

**PHYSICOCHEMICAL AND ELEMENTAL ANALYSIS OF SOIL IN  
MAMBILLA PLATEAU, TARABA STATE, NIGERIA**

**BY**

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FOR THE AWARD OF MASTERS OF TECHNOLOGY  
(M.TECH) DEGREE IN ANALYTICAL CHEMISTRY.**

**JUNE, 2013**

**DECLARATION**

I hereby declare that the thesis titled “Physicochemical and Elemental Analysis of Soil in Mambilla Plateau in Taraba State” have been duly carried out by me under the supervision of Prof. H.M. Maina, Department of Chemistry Modibbo Adama University of Technology, Yola.

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## CERTIFICATION

This is to certify that the research work “Physiochemical and Elemental Analysis of soil in Mambilla Plateau, Taraba State” has been presented by Isaac Ernest (M.Tech/CH/09/0323), Department of Chemistry, ModibboAdama University of Technology, Yola and has been read and approved as meeting part of the requirements for the award of Masters of Technology (M.Tech) Degree Analytical Chemistry.

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## **DEDICATION**

This project work is dedicated to my brother Joel Wakili, who inspired me to further my educational career, for his brotherly guide.

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## **ABSTRACT**

Soil samples from Mambilla Plateau in Taraba State, were analyzed to determine their physicochemical and elemental properties. The physical and chemical parameters determined were colour, pH, conductivity, specific gravity and  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{K}_2\text{O}$ ,  $\text{SiO}_2$ ,  $\text{BaO}$ ,  $\text{CaO}$ ,  $\text{MnO}$ ,  $\text{TiO}_2$ ,  $\text{Cr}_2\text{O}_3$ ,  $\text{CuO}$ ,  $\text{NiO}$  and  $\text{V}_2\text{O}_5$  respectively. The pH ranged between 3.62-4.52, conductivity in turn ranged between 81.79-238.00 and specific gravity also ranged between 2.00-2.50. The percentage elemental content detected for  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{K}_2\text{O}$ ,  $\text{SiO}_2$ ,  $\text{TiO}_2$ ,  $\text{BaO}$ ,  $\text{CaO}$ ,  $\text{MnO}$ ,  $\text{Cr}_2\text{O}_3$ ,  $\text{CuO}$ ,  $\text{NiO}$  and  $\text{V}_2\text{O}_5$  had a mean value of  $16.59\pm 5.11$ ,  $27.69\pm 11.73$ ,  $1.54\pm 0.41$ ,  $37.74\pm 16.95$ ,  $4.00\pm 1.85$ ,  $0.28\pm 0.17$ ,  $0.22\pm 0.17$ ,  $0.23\pm 0.14$ ,  $0.12\pm 0.06$ ,  $0.043\pm 0.05$ ,  $0.11\pm 0.05$  and  $0.15\pm 0.06$  respectively.  $\text{Fe}_2\text{O}_3$  and  $\text{TiO}_2$  were found to be higher, while  $\text{K}_2\text{O}$  was detected in five samples only, and not detected in  $\text{E}_1$ ,  $\text{E}_2$ ,  $\text{N}_2$ ,  $\text{N}_3$ ,  $\text{N}_4$ ,  $\text{S}_1$  and  $\text{S}_3$  locations due to effect of the soil pH.

**Keywords:** Soil, Mambilla Plateau, physicochemical, Mineral, Colour.

## **CHAPTER ONE**

### **INTRODUCTION**

#### **1.1 Background of the Study**

Soil survey generates host of information on soils and other environmental factors. In the past, these soil information's were primarily used to develop land use plans to support increased crop production. However, the use of soil information for non-agricultural purpose is on the increased. There is increased awareness of the use of soil information as an essential input in the evaluation, prediction of minerals deposits and effect of land use on environmental degradation and sustainability. In Nigeria, like most developing countries, one primary constraint to sustainable and successful agricultural programmed is the Lack of knowledge about the soil resources and how to manage them (Rajiet al., 2007).

There is an increasing need for information on soils as a means to produce food. Variation in soil properties due to land use and management and their consequence to the production capacity has been subject of research in the past for a range of climatic and edaphically condition (Fasina, 2004). An assessment of variation in soil properties associated with land use management activities is vital for the selection and establishment of appropriate sustainable practices under different agro-system.

Morphological and physical properties of soils and topographic features of landscapes are important factors that aid the understanding of soil forming factors and processes. Various inferences can be obtained from study of soil morphological and physical properties. Topography modifies soil profile development, consequently understanding the role these properties play should aid in assessing productive value of soil, and developing strategies for its conservation (Owonubiet al., 2007). In the past, agricultural system in Nigeria relied mainly on shifting cultivation to maintain the fertility of the soil through organic matter and plant nutrient build up during fallow periods.

However, due to population pressure and increased in urbanization and being brought under cultivation, while fallow periods are being systematically reduced (Udohet al., 2007). Products of soil are also being lost through erosion, desertification and deforestation. Consequently, output per hectare, especially of food crops, is declining due to continue cultivation which depletes soil nutrients. All these factors have seriously contributed to the current high level of poverty playing the Nation today.

The resulting food crises situation has put pressure on traditional farming systems, conditioning a change from extensive to intensive, land conserving and land augmenting farming system. However, fertilizer hold a key to soil fertility and hence to high agricultural production and productivity. Therefore, in order to derive maximum benefits from fertilizer use, certain policies programmers' and practices have to be fallowed. Therefore a sustainable soil productivity and optimum crop production would demand employing appropriate soil testing programs urgently as a matter of policy. Soil testing is broadly defined as the assessment of chemical properties of a soil for predicting accurately its elemental content and fertilizer requirements (Sabulo and Adepetu, 1987). Soil testing ensures a balanced nutrient application and high yields are guaranteed even under continuous cultivation since the productivity of the soil is preserved. Also it is possible to save up to half the cost of fertilizer by testing the soil properties before deciding on the types of fertilizer and the rate of application.

The effect of soil variability has been extensively studies at both small and large scales (Ogunkule and Clrikezie, 1992). According to Stoltet al., (1993), spatial variability is universal to all soil and can have many possible sources; such sources may include difference in weathering rates, lithology, topography or hydrology. (Mokuna, 1987), has observed that systematic variability is a function of landforms, geomorphic elements, soil forming factors and interactions of these three factors.

Variation in soil properties has been found to have significantly influenced soil management and crop production (Fasina, 2003). Worldwide, soil variability causes farmers yield and complicate the interpretation of agronomic experiment. In Sardauna

Local Government Area of Taraba State (Figure 1.1), information on status as well as the variability of soil physical and chemical properties in this natural architecture and landscaping of the terrain is lacking.

## **1.2 Statement of the Problem**

The Mambilla Plateau is selected for the study, in view of its peerless splendor and natural architecture and landscaping with hilly deep gorges. The climate of the Plateau is comparatively cold, associated with frequent and heavy rainfall due to orographic activities on the Plateau (Zainab, 2011). The vegetation on the plateau comprise low grasses with trees being noticeably absent except for manmade forest (Mambilla Plateau, 2011). There was no known research conducted to assess the nutrient and pollutant elements in this abundant tourism potential. Therefore, it is important to assess the level of some of such elements so as to make appropriate suggestion and recommendations to improve the soil quality for optimum yield of its economic fruits plants such as Carrot, apple, avocados, banana etc and rich tea plantation being processed throughout the year.

## **1.3 Aim and Objectives**

- i. To determine some physical and bulk chemical analyses of major elements and trace elements contents, composition and other characteristic will be performed to measure the expected growth potential of the soil.
- ii. To ascertain the deficiencies and determines toxicities and inhibitions from the presence of non-essential trace minerals.

## **1.4 The major objectives are;**

- 1 To determine the physical parameters in soil.
- 2 To estimate the nutrient and essential elements in soil.
- 3 To determine some trace and toxic elements in soil.
- 4 To compare with results of soils obtained elsewhere.

### **1.5 Significance of the Study**

The results of this study will ascertain the authenticity of high speculation of mineral deposit including gold, silver, precious stones etc. The result from this research would assist in exploiting and preserving the enriching features of the plateau for the enhancement of revenue earning for the state, in particular and the country in general.

### **1.6 The Mambilla plateau**

The Mambila is the highest mountain in West Africa; 2442 metres above sea level and Gashaka-Gumti park covering about 5000 square metres. The Mambilla is a view of splendour. Besides the healing weather, the natural landscaping of the terrain, the mountain has about four untapped waterfalls and match stick-making trees. There are eucalyptus trees, (silver bird), timber plantation and a rich tea plantation, all ready for harvest all through the year. The Plateau is gifted with a year-round winter that peaks and becomes almost unbearable in December (Zainab, 2011).

## CHAPTER TWO

### LITERATURE REVIEW

#### **2.1 Soil Formation**

Geologic processes have brought to Connecticut's surface minerals parent materials in which soil have formed. Rocks are first broken down into progressively smaller particles and mixed with decaying organic materials. Bed rock begins to disintegrate as it is subjected to freezing thawing cycles, rain and other environmental forces. The rock breaks down into parent material which in turn breaks into smaller mineral particles (Birkeland, 1999). The organisms in an area contribute to soil formation by facilitating the disintegration process as they live and adding organic matter to the system when they die. This process continues until soils eventually reach a point where it can support a thick cover of vegetation (USDA, 2010).

#### **2.2 Weathering of Rock**

The weathering processes occur over an extended period of time resulting in soil that varies from the original rock or sediment in its physical and chemical properties. In addition, organic matter from decaying vegetation and micro-organisms is added to the soil and pronounced vertical arrangement of layers develops in the soil, known as a soil profile. Thus soil profile extends from the surface downward to the un-weathered soil parent material.

#### **2.3 Rocks as a Source of Soil Mineral**

Soil consists of layers of mineral constituents of variable thickness, which differ from the parent materials in the morphological, physical, chemical and mineralogical characteristics (Peter, 1999). It is a complex heterogeneous medium comprising mineral and organic solids, aqueous and gaseous components. The minerals present are usually weathered (chemical decomposing) rock fragments and secondary minerals such as clays, hydrous oxide of Fe, Al and Mn and sometime carbonates (usually  $\text{CaCO}_3$ ). The

organic matter comprises living organisms, dead plant material and colloidal humus formed by action of micro-organisms on plant litter. Soil differs from its parent rock due to interaction between lithosphere, hydrosphere and the biosphere. In engineering, soil is referred to as regolith or loose rock material (Boulet al., 1973).

## **2.4 Soil Formation Factors**

Soil formation, or pedogenesis, is the combined effect of physical, chemical, biological and anthropogenic processes on soil parent material. Soil genesis involves processes that develop layers or horizons in the soil profile. These processes involve additions, losses, transformations and translocations of material that compose the soil. Minerals derived from weathered rock undergo changes that cause the formation of secondary minerals and other components that are variably soluble in water, these constituents are moved (translocated) from one area of the soil to other areas by water and animal activity. The alteration and movement of materials within soil causes the formation of distinctive soil horizons. The weathering of bedrock produces the parent material from which soil forms. An example of soil development from bare rock occurs on recent lava flows in warm regions under heavy and very frequent rainfall. In such climates, plants become established very quickly on basaltic lava, even though there is very little organic material. The plants are supported by the porous rocks, as it is filled with nutrient bearing water which carries, for example, dissolved minerals. The developing plant roots, themselves are associated with mycorrhizal fungi, (Van Schollet al., 2000), gradually break up the porous lava and organic matter soon accumulates.

