk density in all pedons. Moisture content was lower in spilled sites, it showed slight variations (CV<15%) at Ugwunagbo unspilled site and Oguta spilled site, moderate variation in Oguta unspilled and high variation in Egbema (spilled and unspilled) and Ugwunagbo spilled. The pH values in all sites ranged from 4.8 – 6.3 indicating that the studied soils were acidic. The spilled pedons in Egbema and Ugwunagbo were more acidic than their unspilled pedons. But the reverse was observed in Oguta sites where the spilled site had a higher pH than the unspilled pedon. Generally, pH in KCl was less than in water. The pH showed slight variability (CV<15%). There was a significant difference between spilled and unspilled pedons. Generally, total nitrogen was highly variable (49 – 92 %), it was higher in spilled pedons of Egbema and Oguta but the reverse was the case in Ugwunagbo. It decrease down the pedons. Available Phosphorus values ranged from low (<5mg/kg) to high (>15mg/Kg). It was highly variable (CV>35%) in most pedons. Available Phosphorus was higher in unspilled than spilled soils. Organic Carbon generally decreased with depth. Values of Calcium and Magnesium were higher in unspilled than in spilled soils. Sodium values were generally low and varied for different pedons. Exchangeable Potassium had higher values in unspilled than spilled soils. Ca:Mg ratio was below 3 in (Egbema-spilled and unspilled, Oguta unspilled and Ugwunagbo spilled and unspilled) indicating unfertile soils. Higher values were recorded for ECEC in unspilled soils than in spilled soils. Values of Base saturation were higher in unspilled than spilled soils and showed a significant difference between spilled and unspilled pedons. Aluminum saturation values were low in the studied soils. Heavy metal values decreased down the pedon and were generally higher in spilled than unspilled soils, but they were within the permissible limit for use. Generally, the magnitude of heavy metals followed Ni > Pb > Cd > Cr for spilled soils and Ni>Pb>Cr>Cd for unspilled soils. Though the detected levels of heavy metals in the studied soil were within the permissible limits, caution should be taken to avoid accumulation and toxicity.

The soils of Ugwunagbo and Egbema were classified as Typic paleudult - Dystric Nitisol (FAO/WRB). While those of Oguta were classified as Typic tropudult - Orthic Acrisol (FAO/WRB).

Keywords: Soil Pollution, Remediation, soil classification, heavy metals, soil physico-chemical properties

CHAPTER I

1.0 Introduction

1.1 Background Information

Soil is a natural body, differentiated into horizons of minerals and organic constituents, usually unconsolidated, of variable depth, which differs from the parent material below in morphology, physical properties and constitution, chemical properties and composition and biological characteristics (Rai, 2002).

Different soils have varying potential to support different land use types. The productivity of a soil depends largely on its Physico-chemical properties which are as a result of the interaction among the soil forming factors during soil formation. Soil formation is a constructive as well as destructive process. Constructive forces develop new chemical compounds, both mineral and organic and provide new distribution or association characteristics, structural properties as well as chemical compositions. Destructive process predominates the physical and chemical breaking down of materials, plants and animal structures, which result in the partial loss of more soluble and volatile product. These factors influence the plant growth in the soil. Where the relative influence of these factors differs, it will give rise to different kinds of soils with different productivity potential.

The mineral composition of soils, the organic matter within it and the environment, all determine the soil's chemical properties. Soils and sediments contain a large variety of organic materials ranging from simple sugars and carbohydrates to the more complex proteins, fats, waxes, and organic acids. Organic matter is characterized by its ability to form complexes, interact with clay minerals, bind particles together and hold water in the soil environment (Brian 2002). As a result of these characteristics, the determination of total organic carbon is an essential part of any site characterization since its presence or absence can influence the reactivity of chemicals in the soil or sediment. Also, the yield of crop depends on fertility and presence of micronutrients and heavy metals in the soil. Therefore in order to derive the maximum benefit from a soil, it is necessary to know its physical and chemical properties. The soil condition is of great importance, because it is a universal medium for plant growth, which supplies essential nutrients to the plants (Pujar *et al.*, 2012).

Soil information gathered by systematic identification, grouping and delineation of different soils is required when sound interpretations towards land use potential are to be made. In addition, climatic and other ecological characteristics as well as socio-economic factors are also important elements in land management. (Msanya *et al.*, 2004)

Pedological characterization provides vital information and knowledge on soil characteristics and gives a clear understanding on soil genesis, morphology, classification and spatial distribution of soils in an area (Msanya et al., 2016; Kalala et al, 2017; Mbaga et al., 2017; Mukungurutse et al., 2018) . Soil characterization data helps in the correct classification of the soil to serve as a basis for a more detailed evaluation of the soil as well as gather preliminary information on nutrient, physical or other limitations needed to produce a capability class for crop production (Msanya et al., 2016; Mukungurutse et al., 2018).

Soil classification is the systematic arrangement of soils into groups or categories based on their characteristics (Karuma et al, 2005; Msanya et al., 2003). Two internationally known soil classification systems have been used to classify soils namely the United States

Department of Agriculture (USDA) Soil Taxonomy and World Reference Base for Soil Resources (WRB). The main purpose of any classification is to establish groups or classes of soils under study in a manner useful for practical and applied purposes in (a) predicting their behavior, (b) identifying their best uses, (c) estimating their productivity and (d) providing objects or units for research and for extending and extrapolating research results.

Environmental degradation from oil extraction in Nigeria is not limited to only one community. In the 3,600 square kilometre Niger Delta- the area where nearly all of Nigeria's oil extraction takes place – an estimated 11 billion gallons of oil have been spilled every year for the past 50 years (Pitkin, 2013).

It is penitent to note that this exploration has also done a lot of damage and released toxicants e.g. heavy metals into the atmosphere and the Soil eventually is at the receiving end. The adverse effect of this generally, and to agriculture in particular, can never be over emphasized. To monitor contamination of soils by heavy metals, the Environmental Protection Agency of the United States (USEPA, 1992) recommends the determination of total contents in soils, extracted with concentrated nitric acid, even if the results often do not represent a good indication of bioavailability to plants. The availability of elements for plants in an agronomic sense can be estimated with reasonable precision by soil analysis, which allows the determination of the degree of deficiency, sufficiency or excess of plant nutrients and other elements. In principle, it can also be used for environmental monitoring, but the diagnosis of toxic levels of nutrients and other elements is seldom a concern in routine soil testing.

1.2 Problem Statement

Pedological information is important to land users especially farmers who use the data to decide on what crops and management practices are best suited for the optimal and sustainable production of crops (Mbaga et al., 2017)

Also, the environmental impacts of the oil and gas industry essentially results from the activities and processes necessary for the successful operations of the oil and gas industry by the multinational oil companies. This had caused a lot of distortions in the soil, flora and fauna, traditional economies (such as farming, fishing, livestock and wildlife production), and social practices of the people of the area (Orta-Martinez and Finer, 2010; Huang *et al*, 2011). It has also enthroned poverty, soil and food contamination and lack of security of human life (Onwuka, 2005; Enemugwem 2009; Ajibade and Awomuti, 2009)

In one of the studies in a community in the Niger Delta Region, Osuji and Onojake (2004) observed that hydrocarbons and heavy metals from crude oil negatively affected flora and fauna, enhanced the absorption and bioaccumulation of heavy metals in plant cells.

There is also a risk of contamination of soils with excess metals because of atmospheric deposition or the use of pesticides or fertilizers that contain considerable amounts of metals. There is a growing concern about the possibility of soil contamination resulting in uptake by plants and the introduction of the elements in vital food chains affecting food safety. Thus knowledge of build-up of metals in the soils of cultivated areas is important to recognize potential ecological problems.

1.3 Objectives of the study

The primary objective of the work was to classify the soils and determine concentration of selected heavy metals (Chromium (Cr), Cadmium (Cd), Nickel (Ni) and Lead (Pb)) in selected soils of the region.

The Specific Objectives include to:

i. determine the physico-chemical properties of soils
ii. investigate the total amount of Heavy metal in the soil (specific reference to Pb, Cr, Ni, Cd).
iii. use the elucidated properties of the soil in classifying the soils using the United States Soil Taxonomy (USDA, 1975) and FAO/UNESCO world soil legend.
iv. correlate the physico-chemical properties and heavy metals in the soil

1.4. Justification of study

Characterization of the soil to know the macromorphological, physical and chemical properties of the soil is very necessary if the soil is to be put into proper use, and classification of soil ensure a method for grouping of soils based on their characteristics. This ensures that the soil is used for its utmost use.

The presence of Heavy metals in the soil from oil pollution is a common sight in the Niger Delta region. Its attendant adverse effect on soil fauna, flora and crop production by altering the soil physico-chemical properties can never be over emphasized. Also the availability of such heavy metals in the soil does not necessarily make it unfit for agricultural production for fear of bio-accumulation in plant tissue as it could be within the permissible limit in other words below the level requiring clean up.

1.5 Scope of the Study

Chapter one deals with general introduction, background information, statement of problem and justification, the other chapters of this work are composed of four (4) chapters. In chapter two (2), a review of key concepts pollution, general forms of pollution, Oil spillage as environmental pollution. It went further to x-ray heavy metals, its fate in the soil environment, movements in soil, their effect on human health, effect on local plant species and horticultural crops, oil spill and soil physico-chemical properties of soil. Finally it looked at the permissible limits and clean-up of heavy metals. Chapter three(3) was concerned with the study area, site, climatic conditions, hydrology of the area and vegetation. It also covers the sampling and laboratory techniques employed in the determination of physico-chemical properties and heavy metals. Chapter four (4) covers the discussion and interpretation of the results obtained. Chapter five (5) gives a summary of the overall work. Using the information, recommendations were made concerning the heavy metals in the studied soils of the Niger Delta region, Nigeria.

CHAPTER II

2.0 Literature Review

2.1.0 Soil Characterization and Classification

Soil characterization provides the information for our understanding of the physical, chemical, mineralogical and microbiological properties of the soils we depend on to grow crops, sustain forests and grasslands as well as support homes and society structures (Ogunkunle, 2005). Soil classification, on the other hand, helps to organize our knowledge, facilitates the transfer of experience and technology from one place to another and helps to compare soil properties. According to Eswaram (1977), some different uses of soil characterization data include to aid in the correct classification of the soil and enable other scientists place the soils in their taxonomies or classification systems and to serve as a basis for more detailed evaluation of the soil as well as gather preliminary information on nutrient, physical or other limitations needed to produce a capability class. A soil characterization study, therefore, is a major building block for understanding the soil, classifying it and getting the best understanding of the environment (Esu, 2004)

Onweremadu, (2007) noted that characterization and classification of soils of any given location help in generating soil and soil related data which are useful in sustainable use of soil resources. Therefore, it is important to characterize and classify soils in a manner that will ease communication and transfer of knowledge about such soils to farmers and other stakeholders (Nuga et al., 2008)

2.1.1.0 Soil Classification Systems

Soils are usually characterized using relevant physical, chemical and morphological properties inherent in them. It has been reported by Idoga et al., (2005) that soil as a natural resource cannot be properly managed without proper understanding of their characteristics.

The most popular soil classification systems in the world are; USDA soil classification system and FAO/WRB (World Reference Base) classification system. In Africa, Ghanaian classification system has been developed. According to Ahukaemere, (2015), some soil properties (Soil drainage, nutrient content of soils, soil colour, soil structure and soil reaction) are influenced by parent material. It is obvious that differences in soil properties are inherently due to differences in the pedogenetic origin (parent material) of the soil and dynamically due to differences in land use and or management practices. Significant relationship between parent material and soil texture, soil reaction, total exchangeable bases, total acidity, soil depth, colour, profile drainage and gravel content have earlier been reported (Esu, 2010).

2.1.1.1 The USDA Soil Classification

Soil taxonomic classifications reflect the dominant Soil Forming Factors active during soil formation at a particular location (Soil Survey Staff, 2015). The USDA system of Soil Taxonomy (soil naming) consists of a hierarchy of six levels. These levels, in order from most general to most specific, are: Order, Suborder, Great Group, Subgroup, family, Series. These categories are defined in the following paragraphs.

Order – Twelve soil orders are recognized. The differences among orders reflect the dominant soil forming processes and the degree of soil formation. Each order is identified by a word ending in 'sol.' An example is Alfisols.

Suborder - Each order is divided into suborders primarily on the basis of properties that influence soil formation and/or are important to plant growth.

Great Group – Each suborder is divided into great groups on the basis of similarities in horizons present, soil moisture or temperature regimes, or other significant soil properties.

Subgroup – Each great group has a ‘typic’ (typical) subgroup which is basically defined by the Great Group. Other Subgroups are transitions to other orders, suborders, or great groups due to properties that distinguish it from the great group.

Family – Families are established within a subgroup on the basis of physical and chemical properties along with other characteristics that affect management.

Series – The series consists of soils within a family that have horizons similar in color, texture, structure, reaction, consistence, mineral and chemical composition, and arrangement in the profile.

2.1.1.2 World Reference base of soils

The classification of soils is based on soil properties defined in terms of diagnostic horizons, diagnostic properties and diagnostic materials, which to the greatest extent possible should be measurable and observable in the field. The WRB comprises two levels of categorical detail:

- the First Level having 32 Reference Soil Groups (RSGs); - the Second Level, consisting of the name of the RSG combined with a set of principal and supplementary qualifiers.

2.2 POLLUTION

The introduction of harmful substances (that cause adverse changes) often referred to as contaminants into the natural environment is termed Pollution (Onweremadu, 2014). The term contamination is in some cases used interchangeably with pollution in environmental chemistry, but the main interest is the harm done to either humans, organisms or environments that are important to human beings. Common soil contaminants include chlorinated hydrocarbons, heavy metals such as chromium, cadmium–found in rechargeable batteries, and lead–found in lead paint, e.t.c. Recycling industrial by-products into fertilizer may result in the contamination of soils with various metals. For instance, the chemical substances entering the soil environment from ordinary municipal landfills and often reaching groundwater, are enormous.

Contamination is the presence of a minor and unwanted constituent in a material, a physical body or the natural environment.

2.3 Forms of pollution

Pollution may take various forms including discharge of deleterious chemical or substances on natural substances. Sometimes it could take the form of even harmful energy such as noise, heat or light.

Generally, foreign substances and energies which contaminate natural resources are termed pollutants. (Onweremadu, 2014) Substances contain some level of impurity; and this may become an issue if the impure chemical is mixed with other chemicals or mixture that causes additional chemical reactions. If the additional chemical reactions are beneficial, the label ‘contaminant’ is replaced with 'reactant' or 'catalyst'. But when they are detrimental, other terms such as 'toxin' or 'poison' are often used. (Onweremadu, 2014).

However, if no remedial action is taken to reduce the accumulation of such contaminants and pollutants, the availability of arable land for cultivation will decrease, this is because of stricter environmental laws limiting food production on contaminated lands. Inorganic and organic contaminants typically found in urban areas are heavy metals and petroleum-derived products, (Onweremadu, 2014).

2.4 Oil spillage as environmental pollution.

Oil spillage is a release of a liquid petroleum hydrocarbon into the environment due to human activity, and is a form of pollution. Presently, in Nigeria, oil spills regularly occur in the oil producing areas of the country, also gases are continually flared in these areas (Abii and Nwosu, 2009). With advanced technology in use in the oil and gas industry, accidents should be less frequent but this certainly has not completely eliminated accidents and vandalizations. (Iturbe *et al* 2008; Ogbu,2008). The term oil spill often refers to marine oil spills, where oil is released into the ocean or coastal waters. Oil spills include releases of crude oil from tankers, offshore platforms, drilling rigs and wells, as well as spills of refined petroleum products (such as gasoline, diesel) and their by-products, and heavier fuels used by large ships such as bunker fuel, or the spill of any oily white substance, refuse or waste oil. Spills may take months or even years to clean up. Oil also enters the marine environment from natural oil seeps. Public attention and regulation has tended to focus most sharply on seagoing oil tankers.

Oil spillage is one of the greatest environmental problem Nigeria is currently battling with especially in the Niger Delta zone. Oil communities have been at the receiving end of this environmental problem.

2.4.1 Sources of Oil Spill

Oil spills may occur for numerous reasons such as equipment failure, disasters, deliberate acts, or human error (Anderson and LaBelle, 2000). Crude oil are exclusively natural products, most of which are produced from artificial wells. Natural seepage of crude oil occurs in various parts of the world, not only on land, but also on the seabed. Seeps emerge through fractures in the crests of folds in rock formations beneath the sea floor that contain oil and gas deposits. Oil and gas tend to rise and become trapped in anticlinal folds in subsea rock strata. Seepage occurs through fracture zones where the folds are truncated at the sea floor.

2.5.0 Heavy Metals

The category of "heavy metals" includes several elements Cd, Cu, Cr, Co, Fe, Hg, Mn, Mo, Ni, Pb, Zn, which are defined by their metallic properties (conductivity, ductility and stability as cations), atomic number greater than 20 and density greater than 5.6 kg/dm^3 .

Their name suggests their inclusion in this category by their specific weight higher than $5mg/cm^3$ and a potentially toxic character upon high concentrations and they can be classified in trace elements category, as in low concentrations they are quality nutrients for plants. Naturally, they show low concentrations in soils, but can occur in high and potentially toxic concentrations as a result of human activities, especially of the uncontrolled type. In this alternative, their excess can cause disturbances in the soil, plants and water and subsequently in the food chain upper links (Shuman 1991).

2.5.1 Fate of Metals in the Soil Environment

According to Shuman 1991, Metals in soils are found in one or more of these several "pools" of the soil, These include:

1. specifically adsorbed on inorganic soil constituents;
2. associated with insoluble soil organic matter;
3. occupying exchange sites on inorganic soil constituents;
4. dissolved in the soil solution;
5. precipitated as pure or mixed solids;
6. present in the structure of secondary minerals; and/or
7. As structural component of primary minerals.

