

**EXPERIMENTAL EVALUATION OF DIFFERENT CLAY
SAMPLES TO DETERMINE ITS SUITABILITY FOR
USE AS CATALYST**

BY

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CERTIFICATION

This is to certify that the project "Experimental evaluation of different clay samples to determine its suitability for use as catalyst" was presented by Youduba Kuro Godspower, for the partial fulfillment of the award of Bachelor Degree in Chemical Engineering (B.Eng).

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19/11/2024

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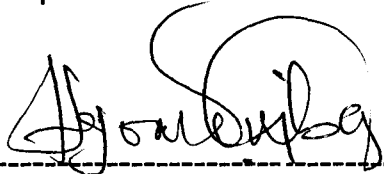
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DEDICATION

This research work is dedicated to my mother who is a personal source of inspiration, an embodiment of love and a cause for my passionate commitment to success.

DECLARATION

This project was carried out by Youduba Kuro Godspower under the supervision of Engr. M. A. Olutoye of Chemical Engineering Department F.U.T. Minna.



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ABSTRACT

The work on experimental determination of clay samples to determine its suitability for use as catalyst has been carried out. To achieve this, clay samples were collected from different locations within Nigeria. The samples were treated by calcination, slides formation and analyzed using the XRD method. On analysis it was observed that the clay samples are composed of alkalis such as sodium, potassium, and alkaline earth such as magnesium, calcium were present in the molecular structure of the clays.

Thus, these constituents of the clay serve as an active site for catalytic reaction.

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CHAPTER ONE

1.0 INTRODUCTION

Apart from petroleum, gas and coal resources, the exploration, mining and exploitation of Nigerian mineral resources have not receive sufficient attention; until recently when the shortage of foreign exchange emphasized the need to integrate backwards, mining of industrial and metallic minerals was generally neglected.

Delving into the geological survey of Nigerian soil, it was reported that, clay as one of the major Nigerian mineral deposit cover an estimate proven reserves of billions of tonnes, and these clay minerals are distributed all over the state in the country (Skinner, 1987).

The use of clays as dated back since about fifteen-century as a result of some properties attributed to it. And as Civilization Progress and technological knowledge too increases, there is an upsurge towards the use of clay locally such as making of cooking pots, building of houses and other local uses (Edward, 1980).

But in modern clay technological advancement, it has taken a new stride and dimension to which clays can be beneficial to humanity.

A lot of project researches had been carried out about the conversions of clay for industrial uses namely:

1. quality control on clays for ceramic glasses for laboratory. (Federal Institute of Industrial Research Centre Oshodi, 1990)
2. Test on Nigerian clay (Bentonite) for uses in drilling mud materials by Petroleum Training Institute Warri Geochemical Division).
3. Nigerian industrial minerals and Clays Chemical and mineralogical composition, specially Kaolin and montmorillonite, University of Port-Harcourt, department of geology.

Having found that much work has been done on clay, through a lot of valuable application of Chemical Engineering in the exploitation of clay as an important mineral.

Nevertheless, since these clays abound in many other parts of the world, the need to further exploit areas to see their viability majorly for catalytic application which is intended in this project research.

1.1 GEOLOGY OF CLAYS

Mode of occurrence: Clay Depths. They are basically two types of clay depths:

1. Primary Clay Depths
2. Secondary Clay Depths

Primary clay depths: These are those depths formed by primary deep seated processes particularly in relation to Igneous Processes.

Secondary clay depths: This is subdivided into two.

- (1) Residual Clay and
- (2) Sedimentary Clay.

Residual clay: These are products of weathering of pre-existing rocks common in tropical environment where intense weathering is common. They are often mixed up with two oxides, Fe_2O_3 and Al_2O_3 ; this can be formed from any rock type e.g. clay formed from Limestone with CaCO_3 . Residual clay are commonly found in Nigeria (Edward A. Keller, 1984)

Sedimentary Clays: These are clays that have been deposited in marine environment, which could have been derived from existing clay or weathering of pre-existing rocks e.g Alluvial clays found at Sagamu in Ogun State used as a substitute for limestone in cement making (Chemical Engineering Journal, 1989).

1.2 THE NATURE OF CLAY

Most clays are crystalline; that is, they have a definite, repeating arrangement of atoms of which they are composed. The majorities are made up of planes of oxygen atoms with silica and aluminum atoms holding the oxygen together by ionic

bonding, which is the attraction of positively and negatively charged atoms.