But even before it does, the predominantly porous broken lava which the plant root grows can be considered a soil. How the soil 'life' cycle process is influenced by at least five classic soil forming factors that are dynamically intertwined in shaping the way soil is developed, they include: parent material, regional climate, topography, biotic potential and the passage of time (Stevens, 2011).

### **2.4.1 Parent Material**

The material from which soil forms is called parent material. It includes: weathered primary bedrock; secondary materials transported from other locations, e.g. colluviums and alluvium deposits that are already present but mixed or altered in other ways-old soil formations, organic material including peat or alpine humus, and anthropogenic materials like landfill or mine waste (Steven, 2011). Few soils form directly from the breakdown of the underlying rocks they develop on. These soils are often called “residual soils” and have the same general chemistry as their parent rocks. Most soils derive from material that has been transported from other locations by wind and water gravity (NASA, 2011). Some of these materials may have moved many miles or only a few feet. Windblown material called loess is commonly in the Midwest of North America and in central Asia and other locations. Glacial till is a component of many soils in the northern and southern latitudes and those formed near large mountain, is the product of glacial ice moving over the ground. The ice can break rock and larger stones into smaller pieces; it also sorts material into different sizes. As glacial ice melts, the melt water also moves and sorts material, and deposits it varying distances from its origin. The deeper sections of the soil profile may have materials that are relatively unchanged from when they were deposited by water, ice or wind.

Weathering is the first stage in the transforming of parent material into soil material. In soil forming from bedrock, a thick layer of weathered material called saprolite may form. Saprolite is the result of weathering processes that include: hydrolysis, chelating from organic compounds, hydration, solution of minerals by water, and physical processes that include freezing and thawing or wetting and drying.

### **2.4.2 Climate**

Soil formation greatly depends on climate and soils from different climatic zones show distinctive characteristics. Temperature and moisture affect weathering and leaching. Wind moves sand and other particles, especially in regions where there is little plant occurring. The type and amount of precipitation influence soil formation by

affecting the movement of ions and particles through the soil, aiding in the development of different soil profiles. Seasonal and daily temperatures of water in weathering parent rock material affect soil dynamics. The cycle of freezing and thawing is an effective mechanism to break up rocks affects biological activity, rates of chemical reactions and types of vegetation cover.

### **2.4.3 Biological Factors**

Plants, animals, fungi, bacteria and humans affect soil formation. Animals and micro-organisms mix soils to form burrows and pores allowing moisture and gases to seep into deeper layers. In the same way, plant roots open channels in the soils especially plants with deep taproots which can penetrate many meters through the different soil layers to bring up nutrients from deeper in the soil. Plants with fibrous roots that spread out near the soil surface, have roots that are easily decomposed, adding organic matter. Micro-organism, including fungi and bacteria, affect chemical exchanges between roots and soil and act as a reserve of nutrients.

Human can impact soil formation by removing vegetation cover, this removal promotes erosion. They can also mix the different soil layers, restarting the soil formation process as less-weathered materials is mixed with diluting the more developed upper layers. Some soils may contain up to one million species of microbes per gram, most of those species being unknown, making soil the most abundant ecosystem on Earth (Copley, 2005).

Vegetation impacts soil in numerous ways, it can prevent erosion by the impact of rain or surface runoff. Plants shade soils, keeping them cooler and slowing evaporation of soil moisture, or plants by way of transportation can cause soils to lose moisture. Plants can form new chemicals which can breakdown or build up soil particles. The type and amount of vegetation depends on climate, land form topography, soil characteristics, and biological factors. Soil factors such as density, depth, chemistry, pH, temperature and moisture greatly affect the type of plants that can grow in a given locations. Dead plants and dropped leaves and stems fall to the

surface of the soil and decomposed. There, organisms feed on them and mix the organic material with the upper soil layers; these added organic compounds become part of the soil formation process (Copley, 2005).

#### **2.4.4 Time**

Time is a factor in the interaction of all the above factors as they develop soil. Over time, soils evolve features interplay with each other. Soil is always changing. It takes about 800 to 1000 years for 2.5cm thick layers of fertile soil to be formed in nature. For example, recently deposited material from a flood exhibits no soil development because there has not been enough time for soil forming activities. The soil surface is buried, and the formation process begins again for this soil. The periods over which changes occur and its multiple influences means that simple soils are rare, resulting in formation of soil horizons. While soil can achieve relative stability in properties for intended periods, the soil like cycle ultimately ends in soil conditions that leave it vulnerable to erosion. Despite the inevitability of soil retrogression and degradation, most soil cycles are long and productive (Copley, 2005).

### **2.5 Characteristics of Soil**

#### **2.5.1 Soil Colour**

Soil colour is often the first impression one has when viewing soil striking colours and contrasting patterns are especially memorable. Soil colour is primarily influenced by soil mineralogy. Many soil colours are due to distribution of colours in a soil profile from chemical and biological weathering, especially reduced reactions. As the primary minerals in soil parent materials weather, the elements combine into new and colourful compounds. Iron forms secondary minerals with a yellow or red colour, organic matter decomposes into black and brown compounds, and manganese, sulfur and nitrogen can form black mineral deposits. These pigments produce various colour patterns due to effects by the environment during soil formation. Aerobic conditions produce uniform or gradual colour changes, while reducing environments results in

disrupted colour flow with complex, mottled patterns and point of colour concentration (Copley, 2005).

### **2.5.2 Soil Structure**

Soil structure is the arrangement of soil particles into aggregates. These may have various shapes, sizes and degrees of development. Soil structure affects aeration, water movement, resistance to erosion and plant root growth. Structure often gives clues to texture, organic matter content, and biological activity, past soil evolution, human use, chemical and mineralogical conditions under which the soil formed. If the soil is too high in clay, adding gypsum, washed river sand and organic matter will balance the composition. Adding organic matter to soil that is depleted in nutrients and too high in sand will boost the quality (Copley, 2005).

### **2.5.3 Soil Texture**

This refers to sand, silt and clay composition. Soil content affected soil behaviour including the retention capacity for nutrient and water (Brown, 2003). Sand and silt are the products of physical weathering, while clay is the product of chemical weathering. Clay content has retention capacity for nutrients and water (Brown, 2003). Clay's soils resist wind and water erosion better than silty and sandy soils, because the particles are more tightly joined to each other. In medium-textured soils, clay is after translocated downward through the soil profile and accumulate in the subsoil (Copley, 2005).

### **2.5.4 Soil Resistivity**

Soil resistivity is a measure of soils ability to retard the conduction of an electric current. The electrical resistivity of soil can affect the rate of galvanic corrosion of metallic structure in contact with the soil. Higher moisture content or increased electrolyte concentration can lower the resistivity and increase the conductivity thereby increasing the rate of corrosion from about 2 to 1000 $\Omega$ .m, but more extreme value are not unusual. (Edwards, 1998).

## 2.6 Soil Properties

The soil is a dynamic system, subject to short-term fluctuations, such as variation in moisture status, pH and redox conditions and also undergoing gradual alternations in response to changes in management and environmental factors. These changes in soil properties could affect the form and bioavailability of metals, and need to be considered in decisions on the management of polluted soil or the use of soils for disposal of waste materials (Bache, 1979).

### 2.6.1 Soil pH

The soil reaction is the pre-eminent factor controlling the chemical behaviour of metal and many other important processes in the soil. However, the pH concept is not as precise for soil as it is for solutions, because of the heterogeneity of soils, the relatively small proportion of solution present in the soil pores of the solid soil and the absorption of cationic  $H^+$  ions on to solid surfaces (Backe and Wild, 1988). The pH of a soil applied to the  $H^+$  ion concentration in the solution present in soil pore which is in dynamic equilibrium with the predominantly negatively charged surfaces of the soil particles. Hydrogen ions are strongly attracted to the surface negative charges, and they have power to replace most other cat ions.

Soil pH is affected by the changes in redox potential which occur in soils that become waterlogged periodically. Reduction conditions generally caused a pH increase, and oxidation brings about a decrease. Variations of up to 2 units can occur over a year in clay soils prone to water logging. Oxidation of pyrite ( $FeS_2$ ) in a soil parent material can cause a marked decrease in pH.

Soils have several mechanisms which serve to buffer pH to varying extents, including hydroxyl aluminium ions,  $CO_2$ , carbonate and cation exchange reactions. However, even with the buffering mechanism, soil pH differs significantly due to localized variations within soil. Soil generally has pH value within the range 4 - 8.5 owing to buffering by Al at the lower end by  $CaCO_3$  at the upper end range (Wild, 1988). Brady (1984), state that the normal pH is 5 - 7 in soil of humid regions and pH 7 -

9 in the soil of arid regions. However, the maximum range of pH conditions found in soils is 2 - 10.5. In a typical temperature environment, soils normally have a pH for most arable crops are 6.5 on minerals soil and 5.5 on peaty soils. The optimum pH for grassland in the UK is 6.0 on mineral soils. Soil pH can be raised by liming, but it is impractical to acidify agricultural soil more alkaline than these (Bache, 1979).

### **2.6.2 Soil Organic Matter**

The main feature which distinguishes soil from regolith (decomposed rock) is the presence of living organisms, organic debris and humus. All soils contain organic matter, although the amount and type may vary considerably. Colloidal soil organic matter has a major influence on the chemical properties of soil, and can be divided into non-humus substances comprise unaltered biochemical's such as amino acids, carbohydrates, organic acid, fats and waxes that hence not changed from the form in which they were synthesized by living organisms. Humus substances are a series of acidic, yellow to black coloured polyelectrolyte of moderately high molecular weight (Chen, 1986). The elemental composition of humus is typically (on an ash-free basis): 44 - 45% C, 3.6 - 5.4% H, 1.8 - 3.6% N and 40.2 - 47% O (White, 1987).

### **2.6.3 Soil Solution**

Soil retains water that can dissolve a range of molecules and ion. These solutions exchange gases with the soil atmosphere, it contain dissolved sugars, fulvic acids and other organic acids, plant nutrients such as gnitrate, ammonium, potassium, phosphate, sulfate and calcium, and micro nutrient such as zinc, iron and copper. These nutrients and humus component, that retain them in its ionic state, solutions that greatly impact plant growth. Soil pH can affect the type and amount of anions and cations that soil solutions contain and the exchange with the soil substrate and biological organism (Dan, 2000).

## 2.7 Classification of Soils

Soil is classified into categories in order to understand relationships between different soils and to determine the usefulness of a soil for a particular use. Based on the idea that soil has a particular morphology based on the materials and factors that form them. In 1960s, a different classification system began to emerge, that focused on soil morphology instead of parental materials and soil-forming factors. Since then it has undergone further modifications, the world Reference Base for soil Resources (WRB) (ICISS, 2007).

## 2.8 Soil Taxonomy

Taxonomy classification system name soil orders ending with the suffix sol. There are 12 soil orders in soil taxonomy.

**Entisol:** Recently formed soils that lack well developed horizons, commonly found on unconsolidated sediment like sand, some have an A horizons on top of bedrock.

**Vertisol:** Inverted soils, they tend to swell when wet and shrink upon drying, often forming deep cracks that surface layer can fall into.

**Inceptisol:** Young soils, they have sub-surface formation but shows little eluviations and alleviation.

**Aridisol:** Dry soils forming under decent conditions. They include nearly 20% of soils on Earth. Soil formation is slow and accumulated organic matter is scarce. They may have subsurface zone (calcic horizons) where calcium carbonate have accumulated from percolating water. Many aridisol soils have well-developed horizon showing clay movement from past periods of greater moisture.