While native metals may be associated with any of the pools depending on the geological history of the area, metals which have been introduced into the environment through anthropogenic activities, are usually associated with the first five pools. The aqueous fractions, and those fractions in equilibrium with this fraction, i.e., the exchange fraction, are of primary importance when considering the migration potential of metals associated with soils.

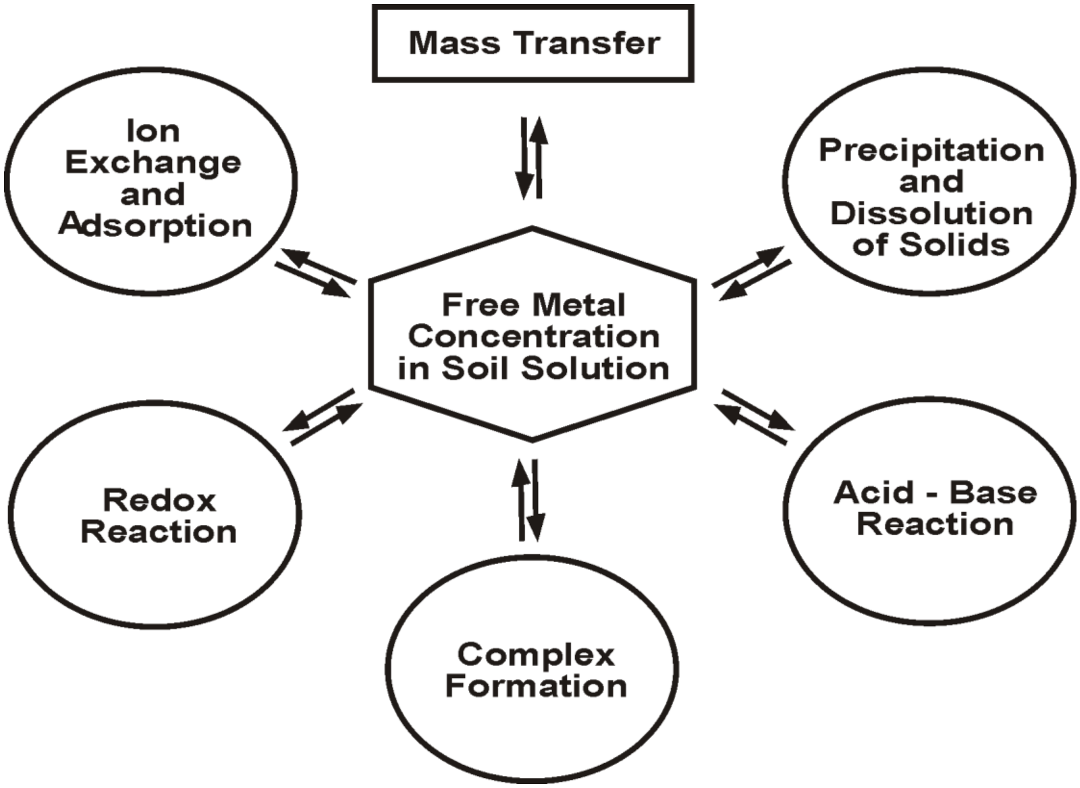

Fig 2.1. Principal controls on free trace metal concentration in soil solution (Mattigod *et al*. 1981).

Multiphase equilibria must be considered when defining metal behavior in soils. Metals in the soil solution are subject to mass transfer out of the system by leaching to ground water, plant uptake, or volatilization, a potentially important mechanism for Hg, Se, and As. Metals also participate in chemical reactions with the soil solid phase. Thus, the concentration of metals in the soil solution, at any given time, is governed by a number of interrelated processes, including inorganic and organic complexation, oxidation-reduction reactions, precipitation/dissolution reactions, and adsorption/desorption reactions. The ability to predict the concentration of a given metal in the soil solution depends on the accuracy with which the multiphase equilibria can be determined or calculated.

2.5.2 Classification of Heavy Metals in Soils

According to Shuman (1991), Metals in soil can be separated into operationally defined fractions that are relevant to the physico – chemical forms. The water – soluble and exchangeable fractions and sometimes the organic fractions are usually considered to be the plant – available forms. They are also the potentially mobile forms in the soil environment and could have harmful impacts on groundwater. Organic acids in the topsoil can solubilize metals in the manganese and iron oxide fractions, thus providing opportunity for them to move downward in the profile and be re-adsorbed on the exchanged complex.

2.5.3 Movement of Heavy Metals in Crude Oil Spilled Soils

Heavy metals' concentration and movement have received much attention with regard to accumulation in soils, uptake by plants, and contamination of water. Environmental hazards derived from heavy metals could be linked closely to their concentration and movement in soil profile because according to Williams and Williams (1996), even slow transport through soil and subsoil materials may eventually increase the content of heavy metals in the ground water.

The use of sequential extraction techniques to separate the soil metals into different forms can be helpful in understanding the processes of metal movement in the soil profile. Crude oil spilled soils are common in Niger Delta area where oil spillages occur as a result of the exploration of oil. The crude oil contain: Lead; Chromium; Cadmium; Iron; Nickel; Vanadium in a very small amount as impurities. Because of the coarse texture nature of soils of Niger Delta, these metals could be transported from the top soil to the subsoil even to the groundwater.

Except for very acidic soil, heavy metals in soil are sparingly and occur prominently in absorbed state or as insoluble compounds. Because of their low solubility, movement of heavy metals in soils has generally been considered either minimal or practically non-existent. Anderson (2007) observed that in natural soil profiles, Cadmium (Cd), Chromium (Cr) and Lead (Pb) are more concentrated in the upper part of the soil profiles. After 14 years of sludge applications, Dowdy *et al*;(1991) found a small movement of Cd and Zn down through the soil profile. The findings of Stevenson and Welch (1999) showed that Pb salt migrated downward to at least a 90cm depth in less than 7 years after Pb acetate was applied to a cropped field. Williams *et al*;(2005) found no significant movement of Cd, Cu, Pb and Zn in soil treated with sludge for 8 years. Chang *et al*; (2004) demonstrated that more than 90% applied heavy metals were found in the top surface (0-15cm) of the soil profile. In natural soil profiles, Cd and Pb accumulated in the upper part of soil profiles and the distribution patterns of these elements were similar to that of organic matter (Anderson, 2007). Dowdy and Volk (1983) demonstrated that the movement of heavy metals in soils could occur in sandy, acidic, low organic matter soil subjected to heavy rainfall or irrigation. These conditions are similar to the soil condition in Niger Delta region.

The soils of Niger Delta area often have coarse texture, low CEC, pH and organic matter content (Ogboi *et al*; 2006). These conditions may adversely affect heavy metals' retention capacity of these soils. Heavy metals in these soils may be proned to downward movement in the soil profiles either slowly by diffusion or much more rapidly because of preferential flow in root channels and voids left by soil fauna. The high annual rainfall in Niger Delta region can facilitate the transport of metal from top soil to subsoil, which could eventually contaminate groundwater in metal contaminated areas.

Heavy metals movement with water in soils requires that the metals be in the soluble phase or associated with mobile particulates. It has been shown that metals such as zinc and copper are complexed strongly by humic acids at pH levels around 4.5 to 5.0 (Waller and Pickering, 1992). Thus, particulate organics could complex metals in the profile where they would be in an exchangeable organic form.

Therefore, movement is essentially related to the physico – chemical forms of the metals in soil because these forms have different potentials for mobilization by inorganic or organic ligands in soil solution (Petruzzelli and Lubrano, 1994 McBridge, 1989 and Shuman, (1996).

2.5.4 Effects of Heavy Metals on human health

In general the toxicity of metal ions to mammalians systems is due to chemical reactivity of the ions with cellular structural proteins, enzymes and membrane system. The target organs of specific metal toxicities are usually those organs that accumulate the highest concentrations of the metal in vivo (Manju, 2015). The target organs and clinical manifestations of chronic exposures to the metal are given in table below. Besides the general toxicities of metals, we are today also concerned with the potential carcinogenicity of metal compounds. Certain metals such as chromium and nickel have been linked with cancers in exposed human populations. Metals have been shown to causes acute as well as chronic poisoning in man and other experimental animals.

Table 2.1 Clinical aspects of chronic toxicities

METAL	TARGET ORGAN	PRIMARY SOURCE	CLINICAL EFFECT
Cadmium	Renal, Skeletal & Pulmonary	Industrial dust, fumes and spilled water and food	Proteinuria, Glucosuria, Osteomalacia, Aminoaciduria, Emphysemia
Chromium	Pulmonary	Industrial dust, fumes and spilled food	Ulcer, Perforation of Nasal Septum, Respiratory Cancer
Lead	Nervous System, Hematopoietic System, Rena	Industrial Dust And Fumes And Spilled Food	Encephalopathy, Peripheral Neuropathy, Central Nervous Disorders, Anemia
Nickel	Pulmonary, Skin	Industrial Dust, Aerosols	Cancer, Dramatis

Source: Manju 2015

The effects of toxic metals (cadmium, chromium, lead, nickel, etc.) on human health and their interactions with essential heavy metals (trace elements) may produce serious consequences (Abdulla and Chimelnicka, 1990; Tokusolu *et al*., 2004). From this viewpoint, metals such as iron, arsenic, lead, mercury, cadmium, chromium and nickel are considered suitable for studying the impact of various foods on human health.

2.5.5 Effect of Oil Pollution on Local Plant Species and Food Crops

Nkwocha and Duru (2010) opined that oil pollution has specific impacts on local plant species as well as on the cultivated food crops.

It was generally observed that the young plants at the impacted sites were more vulnerable to oil pollution than the old plants after a period of fifteen months, even though the latter also showed signs of wilting. Also, nine months after the spills, the young plants, to a large extent showed partial defoliation, leaf loss and sometimes died, Nkwocha and Duru (2010).

It was equally observed that fifteen months after remediation at the spilled sites, recovery rate of plants still remained very slow with none exceeding 2 percent per 6 months.

2.5.6 Effect of Heavy metals on horticultural crops

The effects of heavy metals on some crops have been studied by many researchers.

Ekundayo *et al*. (2001) studied the effect of crude oil spillage on growth, productivity and nutrient uptake of maize (Zea mays L.) The results showed that in crude oil spilled soils, germination was delayed and the germination percentage was significantly affected by oil pollution. Growth was poor in spilled soils using parameters such as plant height; stem girth, ear height, leaf area at four weeks after planting, leaf area at maturity and average length of primary roots as growth indicators. Grain yield was significantly reduced at 95% level of probability when compared with the control. Leaf analysis of the maize plants grown in soils contaminated with crude oil a week before planting (preplant treatment) revealed mean levels of heavy metals which were higher than maximum permissible levels for maize in tropical soils.

Achuba (2006) studied the effect of crude oil contaminated soil at various sublethal concentrations on the growth and metabolism of cowpea (*Vigna unguiculata*) seedlings. The results showed that crude oil induced environmental stress in the seedlings.

Agbogidi *et al*. (2007) results showed that soil treatment with crude oil at four weeks after planting (4WAP), maize died within 24 hours while the plant without crude oil treatment remained intact. The study showed that the time of application of crude oil to soil has a significant effect on growth of the maize.

Eriyameru *et al*. (2007) studied the effect of contaminating soil with Bonny light whole crude, or its fractions on germinating beans (Phaseolus vulgaris L.) and maize (Zea mays L.). The results showed that there was dose dependent reduction in the number of bean or maize seeds that germinated in the contaminated soils compared with the control ($p < 0.05$), with the least number recorded in the 0.3% contaminated soil.

2.5.7 Oil spill and physico-chemical properties of soils

Abii and Nwosu (2009) studied two oil spill affected areas (Ogali and Agonchia) while an unaffected area (Aleto) all in Eleme LGA, Rivers State was used as control. The results showed that there was a significant decrease in the Ca, K, P, (CEC), as well as a significant increase in the sand fraction and Na content of the oil-spill affected soils when compared with the non-affected soil. The results further showed that oil-spill had adversely affected the nutrient level and fertility states of Eleme soil.

2.6 Permissible limits of heavy metals

The United States Environmental Protection Agency (USEPA) and New York Department of Environmental Conservation (NYS DEC) have guidelines for determining the safety of various land uses based on total soil metal concentrations. Table 2.2 shows these limits, which are used to guide clean-up efforts. USEPA levels are used to guide clean-up efforts of contaminated sites while NYS DEC levels are based on removing human health risks; unrestricted use includes agriculture.

In Nigeria, the Federal Environmental Protection Agency have guidelines on permissible limit for heavy metals in the environment. These are captured in Table 2.2 below.

Table 2.2: Federal Environmental Protection Agency (FEPA), New York Department of Environmental Conservation (NYSDEC) and United States Environmental Protection Agency (USEPA) permissible limits

Heavy Metal	**FEPA** (mg/Kg)	**USEPA** (mg/Kg)	**NYS DEC Unrestricted use** (mg/Kg)	
			Unrestricted Use	**Residential Use**
Cadmium	10	70	0.43	0.86
Chromium	30	230	11	22
Lead	50	400	200	400
Nickel	100	1600	72	140

FEPA (1991), USEPA (2002), NYS DEC (2006)

2.7 Cleaning Up Heavy Metals

In soils with elevated heavy metal levels, which may pose higher levels of risk, you should consider whether remedial actions are appropriate, or whether crops should be grown at all. (Vern and Don, 2011)

Remediation deals with the removal of pollutants or contaminants from natural resources. The affected natural resources may include soil, groundwater, surface water sediment, vegetation, rock minerals, wildlife and air. A major aim of remediation is the recovery and general protection of human health and the environment (Onweremadu, 2014). Sometimes, remediation is done in places intended for redevelopment. Remediation goes with an array of regulatory requirements, and its assessments are based on human health and ecological risks.

Several approaches are used in the remediation of spilled soils, ranging from biological, chemical and engineering techniques. Sometimes, it may require a combination of organic and inorganic strategies. For instance the Neapolitan yellow tuff (NYT) was utilized as a component of an organo-mineral sorbent/exchanger soil conditioner with pellet manure (NYT/PM) to reduce the mobility of Cd and Pb and recover plant performance in heavily spilled soils from illegal dumps near Santa Maria La Fossa (Lower Volturno river basin, Campania Region, southern Italy). Pot experiments were performed by adding the NYT/PM mixture (1:1, w/w) to spilled soil at the rates of 0%, 25%, 50% or 75% (w/w). Wheat (Triticum aestivum) was used as the test plant. The addition of organo-zeolite NYT/PM mixture significantly reduced the DTPA (diethylene-triamine-pentaacetic acid)-extractable Cd and Pb from 1.01 and 97.5 mg kg^{-1} in the spilled soil, to 0.14 and 11.6 mg kg^{-1} respectively, in the soil amended with 75% NYT/PM. The best plant response was observed in amended soil systems treated with 25% NYT/PM, whereas larger additions induced plant toxicities due to increased soil salinity.

When a soil on site is found to be contaminated to a depth of several metres and construction work needs to get started in a few months' time, soil replacement is the fastest remedy. However, some of the contaminated areas can be restored by combining modern and age-old methods. This is where plants and their microbial partners may enter the picture now and in the future. This is because heavy metals in soils with residence times of thousands of years present numerous health dangers to higher organisms (Garbisu and Alkorta, 2001). They are also known to decrease plant growth, ground cover and have a negative impact on soil microflora (McGrath *et al*., 2001).

When there is oil spill, certain microorganisms, which degrade it, grow on it, degrading the crude to different components. The disappearance of spilled crude from the environment is attributable to the activities of the microflora. The discovery of the activities of microorganisms in the breakdown of crude oil to less harmful products, gave rise to bioremediation. Bioremediation involves the use of microorganisms to accelerate the natural breakdown of oil into less harmful products (Poly, 1992). Research has demonstrated that plants are effective in cleaning up contaminated soil (Wenzel *et al*., 1999). Phytoremediation is a general term for using plants to remove, degrade, or contain soil pollutants such as heavy metals, pesticides, solvents, crude oil, polyaromatic hydrocarbons, and landfill leacheates e.g. prairie grasses can stimulate breakdown of petroleum products. Also wildflowers were used to degrade hydrocarbons from an oil spill in Kuwait. Hybrid poplars can remove ammunition compounds such as TNT as well as high nitrates and pesticides (Brady and Weil, 1999). Cleanup (or remediation) technologies available for reducing the harmful effects at heavy metal-contaminated sites include;

1. Excavation (physical removal of the contaminated material).
2. Stabilization of the metals in the soil on site.
3. Use of growing plants to stop the spread of contamination or to extract the metals from the soil (phytoremediation).

2.7. 1 Excavation

Excavation and physical removal of the soil is perhaps the oldest remediation method for contaminated soil. It is still in use at many locations, including residential areas contaminated with lead in southwestern Missouri. Advantages of excavation include the complete removal of the contaminants and the relatively rapid cleanup of a contaminated site (Wood, 1997). Disadvantages include the fact that the contaminants are simply moved to a different place, where they must be monitored; also, the risk of spreading contaminated soil and dust particles during removal and transport of contaminated soil; and the relatively high cost. Excavation can be the most expensive option when large amounts of soil must be removed or disposed as hazardous or toxic waste is required.