Three or four planes of oxygen atoms with Intervening silicon and aluminum ions (or others, depending upon the clay) make up a layer. One clay particle is composed of many layers stacked like a deck of cards. A clay particle is called a "Micelle".

AIM

The aim of this research is to determine and to investigate the suitability of some clay samples for use as catalyst.

SCOPE OFWORK

This will involve collecting clay samples, carrying out pre-treatment and analysis.

LIMITATIONS

The clay samples are only from four different areas with two from Niger State.

The analysis of the clay samples were only done using the ray machine of medium size.

The samples are all secondary clays.

CHAPTER TWO

2.0 LITERATURE REVIEW

Clays are in the form of naturally occurring fine-grained minerals, which develops plasticity when mixed with the right proportion of water. It is the part of soil that covers the earth surface, and it is usually found at riverbanks, under the water, sea and deep part of the earth. The mineral content varies from one area to another.

The nature of clays formed by decomposition of Feldspathic rock is as a result of weathering action of wind and rain, and have a general chemical formula of $Al_2O_nSO_2PH_2O$, hence clays belong to aluminum silicate group.

2.1 ORIGIN OF CLAYS

Prior to the X-ray study of mineral compositions clays were incorrectly thought to be just smaller particles of primary minerals, such as small particles of quartz, feldspars, micas, hornblende, or augite. Now clay minerals are known to have specific compositions and are not very similar to the primary minerals, except micas.

Clay minerals are mostly newly formed crystals reformed from the soluble products of the primary minerals and are secondary minerals. Laboratory syntheses of clays have proven

that the kind of clay formed is determined by the proportions of the different ions in the solution during formation.

Removal of some of the soluble products by leaching of the soil solution will reduce clay formation and alter the kind of clay formed. Soils of regions that have hot moist climates, but are not leached excessively because of poor drainage, have large amounts of the primary minerals dissolved, which then crystallize to clays.

These hot, humid, tropical soils therefore tend to be high in percentage clay, even to depths of 5-20m (16.4-65.6ft); other nearly soils have large portions of the weathered primary minerals washed away. Some clays apparently form from slight alteration (selective solubility and rebuilding) of some primary minerals, particularly from the micas such as biotite and muscovite.

2.2 CLAYS CLASSIFICATION

Clays differ very considerably among themselves in structure and in water content. It also differ in workability, plasticity, particle, size distribution and mineralogical composition, and these differences lead to such term as flint clays and the like. Clays are often classified mainly into two ways and are based on mineralogical constituents and properties of clays.

On mineralogical basis, clay minerals are divided into three major groups in accordance with their crystallographic structures.

- 2.2.1 ***Kaolin group:*** This have the simplest clay mineral structures. Their minerals all have the Chemical Formula of $Al_2O_3 \cdot 4SiO_2 \cdot 2H_2O$ and includes, Kaolinite, nacirite, dickite anauxite, halloysite, and livesite.
- 2.2.2 ***Montmorillonite group:-*** These comprises of montmerinite ($Al_2O_3 \cdot 4SiO_2 \cdot n2H_2O$) and others. These minerals have the common properties of absorbing large quantities of water between adjacent layers.
- 2.2.3 ***Illinite or hydromics group:*** This has clay minerals that resembles the micas and they have large spaces which contain cations to keep the charges neutral.

Their finely divided state, however makes many of these cations accessible for exchange.

Unlike montmorillonites, water does not enter into the lattice itself and expand it because the adjacent layers are held together by potassium ions.

The development of the classification of clays by Norton (1952) are as follows:

- 2.2.4. ***White-Burning Clays:*** Commonly used in white ware, consists of Kaolin (residual and sedimentary) and ball clays.

- 2.2.5 **Brick Clays:** These are plastic clays and contain iron-oxide. They are terra-cota clays and face common bricks.
- 2.2.6 **Refractory clays:** These have melting points above 165°C but not necessarily white-burning. They are basically kaolines, fire clays and high alumina clays.
- 2.2.7 **Stone clays:** These are plastic containing fluxes.
- 2.2.8 **Slip clays:** These have more iron oxide content
- 2.2.9 **Heavy clays:** These are product clays, which have low plasticity and containing fluxes. It consists of paving bricks clay and shales.
- 2.20 **Residential clays:** These are those that decomposes at the site of the original rock and are characterized by relatively purity.