**Mollisol:** soft soils with very thick A horizons.

**Spodosol:** Soils produced by podsolization. They are typical soils of cool climates.

**Alfisol:** Soils with aluminum and iron. They have horizons of clay accumulation, and form where there is enough moisture and warmth for at least three months of plant growth.

**Ultisol:** Soil that are heavily leached.

**Oxisol:** Soil with heavy oxides content.

**Andisols:** volcanic soils, which tend to be high in glass content.

**Gelisols:** Permafrost soils.

## 2.9 Soil Mineral Elements

Mineral elements in soils are divided into two groups; the major elements and trace elements.

Major elements: nitrogen (N), phosphorus (P), potassium (K), sodium (Na), calcium (Ca), magnesium (Mg), manganese (Mn), iron (Fe), aluminum (Al), titanium (Ti), and silicon (Si).

Trace elements also include barium (Ba), cerium (Ce), cobalt (Co), chromium (Cr), copper (Cu), gallium (Ga), lanthanum (La), niobium (Nb), nickel (Ni), rubidium (Rb), scandium (Sc), strontium (Sr), rhodium (Rh), uranium (U), vanadium (V), yttrium (Y), zirconium (Zr), zinc (Zn).

Plants obtain carbon, hydrogen and oxygen from the air and water and therefore little control over the availability of these nutrients, which comprise 94% of the dry tissue. The other 12 elements collectively representing 6% of the dry matter however affect crop production when one or more of them are deficient. Plants absorb essential nutrients along with water, directly from one of these components. However, the amount of major nutrients in soil solution at any time is far less than is needed to produce a mature plant. Consequently, the soil solution nutrient level must be constantly, replenished from the organic or inorganic part of soil and from fertilizers or manures added to agricultural soil (Brady and Weil, 2002).

The elements are categorised on the basis of the quantities utilized by plants. Those elements taken up in large amount are often referred to as major (i.e. macro) elements, while those use in very small amounts are term as trace (micro) elements (Landscape America, 2007). Each element has specific role in the physiology and nutrition of crop but they all function in concert to produce optimum yields. They are absorbed primarily in inorganic/ionic forms and utilized at different rates and amounts

interacting mechanically to influence the biochemical and biological characteristics of plants and the final yield (Hanson and Kilmer, 1982).

Deficiency of these minerals does not directly produce symptom, rather the normal plant processes are thrown out of balance, with an accumulation of certain intermediate organic compounds and a shortage of others (Havlin et al., 2003).

## **2.9.1 Major Minerals Elements**

### **2.9.1.1 Nitrogen**

Nitrogen is important for many functions of both plants and animals. It is the major element in amino and nucleic acids, the building blocks of protein, enzymes and genetic cellular materials. It is also a structural part of chlorophyll, the green pigment of the plant that is responsible for photosynthesis (Das, 1997). Other functions of nitrogen include stimulating plants into rapid, vigorous growth, increasing seed and fruit yield and improving the quality of leaf and forage crops. Soil nitrogen is present in elemental, organic or in fertilizers as ammonia type nitrogen and nitrate type nitrogen. Most plants obtain nitrogen in the nitrate form. Urea and sulphate ammonia are unsuitable for hydroponics as they need to undergo conversion from ammonia type by soil bacteria before plants can use them. Plants need more nitrogen in hot climate than the same plants in a cold climate (Kirman, 2007).

Deficiency of nitrogen changes the appearance of leaves from light green to yellow. It also leads to stunted growth and poor fruit development. When there is serious deficiency, the lower leaves dry up and die prematurely (Hanson and Kilmer, 1982).

### **2.9.1.2 Phosphorus**

Phosphorus is an essential part of the process of photosynthesis. All plants require phosphorus during periods of rapid growth. Most annual plants require large amounts of phosphorus during the early age of growth. In most natural ecosystems such as forests and grasslands, phosphorus uptake by plants is constrained by both the

lower quantity of this element in the soils, and by lower solubility of the quantity present (Brady and Weil, 2002). It is a key portion of RNA and DNA molecular containing the coding for protein formation and cellular genetic information. It is bound and stored as phospholipids in cellular membranes and also laid down and stored in seed as phytin (Kirman, 2007). Stored phosphorus in seed is the energy source for its germination and seedling development for the next crop.

The immediate source of phosphorus is that which is dissolved in soil solution. Plants absorb the element primarily as the  $\text{H}_2\text{SO}_4^-$  and  $\text{HPO}_4^{2-}$  ions which are predominant in most soils. A soil solution containing only a few parts per million (ppm) of phosphorus ions is usually considered adequate for plant growth (Uriyoet al., 1983). Established plants such as trees, shrubs and vines, especially those grown in warm climate with longer summer's periods, require the least amount of phosphorus fertilizer.

Deficiency causes stunted growth of plant. Leaves become pale with forward roll and scorched margins. Excess of phosphorus content may cause micronutrient deficiencies especially iron and zinc (Kirman, 2007).

### **Potassium**

Potassium is absorbed by plants in large amounts than any other mineral element except nitrogen. The total potassium content of soils is usually many times greater than the amount taken up by a crop during a growing season and only a small fraction of it is available to plants in most cases. It is as many roles in plant nutrient involving maintenance of electro-neutrality, photosynthesis, enzymes activation, osmotic regulation and photosynthesis transport and storage (Havlinet al., 2003). Potassium has been shown to promote the growth of long strong cotton fibres, increase the shelf life of fruits, stem length and quantity of roses, enhance the green colour and growth of turf grasses and also increases the size and quantity of fruits, grains and vegetable (Uriyoet al., 1983).

Plant deficiency in potassium appears chlorotic, with marginal scorching on the leaves of grasses. Deficiency first appears on the lower leaves because potassium is mobile and

it is redistributed to metabolically active tissue. Root development is also reduced and this in turn reduces the exploitation of the soil for moisture and other nutrients (Hanson and Kilmer, 1982).

### **2.9.1.3 Calcium**

Calcium in soils originated in the rock and minerals from which the soil was formed and its concentration in soil varies. Arid region soils are generally high in calcium content because of low rainfall and its concentration in the soil higher than necessary have little effect on  $\text{Ca}^{2+}$  uptake, because  $\text{Ca}^{2+}$  is generally controlled (Havlin et al., 2003). Increasing the  $\text{Al}^{3+}$  concentration in soil solution reduces  $\text{Ca}^{2+}$  uptake (Benbi and Nieder, 2003).

This element is an essential part of plant cell wall structure. It provides for normal transport and retention of other elements as well as strength in the plant. Its role involves enzymes activations and inhibition, membrane structure and activity cell wall structure and interaction with other elements.

Deficiency of calcium brings about reduced growth or death of growing tips, blossom-end rot of tomato, poor fruit development and appearance in excess, it could cause deficiency of either magnesium or potassium (Brady and Weil, 2002).

### **2.9.1.4 Magnesium**

Magnesium is absorbed by plants as  $\text{Mg}^{2+}$  from the soil solution and the quantity of  $\text{Mg}^{2+}$  taken up by plant is usually less than that of  $\text{Ca}^{2+}$  or part of chlorophyll in all green plants and essential for photosynthesis. It helps to activate many plant enzymes needed for growth. Magnesium concentration also influences the proper formation of cellular organelles, as well as carbohydrates metabolism and protein synthesis (Hanson and Kilmer, 1982). Soil minerals, organic material, fertilizers and dolomitic limestone are sources of magnesium for plants, (Uriyo et al., 1983). With magnesium deficiency, monocots exhibit a light green colour with stripping while dicotyledonous plants exhibits chlorosis, followed by a purpling and necrosis when in excess, the high

concentration of Mg is tolerated in plant, but, imbalance with calcium and potassium may reduce growth (Harbon and Kilmer, 1982).

#### **2.9.1.5 Iron**

Iron is important in chlorophyll production and photosynthesis functioning through ferredoxin. It also functions in the activity of enzymes, namely, catalase, peroxidases and cytochrome oxidase. Only a small portion of the total plant iron is active, while the bulk of it stored as Fe-P protein, phytoferritin (Hanson and Kilmer, 1982). Iron deficiency is most often observed on high pH and calcareous soils in arid regions, but it may also occur on acidic soils that are very low in total iron. Compounds of iron in soil are indicators of drainage and weathering conditions and apart from affecting structure, they are one of the principal cause of soil colour (Hesse, 2002). Its deficiency causes initial distinct yellow or white areas between veins o young leaves leading to spots of dead leaf tissues. In excess, there is possible bronzing of leaves with tiny brown spots (Kirman, 2007).

#### **2.9.1.6 Manganese**

Manganese is absorbed by plants in the form of  $Mn^{2+}$ . It may be used to substitute magnesium by activating certain phosphate transferring enzymes, which in turn affect many metabolic processes. Manganese availability is closely related to the degree of soil acidity (Uriyoet al., 1983). Leaves of monocots that are manganese deficient are light green and develop an alternate intervenial stripping. Deficiency in dicots shows up in new leaves, with clitorises or bleaching between the veins. Chloroplasts in manganese deficient plants are neither properly nor do they function properly. With excess of manganese, older leaves develop brown spots surrounded by a chlorotic circle or zone (Kirman, 2007).

### **2.9.1.8 Aluminium**

Aluminium is the most commonly recurring metallic element, comprising 7.5 to 8.1% of the earth crust. It is a major component of almost all common inorganic soil particles with the exception of quartz, sand, clay fragments, and ferromanganiferous concentrations (Ecological Soil Screening Level, 2002). Aluminium is very rare in its free form and contributes greatly to properties of soil, where it is present mainly as insoluble aluminium hydroxide (Lenntech, 2008). Aluminium toxicity occurs in strongly acid soils of pH below 5.0 and excess aluminium accumulation in roots reduces their capacity for translocations of phosphorus and hinders uptake of copper by plants (Onanuga, 2008).

## **2.9.2 Trace Minerals Elements**

### **2.9.2.1 Sulphur**

In most soils, sulphur is present primarily in the organic fraction which becomes available upon decomposition of organic matter and crop residue. It plays a major role in protein synthesis through the amino acids cystine and methionine. It is taken up primarily in form of  $\text{SO}_4^{2-}$  ions and is reduced and assembled into organic compounds. It is also present in glycosides which give the characteristic odour and flavour in mustard, onion and garlic plants (Das, 1997). It is required in modulation and nitrogen fixation in legumes. Sulphur deficiency causes stunted to tall spindly growth, initial yellowing of younger leaves spreading to the whole plant and also reduced amount of seed or crops. Excess of sulphur may cause premature dropping of leaves (Kirman, 2007).

### **2.9.2.2 Copper**

Copper is essential for growth and activates many enzymes in plants. It is associated with the organelles, such as the chloroplasts. It is required for plastocyanin, a chloroplast protein. It is also essential for the formation of several oxidase enzymes including cytochrome oxidase, ascorbate oxidase and polyphenol oxidase. Excess copper can be toxic to plants and this is liable to occur where copper compounds are applied as fungicides, particularly on acid soils (Hesse, 2002).

It deficiency in grasses such as corn shows up initial in young leaves, with protein synthesis and causes a building of soluble nitrogen compounds. In excess, symptoms appears in young tissues and include dark green leaves followed by induced iron chlorosis in which the leaves may appear nearly white, thick, short, or barbed-wire looking roots (Kirman, 2007).

### **2.9.2.3 Zinc**

Zinc is essential for plant growth because it controls the synthesis of idealistic acid, which dramatically regulates plant growth (Hanson and Kilmer, 1982). It arts as a chemical catalyst, assisting ssother elements to perform their functions. Its deficiency causes interveinal yellowing on young leaves and also reduced leaf size. Excess of it may cause iron deficiency in some plants (Kirman, 2007).