2.7.2 Stabilizing Metals in the Soil

Heavy metals can be left on site and treated in a way that reduces or eliminates their ability to adversely affect human health and the environment. This process is sometimes called stabilization. Eliminating the bioavailability of heavy metals on site has many advantages over excavation. One way of stabilizing heavy metals consists of adding chemicals to the soil that cause the formation of minerals that contain the heavy metals in a form that is not easily absorbed by plants, animals, or people. This method is called in situ (in place) fixation or stabilization. This process does not disrupt the environment or generate hazardous wastes. Instead, the heavy metal combines with the added chemical to create a less toxic compound. The heavy metal remains in the soil, but in a form that is much less harmful. One example of in situ fixation of heavy metals involves adding phosphate fertilizer as a soil amendment to soil that has high amounts of the heavy metal lead. Chemical reactions between the phosphate and the lead causes a mineral to form called lead pyromorphite. Lead pyromorphite and similar minerals called heavy metal phosphates are extremely insoluble. This means the new minerals cannot dissolve easily in water (Lambert *et al*., 1997). This has two beneficial effects.

1. The minerals (and the heavy metals) cannot be easily spread by water to pollute streams, lakes, or other groundwater.

2. Also the heavy metal phosphates are less likely to enter the food chain by being absorbed into plants or animals that may eat soil particles.

This method is relatively rapid and takes about the same amount of time as excavation.

2.7.3 Use of Plants for cleanup of heavy metals

Research has demonstrated that plants are effective in cleaning up contaminated soil (Wenzel *et al*., 1999). Phytoremediation is a general term for using plants to remove, degrade, or contain soil pollutants such as heavy metals, pesticides, solvents, crude oil, polyaromatic hydrocarbons, and landfill leacheates. For example, prairie grasses can stimulate breakdown of petroleum products. Wildflowers were recently used to degrade hydrocarbons from an oil spill in Kuwait. Hybrid poplars can remove ammunition compounds such as TNT as well as high nitrates and pesticides (Brady and Weil, 1999).

Growing plants can help contain or reduce heavy metal pollution. It has the advantage of relatively low cost and wide public acceptance (Schnoor, 1997). It can be less than a quarter of the cost of excavation or in situ fixation. Phytoremediation has the disadvantage of taking longer time to accomplish than other treatment. Plants can be used in different ways. Sometimes a contaminated site is simply revegetated in a process called phytostabilization. The plants are used to reduce wind and water erosion that spread materials containing heavy metals. In one example, grass or tree buffers could reduce sediment loss from the chat piles at a contaminated site in Galena, Kansas, anywhere from 18% to 25% (Green, *et al*. 1997). If all of the ground could be revegetated, sediment loss could be cut by approximately 70%. However, it would be necessary to find plants that could tolerate high levels of heavy metals.

Another way plants can be used to clean up heavy-metal contaminated soil is called phytoextraction. Some plant species can take up heavy metals and concentrate them in their tissue. The plants can be harvested and the contaminated plant material disposed of safely. Sometimes soil amendments are added to the soil to increase the ability of the plants to take up the heavy metals. One type of plant used for this purpose is called Indian mustard. This plant has been used to extract lead from soil and reduce lead contamination at various contaminated sites. Other plants that may be used for phytoextraction include alfalfa, cabbage, tall fescue, juniper, and poplar Trees. (Lambert *et al*, 2005).

Furthermore, plants are used to treat heavy metal contamination through rhizofiltration (EPA, 2000). In this method, heavy metals are removed directly from water by plant roots. The plants are grown directly in water or in water rich materials such as sand, using aquatic species or hydroponic methods. In field tests sunflowers on floating rafts have removed radioactive metals from water in ponds at Chernobyl, and other plants removed metals from mine drainage flowing through diversion troughs (USEPA, 2000). Plants used for phytoextraction may accumulate high concentrations of metals. Fences or other ways to limit access to people and animals, and disposal of plant matter as special waste is some-times necessary.

CHAPTER III:

3.0 Materials and Methods

3.1.0 Study Area:

The study was conducted in some states (Imo and Abia) of the Niger Delta region of Nigeria, In Imo State, samples were collected from Obokofia (Lat $5^0$29'31.946 and $5^0$41' and Long. $6^0$37' and $6^0$49') in Ohaji – Egbema (between Lat $5^0$21' and $5^0$41' and Long. $6^0$37' and $6^0$49'), having an altitude not exceeding 125m above sea level (Orajaka, 1975) and Oguta LGAs (between Lat $5^0$38' and $5^0$42' and Long. $6^0$65' and $6^0$85') having an altitude not exceeding 25m above sea level, while in Abia, samples were collected from Ugwunagbo Local Government Area (between Lat $4^0$48' and $5^0$25' and Long. $7^0$00' and $7^0$25') with an altitude of not exceeding 40m above sea level.

TABLE 3.1: Geographical coordinates of sampled sites.

LOCATION	STATUS	LATITUDE	LONGITUDE
Ugwunagbo	Spilled	5^{0}0’5.294”N	7^{0}18’43.748” E
	Unspilled	5^{0}0’9.295”N	7^{0}18’37.221”E
Oguta	Spilled	5^{0}41’37.054”N	6^{0}47’55.29E
	Unspilled	5^{0}41’40.129”N	6^{0}47’42.312E
Egbema	Spilled	5^{0}29’3.935”N	6^{0}47’29.18”E
	Unspilled	5^{0}28’31.946”N	6^{0}47’6.933”E

3.1.1 Geology and Geomorphology:

The lithology of Imo state and Abia are similar and composed predominantly of Benin formation, Imo Shale, Nsukka Formation, Bende, Ogwashi formation and Alluvium, Igali Sandstone (Onwuremadu, 2007). The Soils are basically Ultisols formed on 6 lithological materials namely Alluvium, Coastal Plain sands (Benin formation), Lower coal measures (Mamu formation), upper coal measures (Nsukka formation), shale (Bende-Ameke formation) and false bedded sandstone (Orajaka, 1975).

The selected locations (Egbema, Oguta and Ugwunagbo) are of Benin formation.

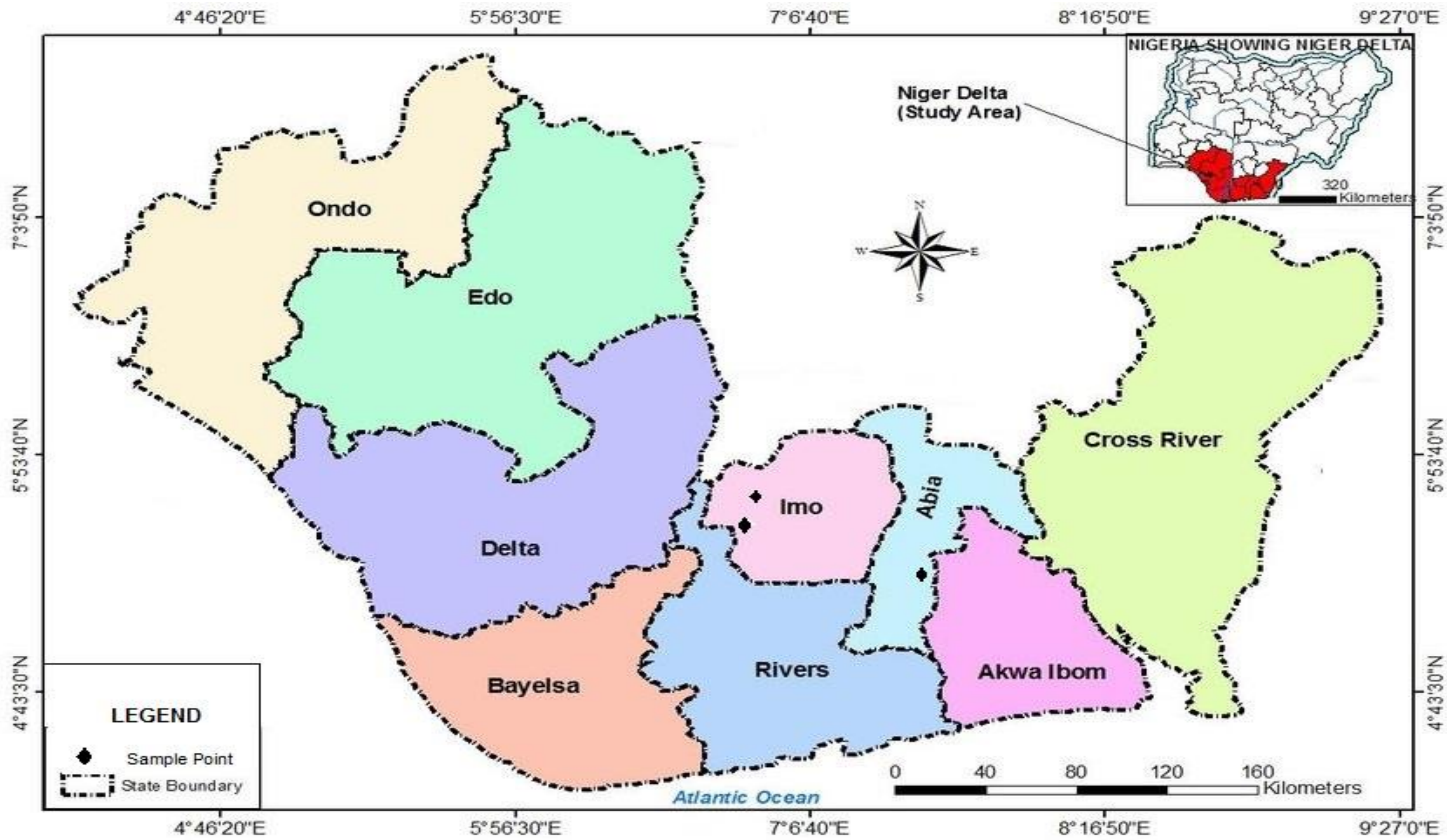

Figure 3.1 Map of Niger Delta Region showing the sample points

3.1.2 Climate / vegetation

The study areas have humid tropical climate, the rainy season in Imo and Abia States starts in February- March and last till November. It has bi-modal rainfall pattern with a mean annual rainfall of about 2500mm in Imo(Obi and Salako, 1995) while it varies from 1900mm – 2200mm in Abia State. Mean annual temperature is about 28^0C in Imo and 27^0C in Abia. Rainforest vegetation predominates in both states though slightly altered by anthropogenic activities. Staple food farming, Fishing, palm oil processing and Hunting are basic socio – economic activities of these areas. Some dominant tree crops found include Dacryodes edulis, Pentaclethra macrophylla, Raffia spp., Elaeis guineensis and citrus spp while staple food include cassava yam, maize, cocoyam and vegetables

3.2.0 Field Study

A reconnaissance visit was made to the locations and sample sites identified before the actual field work. A Handheld Global Positioning System (GPS) receiver, Core Samplers, Munsell Colour Chart and Rolling tape were used to take location coordinates, collect samples for bulk density, determine soil colour and thickness of each horizon respectively.

Samples were collected from three (3) locations. A total of Six (6) profile pits were sunk i.e. two (2) at each location.

One pit was sunk at spilled site while the second was sunk as a control on an unspilled area at each location.

Description of each pit was done according to FAO guidelines for soil description (FAO, 2006) and samples were collected according to genetic horizons from the deepest horizon, this is to avoid contamination. Samples collected were bagged in properly labelled cellophane bags then transported to the laboratory where they were air dried, gently crushed and sieved using a 2 mm sieve in preparation for analysis.

3.3.0 Laboratory Analyses

3.3.1.0 Soil Physical Properties

3.3.1.1 Particle Size Distribution:

Particle Size Distribution (Mechanical analysis) was determined by the hydrometer method as described by Gee and Or (2002), using Sodium hexametaphosphate (calgon) as dispersant. The result was expressed in gkg^{-1}.

The textural class was determined using the Soil Textural Triangle. (Loganathan, 1984).

3.3.1.2 Soil Bulk density:

Bulk Density (BD) was determined by the core method according to Grossman and Reinsch, (2002).

The Bulk Density was calculated as Mass of oven dried soil (g) per bulk volume of soil (cm^3) which is equivalent to the volume of core. The mathematical relationship is given as

$$\frac{m_3 - m_1}{\pi r^2 h}$$

Where m_3 *= Mass of crucible + Oven dried Soil*

m_1 *= Mass of empty crucible*

r *= Radius of the core sampler*

h *= height of the core sampler*

It was expressed in gcm^{-3}.

3.3.1.3 Moisture Content:

The moisture content was determined via the use of core samplers. Samples were collected from genetic horizons of each pedon, preserved in a polythene, taken to the laboratory, poured in a crucible and weighed, this is recorded as m_2. The empty crucible was initially weighed and recorded as m_1. The crucible with its content were oven dried at a temperature of 105°C until constant weight was achieved, this is recorded as m_3. Thus the Gravimetric moisture content (Obi, 1990) was calculated from this relationship

$$\frac{m_2 - m_1}{m_3 - m_1} X100$$

Where m_3 = *Mass of crucible + Oven dried Soil*
m_2 = *Mass of crucible + Soil + Water*
m_1 = *Mass of empty crucible*

It was expressed in percentage

3.3.1.4 Total Porosity:-

Total porosity was calculated from the relationship between bulk density and particle density. Whole particle density was taken as 2.65g/cm^3.

$$f = (1 - \frac{pb}{ps}) X100$$

Where f = Porosity
pb = bulk density (g/cm^3)
ps = Particle density (assumed to be 2.65g/cm^3)

3.3.1.5 Silt / Clay Ratio:

This was calculated by dividing the quantity of silt by that of clay as follows.

$$Silt / Clay Ratio = \frac{Silt}{Clay}$$

Where Silt = Amount of Silt (g/kg)
Clay = Amount of Clay (g/kg)

3.3.2.0 Soil Chemical Properties

3.3.2.1 Soil pH:

pH reading was taken in water and Potassium Chloride (KCl) using a pH meter in a soil/liquid ratio of 1:2.5 (Hendershot *et al*., 1993)

3.3.2.2 Soil Organic Carbon:

It was determined by dichromate wet oxidation (Walkley - Black) method as described by Nelson and Sommers (1982). The values of organic carbon gotten was multiplied by 1.724 (Van Bemmelen factor) to obtain the values of Soil Organic Matter (SOM)

3.3.2.3 Available phosphorus:

Available Phosphorus was determined using Bray II solution method according to Olsen and Sommers, 1982 where the available P was extracted using the molybdateblue colorimetric method.

3.3.2.4 Exchangeable Bases:

Exchangeable bases (Ca, Mg, K and Na) were extracted using 1N ammonium acetate (1N NH_4OAC), Calcium(Ca) and Magnesium (Mg) were determined by EDTA titration while Exchangeable Potassium and Sodium were determined by flame photometery (Jackson, 1962; Thomas, 1982).

3.3.2.5 Total Nitrogen:

Nitrogen was determined by Kjeldahl digestion method using conc. H_2SO_4 and a sodium copper sulphate catalyst mixture (Jackson, 1962; Bremner and Mulvaney 1982).

3.3.2.6 Exchangeable Acidity (Al^{3+} + H^+):

It was determined by titration as described by (Mclean 1982). It was extracted with one normal potassium chloride solution. The exchangeable hydrogen was obtained by subtracting exchangeable aluminium from the exchangeable acidity.

3.3.2.7 Cation Exchange Capacity (CEC):

It was determined by 1N ammonium acetate extraction method (Ciesielski *et al*, 1997).

3.3.2.8 Effective Cation Exchange Capacity (ECEC):

It was derived by the summation of the total exchangeable bases (TEB) and exchangeable acidity (Al^{3+} + H^+) (Brady and Weil, 2010).

3.3.2.9 Percentage Base Saturation (%BS):

It was calculated as

$$\%BS = \frac{TEB}{ECEC} X100$$

Where %BS = Percentage base saturation

TEB = Total exchangeable basic cations

ECEC = Effective Cation exchange capacity

3.3.2.10 Aluminium Saturation (Al Sat.):

It was calculated as

$$\%Al.Sat = \frac{Exch.Al^{3+}}{ECEC} X100$$

Where Al. Sat = Aluminium saturation

Exch. Al = Exchangeable Aluminium

ECEC = Effective cation exchange capacity

3.3.2.11 Carbon/Nitrogen Ratio:

This was calculated as

$$C/N..Ratio = \frac{OC}{TN}$$

Where C/N ratio = Carbon Nitrogen ratio
OC = Organic carbon
TN = Total nitrogen

3.3.2.12 Calcium/Magnesium Ratio:

This was calculated as

$$Ca / Mg..Ratio = \frac{Ca}{Mg}$$

Where Ca/Mg ratio = Calcium Magnesium ratio
Ca = Calcium
Mg = Magnesium

3.3.3 Heavy metals determination

One (1) gram of the sieved soil sample was digested using nitric / perchloric acid as described by Odu *et al*. (1986). The concentration of some heavy metals (Chromium(Cr), Cadmium(Cd), Lead(Pb) and Nickel(Ni)) were determined using atomic absorption spectroscopy following the procedure outlined in American Public Health Association (1992).

3.4.0 Data Analysis

Soil data obtained for physico-chemical properties and heavy metal analyses were subjected to T-Test to ascertain differences between spilled and unspilled soils. Coefficient of variation was carried out to determine the vertical variations of the properties of the different soil horizons, while correlation analysis was done to determine the functional relationship between some soil properties and the heavy metals. The result of the coefficient of variation were ranked using the ranking of wilding 1985 where CV ≤ 15% = low variation, >15 and ≤ 35% = moderate variation and >35 = high variation.

The mean was determined for all data derived from each profile pit using SPSS version 21.

3.5. Soil Classification

The soils were classified according to United States Soil taxonomy (USDA, 2010) and correlated with World Reference Base (FAO/UNESCO).

CHAPTER IV

4.0: Results and Discussion

4.1 Morphological Characteristics

Results of some macromorphological characteristics of the studied soils are shown in Tables 4.1a – 4.1c

The soil drainage were classified as well drained for all locations, this could be inferred from the macro porosity of the sand particles which is the dominant particle size fraction in the top horizon (Nkwopara *et al*, 2012).