These classes of clays possesses good plasticity but are relatively improve, since during their passage they would have been contaminated with some impurities present in other rocks, which similarly decompose.

Since clays are inorganic earthly materials, which becomes plastic and sticky when wet and dries to a fairy hard and rigid mass. The bounding power of a clay has been defined as the degree of cohesiveness it exhibits in holding the grains of sand together.

2.3 NIGERIAN CLAYS

As we all know that clays could be found in almost different parts of the world, and it varies from nation to nation, country to country.

Nigeria is not left out amongst the nations where different types of clay are useful for both chemically, physically and human use. A good number of clay deposits in Nigeria has been chemically analyzed and their properties has been investigated to see their usefulness in nation building (M.J Smith, 1981).

Some of the known deposits of clay include, Sabo Gida, Osi-Opin Clay, Isolo-Sopin red clay, Chanchaga Clay, (Niger State) Kankara clays, Giro clays to name but a few.

2.4 CHEMICAL COMPOSITION OF CLAYS

Clays are known to be composed of various compounds that in one way or the other affect the micro logical structure of its properties (catalytic).

Therefore, this section explains the aims and the identification of various component effects of the individual compound on clay properties.

2.4.1 Silica

This occurs in clays either in free state, as aquatic or other forms of crystalline silica, or as hydrated amorphous compound

in the colloidal state. It also occurs in combination with alumina in form of fluxes and feldspar, mica or other alumina silicates.

2.4.2 Alumina

This occurs in clays not only in form of clay mineral, but also in Feldspar, mica and other alumina-silicates.

2.4.3 Alkali Compounds

These are present mainly as silicates or alumina silicates, they also occur in the form of soluble salts, such as sodium chloride.

These compounds are commonly referred to as mixes.

2.4.4 Magnesium Minerals

This occurs to a small extent in most clays as magnesite (MgCO_3) Magnesium compound acts as Fluxes.

2.4.5 Titanium Compound

Titanium compounds with the formula T_2O_3 are very resistance to the action of weather. Titanium compounds remain unaltered in clay which acts as feasible flux; although they are not active below 115°C .

2.4.6 Iron Compound

These include ferric and ferrous oxides, ferric silicate and sulphates. The principal effects of iron compounds in clay are that they affect an alteration in colour.

2.4.7 Calcium Compounds

Calcium compounds readily found in clay are calcite (CaCO_3) and calcium silicates. They combine with alumina and silicates to form low melting point admixtures.

2.4.8 Organic Matter

This is present in various properties according to the mode of formation and the condition to which the clay has been exposed. It is derived mainly from the deposit of clay on organic matter on clay beds with carbonaceous materials been gradually incorporated into the clay.

2.4.9 Moisture and Colloidal Waste

These may also be conveniently regarded as a mineral and it is present in clays in variety of forms as:

- a. Hygroscopic; waste and moisture in which its amounts depends on surface area of the clay.
- b. **Absorbed water:** this is also associated with the exchangeable cation in certain minerals.

2.5.1 Structure of Clays

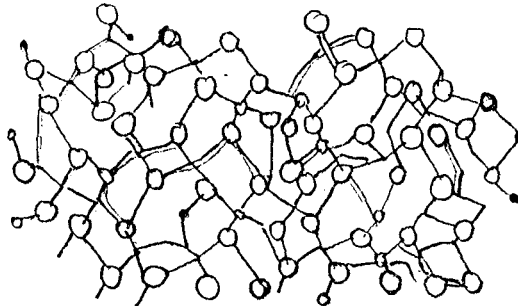
Clays are crystalline in structure, the chemical analysis of clays shows them to be composed essentially of silica, alumina and water with appreciable amounts of iron alkalis, and the lattice

of most of the clay, minerals are composed of two structures i.e. structural units.

The first units consist of closely packed six oxygen atom or hydroxyl in the basal planes of an Octahedral structure.

Aluminum (Al), Iron (Fe), Magnesium (Mg) atoms are embedded in the octahedral positions, so that they are equidistant from six-oxygen or hydroxyls in the base plane.

Fig. 1 shows a typical structure of clay



When aluminum is present, only two thirds of the possible position required to balance the structure are filled. The structure containing aluminum ion is called the gibbsite structure and has the formula $\text{Al}_2 (\text{OH})_6$. When magnesium is present, all the position are filled resulting in a balanced structure with formula $\text{Mg}_3 (\text{OH})_6$.