### **2.9.2.4 Molybdenum**

Molybdenum is necessary in both plant and animal nutrition. Generally when soil pH is 6.0 or higher, its deficiency will not occur. It functions largely in the enzyme systems of nitrogen fixation and nitrate reduction in soils and plants (Brady and Weil, 2002). Molybdenum is strongly absorbed by Fe/Al oxides, a portion which becomes unavailable to the plants. Soils that are high in amorphous Fe/Al oxide tend to be low in available molybdenum (Havlinet al., 2003). Its deficiency makes leaf edges to curl upward on the whole plant and also 'whip tail' in cauliflowers and 'blossom and rot' in tomatoes. Excess of it turns leaves to intense yellow or purple colour (Kirman, 2007).

## **2.10 Heavy Metals Contamination in the Soil**

Soil contamination is the presence of man-made chemical or other alteration to the natural soil environment. Soil contamination may disrupt the delicate balance of physical, chemical processes, which are the basis of soil fertilizer (Bolt, 1991). This contamination typically arises from rapture of underground storage tanks, application of pesticides and herbicides, direct discharge of industrial wastes to the soil(Soil

Contamination, 2010). Pollution of soil by heavy metals and organic chlorine compounds may inhibit microbial enzymes activity and decrease the diversity of population of soil flora and fauna. Most soil are capable to some degree, of sorting out and neutralizing many contaminants and pollutant to harmless level through chemical (abiotic) and biochemical (biotic) processes (Pierzynskiet al., 2005).

### **2.10.1 Sources of Heavy Metal Pollution in Soil**

- 1. Atmospheric Pollution from Motor Vehicles:** The use of leaded petrol has been responsible for the global dispersion of Pb aerosols.
- 2. The Combustion of Fossil Fuel:** This result in dispersion of many elements in the air oven a large area.
- 3. Agricultural Fertilizers and Pesticides:** Several of these including phosphatic fertilizer, stages from iron manufacture, pesticides and herbicides contain various impurities.
- 4. OrganicManures:** These include pig and poultry manures which contain high concentration of effluence. Sewage sludge's usually contain relatively high concentration of several metals.
- 5. Disposal of Urban and Industrial Waste:** The careless (unauthorized) dumping or disposal of metal-containing items, ranging from miniature dry cell batteries (Ni, Cd and Hg) give rise to heavy metal concentration in soil.
- 6. Mining and Smelting of Non-Ferrous Metals:** These processes disperse metals in dusts, effluents and seepage water. In the right concentrations, many metals are essential to life and in excess these same elements can be poisonous, similarly, chronic low exposure to heavy metals can have serious health effects on the long run. The main threats to human well-being are associated with lead, arsenic cadmium and mercury (Alloway, 1990).

### **2.10.2 Arsenic**

Arsenic is the most common cause of acute heavy metal poisoning in adults. Arsenic is released into the environment by smelting process of copper, zinc and lead, as well as the manufacturing processes of chemicals and glasses. Arsenic gas is a common by product produced by the manufacturing of pesticides that contain arsenic. Arsenic may also be found in water supplies worldwide. Human are primarily exposed to arsenic through drinking water and foods. Arsenic can occur in agricultural soil in some regions, as a consequence of the use of arsenic containing pesticide and herbicides. Inorganic arsenic is more toxic than organic forms, and its effects, peripheral neuropathy and kidney damage (Kahakachchiet al., 2004).

### **2.10.3 Lead**

Lead is nearly always present in soils although in small amounts. The solubility of lead in soil increase with acidity and in acid soils it is accumulated by plants. Food plants may acquire toxic (to the consumer) amounts of lead. Nowadays, there is a danger of land near motorways becoming contaminated with lead from exhaust fume (Hesse, 2002).

### **2.10.4 Cadmium**

Cadmium is a borderline metal and one of the most toxic metals. It is believed to be the dangerous form of trace elements contamination in soil. Cadmium exposure occurs mainly through cereals and vegetables grown on soils contaminated by mining activities and use of phosphorus fertilizers. Shell fish and animal organs also contain high level of cadmium. Cadmium accumulates in the kidney and is implicated in a range of kidney disease (Onanuga, 2008).

### **2.10.5 Mercury**

Mercury is the only metal in liquid form in nature. It can volatilize and be transported through the atmosphere. Certain micro-organisms can mutilate it and aid its chemical form (Bolt et al., 1991). At level well below WHO limits, it can damage the total and embryonic nervous system with consequent learning difficulties, poor memory and shortened attention spans (Jorgensen et al., 1997).

### **2.10.6 Principle Method of Determination Using X-ray Fluorescence**

A secondary fluorescence photon is emitted by the sample, under the bombardment of primary photons emitted by the X-ray tube (XRF principle). Secondary photons are emitted in all directions, but we consider here only the directions which bring them to the detector.

The secondary photon enters the detector. It is converted to an electrical impulse. The height (amplitude) of this pulse is directly proportional to the energy of the photon. The electrical impulse is processed by the Multi-channel analyzer. This unit determines (measures) the pulse height (via a very fast analog-to-digital converter). The pulse's height is converted into a specific channel address. The channel memory of concern (in this example, the channel dedicated to the "red energy photons" is then incremented.

## **2.11 Soil Uses**

### **2.11.1 Agriculture**

Soil is used in agriculture, where it serves as the primary nutrient base for plants, however, as demonstrated by hydroponics, it is not essential to plant growth if the soil-contained nutrients could be dissolved in solutions, soil provides minerals and water to plant. The types of soil used in agriculture (among other things, such as the purported level of moisture in soil) vary with respect to species of plants that are cultivated (Ponge and Jean Francois, 2003).

### **2.11.2 Mining and Construction**

Soil material is a critical component in the mining and construction industries. Soil serves as a fundamental for most construction projects. Massive volumes of soil can be involved in surface mining, road building and dam construction. Earth sheltering is the architectural practice of using soil for external thermal mass against building walls (De Deynet al., 2005).

### **2.11.3 Environmental Protection**

Soil resources are critical to the environment, as well as to food and fiber production. Soil absorbs rainwater and releases it later, thus preventing floods and drought. Soil cleans the water as it percolates. Soil is the habitat for many organisms, the major part of known and unknown biodiversity is in the soil, in the form of invertebrates (earthworms, woodlice, millipede, centipedes, snails, slugs, mites, springtails, enchytracids, nematodes, protists), bacteria, archaea, fungi and algae; and most organisms living above ground have part of them (plants) or spend part of their life cycle (insects) below ground and below-ground biodiversities are tightly interconnecting (De Deynet al., 2005), making soil protection of paramount importance for any restoration or conservation plan.

### **2.11.4 Waste Management and Consumption**

Waste management often has a soil component. Septic drain fields treat septic tank effluent using aerobic soil processes. Landfills use soil for daily cover. Land application of waste water relies on soil biology to aerobically treat BOD. Organic soils, especially peat, serves as significant fuel resources, such as sphagnum bogs, are now protected because of patrimonial interest. Both animals and human in many cultures occasionally consume soil, together with their preferred food (tree foliage and fruits), in order to alleviate tannin toxicity, (Enzweileret al., 1999).

### **2.11.5 Purification Process**

Soils filter and purify water and affect its chemistry. Rain water and pooled water from ponds, lakes and rivers percolate through the soil horizons and the upper rock strata, thus becoming groundwater. Pests and pollutants, such as persistent organic pollutants, such as persistent organic pollutants (chlorinated pesticides, polychlorinated biphenyls), oils (hydrocarbons), heavy metals (lead, zinc, cadmium), and excess nutrients (nitrate, sulfates, phosphates) are filtered out by the soil (Kohne, 2009). Soil organisms metabolize them or immobilize them into their biomass and necromass (Diplock et al., 2008), thereby incorporating them into stable humus, (Moeckel et al., 2008). The physical integrity of soil is also a prerequisite for avoiding landslides in rugged landscapes (Rezaei et al., 2009).

## **2.12 Degradation of Soil**

Land degradation is a human induced or natural process which impairs the capacity of land to function. Soils are the critical component in land degradation when it involves acidification, contamination, desertification, erosion and salination. While soil acidification of alkaline soil is beneficial, it degrades land when soil acidity lowers crop productivity and increases soil vulnerability to contamination and erosion. Soil is often initially acid because their parent materials were acid and initially low in the basic cations (calcium, magnesium, potassium and sodium). Acidification occurs when these elements are removed from the soil profile by normal rainfall, or the harvesting of forest or agricultural crops. Soil acidification is accelerated by the use of acid forming nitrogenous fertilizers and by the effects of acid precipitation (Jorgensen et al., 1997).

### **2.12.1 Soil Pollution**

Contamination at low level is often within soil capacity to treat and assimilate. Many waste treatment processes rely on this treatment capacity. Exceeding treatment capacity can damage the soil biota and limit soil function. Derelict soils occur where industrial contamination or other development activity damages the soil to such a degree that the land cannot be safely or productively. Remediation of derelict soil uses

principles of geology, physics, chemistry and biology to degrade, attenuate, isolate or remove soil contaminants to restore soil functions and values. Techniques include leaching, air sparging, chemical amendments, phytoremediation, bioremediation and natural attenuation (Jorgensen et al., 1997).

### **2.12.2 Desertification**

This is an environmental process of ecosystem degradation in arid, semi-arid regions, often caused by human activity. It is a common misconception that droughts cause desertification. Droughts are common in arid and semi-arid lands. Well-managed land can recover from drought when the rain returns. Soil management tools include maintaining soil nutrient and organic matter levels, reduced tillage and increased cover. These practices help to control erosion and maintain productivity during periods when moisture is available. Continued land abuse during droughts, however, increases land degradation (Jorgensen et al., 1997).

### **2.12.3 Soil Erosion**

Soil erosional loss is caused by wind, water, ice and movement in response to gravity. Although the process may be simultaneous, erosion is distinguished from weathering. Erosion is an intrinsic natural process, but in many places it is increased by human land use, poor land use practices including deforestation, overgrazing and improper construction activity. Improved management can limit erosion by using techniques like limiting disturbance during erosion prone periods intercepting runoff, terrace-building, use of erosion-suppressing cover materials, and planting trees or other soil building plants.

Soil piping is a particular form of soil erosion that occurs below the soil surface. It is associated with levee and dam failure, as well as sink hole formation. Turbulent flow removes soil starting from the mouth of the seep flow and subsoil erosion advances up gradient (Jones et al., 1976). The term sand boil is used to describe the appearance of the discharging end of an active soil pipe (Dooley, 2006).

#### **2.12.4 Soil Salinity**

This is the accumulation of free salts to such an extent that it leads to degradation of soils and vegetation. Consequences include corrosion damage, reduced plant growth, erosion due to loss of plant cover and soil structure, and water quality problems due to sedimentation. Salination occurs due to combination of natural and human caused processes. An arid conditions favour salt accumulation. This is especially apparent when soil parent material is saline. Irrigation of arid lands is especially problematic (ILRI, 1989). All irrigation water has some level of salinity. Irrigation, especially when it involves leakage from canals and over irrigations in the field, often raises the underlying water table. Rapid salination occurs when the land surface is within the capacity fringes of saline groundwater. Soil salinity control involves water table control and flushing with higher levels of applied water in combination with tile drainage or another form of subsurface drainage (Drainage, 1993).

## CHAPTER THREE

### MATERIALS AND METHODS

#### 3.1 Study Area

The study was carried out at Mambilla Plateau found in the south-eastern part of Taraba state of Nigeria (see Figure 3.1 and 3.2), under Sarduna Local Government Area. The Mambilla Plateau measure about 96km (60m) along its curved length it is 40km (25m) wide and bounded by an escarpment that is about 900m (2,953 ft) high in some places. The plateau covers area of over 9,389 square kilometres (3,625 sq mi) with daytime temperature hardly exceeds 25<sup>0</sup>C (77.0<sup>0</sup>F).