All soils of both spilled and unspilled sites were deep (greater than 180cm) and there was no sharp distinction in drainage.

4.1.1 Soil colour (moist)

Soil colour of spilled pedon in Egbema varied from very dark grey brown (10YR3/2) in the uppermost horizon to Strong brown (7.5YR5/6) with increase in depth, while spilled soils of Oguta varied from Dark Brown (7.5YR3/2) to yellowish red and spilled soils of Ugwunagbo varied from greyish brown (10YR5/2) to yellow brown(7.5YR5/4). Whereas the unspilled soils varied from Dark brown (10YR3/3) to Red (2.5YR4/6) in Ugwunagbo, Dark brown (10YR4/2) to Red (2.5YR5/8) in Egbema sites and from Dark Reddish brown (5YR3/2) to Red (2.5YR4/8) at Oguta site.

TABLE 4.1(a): Selected macromorphological characteristics of studied soils

Horizon	Depth	Colour Matrix (Moist)	Textural Class	Structure	Consistence	Roots	Boundary
EGBEMA (POLUTED)							
A	0 -19	Very Dark greyish Brown (10YR3/2)	LS	Weak very fine granular	Very Friable	Common Fine	Gradual Smooth
E	19 – 23	Dark Brown (10YR3/3)	SL	Weak Moderate SBK	Friable	Many fine	Clear Smooth
Bt_1	23 – 60	Yellowish Brown (10YR5/4)	SC	Moderate Fine SBK	Friable	Very Few fine	Gradual Smooth
Bt_2	60 – 118	Yellowish Brown (10YR5/6)	C	Moderate Medium SBK	Friable	Very few fine	Clear Smooth
Bt_3	118 – 212	Strong Brown (7.5YR5/6)	C	Moderate Medium SBK	Friable	Very Few fine	
EGBEMA (UNPOLUTTED)							
A	0 -15	Dark Brown (10YR4/2)	S L	Weak Fine Single grained	Very Friable	Many Fine	Clear smooth
AB	15 – 54	Yellowish Brown (10YR5/4)	SCL	Weak Fine Single grained	Friable	Many fine to medium	Clear wavy
Bt_1	54 – 90	Yellowish Brown (10YR5/6)	SC	Weak Fine granular	Friable	Few fine and medium	Abrupt wavy
Bt_2	90 – 160	Red (2.5YR4/6)	C	Weak Fine SBK	Friable	Very few fine and medium	Abrupt wavy
Bt_3	160 – 210	Red (2.5YR5/8)	C	Moderate fine SBK	Friable	Few fine	

SBK= Subangular Blocky, SL=Sandy Loam, SCL = Sandy Clay Loam, SC = Sandy Clay, C = Clay

TABLE 4.1(b): Selected Macromorphological Characteristics of studied soils contd

Horizon	Depth	Colour Matrix (Moist)	T C*	Structure	Consistence	Roots	Boundary
OGUTA (SPILLED)							
A	0 -18	Dark Brown (7.5YR3/2)	SL	Weak very fine granular	Friable	Many fine to Medium	Smooth Gradual
AB	18 – 52	Dark Brown (2.5YR5/4)	SCL	Moderate very fine granular	Friable	Many fine to Medium	Smooth abrupt
Bt_1	52 – 99	Yellowish Red (5YR5/6)	SCL	Moderate to very Fine granular	Friable	Common and fibrous	Gradual Smooth
Bt_2	99 – 190	Yellowish Red (5YR5/8)	SL	Weak very fine granular	Friable	Many fine and Medium	
OGUTA (UNSPILLED)							
A	0 -16	Dark Reddish Brown (5YR3/2)	SL	Weak fine Single grained	Very Friable	Many Fine & Medium	Clear Wavy
AB	16 – 67	Dark Red (7.5YR4/4)	SL	Weak fine granular	Friable	Many Fine & Medium	Abrupt Wavy
Bt_1	67 – 125	Red (2.5YR4/6)	SCL	Moderate to Fine granular	Friable	Fine fibrous and Medium	Clear Smooth
Bt_2	125 – 186	Red (2.5YR4/8)	SL	Weak Fine granular	Friable	Very Few fine	

SL=Sandy Loam, SCL = Sandy Clay Loam

TABLE 4.1(c): Selected Macromorphological Characteristics of studied soils contd

Horizon	Depth	Colour Matrix (Moist)	T C*	Structure	Consistence	Roots	Boundary
UGWUNAGBO (SPILLED)							
A	0 -12	Greyish Brown (10YR5/2)	SL	Weak fine SBK	Friable	Many fine and Medium	Clear smooth
AB	12 – 25	Dark Greyish Brown (10YR4/2)	SL	Weak Fine to Medium SBK	Friable	Many fine and Medium	Clear smooth
Bt_1	25 – 55	Yellowish Brown (10YR5/4)	SCL	Weak fine SBK	Friable	Fine fibrous and medium	Gradual Clear
Bt_2	55 – 91	Yellowish Brown (10YR5/4)	SCL	Weak Fine SB	Friable	Few fine and medium	Clear smooth
Bt_3	91 – 186	Yellowish Brown (10YR5/4)	SCL	Weak Fine SBK	Friable	Very few fine	
UGWUNAGBO (UNSPILLED)							
A	0 -7	Dark Brown (10YR3/3)	SL	Weak fine granular	Loose	Many Fine to Medium	Gradual smooth
AB	7 – 30	Dark Red (7.5YR4/4)	SL	Weak Fine SBK	Friable	Many Fine to Medium	Gradual smooth
Bt_1	30 – 101	Yellowish Red (5YR4/6)	SL	Moderate Fine SBK	Friable	Common and fibrous	Gradual smooth
Bt_2	101 – 190	Red (2.5YR4/6)	SL	Moderate Medium SBK	Friable	Many fine and Medium	

SBK= Subangular Blocky, SL=Sandy Loam, SCL = Sandy Clay Loam,

4.1.2 Soil structure

The soil structure ranged from very weak fine granular to moderate medium subangular blocky in Egbema and Oguta spilled soils whereas their unspilled counterparts were predominantly very fine single grained. The soils of Ugwunagbo were predominated with weak fine subangular blocky structure.

4.1.3 Soil Consistence

The consistence of the soils were very friable (Oguta-unspilled, Egbema-spilled and unspilled), friable (Oguta-spilled and ugwunagbo-spilled) and loose (ugwunagbo-unspilled) in the surface but were generally friable in the subsurface horizon.

4.1.4 Root abundance

There were generally fine and medium roots in pedons of the studied soils.

4.1.5 Boundary

The boundary ranged from gradual smooth to Clear smooth. But had abrupt wavy in the unspilled sites of Imo State (Egbema and Oguta). While it was clear smooth for Ugwunagbo spilled and Gradual smooth for its unspilled counterpart.

4.2 Physical Properties

Results of the physico-chemical properties of the studied soils are shown in Tables 4.2 and 4.3. Data on physical properties are shown o Table 4.2. There were no differences in textures of spilled and unspilled soils at all sampled locations.

TABLE 4.2(a): Physical Properties of Studied Soils.

Horizon	Depth	Sand gkg^{-1}	Silt gkg^{-1}	Clay gkg^{-1}	T C	BD gcm^{-3}	Total Porosity (%)	MC	Silt/Clay RATIO
EGBEMA (SPILLED)									
A	0 – 19	750	120	130	SL	1.20	54	10	0.92
E	19 – 23	710	130	160	SL	1.27	52	15	0.81
Bt1	23 – 60	490	90	420	SC	1.36	48	24	0.21
Bt2	60 – 118	370	80	540	C	1.49	44	26	0.15
Bt3	118 – 212	370	50	580	C	1.55	42	27	0.09
	Mean	**538**	**94**	**366**		**1.374**	**48**	**20.4**	**0.44**
	SD	**183**	**32**	**210**		**0.15**	**5**	**8**	**0.40**
	CV%	**34**	**34**	**57**		**11**	**11**	**37**	**91**
EGBEMA (UNSPILLED)									
A	0 – 15	750	130	120	SL	1.17	56	12	0.92
AB	15 – 54	630	160	210	SCL	1.21	54	14	0.76
Bt1	54 – 90	480	110	410	SC	1.27	52	25	0.27
Bt2	90 – 160	360	80	560	C	1.38	48	32	0.14
Bt3	160 – 210	310	80	600	C	1.48	44	39	0.13
	Mean	**506**	**110**	**382**		**1.30**	**51**	**24.4**	**0.45**
	SD	**184**	**34**	**211**		**0.13**	**5**	**12**	**0.37**
	CV%	**36**	**30**	**54**		**9**	**9**	**47**	**83**

SD= Standard Deviation, TC= Textural Class, BD=Bulk Density, MC= Moisture Content, SL=Sandy Loam, SC=Sandy Clay, SCL=Sandy Clay Loam, C=Clay, CV ≤ 15 = low variation, CV >15 and ≤ 35 = Medium variation, CV > 35 = High variation

TABLE 4.2(b): Physical Properties of Studied Soils contd.

Horizon	Depth	Sand gkg^{-1}	Silt gkg^{-1}	Clay gkg^{-1}	T C	BD gcm^{-3}	Total Porosity (%)	MC	Silt/Clay RATIO
OGUTA (SPILLED)									
A	0 – 18	810	40	150	SL	1.22	54	15	0.27
AB	18 – 52	680	50	270	SCL	1.29	51	18	0.19
Bt1	52 – 99	670	40	290	SCL	1.46	45	20	0.14
Bt2	99 - 190	760	50	190	SL	1.52	43	19	0.26
	Mean	**730**	**45**	**225**		**1.37**	**48**	**18**	**0.21**
	SD	**67**	**6**	**66**		**0.14**	**5**	**2**	**0.06**
	CV%	**9**	**12**	**29**		**10**	**11**	**12**	**29**
OGUTA (UNSPILLED)									
A	0-16	790	50	160	SL	1.2	55	17	0.31
AB	16-67	700	110	190	SL	1.25	53	21	0.58
Bt1	67-125	660	80	260	SCL	1.38	48	25	0.31
Bt2	125-198	750	60	190	SL	1.49	44	19	0.32
	Mean	**725**	**75**	**200**		**1.33**	**50**	**20.5**	**0.38**
	SD	**57**	**26**	**42**		**0.13**	**5**	**3**	**0.13**
	CV%	**8**	**35**	**21**		**10**	**10**	**17**	**35**

SD= Standard Deviation, TC= Textural Class, BD=Bulk Density, MC=Moisture Content, SL=Sandy Loam, SC=Sandy Clay, SCL=Sandy Clay Loam, C=Clay, CV ≤ 15 = low variation, CV >15 and ≤ 35 = Medium variation, CV > 35 = High variation

TABLE 4.2(c): Physical Properties of Studied Soils contd.

Horizon	Depth	Sand gkg^{-1}	Silt gkg^{-1}	Clay gkg^{-1}	T C	BD gcm^{-3}	Total Porosity (%)	MC	Silt/Clay RATIO
UGWUNAGBO (SPILLED)									
A	0-12	740	140	120	SL	1.19	55	11	1.17
AB	12-25	710	150	140	SL	1.26	52	15	1.07
Bt1	25-55	550	180	270	SCL	1.48	44	18	0.67
Bt2	55-91	510	210	280	SCL	1.51	43	19	0.75
Bt3	91-186	450	230	320	SCL	1.56	42	23	0.72
Mean		**592**	**182**	**226**		**1.40**	**47**	**17.2**	**0.87**
SD		**127**	**38**	**90**		**0.16**	**6**	**4**	**0.23**
CV%		**21**	**21**	**40**		**12**	**13**	**26**	**26**
UGWUNAGBO (UNSPILLED)									
A	0-7	750	150	100	SL	1.17	56	17	1.50
AB	7-30	750	120	130	SL	1.28	52	19	0.92
Bt1	30-101	700	130	170	SL	1.36	49	22	0.76
Bt2	101-190	710	130	160	SL	1.49	44	20	0.81
Mean		**727.50**	**132.50**	**140.00**		**1.33**	**50**	**19.50**	**0.95**
SD		**26**	**13**	**32**		**0.13**	**5**	**2**	**0.34**
CV%		**3**	**8**	**20**		**10**	**9**	**9**	**29**

SD= Standard Deviation, TC= Textural Class, BD=Bulk Density, MC=Moisture Content, SL=Sandy Loam, SC=Sandy Clay, SCL=Sandy Clay Loam, C=Clay, CV ≤ 15 = low variation, CV >15 and ≤ 35 = Medium variation, CV > 35 = High variation

Low clay characterized the epipedons at all sites which could be accorded to high rainfall thus eluviation of clay particles from the top soil horizon.

Although, there were slight differences in the textures, yet, from the T-Test (Table 4.5), there was no significant difference between soil textures of spilled and unspilled pedons, while observesd slight differences in particle size distribution could be attributed to intra-pedal properties such as infiltration rates, moisture content and drainage of the soils.

4.2.1 Soil texture

The soils of Egbema ranged from Sandy Loam (SL) to Clay (C). Soils of Oguta were Sandy Loam (SL) with Sandy clay loam (SCL) at the boundaries between A and B horizons, there is no particular trend for Clay distribution down the profile for Oguta profiles. Ugwunagbo had Sandy Loam (SL) and Sandy clay loam (SCL) texture for the spilled site, while the unspilled site was basically Sandy Loam (SL).

Spilled Soils of Oguta recorded the highest value of total sand (810 gkg^{-1}) and the spilled soils of Ugwunagbo recorded the lowest value for total sand (740 gkg^{-1}).

The sand and silt fractions decreased with depth down the pedons for Egbema locations whereas Oguta and Ugwunagbo had no definite trend in distribution down the pedon for both spilled and unspilled sites.

Soils of Egbema varied from Sandy Loam (SL) to Clay (C) for both spilled and unspilled sites, Oguta was Sandy Loam at epipedons, but had Sandy Clay Loam midway and Sandy Loam down the pedons for both spilled and unspilled pedons. Ugwunagbo soils had Sandy Loam for epipedons of both spilled and unspilled pedons, but down the profile (spilled), there was a change to Sandy Clay Loam. whereas the unspilled pedon, was Sandy Loam all through. The sandy nature of all pedons could be attributed to the nature of the parent materials (Subrecent alluvium and coastal plain sands) from which they were derived.

Sand particles slightly varied in Oguta pedons(CV spilled = 9% and CV unspilled=8%) and Ugwunagbo Unspilled (CV=3%), moderately varied in Egbema spilled(CV = 34%) and Ugwunagbo spilled(CV = 21%) whereas it showed high variation in Egbema Unspilled pedon (CV=36%).

4.2.2 Bulk Density

Bulk density increased down the pedon for both spilled and unspilled sites at all locations. It ranged from 1.17gcm^{-3} (Egbema Unspilled and Ugwunagbo Unspilled) to 1.56 gcm^{-3} (Ugwunagbo spilled). These values were in consonance with the values of Savalia *et al* (2009), who noted that bulk density of clay loam and silty loam ranged from 1.1 – 1.4 g/cm^3, clayey soils range from 1.0 – 1.3 g/cm^3 and that of loam, sandy loam and sand ranged from 1.3 – 1.7 g/cm^3. Soils with high porosity have lower bulk densities than those that are more compacted and have less porosity (Brady and Weil, 2010). Generally spilled pedons had higher bulk densities than unspilled ones. This was corroborated by the report of Oyem and Oyem(2013) who reported that bulk density values from their control sites were lower than the spilled sites. The higher bulk densities in spilled soils could be attributed to the impacts of exploration activities. E.g oil spill can clog the soil pores, also the used oh heavy duty machines. Values of bulk densities were lower than values reported by Nkwopara *et al* (2012) who recorded a range of 1.21 – 1.75 gcm^{-3} for spilled soils and 1.31 – 1.75 gcm^{-3} for unspilled soils.

The critical value of bulk density for restricting root growth varies with soil type (Hunt and Gilkes,1992) but in general bulk densities greater than 1.6g/cm^3 tend to restrict root growth (McKenzie *et al*., 2004) thus these soils studied were classified as satisfactory and below the critical values for root restrictions. Though there were differences in values observed, there was no statistical difference between spilled and unspilled pedons as evident from the t-test as shown in Table 4.5 (a).

Bulk density generally showed slight variability (CV <15%) in all pedons (both spilled and unspilled)

4.2.3 Total Porosity

Total Porosity decreased with depth at all studied sites. It was higher in unspilled pedons with values of 51%, 50% and 50% for Egbema, Oguta and Ugwungbo respectively than the spilled pedons (48%, 48% and 47% for Egbema, Oguta and Ugwunagbo respectively). This was corroborated by Owetola (2013) who recorded higher values of total porosity in their control site in comparison to the spilled sites. Total porosity showed slight variability in all the pedons (11%, 9%, 11%, 10%, 13% and 9% in Egbema spilled, Egbema unspilled, Oguta spilled, Oguta unspilled, Ugwunagbo spilled and Ugwunagbo Unspilled respectively). Though there were differences in results obtained, but from the results of the t-test in Table 4.5 (a), there was no significant difference ($p>0.05$) between values of spilled and unspilled pedons.

4.2.4 Moisture content

Moisture content was lower at the epipedons in all the pedons. Results showed that values of moisture content was higher in unspilled pedons than spilled pedons. This result was in consonance with the reports of Ohanmu *et al* (2018), who reported that there was a decrease in moisture content with crude oil pollution, and the reduction is inversely related to the level of pollution. Moisture Content showed high variability in Egbema pedons (spilled-37% and unspilled-47%), moderately variable in Oguta Unspilled (17%) and Ugwunagbo spilled (26%) and slightly variable in Ugwunagbo unspilled (9%) and Oguta spilled (12%). T-test result in Table 4.5 (a) showed no significant difference between spilled and unspilled pedons.

4.2.5 Silt / Clay Ratio

For Silt/Clay ratio, there was a decrease with increase in depth for the soils of Egbema and Ugwunagbo, but there was no particular trend for the soils of Oguta. The silt/clay ratio was generally low (<1) and according to Essoka and Esu (2000), it indicates that the soils are highly weathered; this could be attributed to the prevailing weather conditions of high rainfall intensity and high temperature which favours weathering.

4.3.0 Chemical properties of studied soils

Results of some chemical properties of the studied soils are shown in Table 4.3.

4.3.1 Soil pH

Soil pH measured in distilled water was higher than pH measured in 1NKCl, this resulted to a negative pH ($pH_{KCl} - pH_{Water}$), which indicates the dominance of silicate clay instead of sesquioxides, also a negative charge on the adsorption complex of the studied soils (Woods *et al*,. 2008). Generally, the soil pH was low. This could be attributed to high amount of rainfall that had leached basic cations thus the exchange complex of the soils is dominated by acidic cations.

Soils from spilled pedon at Oguta showed higher pH values than the unspilled pedons.

TABLE 4.3(a): Chemical properties of studied soils

Horizon	Depth (Cm)	pH H_2O	pH KCl	OC g/Kg	OM g/Kg	TN g/Kg	Av. P mg/Kg	Ca	Mg	K	Na Cmol/Kg	TEB	CEC	ECEC	BS %	H^+	Al^{3+}	TEA	Al sat %	Ca/Mg
EGBEMA (SPILLED)																				
A	0 – 19	5.3	4.4	16	27.6	0.4	3.3	0.9	0.41	0.51	0.1	1.92	5.8	4.29	33	1.15	1.22	2.37	21	2.20
E	19 – 23	4.8	4	9	15.5	0.1	1.1	0.71	0.21	0.4	0.06	1.38	4.3	2.89	32	0.38	1.13	1.51	26	3.38
Bt1	23 – 60	5	4.2	5	8.6	0.1	3	0.83	0.34	0.42	0.1	1.69	4.6	3.71	37	0.16	1.86	2.02	40	2.44
Bt2	60 – 118	4.9	4.1	3	5.2	0.1	2.8	1.1	0.38	0.43	0.13	2.04	5.5	4.09	37	0.11	1.94	2.05	35	2.89
Bt3	118 – 212	4.8	4.1	2	3.5	0.1	2.1	1.3	0.39	0.48	0.14	2.31	7.9	5.4	29	0.96	2.13	3.09	27	3.33
Mean		**4.96**	**4.16**	**7**	**12.1**	**0.16**	**2.46**	**0.97**	**0.35**	**0.45**	**0.11**	**1.88**	**1.88**	**4.08**	**33.6**	**0.55**	**1.66**	**2.21**	**29.8**	**2.85**
SD		**0.21**	**0.15**	**6**	**10**	**0.13**	**0.88**	**0.23**	**0.08**	**0.05**	**0.03**	**0.35**	**1**	**0.91**	**3**	**0.48**	**0.45**	**0.58**	**8**	**0.53**
CV%		**4**	**4**	**81**	**81**	**84**	**36**	**24**	**23**	**10**	**30**	**21.75**	**25**	**22**	**10**	**86**	**27**	**56.5**	**26**	**19**
EGBEMA (UNSPILLED)																				
A	0 – 15	5.6	5.2	22	37.9	0.6	5.2	1.21	0.66	0.9	0.3	3.07	7.2	5.7	54	1.23	1.4	2.63	25	1.83
AB	15 – 54	5.4	5.1	19	32.8	0.3	4.5	1.1	0.45	0.6	0.15	2.30	5.6	4.6	50	1.2	1.1	2.30	24	2.44
Bt1	54 – 90	5.1	4.9	12	20.7	0.4	4.1	0.96	0.41	0.52	0.14	2.03	5	4.13	49	1	1.1	2.10	27	2.34
Bt2	90 – 160	5	4.5	8	13.8	0.2	3.5	1.16	0.4	0.6	0.17	2.33	4.4	4.09	57	0.84	0.92	1.76	22	2.9
Bt3	160 – 210	4.8	4.3	5	8.6	0.2	2.8	1.3	0.42	0.48	0.6	2.80	6.2	5.21	53	1.1	1.31	2.41	25	3.10
Mean		**5.18**	**4.8**	**13.2**	**22.8**	**0.34**	**4.02**	**1.15**	**0.47**	**0.62**	**0.27**	**2.51**	**2.51**	**4.75**	**52.6**	**1.07**	**1.17**	**2.24**	**24.6**	**2.52**
SD		**0.3**	**0.4**	**7**	**12.4**	**0.2**	**0.9**	**0.1**	**0.1**	**0.2**	**0.2**	**0.4**	**1**	**0.7**	**3**	**0.2**	**0.2**	**0.3**	**2**	**0.5**
CV%		**6**	**8**	**55**	**55**	**49**	**23**	**11**	**23**	**27**	**72**	**33.25**	**33.25**	**15**	**6**	**15**	**16**	**15.50**	**7**	**20**

SD = Standard Deviation, OC=Organic Carbon, OM=Organic Matter, TN=Total Nitrogen, Av. P=Available Phosphorus, BS=Base Saturation, Ca=Calcium, CEC=Cation Exchange Capacity, ECEC=Effective Cation Exchange Capacity, Al Sat = Aluminium Saturation

TABLE 4.3(b): Chemical properties of studied soils contd.

Horizon	Depth (Cm)	pH H$_2$O	pH KCl	OC g/Kg	OM g/Kg	TN g/Kg	Av. P mg /Kg	Ca	Mg	K	Na Cmol /Kg	TEB	CEC	ECEC	BS %	H^+	Al^{3+}	TEA	Al sat %	Ca/ Mg
OGUTA (SPILLED)																				
A	0 – 18	6.3	5.4	30	51.7	1	12.8	1.11	0.41	0.1	0.03	1.65	9.2	5.75	18	1.8	2.3	4.10	25	2.71
AB	18 - -52	4.9	4	9	15.5	0.5	10.2	1.2	0.3	0.13	0.03	1.66	7.2	5.97	23	1.2	3.11	4.31	43	4.00
Bt1	52 – 99	5.1	4.2	8	13.8	0.3	4.8	1.01	0.31	0.14	0.06	1.52	6.8	4.79	22	0.96	2.31	3.27	33.9	3.26
Bt2	99 - 190	5	4.2	4	6.90	0.1	2	0.61	0.11	0.14	0.06	0.92	2.3	2.03	39	0.01	1.1	1.11	47.8	5.55
Mean		**5.33**	**4.5**	**12.8**	**22.0**	**0.48**	**7.45**	**0.98**	**0.28**	**0.13**	**0.05**	**1.44**	**6.38**	**4.64**	**25.5**	**0.99**	**2.21**	**3.20**	**37.43**	**3.88**
SD		**0.66**	**0.64**	**12**	**20**	**0.39**	**5**	**0.26**	**0.13**	**0.02**	**0.02**	**0.35**	**3**	**2**	**9**	**0.74**	**0.83**	**1**	**10**	**1**
CV%		**12**	**14**	**92**	**92**	**81**	**66**	**27**	**44**	**15**	**39**	**31**	**46**	**39**	**36**	**75**	**38**	**57**	**27**	**32**
OGUTA (UNSPILLED)																				
A	0-16	5.80	5.20	35.00	60.34	1.20	15.20	1.24	0.62	0.34	0.08	2.28	10.20	6.79	33.58	2.11	2.40	4.51	35.35	2.00
AB	16-67	5.40	4.80	29.00	50.00	0.70	13.70	1.17	0.48	0.25	0.06	1.96	9.57	5.74	34.15	1.77	2.01	3.78	35.02	2.44
Bt1	67-125	5.10	4.10	22.00	37.93	0.20	6.40	1.03	0.50	0.19	0.03	1.75	7.86	4.71	37.15	1.24	1.72	2.96	36.52	2.06
Bt2	125-198	4.90	4.00	17.00	29.31	0.20	3.20	0.80	0.30	0.11	0.02	1.23	4.30	3.15	39.05	0.80	1.12	1.92	35.56	2.67
Mean		**5.30**	**4.53**	**25.75**	**44.39**	**0.58**	**9.63**	**1.06**	**0.48**	**0.22**	**0.05**	**1.81**	**7.98**	**5.10**	**35.98**	**1.48**	**1.81**	**3.29**	**35.61**	**2.29**
SD		**0.39**	**0.57**	**8**	**14**	**0.48**	**6**	**0.19**	**0.13**	**0.10**	**0.03**	**0.44**	**3**	**2**	**3**	**0.58**	**0.54**	**1**	**0.64**	**0.32**
CV%		**7**	**13**	**31**	**31**	**83**	**60**	**18**	**28**	**44**	**58**	**37**	**33**	**30**	**7**	**39**	**30**	**35**	**2**	**14**

SD = Standard Deviation, OC=Organic Carbon, OM=Organic Matter, TN=Total Nitrogen, Av. P=Available Phosphorus, BS=Base Saturation, Ca=Calcium, CEC=Cation Exchange Capacity, ECEC=Effective Cation Exchange Capacity, Al Sat = Aluminium Saturation

TABLE 4.3(c): Chemical properties of studied soils contd.

Horizon	Depth (Cm)	pH H_2O	pH KCl	OC g/Kg	OM g/Kg	TN g/Kg	Av. P mg /Kg	Ca	Mg	K	Na Cmol /Kg	TEB	CEC	ECEC	BS %	H^+	Al^{3+}	TEA	Al sat %	Ca/ Mg
UGWUNAGBO (SPILLED)																				
A	0-12	5	4.1	24	41.4	1.6	30	1.31	0.7	0.3	0.04	2.35	8.4	5.14	27	0.18	2.61	2.79	31	1.87
AB	12-25	4.9	4	14	24.1	1.1	5.4	1.02	0.51	0.2	0.03	1.76	6	4.64	29	0.54	1.9	2.44	32	2.00
Bt1	25-55	4.8	4	7.8	13.5	0.7	6.3	1.11	0.41	0.16	0.04	1.72	5.8	3.89	29	0.19	2	2.19	34	2.71
Bt2	55-91	5	4.2	5.4	9.31	0.4	6.8	2.3	0.61	0.19	0.05	3.15	5.2	4.35	60	0.14	1.1	1.24	21	3.77
Bt3	91-186	5.2	4.3	0.9	1.55	0.2	8.2	2.41	0.7	0.2	0.06	3.37	5.8	4.47	58	0.12	0.98	1.10	16.9	3.44
Mean		**4.98**	**4.10**	**10.40**	**18.00**	**0.80**	**11.34**	**1.63**	**0.59**	**0.21**	**0.04**	**2.47**	**6.24**	**4.50**	**40.60**	**0.23**	**1.72**	**1.95**	**26.98**	**2.76**
SD		**0.15**	**0.13**	**9**	**15**	**0.56**	**10**	**0.67**	**0.13**	**0.05**	**0.01**	**0.77**	**1**	**0.45**	**17**	**0.17**	**0.68**	**0.75**	**8**	**0.84**
CV%		**3**	**3**	**86**	**86**	**70**	**92**	**41**	**22**	**25**	**26**	**29**	**20**	**10**	**42**	**74**	**39**	**56.50**	**28**	**31**
UGWUNAGBO (UNSPILLED)																				
A	0 – 18	5.4	4.7	22.6	38.9	1.4	35	2.11	0.96	0.28	0.02	3.37	13.6	5.34	63	0.16	1.81	1.97	33	2.20
AB	18 - -52	5.1	4.5	14.2	24.5	0.9	11	1.16	0.42	0.18	0.02	1.78	11.8	3.76	47	0.22	1.76	1.98	47	2.76
Bt1	52 – 99	5	4.3	7.3	12.6	0.5	12.6	1.91	0.6	0.22	0.02	2.75	14.8	3.96	69	0.1	1.11	1.21	28	3.18
Bt2	99 - 190	4.8	4.3	2.1	3.6	0.1	9.2	1.72	0.58	0.19	0.01	2.50	13.2	3.36	74	0.08	0.78	0.86	23	2.97
Mean		**5.08**	**4.5**	**11.55**	**19.91**	**0.73**	**16.95**	**1.73**	**0.64**	**0.22**	**0.02**	**2.61**	**13.35**	**4.11**	**63.25**	**0.14**	**1.37**	**1.51**	**32.75**	**2.78**
SD		**0.25**	**0.19**	**9**	**15**	**0.56**	**12**	**0.41**	**0.23**	**0.04**	**0.01**	**0.66**	**1**	**0.86**	**12**	**0.06**	**0.50**	**0.56**	**10**	**0.42**
CV%		**4.27**	**3.7**	**77**	**77**	**77**	**72**	**24**	**36**	**21**	**29**	**28**	**9**	**21**	**19**	**45**	**37**	**41**	**31**	**15**

SD = Standard Deviation, OC=Organic Carbon, OM=Organic Matter, TN=Total Nitrogen, Av. P=Available Phosphorus, BS=Base Saturation, Ca=Calcium, CEC=Cation Exchange Capacity, ECEC=Effective Cation Exchange Capacity, Al Sat = Aluminium Saturation

This is in agreement with earlier observations by Osuji and Onojake (2006) who reported higher pH values in spilled pedons when compared with unspilled pedons, whereas spilled pedons of Egbema and Ugwunagbo were more acidic when compared to the unspilled pedons. This later observation was in consonance with the observations of Nkwopara *et al* (2012) who reported low pH value for oil affected pedons.

pH values slightly varied in all sites studied with values (CV=4%, 8%, 14%, 13%, 3%, and 4%) for Egbema spilled and Unspilled, Oguta spilled and unspilled, Ugwunagbo spilled and unspilled respectively. The t-test results in Table 4.5 (b) show that there was a significant difference in pH(KCl) between spilled and unspilled pedons, thus pollution affects the pH of soils.

4.3.2 Organic Carbon

Organic carbon (OC) generally decreased with depth in all pedons, thus higher values were recorded in surface horizon of all pedons. The values ranged from 16 – 35g/kg, with mean values of (7 g/kg, 12.8 g/kg, 10.40 g/kg) for spilled and (13.2 g/kg, 25.75 g/kg and 11.55 g/kg) for unspilled in Egbema, Oguta and Ugwunagbo respectively. Organic Carbon distribution varied with locations. Soils from spilled locations in Egbema and Oguta showed lower Organic Carbon than soils from unspilled sites. This was in consonance with the observations of Nkwopara *et al*, (2012); who opined that low Organic Carbon content of spilled sites could be attributed to longer time of pollution as the amount of carbon reduces with time. Spilled soils from Ugwunagbo showed higher Organic Carbon content than unspilled soils. This situation could be viewed as crude oil filling the pore spaces thus denying microbes of oxygen hence they die as most are aerobic. Sequel to this, the organic matter decomposition slows down and there is bound to be an increase in organic matter of spilled soils. This is in line with the findings of Essien and John (2010), who revealed an increase of up to 51% in spilled soils. Generally, Sim (1990) attributed low organic matter in the tropics to high temperatures which hasten mineralization process in organic matter. Organic carbon highly varied in all studied pedons having Coefficient of Variability (CV) values >55% (Table 4.3 (a,b,c)). T-test results in Table 4.5 (b) showed that there was a significant difference in organic Carbon (OC) for spilled and unspilled soils at a probability level of 0.05, thus pollution significantly affects OC composition in soils.

4.3.3 Total Nitrogen

The values of Total Nitrogen were generally low (0.4 – 1.6g/kg) in the studied soils. According to Opukiri *et al* (1991) this could be attributed to leaching after heavy rainfall.

From the ratings of Beernaert and Bitondo (1992), the value of Total Nitrogen (TN) was very low (<1g/kg) in Egbema soils and Low (1 – 2g/kg) in soils of Oguta and Ugwunagbo pedons. Between spilled and unspilled soils, Total Nitrogen was higher in unspilled soils at Egbema and Oguta whereas it was lower in unspilled soils of Ugwunagbo.

The higher TN in spilled soils could be attributed to C:N imbalance created by the presence of petroleum hydrocarbons which can inhibit the activity of nitrogen-fixing bacteria and other microbial populations involved in nitrogen transformations (Ayotamuno *et al*, 2006). As a result, nitrogen may accumulate in the soil in its organic or inorganic forms, leading to higher TN levels. But with time, there is a reduction in the oil concentration and thus possible increased microbial activity which is what could have accounted for the situation in Ugwunagbo soils where the Total Nitrogen was lower in spilled pedons than their unspilled counterparts (Ayotamuno *et al*, 2006). Generally, the amount of Total Nitrogen decreased down the pedon for all three (3) locations this was corroborated by Zhijing *et al* (2013). There was no significant difference between spilled and unspilled pedons.

Total Nitrogen highly varied in all studied pedons having Coefficient of Variability (CV) values of 84%, 49%, 81%, 83%, 70% and 77% for Egbema spilled and Unspilled, Oguta spilled and Unspilled, Ugwunagbo spilled and Unspilled respectively.

4.3.4 Available Phosphorus

According to the ratings of Beernaert and Bitondo, (1992), Available Phosphorus of the soils were classified as low (<5mg/kg) for Egbema, medium (5-15mg/kg) for Oguta and high (>15mg/kg) for Ugwunagbo. Available Phosphorus decreased down the pedon at most sites (Unspilled site at Egbema, spilled and unspilled pedons at Oguta) but did not show any particular trend down the profile at other sites (Ugwunagbo-spilled and unspilled and Egbema spilled). Though, available phosphorus was higher in unspilled soils (4.02 $mgKg^{-1}$, 9.63 $mgKg^{-1}$, 16.95 $mgKg^{-1}$) than their spilled counterparts (2.46 $mgKg^{-1}$, 7.45 $mgKg^{-1}$, 11.34 $mgKg^{-1}$) for Egbema, Oguta and Ugwunagbo respectively, the difference was not significant (Table 4.5b). This corroborates Abii and Nwosu (2009) who reported lower Available P in spilled pedons. Nnaji *et al*, (2002) opined that the low available P could be attributed to low pH of spilled soils resulting in P-fixation.