#### 3.2 Sampling

Sampling was done by digging, after clearing with cutlass, at 5 - 25cm depth using hoe and a ruler for the measurement. For each location, three samples weighing about 500 g were collected at three points. This was mixed, dried and quartered to obtain a representation of that location, and finally introduced into a polyethylene bag, after being ground using mortar to pass through a 2mm sieve.

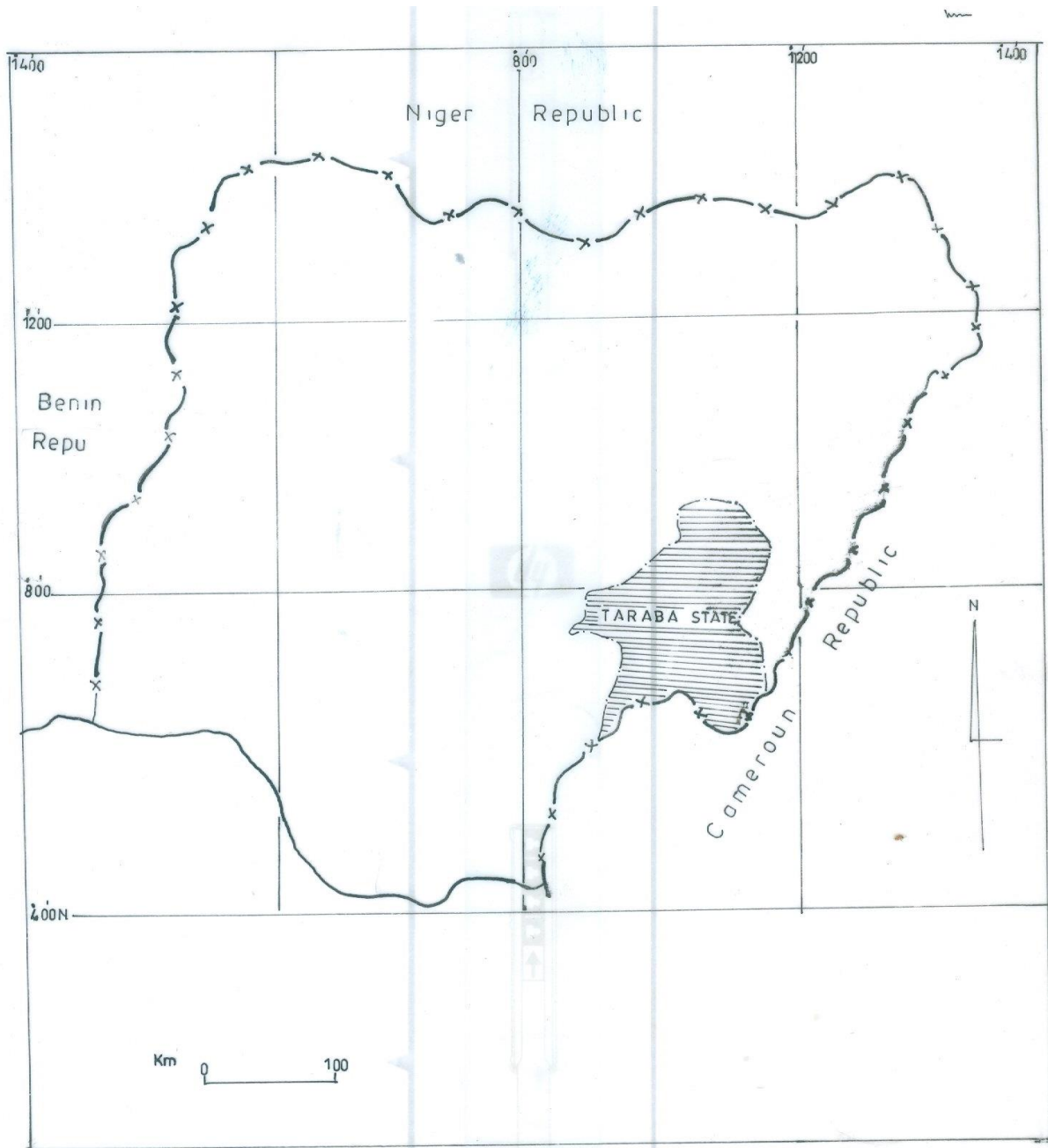


Fig. 3.1 Map of Nigeria Showing Taraba State

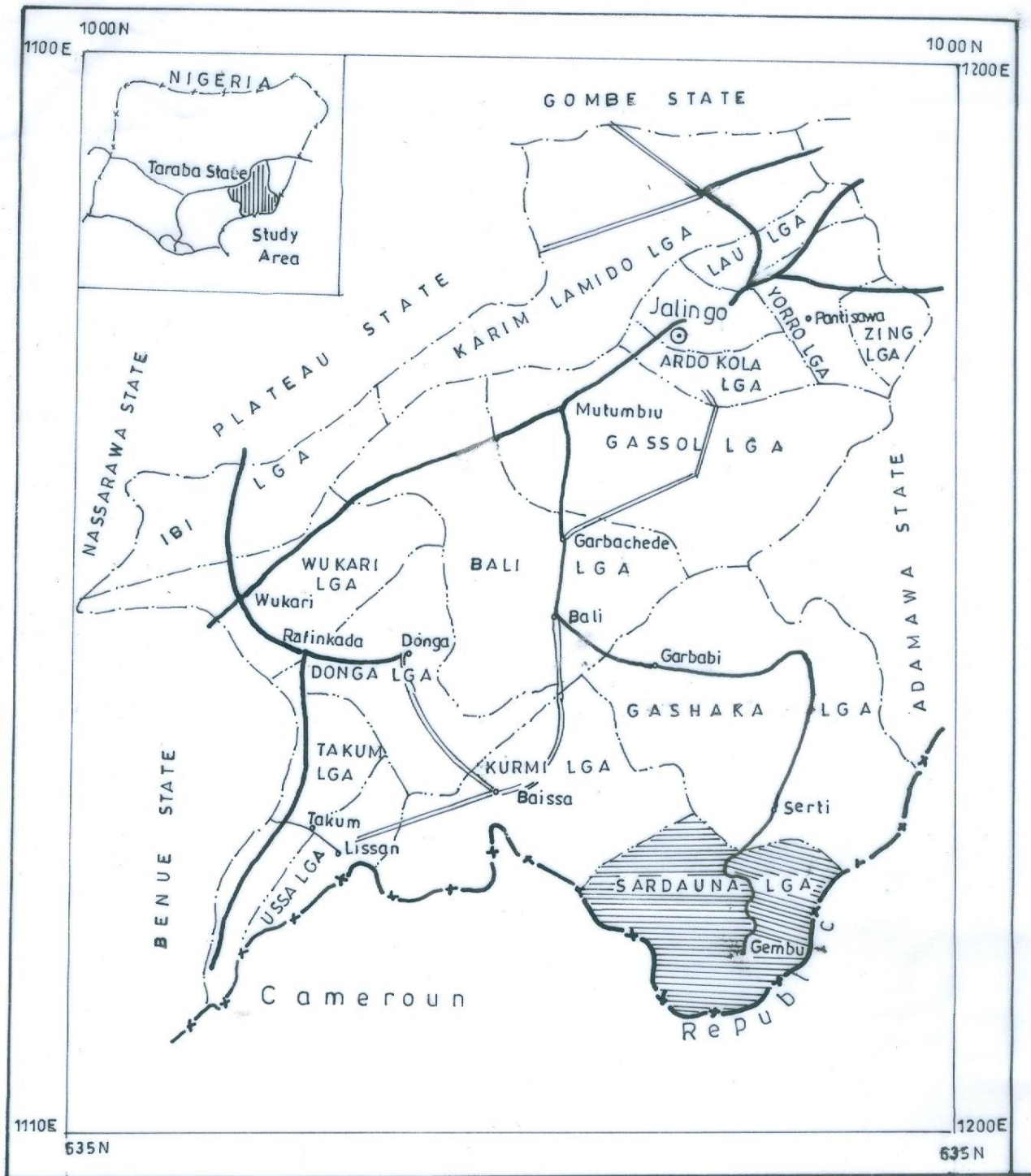


Fig 3.2 Map of Taraba State Showing the Study Area

Samples of soils were collected starting from the foot of the mountain as indicated in Figure 3.3 (Mayo-Selbe Village), at a distance of 3km interval moving through hilly with deep gorges from one panoramic view to the other, to the first tip settlement (Maisamari village). The sampling collections then proceeded from the first settlement to Ngoroje town where soil samples were randomly collected from 3 villages. Samples collection extended from Ngoroje to Mayo Ndaga (eastern part of the plateau), through 7 villages which finally acute at Gembu Town (southern part of the plateau), the headquarters of Sardauna local government area. A total of 12 samples were collected and labeled as

N<sub>1</sub>:Mayoselbe Village:

N<sub>2</sub>:Maisamari Village

N<sub>3</sub>:Yelwa Village

N<sub>4</sub>:Nguroje Town

E<sub>1</sub>:Kakara Village

E<sub>2</sub>:TungaShaibu Village

E<sub>3</sub>:Papa village

E<sub>4</sub>:Mayondaga Town

S<sub>1</sub>:Lekkitaba Village

S<sub>2</sub>:Gembu Town

S<sub>3</sub>:Mayodule Village

S<sub>4</sub>:MbamgaTown

### **3.3 Analytical Studies of Soil**

#### **3.3.1 Determination of Soil Colour**

The colour of each soil sample was determined by comparing the soil colour in its field condition to a series of colour clips in a booklet called soil colour chart (Munsell, 1973). Each chip will describe uniquely by position on three axes of colour; hue or spectral colour, value or degree of darkness, and chroma, purity or strength of colour (Tel and Hagarty, 1984). The soil sample paste was held behind the page of the chips

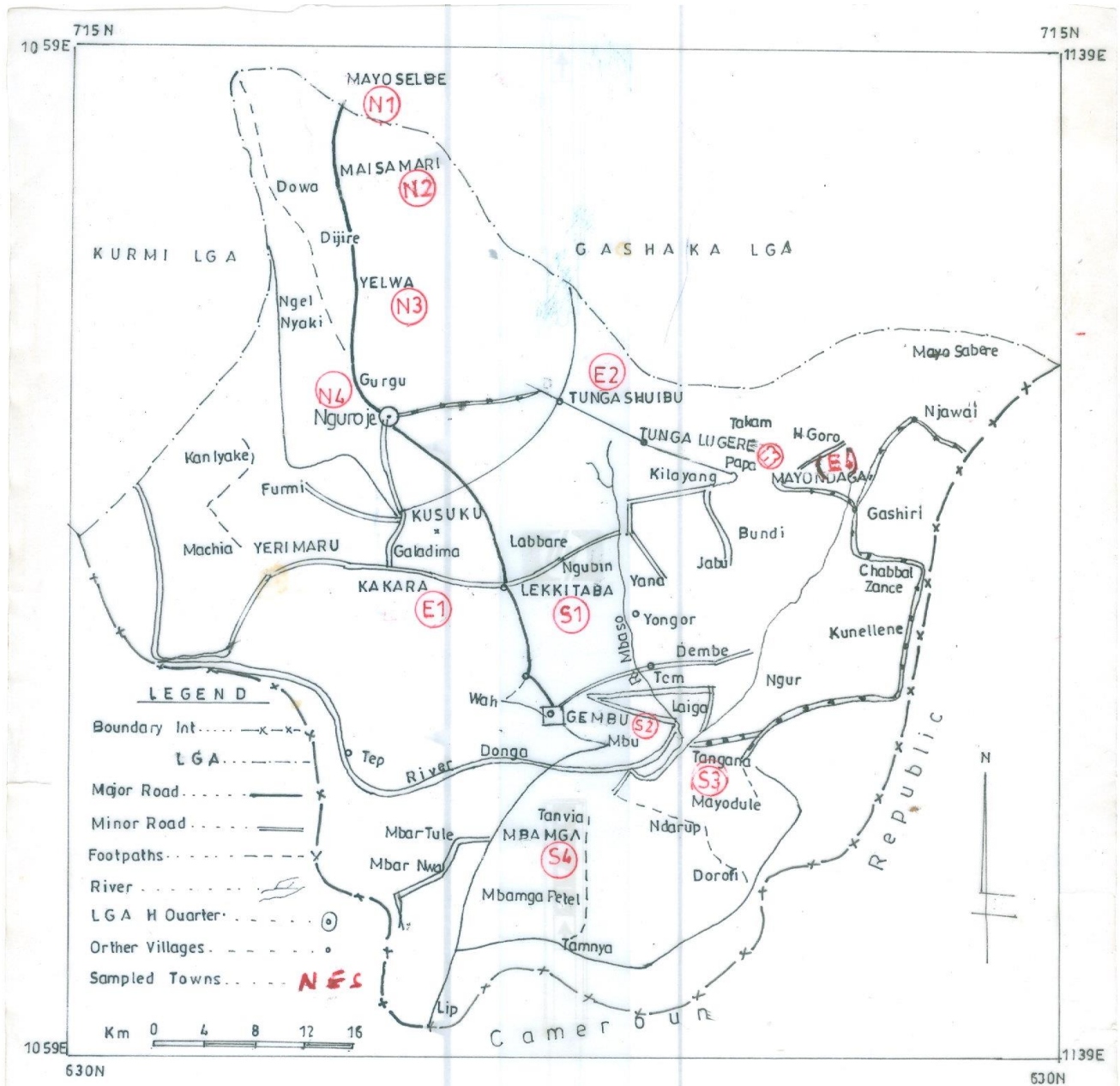


Fig 3.3 Map of Study Area indicating sampling locations

solute so that the soil colour matches any of any of the colours of chips seen through the holes punched on the page then, the colour was then recorded as its colour both in name and standard notation.

### 3.3.2 pH Measurement

About 5g of each air-dried and sieved soil sample was used, 40cm<sup>3</sup> distilled water was then added and stirred with glass rod for 15 minutes for equilibration of the contents. The pH meter was standardized using standard buffer solution. The pH of the aqueous sample suspension was read off after inserting the sensio pH meter electrode into each soil suspension and gently swirling the breaker so as to ensure accurate reading. The reading was taken after it had attained stability

### 3.3.3 Conductivity Measurement

The aqueous sample that was prepared and used for the pH measurement was also used for conductivity measurement. The sensio conductivity metre was checked and the electrode cleaned using distilled water and was dipped into the aqueous sample solution. The reading was taken when pointer becomes steady.

### 3.3.4 Specific Gravity Measurement

The specific gravity of the sample was determined using a specific gravity bottle as describe by a bureau of Indian standards (IS2720, 1980). 12.5 grams of the sample was used. The specific gravity of the sample was calculated from (BIS, 1980).

$$S. G = \frac{(W_2 - W_1)}{[(W_4 - W_1)(W_3 - W_2)]}$$

where:  $W_1$  weight of bottle  
 $W_2$  weight of bottle + sample  
 $W_3$  weight of bottle + sample + water  
 $W_4$  weight of bottle + water

### 3.3.5 X-Ray Fluorescence Analysis

The concentrations of Ca, Mg, Al, Ti, V, Si, S, P, K, Na, As, Cu, Ni, Cr, Fe, Mn, Pb, Zn, Fe, Hg, Au, Se and Mo in the soil samples were determined using X-ray fluorescence (XRF) spectrophotometer as described below.

Each soil sample Pellets of 19mm diameter was prepared from 5.0g powder, mix with 1.0g cellulose flakes binder and palletized at a pressure of 10 ton/inch in a palletizing machine. The palletized samples were stored in a desecator for analysis. The machine (XRF) was switched on and allowed to warm up for 2 hours. Finally, appropriate programs for the various elements of interest were employed to analyze the sample materials for their presence or absence. The measurement was performed using rhodium as the excitation source. The fluorescent x-ray emitted was sent to the Si (Li) detector, coupled to a computer controlled ADC - card.

Since each element has a characteristic and identifiable x-ray signature, the presence and concentration of each element of interest within the sample was determined by looking at specific part of the spectrum and counting the number of the pulses registered. A computer will be used to perform the analysis of the data.

## CHAPTER FOUR

### RESULTS AND DISCUSSION

#### 4.1 Physical Properties of Soil Samples.

The results for the analysis of the physical properties of the soil samples are presented in Table 4.1

##### 4.1.1 Soil Colour

Sample E<sub>3</sub> had brown colour representing a single colour, sample E<sub>1</sub> and N<sub>2</sub> were dark reddish brown while sample E<sub>4</sub> was dark reddish gray; sample E<sub>2</sub> and N<sub>4</sub> were reddish brown in colour. The brown colours of the soil were perhaps due to the high magnetite content in the soil (Sakar et al., 2001). Sample S<sub>3</sub> shows gray colour and N<sub>1</sub> shows light gray colour. However sample N<sub>3</sub> and S<sub>2</sub> were yellowish red; S<sub>1</sub> and S<sub>4</sub> were weak red and strong brown respectively. The yellowish, reddish colours shown by the samples may be due to high oxidized iron oxides (Brady and Weil, 2002).

The development and distribution of colours are due to the extensive and various iron minerals as it result from chemical and biological weathering, especially redox reaction. The primary minerals in soil parent material are weathered, the elements combine into new and colourful compounds, iron forms secondary minerals with a yellow or red colour, organic matter decomposes into black and brown compound (USDA, 2008).

##### 4.1.2 pH of the Soil Samples

The pH analysis of samples conducted confirmed that the soils are slightly acidic, with a mean value of  $4.13 \pm 0.22$  in a range of 3.62 - 4.52. This verifies that the parent's materials of these samples were low in soluble ions (calcium, magnesium, potassium and sodium), water moving through the soil removes the bases and substitutes them with hydrogen ions making the soil acidic and unsuitable for Agriculture (Michael, 2003).

### **4.1.3 Electrical Conductivity**

The electrical conductivity of most of the sample were somewhere less than 200 $\mu$ S/cm, meaning there are no enough essential nutrients available in the soil to support plants. This could perhaps show a sterile soil with little microbial activity (Eshanic et al., 2006). Sample N<sub>1</sub> was exceptional of all samples with electrical conductivity above 200 $\mu$ S/cm.

**Table 4.1 Physical Properties of Soil Samples**

<b>Physical Properties of Soil Samples</b>					
<b>Sample Code</b>	<b>Soil Colour</b>	<b>Standard Notation</b>	<b>Specific Gravity</b>	<b>pH</b>	<b>Conductivity</b>
E <sub>1</sub>	drb	5YR3/4	2.21	4.12	93.00
E <sub>2</sub>	rb	5YR 5/4	2.12	4.26	89.30
E <sub>3</sub>	b	7.5YR 4/4	2.33	4.24	153.60
E <sub>4</sub>	drg	5YR 4/2	2.50	4.11	136.70
<b>RANGE</b>			<b>2.12-2.50</b>	<b>4.11-4.26</b>	<b>93.00-153.60</b>
<b>MEAN</b>			<b>2.29<sub>±</sub>0.14</b>	<b>4.18<sub>±</sub>0.07</b>	<b>118.15<sub>±</sub>29.68</b>
N <sub>1</sub>	lg	10YR7/2	2.35	4.52	238.00
N <sub>2</sub>	drb	5YR 3/4	2.00	3.93	183.20
N <sub>3</sub>	yr	5YR5/6	2.09	4.09	174.30
N <sub>4</sub>	rb	5YR5/4	2.31	3.62	93.00
<b>RANGE</b>			<b>2.00-2.31</b>	<b>3.62-4.52</b>	<b>93.00-238.00</b>
<b>MEAN</b>			<b>2.19<sub>±</sub>0.15</b>	<b>4.04<sub>±</sub>0.32</b>	<b>172.13<sub>±</sub>51.78</b>
S <sub>1</sub>	wr	2.5YR5/2	2.24	4.29	81.79
S <sub>2</sub>	yr	5YR5/6	2.36	4.30	101.90
S <sub>3</sub>	g	5YR 6/1	2.39	4.16	120.10
S <sub>4</sub>	sb	7.5YR5/6	2.37	3.96	100.20
<b>RANGE</b>			<b>2.24-2.39</b>	<b>3.96-4.30</b>	<b>81.79-120.10</b>
<b>MEAN</b>			<b>2.34<sub>±</sub>0.09</b>	<b>4.18<sub>±</sub>0.13</b>	<b>100.10<sub>±</sub>13.56</b>
<b>RANGE</b>			<b>2.00-2.50</b>	<b>3.62-4.52</b>	<b>18.79-238.0</b>
<b>OVERALL MEAN</b>			<b>2.27<sub>±</sub>0.14</b>	<b>4.13<sub>±</sub>0.22</b>	<b>130.42<sub>±</sub>46.17</b>

b= brown; drb=dark reddish brown; drg=dark reddish gray; g=gray; lg=light gray; sb=strong brown; rb=reddish brown;wr= weak red; yr= Yellow red;

#### **4.1.4 Specific Gravity**

The specific gravity of the samples was in a range of 2.00 – 2.500g/cm<sup>3</sup>. This result confirms the soil is made up of mineral particles with very low organic matter content (USDA, 2008).

#### **4.2. Chemical Properties**

All metal contents detected in the samples of the study locations are grouped into major, minor and trace elements in their compound form (Appendix 2 - 4).

##### **4.2.1 Major Elements**

The major elements detected in samples were AL<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, K<sub>2</sub>O, SiO<sub>2</sub> and TiO<sub>2</sub> (Table 4.2). The order of their concentrations is SiO<sub>2</sub>>FeO<sub>3</sub>>AL<sub>2</sub>O<sub>3</sub>>TiO<sub>2</sub>>K<sub>2</sub>O with concentration of Fe<sub>2</sub>O<sub>3</sub> highest than SiO<sub>2</sub> in some cases.

##### **4.2.2 Aluminium**

The concentration of aluminium ores in the samples was in a range of 9.00 – 27.00% with a mean value of 16.59±5.11, this was observed to be normal as reported by Agronomic Library, (2008). It reduces plant growth on arid soil, although generally harmless to plant in pH neutral soils. The concentration in arid soils of toxic Al<sup>3+</sup>cations increases and disturbs root growth and function (Horst et al., 1995).

##### **4.2.3 Iron**

Iron content in the samples was observed to be high, within the range of 10.06-46.5% as reported by Havlin et al., (2003) it indicates the availability of hematite (dark reddish) in soil due to effect of the soil pH. Higher soil pH reduces iron availability, while arid soils increase iron availability. The higher pH effect in soil increased in water logged, compacted or other poorly aerated soils. One factor to this effect is the presence of higher carbonate in the soil (dark brown soil), which also plays a role in water logged soils and in the root rhizosphere reaction to certain other nutrient and fertilizer source (Spectrum Analytical, 2011).