4.3.5 Exchangeable Bases

Calcium (Ca) and Magnesium(Mg) were very low at five (5) sites (Egbema spilled and unspilled, Oguta spilled and unspilled and ugwunagbo spilled) but was low at Ugwunagbo unspilled site. Generally, values of Ca and Mg were higher in unspilled soils than spilled soils. This corroborates the findings of Abii and Nwosu (2009). Potassium (K) and Sodium (Na) were higher spilled pedons than unspilled pedons.

Exchangeable calcium had higher values in the unspilled pedons when compared with their spilled counterparts having mean values of 0.97 cmol/Kg, 1.15 cmol/Kg, 0.98 cmol/Kg, 1.06 cmol/Kg, 1.63 cmol/Kg and 1.73 cmol/Kg in Egbema (Spilled and

Unspilled), Oguta (Spilled and Unspilled), Ugwunagbo (Spilled and Unpollluted) respectively. This is in agreement with the findings of Abii and Nwosu (2009) who opined that Ca^{2+}, K^{+} and Available P decrease with oil pollution. Beernaert and Bitondo (1992) rated exchangeable calcium as Very Low (< 2cmolkg^{-1}). This suggests that the values obtained for these soils were generally very low and could be attributed to the parent material from which these soils where formed. This finding corroborated the works of Onweremadu *et al* (2011) who reported low calcium for Coastal plain sands and Subrecent alluvium in Southeastern Nigeria. Generally, there was no particular trend for calcium in the soil studied with profile depth. Calcium was highly variable (CV=41%) in Ugwunagbo spilled, slightly variable (CV=11%) in Egbema Unspilled and moderately variable (15% < CV < 35) for other pedons.

Exchangeable magnesium had higher values in the unspilled pedons when compared with the spilled soils. Exchangeable magnesium had mean values of 0.35 cmol/Kg, 0.47 cmol/Kg, 0.28 cmol/Kg, 0.48 cmol/Kg, 0.59 cmol/Kg and 0.64 cmol/Kg in Egbema (Spilled and Unspilled), Oguta (Spilled and Unspilled), Ugwunagbo (Spilled and unspilled) respectively which according to the ratings of Beernaert and Bitondo (1992), suggests that the values obtained from the samples varied from very low (<0.5 cmolkg^{-1}) in Egbema and Oguta (Imo) to low (0.5 – 1.5 cmolkg^{-1}) in Ugwunagbo (Abia). This was explained by Narayanan (2011) who opined that the continuous percolation of water for a very long time, could result in leaching of relatively soluble bases, alkaline earth metals and some silica. Generally, there was no particular trend for magnesium in the studied soils down the pedon. Values were highly variable in Oguta spilled (CV = 44%) and Ugwunagbo unspilled(CV = 36%) but moderately variable (23%, 23%, 28% and 22%) for other pedons (Egbema spilled and unspilled, Oguta Unspilled, Ugwunagbo spilled) respectively.

Exchangeable sodium had higher values in the unspilled pedons when compared with their unspilled pedons in Egbema pedons, but was higher in spilled than in unspilled pedon for Oguta and Ugwunagbo. Sodium had mean values of 0.11 cmol/Kg, 0.27 cmol/Kg, 0.05 cmol/Kg, 0.0.05 cmol/Kg, 0.04 cmol/Kg and 0.02 cmol/Kg in Egbema (Spilled and Unspilled), Oguta (Spilled and Unspilled), Ugwunagbo (Spilled and Unapollluted) respectively. Beernaert and Bitondo (1992) classified exchangeable sodium as Very Low (< 0.1), Low (0.1 – 0.3), Medium (0.3 – 0.7), High (0.7 – 2.0) and very high (> 2) cmol/Kg. This suggests that the values obtained from the studied samples varied from very low in Oguta and Ugwunagbo to low in Egbema. The low sodium content implies that the soils are not sodic. Samples were highly variable in Egbema Unspilled (CV=72%), Oguta spilled (CV=39%) and Unspilled (CV=58%) while it was moderately variable in other pedons (Egbema spilled=30%, Oguta spilled=39%, Ugwunagbo spilled=26% and Unspilled=29%).

Exchangeable potassium had higher values in the unspilled than spilled pedons. Mean values of spilled pedons(0.45 cmol/Kg, 0.13 cmol/Kg, 0.21 cmol/Kg) and unspilled

(0.62 cmol/Kg, 0.22 cmol/Kg, and 0.22 cmol/Kg) were obtained in Egbema, Oguta and Ugwunagbo respectively. The lower concentration in spilled pedon is in consonance with the works of Abii and Nwosu (2009) who reported that there was a significant decrease in potassium concentration in spilled soils. The ratings of Beernaert and Bitondo (1992) classified exchangeable potassium as Very Low (< 0.1), Low (0.1 – 0.3), Medium (0.3 – 0.6), High (0.6 – 1.2) and very high (> 1.2) cmol/Kg. This suggests that the values obtained from the soils studied varied from low in Oguta and Ugwunagbo through medium (Egbema Spilled) to High (Egbema unspilled). It shows slight variability in Egbema spilled (10%) and Oguta spilled (15%), moderate variability in Egbema unspilled (27%), Ugwunagbo spilled (25%) and unspilled (21%), while it was highly variable in Oguta Unspilled (58%).

4.3.6 Cation Exchange Capacity

Cation Exchange Capacity recorded higher values in the unspilled pedons than their spilled counterparts having mean values of 1.88 cmol/Kg, 2.51cmol/Kg, 6.38cmol/Kg, 7.98 cmol/Kg, 6.24 cmol/Kg and 13.35cmol/Kg in Egbema (spilled and unspilled), Oguta (spilled and unspilled), Ugwunagbo (spilled and unspilled) respectively. Beernaert and Bitondo (1992) classified Cation Exchange Capacity as Very Low (< 20), Low (21 – 40), Medium (41 –60), High (61 – 80) and very high (81 – 100) cmol/Kg. Thus, the values obtained from the studied soils were generally very low. The values of

CEC of the studied soils showed high variation in Oguta (spilled =46%), moderate variation in Egbema (spilled=25%, unspilled = 33%), Oguta (unspilled = 33%) and Ugwunagbo spilled(CV=20%) , and slight variation in Ugwunagbo unspilled(CV = 9%). T-test results in Table 4.5(b) showed a significant difference between CEC for spilled and unspilled pedons at 0.05 probability level.

4.3.7 Effective Cation Exchange Capacity

Effective Cation Exchange Capacity recorded higher values in the unspilled pedons than their spilled counterparts having mean values of 4.08 cmol/Kg, 4.75 cmol/Kg, 4.64 cmol/Kg, 5.10 cmol/Kg, 4.50 cmol/Kg and 4.71 cmol/Kg in Egbema (Spilled), Egbema (Unspilled), Oguta (Spilled), Oguta (Unspilled), Ugwunagbo (Spilled) and Ugwunagbo (Unspilled) respectively. This agrees with Abii and Nwoso (2009) who opined that pollution causes a significant decrease in effective cation exchange capacity. Esu (1999) classified ECEC as Low(< 6), Medium (6 – 12) and High (> 12) cmol/Kg. This suggests that the values obtained from the studied samples are low in all locations. This could be not only due to heavy rainfall that characterizes the study area and thus leaching of basic cations, but also the parent material from which the soils were derived. This result is in agreement with the reports of Onweremadu *et al*. (2011) who also reported low ECEC for sub recent alluvium and coastal plain sands in Southeastern Nigeria. There was no particular trend for ECEC with increase in depth for individual pedon.

4.3.8 Base Saturation

Percentage Base saturation had higher values in the unspilled pedons than spilled pedons, having mean values of 33.6 %, 52.6 %, 25.5 %, 35.98%, 40.60% and 63.25% in soils of Egbema (Spilled), Egbema (Unspilled), Oguta (Spilled), Oguta (Unspilled), Ugwunagbo (Spilled) and Ugwunagbo (Unpollluted) respectively. Food and Agricultural organization (FAO), (2004) classified Base saturation as very low (0 – 20%), low (20 – 40%), medium (40 – 60%), high (60 - 80) and very high (>80). From these, the values obtained from the studied samples could be classified as low (Egbema(spilled), Oguta (spilled and Unspilled)), medium (Egbema (Unspilled) and Ugwunagbo (spilled)) and High (Ugwunagbo Unspilled). The low Base Saturation could be attributed to leaching due to heavy rainfall that lasts for up to 9 months and estimated to about 2400mm (Oti, 2002). Base Saturation values showed slight variation in Egbema (spilled CV=10% and Unspilled CV=6%) and Oguta Unspilled (CV=7%), moderate variation in Ugwunagbo unspilled (19%) and high variation in Oguta spilled soils (CV=36%) and Ugwunagbo spilled (CV = 42%). Results of T-test (Table 4.5 (b)) showed that there was significant difference in Base Saturation between spilled and unspilled pedons thus indicating that crude oil pollution influenced the base saturation of a soil.

4.3.9 Aluminium saturation

Aluminium saturation recorded mean values of 29.8 %, 24.6 %, 37.43 %, 35.61%, 26.98% and 32.75% in soils of Egbema (Spilled), Egbema (Unspilled), Oguta (Spilled), Oguta (Unspilled), Ugwunagbo (Spilled) and Ugwunagbo (Unspilled) respectively. The values are considered generally low as they are below 50% (Onweremadu *et al.,* 2011).

4.3.10 Calcium / magnesium ratio

The Ca:Mg ratio had mean values for spilled(2.85, 3.88, 2.76) and for unspilled(2.52, 2.29, 2.78) in soils of Egbema, Oguta and Ugwunagbos respectively. The normal range of Ca:Mg ratio in soils is 3 – 5, values less than 3 will result to Ca and P inhibition thus Ca deficiency while values greater than 5 result to Mg and P inhibition (Udo *et al*., 2009). Thus the results obtained indicate an unfertile soil. This was corroborated by Landon (1991) who reported ratios less than 3 .0 as typical for unfertile soils.

4.4.0 Concentration of heavy metals in the studied soils

Tables 4.4 show the concentration of heavy metal in studied soils. Generally, there was a higher amount of heavy metal in spilled soils at the three locations than unspilled soils. This is in consonance with the findings of Nkwopara *et al* (2012) who reported higher

TABLE 4.4(a): Heavy metal concentration of studied soils

Horizon	Depth *(Cm)*	Pb *mg /Kg*	Cr *mg /Kg*	Cd *mg /Kg*	Ni *mg /Kg*
EGBEMA (SPILLED)					
A	0 – 19	0.18	0.09	0.02	5.56
E	19 – 23	0.08	0.07	0.01	2.23
Bt1	23 – 60	0.09	0.08	0.008	2.82
Bt2	60 – 118	0.03	0.02	0.003	0.48
Bt3	118 – 212	0.01	0.01	0.001	0.15
	Mean	**0.08**	**0.05**	**0.01**	**2.25**
	SD	**0.066**	**0.036**	**0.007**	**2**
	CV%	**85**	**68**	**89**	**97**
EGBEMA (UNSPILLED)					
A	0 – 15	0.12	0.06	0.015	4.22
AB	15 – 54	0.1	0.04	0.009	3.61
Bt1	54 – 90	0.07	0.02	0.006	2.72
Bt2	90 – 160	0.04	0.02	0.002	1.13
Bt3	160 – 210	0.02	0.01	0.001	0.10
	Mean	**0.07**	**0.03**	**0.007**	**2.36**
	SD	**0.041**	**0.020**	**0.006**	**2**
	CV%	**59**	**67**	**86**	**73**

Pb = Lead, Cr = Chromium, Cd = Cadmium, Ni = Nickel, SD= Standard Deviation,
CV ≤ 15 = low variation, CV >15 and ≤ 35 = Medium variation, CV > 35 = High variation

TABLE 4.4(b): Heavy metal concentration of studied soils contd.

Horizon	Depth *(Cm)*	Pb *mg /Kg*	Cr *mg /Kg*	Cd *mg /Kg*	Ni *mg /Kg*
OGUTA (SPILLED)					
A	0 – 18	0.41	0.12	0.046	7.88
AB	18 - -52	0.29	0.07	0.018	4.10
Bt1	52 – 99	0.08	0.03	0.010	1.80
Bt2	99 - 190	0.02	0.01	0.003	0.16
Mean		**0.20**	**0.06**	**0.019**	**3.49**
SD		**0.18**	**0.05**	**0.02**	**3**
CV%		**91**	**84**	**98**	**96**
OGUTA (UNSPILLED)					
A	0-16	0.12	0.05	0.011	2.08
AB	16-67	0.08	0.03	0.008	2.10
Bt1	67-125	0.05	0.01	0.005	1.20
Bt2	125-198	0.01	0.01	0.001	0.12
Mean		**0.07**	**0.03**	**0.006**	**1.38**
SD		**0.047**	**0.019**	**0.004**	**0.936**
CV%		**72**	**77**	**68**	**68**

Pb = Lead, Cr = Chromium, Cd = Cadmium, Ni = Nickel, SD= Standard Deviation,
CV ≤ 15 = low variation, CV >15 and ≤ 35 = Medium variation, CV > 35 = High variation

TABLE 4.4(c): Heavy metal concentration of studied soils contd.

Horizon	Depth *(Cm)*	Pb *mg /Kg*	Cr *mg /Kg*	Cd *mg /Kg*	Ni *mg /Kg*
UGWUNAGBO(SPILLED)					
A	0-12	0.29	0.09	0.03	4.70
AB	12-25	0.22	0.06	0.02	3.80
Bt1	25-55	0.09	0.02	0.008	2.10
Bt2	55-91	0.03	0.02	0.003	0.18
Bt3	91-186	0.01	0.01	0.001	0.02
	Mean	**0.01**	**0.04**	**0.01**	**2.16**
	SD	**0.122**	**0.034**	**0.012**	**2**
	CV%	**95**	**85**	**99**	**97**
UGWUNAGBO (UNSPILLED)					
A	0 – 18	0.11	0.03	0.020	2.12
AB	18 - -52	0.09	0.02	0.010	1.85
Bt1	52 – 99	0.06	0.01	0.010	0.42
Bt2	99 - 190	0.01	0.01	0.010	0.08
	Mean	**0.07**	**0.02**	**0.013**	**1.12**
	SD	**0.043**	**0.010**	**0.005**	**1**
	CV%	**64**	**55**	**40**	**91**

Pb = Lead, Cr = Chromium, Cd = Cadmium, Ni = Nickel, SD= Standard Deviation,
CV ≤ 15 = low variation, CV >15 and ≤ 35 = Medium variation, CV > 35 = High variation

values of heavy metals in soils affected by pollution than in unspilled soils. The heavy metals decreased down the pedon, thus corroborating Osuji and Onojake(2006). The higher concentrations of these heavy metals at the epipedons could be attributed to the fact that metals in spilled soils, penetrate only a little below 10cm depth even after many years. (Smith *et al*, 1999).

According to Ahalya *et al* (2003), crude Oil contains Mercury, Lead, Vanadium and Chromium which could possibly have been added to the soil during the process of oil spillage, thus this could be said to account for the higher concentrations of these heavy metals in spilled pedons than the unspilled pedons.

The concentration of Lead, Nickel, Chromium and Cadmium was very low and within the permissible limits for unrestricted use when compared with both local and international standards.

The magnitude of the Heavy metals followed the following trend Ni > Pb > Cd > Cr for all spilled soils and Ni > Pb > Cr > Cd for unspilled soils.

4.4.1 Concentration of Lead (Pb)

Lead concentration reduces with increase in depth for all studied pedons, showing high variability (CV > 35%) with depth. The concentration of Lead in the studied soils was 0.08 mg/Kg, 0.20 mg/Kg and 0.01 mg/Kg for the spilled soils of Egbema, Oguta and Ugwunagbo respectively. These concentration were higher in comparison to unspilled

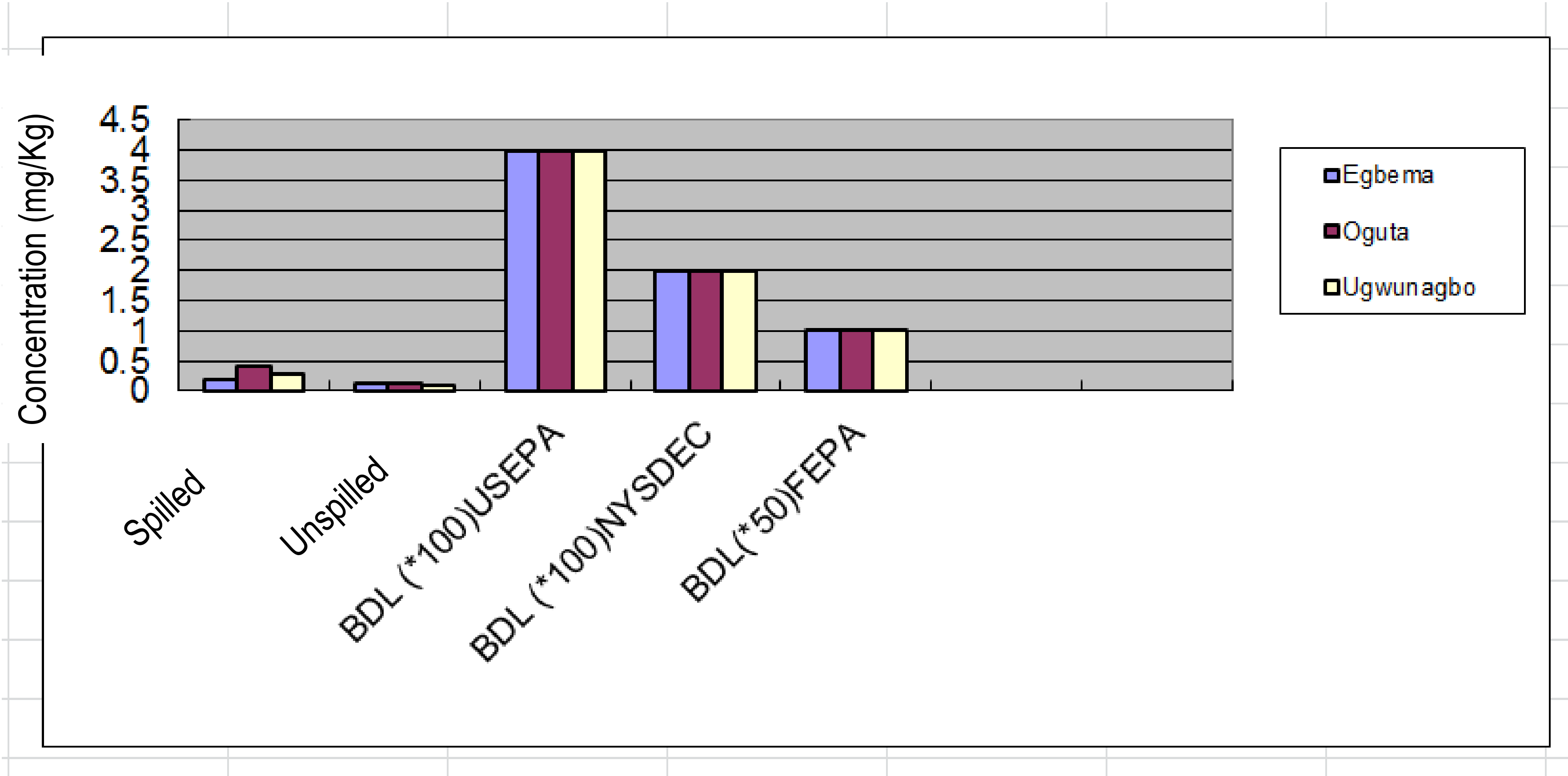

Figure 5. Lead level in spilled and unspilled soils compared with standards

pedons (0.07 mg/Kg, 0.07 mg/Kg, 0.07 mg/Kg). The values were less when compared with the level requiring clean-up (400mg/Kg) as stated by United States Environmental Protection Agency and the level permitted for unrestricted use, including agriculture (200mg/Kg) by NYS DEC (Vern and Don, 2011). Also it was below the Federal Environmental Protection Agency standard (50 mg/Kg) in Nigeria requiring clean-up. (FEPA, 1991).

Comparing the different locations (Egbema, Oguta and Ugwunagbo) using mean value, Lead concentration could be summarized as Oguta (0.20 mg/Kg) > Egbema (0.08 mg/Kg) > Ugwunagbo (0.01 mg/Kg) for spilled pedons but was same for unspilled pedon (0.7mg/Kg).

4.4.2 Concentration of Chromium (Cr)

Chromium decreased with increase in depth. The concentration of Chromium in all soils ranged from 0.04 mg/Kg to 0.06 mg/Kg in spilled soils. Chromium values were higher for spilled soils when compared against the unspilled (0.02 – 0.03 mg/Kg). This is in consonance with the results of Nkwopara *et al* (2012) who reported higher values of Chromium in spilled than unspilled soils. But are less when compared with the level requiring clean-up as stated by United States Environmental Protection Agency (230mg/Kg) and the level permitted for unrestricted use i.e including agriculture by NYS DEC (11mg/Kg) (Vern and Don, 2011). Also it was below the Federal

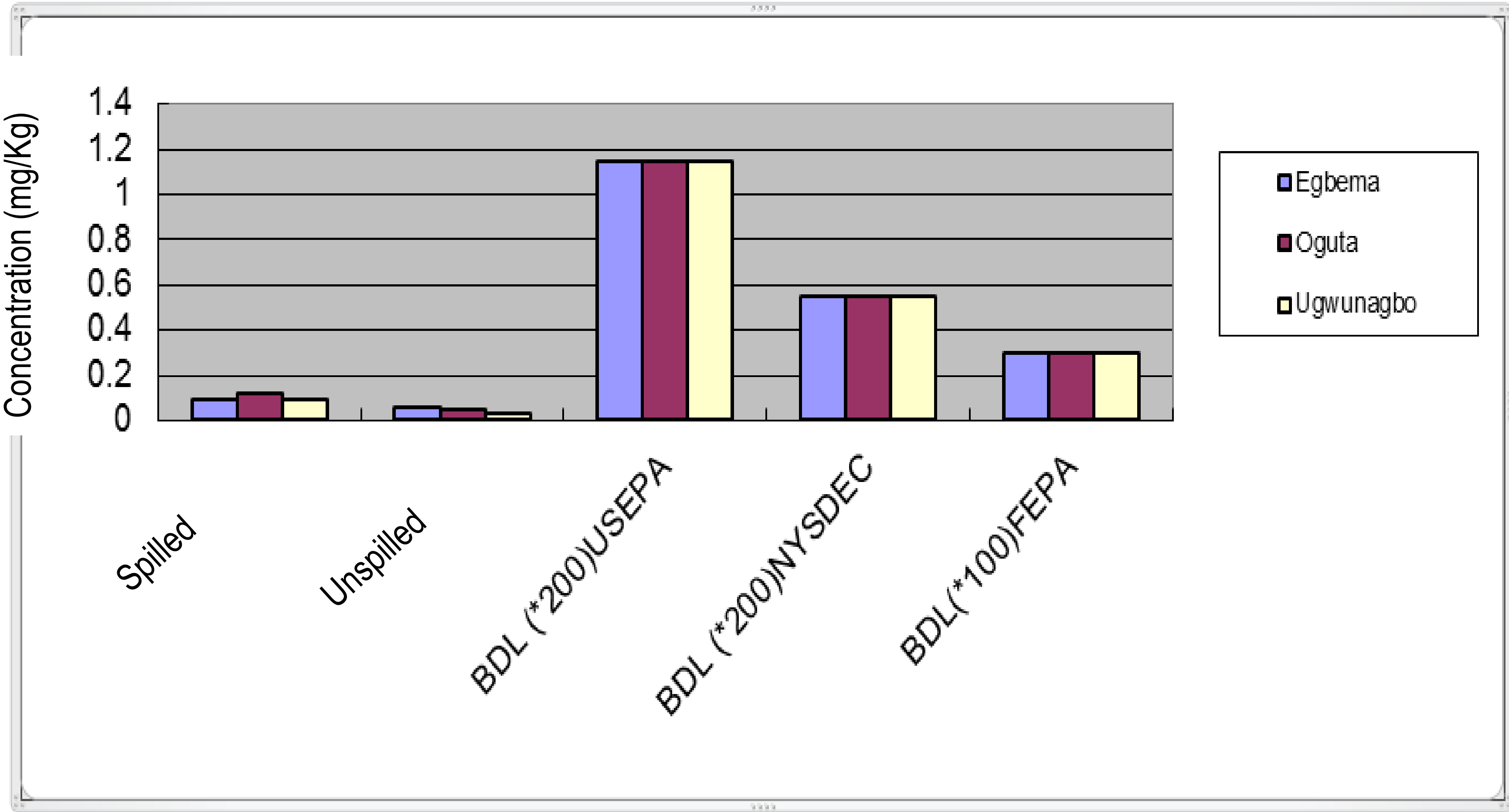

Figure 6. Chromium level in spilled and unspilled soils compared with standards

Environmental Protection Agency standard (30 mg/Kg) in Nigeria requiring clean-up.(FEPA, 1991)

Chromium values varied highly (CV > 35 %) in all studied pedons. There was significant difference ($p<0.05$) between spilled and unspilled pedons. This was evident from the T-test result in Table 4.5(c).

On comparing the different locations, the trend of chromium level can be summarized as Oguta (0.06 mg/Kg) > Egbema (0.05 mg/Kg) > Ugwunagbo (0.04 mg/Kg) for spilled pedons and Oguta (0.03 mg/Kg) = Egbema (0.03 mg/Kg) > Ugwunagbo (0.02 mg/Kg) for spilled pedons

4.4.3 Concentration of Cadmium (Cd)

The concentration of Cadmium in the studied soils ranged from 0.01 – 0.019 mg/Kg for spilled soils. Cadmium values decreased with increase in depth. These were higher when compared to unspilled pedons (0.006 – 0.01 mg/Kg). This is in consonance with the reports of Kirkham (2006), Nkwopara *et al* (2012) and Ihem *et al* (2015), who reported higher levels of cadmium in spilled soils over unspilled soils. Also Onweremadu and Duruigbo reported similarly a higher value of cadmium in spilled soils than unspilled ones. Values of Cadmium showed high variability (CV > 35%) in all studied soils. From the result of the T-test (Table 4.5(c)), there was no significant difference p(0.05) between spilled and unspilled pedons.

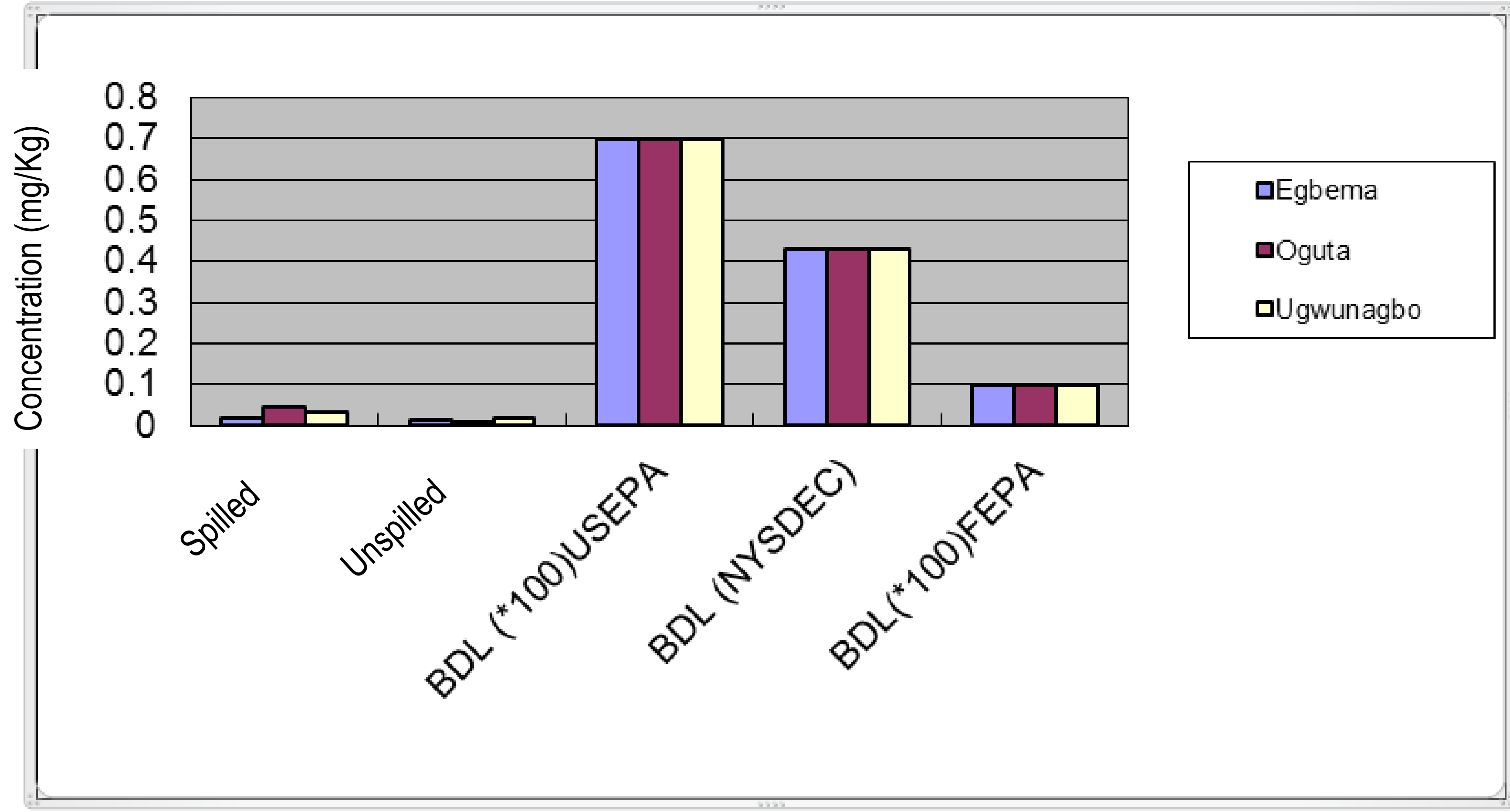

Figure 7. Cadmium level in spilled and unspilled compared with standards

Cadmium values were less when compared to the level requiring clean-up as stated by United States Environmental Protection Agency (70mg/Kg) and the level permitted for unrestricted use, including agriculture by NYSDEC (0.43mg/Kg) (Vern and Don, 2011). Also it was below the Federal Environmental Protection Agency standard (10 mg/Kg) in Nigeria requiring cleanup.(FEPA, 1991).

A comparison between the different locations, showed that the cadmium level of Oguta (0.019 mg/Kg) > Egbema (0.01 mg/Kg) = Ugwunagbo (0.01 mg/Kg) for spilled pedons and Ugwunagbo (0.013 mg/Kg) > Egbema (0.007 mg/Kg) > Oguta (0.06 mg/Kg) for unspilled pedons

4.4.4 Concentration of Nickel

The concentration of heavy metals is given in Tables 4.4(a,b,c).

Generally, Nickel decreased down the profile, the concentration of Nickel in all soils ranged from 5.56 - 7.88 mg/Kg in the epipedon for spilled soils. Though, these were higher when compared to the unspilled soils (2.08 – 4.22 mg/Kg), there was no significant difference between spilled and unspilled soils based on the T-test (Table 4.5 (c)). The non-significant higher values associated with the spilled soils is in agreement with the findings of Ihem *et al* (2015), who reported non-significant higher concentrations of Nickel in oil-spilled soils than the unspilled soils.

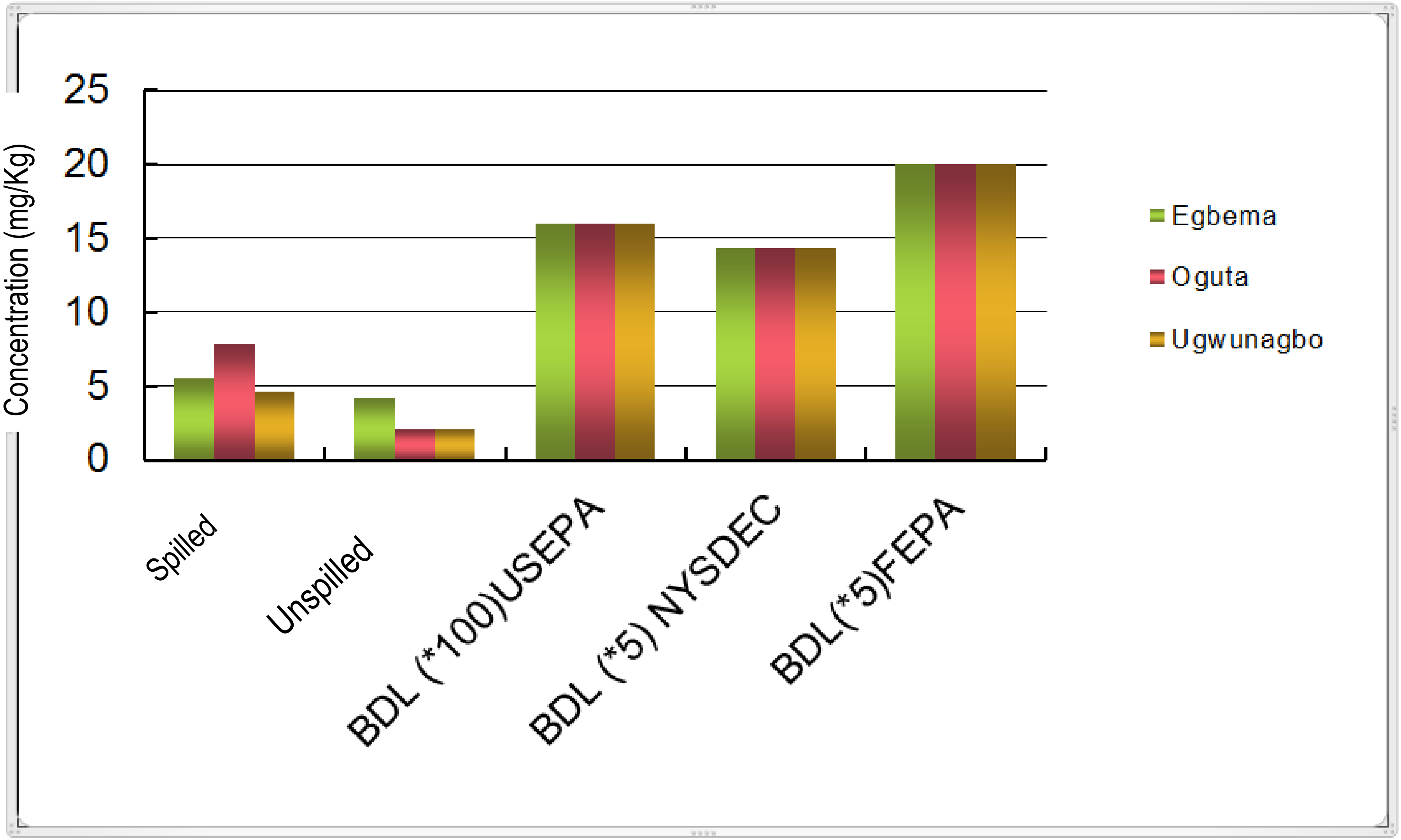

Figure 8: Nickel level in spilled and unspilled compared with standards

The values of Nickel showed high variation (CV > 35%) in all studied soils. Nickel values are far below the level requiring clean-up as stated by US EPA (1600mg/Kg) and the level permitted for unrestricted use i.e including agriculture by NYS DEC (72mg/Kg) (Vern and Don, 2011). Also it was below the Federal Environmental Protection Agency standard (100 mg/Kg) in Nigeria requiring cleanup (FEPA, 1991). Nkwopara *et al* (2012) also reported levels of Nickel that were below the levels requiring cleanup.