#### **4.2.4 Potassium**

Potassium ores were detected in five samples only, namely, E<sub>3</sub>(1.50), E<sub>4</sub>(1.10), N<sub>1</sub>(2.10), S<sub>2</sub>(1.10) and S<sub>4</sub>(1.90) with a mean value of 1.54.

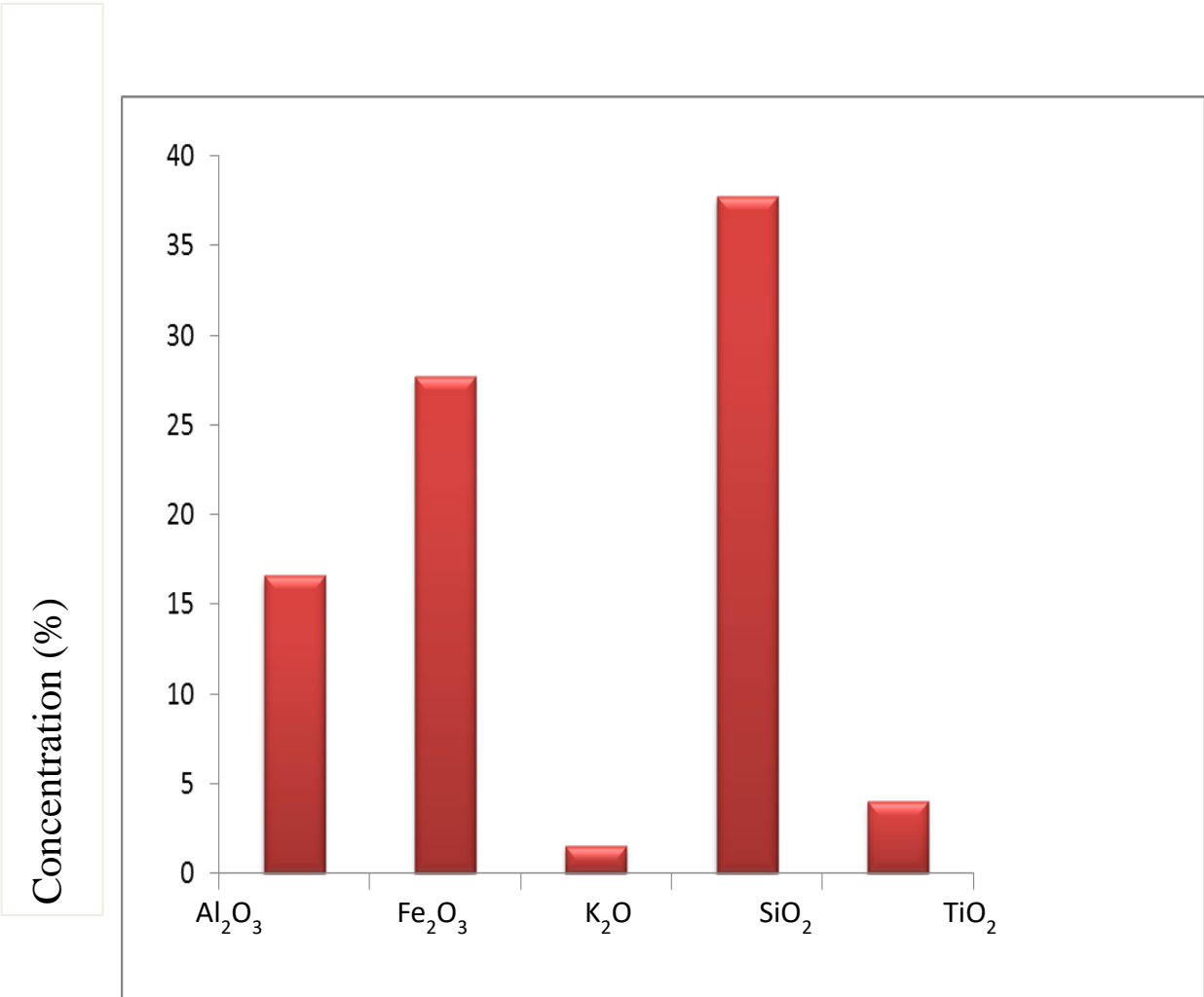
The absence or loss of this soil potassium may occur in soil due to leaching or erosion. Parent materials composition has a direct impact on soil properties; especially soils developed over sand stone are low in soluble bases and coarse in texture which facilitates leaching (Micheal, 2011).

#### **4.2.5 Silicon**

The percentage availability of silicon ores was satisfactory as the most abundant element on the earth crust as reported by Havlin et al., (2003). Low percentage were recorded in few samples, this may be as a result of elemental percentage competitiveness with other most available element such as aluminium and iron (Appendix 2).

#### **4.2.6 Titanium**

Titanium ores were highly detected within a range of 1.5–7.89% and a mean value of 4.00±1.85. This indicates the presence of umenite ore, the most widely spread titanium chloride bearing ore around the world (Earnshaw, 1984).



### **4.3.0 Minor Elements**

The detectable minor elements in the analyzed samples were BaO, CaO and MnO (Appendix 3). Calcium occurs largely, followed by manganese and then Barium, was only detected in seven samples.

#### **4.3.1 Barium**

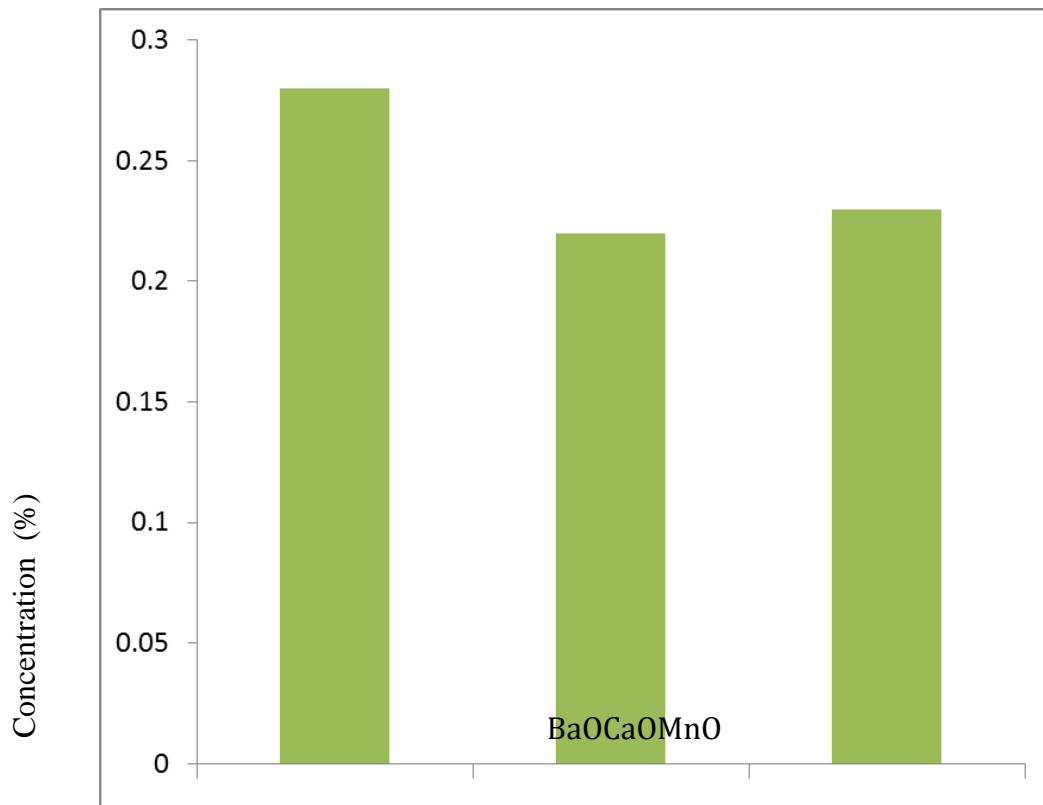
The concentration of Barium oxide in the seven detectable samples ranged from 0.02–0.48% with a mean value of  $0.28 \pm 0.17$ , it was lower compared to concentration detected by Shacklette and Boerngen (1984). No trace was recorded in the other five samples. Barium become soluble at lower pH and is leached from water shed soil by acidic deposition (Heart and Lungrish, 2003).

#### **4.3.2 Calcium**

The concentration of calcium in all the samples was significantly found to be in the range of 0.01–0.67% and a mean value of  $0.22 \pm 0.17$  as compare to work carried out by Onanuga (2008). As concentration become lower in the samples due to effect of the soil pH (spectrum analytical 2011). Acid soils have less calcium, and high pH soil normally have more. As the soil pH increases above 7.2, due to addition of soil calcium, the additional “free” calcium is not absorbed into the soil. Much of the free calcium forms nearly insoluble compounds with other elements such as phosphorus (Spectrum Analytical, 2011).

#### **4.3.3 Manganese**

The concentration of detectable manganese in the samples was within the range of 0.04–0.55% and a mean value of  $0.23 \pm 0.14$ , as compare to work reported by Saxena (1990). The concentration is said to be higher in soil sample with low pH value which increases its availability even to the point of toxicity (Spectrum analytical, 2011). Manganese can also be “tied” by organic matter such that high organic matter can be manganese deficient (Spectrum Analytical, 2011).



#### **4.4. Trace Elements**

The trace metals analyzed in this study were  $\text{CrO}_3$ ,  $\text{CuO}$ ,  $\text{NiO}$  and  $\text{V}_2\text{O}_5$ , in their compound state. Other metals like  $\text{Pb}$ ,  $\text{Cd}$ ,  $\text{Hg}$  etc were not detected in any of the samples. The absence of these elements might be due to their concentration below detection limit of the instrument used for the determination. The order of these detectable trace element is  $\text{V}_2\text{O}_5 > \text{CrO}_3 > \text{NiO} > \text{CuO}$ .

##### **4.4.1 Chromium**

The concentration of chromium ores detected in the samples was in the range of 0.04–0.25% and a mean value of  $0.12 \pm 0.06$  (Appendix 4), which falls within normal range as indicated by Saxena (1990). This suggests that the soils are not contaminated by the elements, and their content could be attributed to the parent materials of the soils.

##### **4.4.2 Copper**

Copper oxide was also detected in seven samples only with a range of 0.001 – 0.17 and a mean value of  $0.043 \pm 0.05$ . No detection was recorded in  $\text{E}_3$ ,  $\text{N}_1$ ,  $\text{N}_2$ ,  $\text{N}_3$  and  $\text{N}_4$  Samples. The availability of copper in soil is also the function of the soil pH and texture (George and Michael, 2009). The finer-texture mineral soils generally contain the highest amount of copper, the lowest concentrations are associated with the organic or peat soils. As soil pH increases, the availability of these nutrients decreases (George and Michael, 2009).

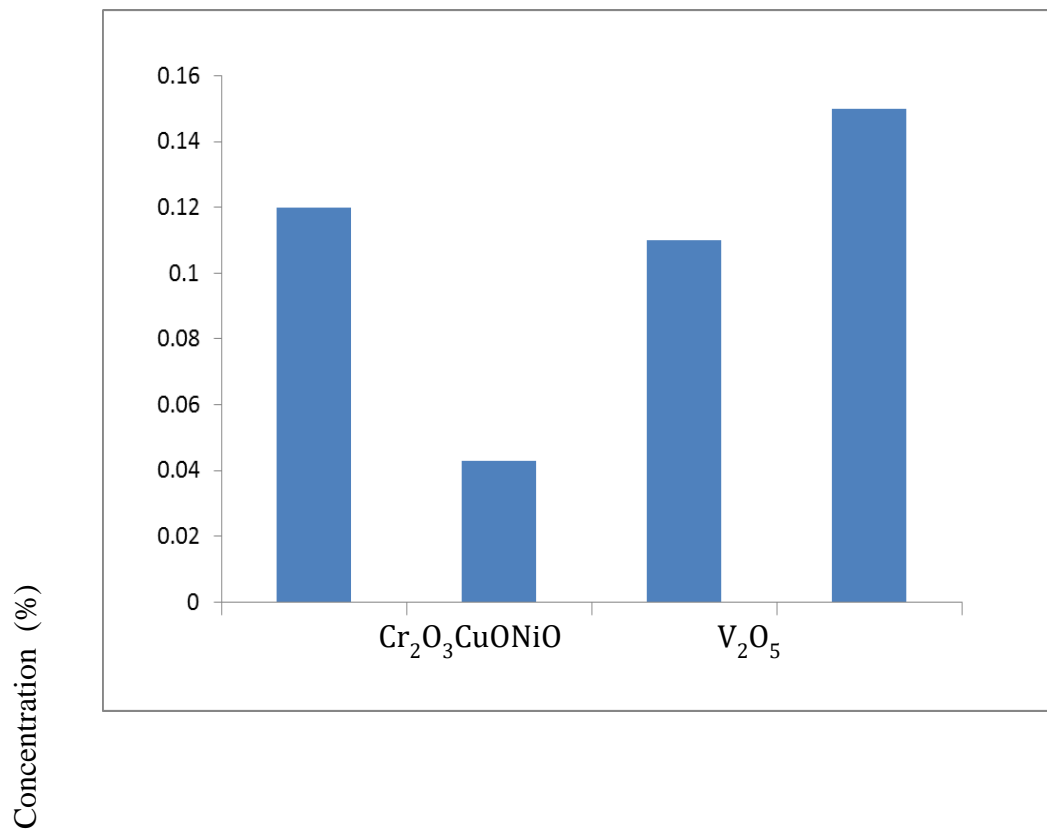
##### **4.4.3 Nickel**

Nickel oxide was also detected in all samples in a range of 0.03 – 0.23%, and a mean value of  $0.11 \pm 0.05$ . The concentration was higher in most samples analyzed and low in few, this is probably due to liming which rapidly decreased nickel (Lenntech, 2008).

##### **4.4.4 Vanadium**

The concentration of Vanadium ores detected in the samples was in a range of 0.1 – 0.23% with a mean value of  $0.14 \pm 0.06$ . Its occurrence could be as a result of it bounding

nature with other minerals like bauxite and carnotile (Lenntech, 2008). Vanadium occurs in carbon containing deposits such as crude oil, oil shale and tar sand.



## CHAPTER FIVE

### CONCLUSION AND RECOMMENDATIONS

#### 5.1 Conclusion

The mean values of the physical parameters of the soil sample studied namely colour, pH, Specific gravity and conductivity has upheld that the plateau is developed on a highly weathered soil. This tends to decrease its organic matter content owing to the soil pH which has greatly decreased the availability of soluble bases (oxides of calcium magnesium potassium etc) in the soil.

Initially, as soil particles begin to weather, primary minerals release nutrients into the soil. As these particles decrease in size, the soil also retains greater amounts of nutrients. Ultimately, however, the capacity to hold and retain nutrients is generally reduced in highly weathered soils, since most nutrients have been lost due to leaching (UOH, 2013).

Most of the trace elements analyzed were absent in most of the soil samples which may be due to their trace amounts in nature below the detection limit of the instrument used for determination (XFR) and or scavenging capabilities of clay minerals and the oxide of iron, aluminum and manganese (Onanuga, 2008).

It can be concluded that the soil form on the plateau is slightly acidic and therefore attributed to the low concentration of chemical minerals. However the high iron oxide content also testifies the colour depicted by the soil. Indicating the presence of hematite, goethite, magnetite, maghemite, lepidocrocite and ferrihydrite.

#### 5.2 Recommendations

1. Rock samples should be assessed for important minerals such as gold, silver, platinum etc, and radioactive elements such as Uranium and Plutonium.
2. X-Ray diffraction method should be employed to assess the structure of some important minerals.
3. Toxic elements such as lead, cadmium etc, should be assessed to enable us know their levels in the soil.

### **5.3 Contribution to Knowledge**

1. The acidity nature of soil has being identified to be responsible for the poor yielding in this Area.
2. The discovery of titanium ores deposits can be exploited for industrial purposes.

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## Appendix 1 - International Standards for Metals in Soil

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Metals	Normal Range (%)
Ca	0.1 - 1.5
Mg	0.1 - 4
Na	0.1 - 1
K	0.5 - 2.5
Fe	0.7 - 55
P	0.005 - 0.15
Si	23 - 35
Se	$1 \times 10^{-5} - 2 \times 10^{-4}$
Mn	$2 \times 10^{-3} - 0.1$
Mo	$2 \times 10^{-5} - 2 \times 10^{-4}$

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\*Sources: Havlin et al. (2003)

**Appendix 2: Concentration of Major Elements (%)**

<b>Sample Code</b>	<b>Al<sub>2</sub>O<sub>3</sub></b>	<b>Fe<sub>2</sub>O<sub>3</sub></b>	<b>K<sub>2</sub>O</b>	<b>SiO<sub>3</sub></b>	<b>TiO<sub>2</sub></b>
E <sub>1</sub>	16.00	37.10	0.001	19.00	4.47
E <sub>2</sub>	19.00	30.28	0.001	41.70	3.14
E <sub>3</sub>	20.00	24.22	1.50	48.50	4.05
E <sub>4</sub>	14.00	29.80	1.10	32.00	4.20
<b>RANGE</b>	<b>14.00-20.00</b>	<b>24.22-37.10</b>	<b>0.001-1.50</b>	<b>19.00-48.50</b>	<b>3.14-4.47</b>
<b>MEAN</b>	<b>17.25±2.38</b>	<b>30.35±4.57</b>	<b>1.30±0.20</b>	<b>35.25±11.06</b>	<b>3.97±.050</b>
N <sub>1</sub>	11.00	17.50	2.10	51.70	3.06
N <sub>2</sub>	12.00	46.50	0.001	10.00	7.36
N <sub>3</sub>	17.00	44.54	0.001	26.90	5.67
N <sub>4</sub>	9.00	39.70	0.001	11.00	7.89
<b>RANGE</b>	<b>9.00-17.00</b>	<b>17.50-46.50</b>	<b>0.001-2.10</b>	<b>10.00-51.70</b>	<b>3.06-7.89</b>
<b>MEAN</b>	<b>12.25±2.95</b>	<b>37.06±11.56</b>	<b>2.10±0.00</b>	<b>24.90±21.23</b>	<b>6.00±1.89</b>
S <sub>1</sub>	27.10	10.06	0.001	54.60	1.59
S <sub>2</sub>	12.00	21.00	1.10	53.00	2.10
S <sub>3</sub>	23.00	18.17	0.001	43.30	2.50
S <sub>4</sub>	19.00	13.40	1.90	61.20	1.97
<b>RANGE</b>	<b>12.00-27.10</b>	<b>10.06-21.00</b>	<b>0.001-1.90</b>	<b>43.30-61.20</b>	<b>1.59-2.50</b>
<b>MEAN</b>	<b>20.28±5.80</b>	<b>15.66±4.05</b>	<b>1.50±0.40</b>	<b>53.03±6.40</b>	<b>2.04±0.33</b>

**Detection limit < 0.001**

**Appendix 3: Concentration of Minor Elements (%)**

<b>Sample code</b>	<b>BaO</b>	<b>CaO</b>	<b>MnO</b>
E <sub>1</sub>	0.34	0.67	0.23
E <sub>2</sub>	0.40	0.27	0.26
E <sub>3</sub>	0.001	0.13	0.22
E <sub>4</sub>	0.17	0.01	0.23
<b>RANGE</b>	<b>0.001-0.70</b>	<b>0.01-0.67</b>	<b>0.20-0.26</b>
<b>MEAN</b>	<b>0.30<sub>±</sub>0.10</b>	<b>0.27<sub>±</sub>0.05</b>	<b>0.24<sub>±</sub>0.02</b>
N <sub>1</sub>	0.001	0.27	0.22
N <sub>2</sub>	0.001	0.18	0.38
N <sub>3</sub>	0.001	0.14	0.55
N <sub>4</sub>	0.001	0.20	0.44
<b>RANGE</b>	<b>0.001</b>	<b>0.14-0.27</b>	<b>0.22-0.55</b>
<b>MEAN</b>	<b>0.001</b>	<b>0.20<sub>±</sub>0.04</b>	<b>0.40<sub>±</sub>0.12</b>
S <sub>1</sub>	0.02	0.37	0.07
S <sub>2</sub>	0.04	0.10	0.04
S <sub>3</sub>	0.48	0.17	0.05
S <sub>4</sub>	0.48	0.10	0.06
<b>RANGE</b>	<b>0.02-0.48</b>	<b>0.10-0.37</b>	<b>0.04-0.07</b>
<b>MEAN</b>	<b>0.26<sub>±</sub>0.23</b>	<b>0.19<sub>±</sub>0.11</b>	<b>0.06<sub>±</sub>0.01</b>

**Detection Limit < 0.001**

**Appendix 4: Concentration of Trace Elements (%)**

<b>Sample Code</b>	<b>Cr<sub>2</sub>O<sub>3</sub></b>	<b>CuO</b>	<b>NiO</b>	<b>V<sub>2</sub>O<sub>5</sub></b>
E <sub>1</sub>	0.16	0.10	0.11	0.23
E <sub>2</sub>	0.13	0.10	0.12	0.15
E <sub>3</sub>	0.11	0.001	0.08	0.18
E <sub>4</sub>	0.21	0.17	0.23	0.10
<b>RANGE</b>	<b>0.11-0.21</b>	<b>0.001-0.17</b>	<b>0.08-0.23</b>	<b>0.10-0.23</b>
<b>MEAN</b>	<b>0.15<sub>±</sub>0.04</b>	<b>0.12<sub>±</sub>0.03</b>	<b>0.14<sub>±</sub>0.06</b>	<b>0.17<sub>±</sub>0.05</b>
N <sub>1</sub>	0.08	0.001	0.10	0.07
N <sub>2</sub>	0.13	0.001	0.12	0.20
N <sub>3</sub>	0.13	0.001	0.17	0.20
N <sub>4</sub>	0.25	0.001	0.19	0.29
<b>RANGE</b>	<b>0.08-0.25</b>	<b>0.001</b>	<b>0.10-0.19</b>	<b>0.07-0.29</b>
<b>MEAN</b>	<b>0.15<sub>±</sub>0.06</b>	<b>0.001</b>	<b>0.15<sub>±</sub>0.04</b>	<b>0.19<sub>±</sub>0.08</b>
S <sub>1</sub>	0.04	0.02	0.08	0.08
S <sub>2</sub>	0.06	0.04	0.04	0.09
S <sub>3</sub>	0.05	0.01	0.03	0.10
S <sub>4</sub>	0.10	0.07	0.08	0.10
<b>RANGE</b>	<b>0.04-0.10</b>	<b>0.01-0.07</b>	<b>0.03-0.08</b>	<b>0.08-0.10</b>
<b>MEAN</b>	<b>0.06<sub>±</sub>0.02</b>	<b>0.07<sub>±</sub>0.11</b>	<b>0.06<sub>±</sub>0.02</b>	<b>0.09<sub>±</sub>0.01</b>

**Detection Limit > 0.001**