Comparing the different locations (Egbema, Oguta and Ugwunagbo) using mean value, Nickel concentration could be summarized as Oguta (3.49 mg/Kg) > Egbema (2.25 mg/Kg) > Ugwunagbo (2.16 mg/Kg) for spilled pedons and Egbema (2.36 mg/Kg) > Oguta (1.38 mg/Kg) > Ugwunagbo (1.12 mg/Kg).

TABLE 4.5 (a): T-Test result for physical properties of studied soils

	Sand	Silt	Clay	BD	TP	MC	Silt/Clay
Spilled	612	111	275	1.38	47	18	0.53
Unspilled	641	106	250	1.31	50	21	0.59
p-value	0.62	0.82	0.68	0.21	0.19	0.21	0.67
Remarks	NS	NS	NS	NS	NS	NS	NS

BD = Bulk Density, MC = Moisture Content, TP = Total Porosity, NS = Not Significant

TABLE 4.5 (b): T-Test result for chemical properties of studied soils.

	pHWat	pHKCl	OC	TN	AvP	Ca	Mg	K	Na	TEB	CEC	ECEC	BS	H	Al	TEA	AlSat	Ca/Mg
Poll	5.07	4.22	9.86	0.48	7.05	1.20	0.41	0.27	0.07	1.96	6.05	4.39	33.78	0.56	1.83	2.39	30.97	3.11
Unpoll	5.18	4.60	16.55	0.53	9.7	1.29	0.52	0.37	0.12	2.31	8.74	4.66	50.76	0.91	1.42	2.33	30.50	2.53
p-value	0.41	0.02	0.06	0.76	0.4	0.62	0.10	0.18	0.24	0.15	0.03	0.52	0.002	0.14	0.07	0.88	0.88	0.05
Remarks	NS	*	*	NS	NS	NS	NS	NS	NS	NS	*	NS	*	NS	*	NS	NS	*

*BD = Bulk Density, MC = Moisture Content, TP = Total Porosity, NS = Not Significant, * = Significant at 0.05 probability level*

TABLE 4.5 (c): T-Test result for heavy metal concentration in studied soils.

	Pb	**Cr**	**Cd**	**Ni**
Spilled	0.13	0.05	0.012	2.57
Unspilled	0.06	0.02	0.008	1.67
p-value	0.09	0.03	0.24	0.24
Remarks	NS	*	NS	NS

*BD = Bulk Density, MC = Moisture Content, TP = Total Porosity, NS = Not Significant, * = Significant at 0.05 probability level*

4.5 Simple correlation

The results of the physico-chemical properties of the soils and heavy metals were subjected to simple correlation analysis. The result is shown in Table 4.6.

Bulk Density positively correlated with Moisture Content ($p <0.05$) in spilled soils of Egbema and Ugwunagbo while Bulk Density significantly correlated to Moisture content ($p < 0.01$) in Egbema spilled soil, this was corroborated by Nkwopara *et al* (2012). There was also correlation between Cadmium and Organic Carbon in Egbema spilled soils.

Lead correlated negatively with TEB in spilled soils of Egbema and Ugwunagbo. This is in agreement with the observations of Rolka and Wyszkowski (2021). Soil pH positively correlated with Lead, Chromium, Cadmium and Nickel in Oguta and Egbema spilled and Oguta unspilled soils. ECEC negatively correlated with Cadmium, Nickel, Chromium and Lead in Egbema spilled soils but positively in other studied soils. ECEC correlated with Lead in Oguta unspilled soils ($p <0.01$).

TABLE 4.6: Simple correlation coefficient (r) among soil properties and heavy metals

Soil Properties	**Site 1**	**Site 2**	**Site 3**	**Site 4**	**Site 5** *mg /Kg*	**Site 6**
BD and MC	0.94*	0.98**	0.86	0.30	0.95*	0.69
Cd and TEB	- 0.41	0.31	0.70	0.87	- 0.63	0.78
Cr and TEB	- 0.69	0.46	0.75	0.87	- 0.44	0.38
Pb and TEB	-0.39	0.18	0.77	0.99*	- 0.61	0.22
Ni and TEB	- 0.38	0.06	0.75	0.95*	-0.73	0.10
Cd and TEA	-0.28	0.64	0.72	0.82	0.96*	0.55
Cr and TEA	- 0.52	0.57	0.79	0.91	0.86	0.87
Pb and TEA	- 0.26	0.50	0.82	0.99**	0.94*	0.95
Ni and TEA	-0.22	0.42	0.78	0.96*	0.98*	0.99*
Cd and ECEC	-0.34	0.49	0.72	0.84	0.50	0.96*
Cr and ECEC	-0.59	0.55	0.78	0.91	0.66	0.86
Pb and ECEC	- 0.31	0.35	0.82	0.99**	0.51	0.79
Ni and ECEC	-0.29	0.24	0.78	0.96*	0.38	0.71
Cd and OC	0.99**	0.92*	0.98*	0.74	0.97*	0.83
Cr and OC	0.79	0.96*	0.93	0.96*	0.98*	0.97*
Pb and OC	0.95	0.99**	0.85	0.99*	0.97*	0.95*
Ni and OC	0.93*	0.98*	0.92	0.93	0.94*	0.95*
Pb and pH(Water)	0.89*	0.99**	0.71	0.99*	- 0.35	0.94
Pb and pH(KCl)	0.79	0.98**	0.67	0.96*	- 0.61	0.86
Cr and pH (Water)	0.65	0.98**	0.81	0.97*	- 0.20	0.94
Cr and pH(KCl)	0.53	0.87	0.78	0.99*	- 0.45	0.99*
sCd and pH (water)	0.84	0.92*	0.89	0.68	- 0.36	0.86
Cd and pH (KCl)	0.73	0.93*	0.87	0.61	- 0.62	0.87
Ni and pH (water)	0.89*	0.96**	0.83	0.85	- 0.47	0.89
Ni and pH (KCl)	0.80	0.99**	0.80	0.87	- 0.72	0.94

** = Significant at 0.05 Level, ** = Significant at 0.01 Level, Site 1=Egbema Spilled, Site 2 = Egbema Unspilled, Site 3 = Oguta Spilled, Site 4 = Oguta Unspilled, Site 5= Ugwunagbo Spilled, Site 6 = Ugwunagbo Unspilled*

4.6 Classification of soils

The morphological characteristics and results gotten from the analysis of the soil samples were used to classify the six (6) pedons in accordance with USDA Soil Taxonomic system and correlated with the World Reference Base (FAO,2006). Pedons were placed in a particular order, suborder, great groups and sub group based on the presence or absence of diagnostic horizons, properties associated with wetness (Soil Moisture Regime), horizon features and their arrangement and whether they represent or not the central concepts of the great group, intergrades or transitional forms respectively.

Order: At the order level; Results showed that the study areas had high sand content which generally decreased with profile depth, irrespective of the study site. All six (6) pedons showed evidence of argillic (Bt) horizon, and low cation exchange capacity which placed them in the Ultisols order (Soil Survey Staff, 2010). Previous works of Kang and Juo (1979) described soils in this area as "Low Activity Clay" (LAC) soils due to their low ECEC, and Onweremadu (2007) classified the soils of the area as Ultisols, hence they are placed in the Ultisols order.

Sub Order: At the sub-order level, all soils had udic moisture regime in addition to the low CEC, they were freely drained and no evidence of water accumulation. Egbema spilled pedon had greyish brown (10YR3/2) surface horizon resting on a Yellowish brown (10YR5/4) subsurface horizon, Egbema Unspilled pedon showed a dark brown (10YR4/2) on the surface horizon and a yellowish brown (10YR5/4) subsurface horizon, Oguta spilled pedon has a dark brown (7.5YR3/2) surface horizon upon a Yellowish red (5YR5/8) subsurface horizon, Oguta Unspilled pedon showed a dark reddish brown (5YR3/2) surface horizon resting on a dark red

(7.5YR4/4) subsurface argillic horizon, Ugwunagbo spilled pedon has a greyish brown (10YR5/2) surface horizon resting on a yellowish brown (10YR5/4) subsurface horizon and Ugwunagbo unspilled pedon had a dark brown (10YR3/3) surface horizon over a red (2.5YR4/6) subsurface horizon. Thus, they were classified as Udults.

Great Groups; At the great group level, Soils of Ugwunagbo spilled and Egbema (spilled and unspilled) had no decrease in clay content with increasing depth in addition to the absence of a densic or lithic contact within 150cm depth (Soil Survey Staff, 2010). Also, Ugwunagbo unnspilled soil showed an argillic horizon with clay content that decreased by not up to 20% within a depth of 150cm (Soil Survey Staff, 2010) and thus the soils (Ugwunagbo and Egbema both spilled and unspilled) qualified as Paleudults.

Oguta soils (spilled and unspilled) showed a significant decrease in clay from maximum amount by >20 % of that maximum amount within 150cm of the soil surface. It shows a layer of maximum clay contecnt. Furthermore, there was no evidence of eluviation in the form of skeletans on ped surfaces. Thus they were classified as Tropudults.

Sub Groups: The tropudults (Oguta spilled and unspilled) did not show evidence of saturation with water in the mottled zone that will be artificially drained and do not have cracks at some time of the year that are 1 cm wide at a depth of 50 cm which extends to the soil surface. Thus they were classified as Typic tropudults.

The Paleudults (Egbema and Ugwunagbo both spilled and unspilled) have no plinthite in the epi-pedon thus they were classified as Typic Paleudult (Soil Taxonomy) and Dystric Nitisol (FAO/WRB).

The tropudults were classified as Typic Tropudult (Soil Taxonomy) and Orthic Acrisol (FAO/WRB).

CHAPTER V:

5.0 Summary, Conclusion and Recommendation

5.1 Summary

Field investigations was carried out in three (3) locations (Egbema, Oguta and Ugwunagbo) in the Niger Delta region Nigeria to characterize and classify the spilled and unspilled soils of the area and their heavy metals concentration. A total of six (6) profiles were sunk in the three locations, two in each, representing spilled and unspillled fields. Samples were collected from genetic horizons, prepared and analysed following standard procedures.

The studied soils were generally deep (greater than 180 cm) and well drained with friable consistence. There were generally fine to medium roots. There was no sharp difference in texture between spilled and unspilled pedons. The value of clay content and bulk density increased with depth in all pedons with spilled pedons having higher bulk density. The soils were basically sandy loam and this could be attributed to the nature of their parent material (Sub recent alluvium and coastal plain sands). The silt/clay ratio decreased as you go deeper in the profile pit and was generally low (<1) which implies that the soils are highly weathered. pH was generally low. Organic Carbon decreased with depth. Total Nitrogen and Available Phosphorus were higher in unspilled soils than spilled soils. Exchangeable Ca^{2+}, Mg^{2+}, Na^{+}, K^{+}, ECEC and Percentage base saturation recorded higher values in unspilled pedons than in spilled pedons. The value of sodium ranged from low to very low indicating that the soil is not sodic. The Ca:Mg ratio was low indicating an unfertile soil. Soil pH and Base Saturation slightly varied (CV < 15%), Organic Carbon and Total Nitrogen were highly variable (CV >35) at Egbema and Ugwunagbo.

Bulk Density positively correlated Moisture content ($p<0.05$) in spilled soils of Egbema and Ugwunagbo. There was a significant correlation between Cadmium and Organic Carbon in Egbema spilled soils. Soil pH positively correlated with Lead, Chromium, Cadmium and Nickel in Oguta and Egbema spilled soils.

The concentration of heavy metals followed the following trend Ni > Pb > Cd > Cr in spilled pedons and Ni > Pb > Cr > Cd in unspilled pedons. The heavy metals (Ni,Cd,Cr and Pb) were higher in the spilled pedons than in their unspilled counterparts but below standards (FEPA, NYSDEC, USEPA) requiring cleanup.

The soils were classified based using USDA Soil taxonomy and FAO/WRB. Soils from Oguta soils were classified as Typic tropudult (Soil Taxonomy) and Orthic Acrisol (FAO/WRB). Egbema and Ugwunagbo soils both spilled and unspilled were classified as Typic paleudult (Soil Taxonomy) and Dystric Nitisol (FAO / WRB).

5.2 Conclusion

This work aimed to determine the physico-chemical properties of soils, investigate the total amount of heavy metals (specifically Pb, Cr, Ni, Cd) in the soil, use the elucidated properties of the soil to classify it according to the United States Soil Taxonomy (USDA, 1975) and correlate it with FAO/UNESCO world soil legend, and establish correlations between the physico-chemical properties and heavy metal concentrations in the soil.

Through selected analysis, these objectives were achieved and valuable insights into the soil characteristics and heavy metal content were obtained which include the values of clay content and bulk density increased with depth in all pedons with spilled pedons having higher bulk density. The soils were basically sandy loam and this could be attributed to the nature of their parent material (Sub recent alluvium and coastal plain sands). The silt/clay ratio decreased as you go deeper in the profile and was generally low (<1) implying highly weathered soils. pH was generally low. Organic Carbon decreased with depth. Total Nitrogen and Available Phosphorus were higher in unspilled soils than spilled soils. Exchangeable bases and

Percentage base saturation recorded higher values in unspilled pedons than in spilled pedons. The soils were not sodic. The Ca:Mg ratio was low indicated an unfertile soil. There were slight variations in Soil pH and Base Saturation while Organic Carbon and Total Nitrogen were highly variable (CV >35) at Egbema and Ugwunagbo.

Also, the investigation into the total amount of heavy metals, specifically Pb, Cr, Ni, and Cd, has shed light on the presence and concentrations of these potentially hazardous elements in the soil. Though present, the values were below critical levels requiring clean up as described by both local (Federal Environmental Protection Agency (FEPA)) and international (United States Environmental Protection Agency (USEPA) and for Agricultural purposes as specified by New York Department of Environmental Conservation (NYSDEC)) standards, thus indicative of non-contamination.

Moreover, the classification of soils using the United States Soil Taxonomy and FAO/UNESCO world soil legend provides a standardized framework for categorizing and characterizing soil types based on their properties. Soils from Oguta were classified as Typic tropudult - Orthic Acrisol while Egbema and Ugwunagbo soils were classified as Typic paleudult - Dystric Nitisol.

Lastly, by correlating the physico-chemical properties and heavy metal concentrations in the soil, we have identified that there was better positive correlation between the studied heavy metals (Ni, Pb, Cr and Cd) and each of TEA, ECEC and pH in all pedons.

5.3 Recommendations.

From the analysis, there was presence of heavy metals in the studied soils though the concentration of heavy metals in the studied soils are generally low (both spilled and unspilled soils), thus the presence of heavy metals in the studied soil is not a limitation to the use of the soils e.g for agriculture.

5.4 Contributions to knowledge.

The primary aim of this work is to classify the soils and determine the presence and concentration of heavy metals (Lead (Pb), Chromium (Cr), Cadmium (Cd) and Nickel (Ni)) in selected soils of the region.

There has been research on some other soils of the region. In this study, the researcher characterized the soils to understand the morphological, physical and chemical properties of the soil and so advice with precision and certainty possible uses for the soils. Also the soils were classified according to USDA and correlated with WRB thus giving room for standard information sharing on the properties and capabilities of the soil. Furthermore, the concentration of heavy metals in the soil and from comparison with both local and international standards, one could conclude that the soils can be put into agricultural production without fear of heavy metal poisoning. As the level of the heavy metals in the studied soils were within the permissible level for all uses.

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APPENDICES

Appendix 1 Critical values of nutrients and soil properties

Properties	Critical levels				
	Very Low	Low	Medium	High	Very high
OM %	< 1	1-2	2-4.2	4.2-6	> 6
Total N g/Kg	< 0.5	0.5-1.25	1.25-2.25	2.25 – 3.0	> 3.0
C/N	<10 = good, 10 – 14 = medium and >14 = poor				
Ca cmol/Kg	< 2	2-5	5 – 10	10 – 20	> 20
Mg cmol/Kg	< 0.5	0.5-1.5	1.5 – 3	3 -8	> 8
K cmol/Kg	< 0.1	0.1-0.3	0.3 – 0.6	0.6-1.2	> 1.2
Na cmol/Kg	< 0.1	0.1-0.3	0.3 – 0.7	0.7-2.0	> 2.0
Olsen-P mg/Kg		< 5	5 – 15	> 15	
pH	5.3 – 6.0 = moderately acid; 6.0-7.0=slightly acid; 7.0-8.5=moderately alkaline				
ESP %		< 2	2 – 8	8-15	15-27
CEC_7 cmol/kg	0-20	21 – 40	41 – 60	61-80	81-100

Adapted from Beernart and Bitondo (1992)

Appendix 2 Grouping coefficient of variation into variability classes

CV (%)	Variability grouping (class)
<15	Slightly Variable
15<CV≤ 35	Moderately Variable
CV >35	Highly Variable

Wilding (1985)