The second unit has the silica. Structure made of units of tetrahedrons in which the silicon atom is centrally placed equidistant from the four oxygen or hydroxyl needed to balance the structure from six oxygen or hydroxyl in the base planes.

2.5.1. *Structural Chemistry of Clay Minerals*

As indicated earlier, clays can exist in crystalline, structurally disordered, or amorphous form. The amorphous state generally has no recognizable shape or geometric internal arrangement of atoms. Depending on the degree of sophistication in methods for analysis, a sharp distinction does not exist between crystalline and amorphous states.

In soil science, clay is considered amorphous if it is amorphous to X-ray diffraction analysis (i.e. lacks regularity in internal atomic arrangement as reflected by a featureless diffractogram). In crystals, the atomic arrangement may be repeated in a regular three-dimensional pattern.

However, in amorphous materials, such as glass, the chemical bonding of the component atoms is, perhaps the only unit repeating itself. The spatial arrangement of atoms producing the building unit of a crystal is called the unit cell.

The latter exhibits a complete group pattern of atoms that repeats itself in three directions in space according to the x, y and z axis respectively.

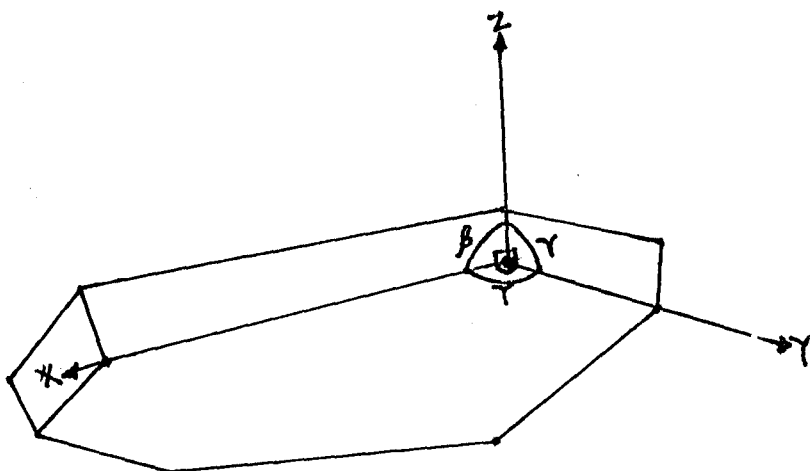


FIG 2

Fig 2. Crystal structure, exhibiting a group pattern of atoms repeating itself in three directions in space, according to the x, y, z axes respectively.

The atomic groups in a crystal lattice can be arranged in plans at equal spacing along the crystallographic direction.

Silicates are built around a silica tetrahedron in which each oxygen atom receives one valency from the silicon atom. To satisfy its divalent requirement, the oxygen atoms can be linked to other cations or to a silicon atom of an adjacent silica tetrahedron.

The linkage of silica tetrahedral yields five groups of structural arrangements of silicates: Island, isolated group, chain, sheet and frame work structure. Silicate clay minerals are characterized by a sheet structure. In contrast with the other silicates, the structure of clay is not a three-dimensional network of simple linkages of silicon-oxygen of silica tetrahedral and Aluminum, magnesium Octahedral sheets.

The sheets are developed by the linkage of three oxygen in each tetrahedron with adjacent silica tetrahedral units. The silica tetrahedron are arranged in hexagonal rings.

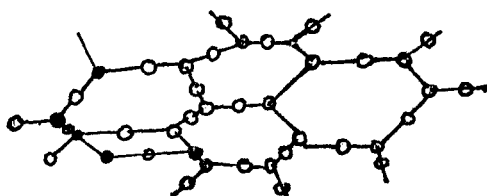
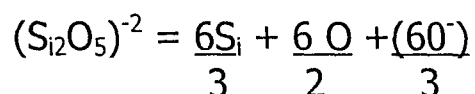


Fig. 3. A silica tetrahedra structure arranged in a hexagonal rings.

The sheet can extend indefinitely in a two dimensional direction, according to the two planes a and b, or parallel to the plane of the paper in this book, which is the reason for the platelike nature of clays. The composition of each ring, or the lowest unit of the sheet is,



in such a network silica tetrahedra, one oxygen in each tetrahedron remains electrically unbalanced.

To satisfy the divalent requirement, the latter is linked to Aluminum in octahedral coordination.

By such a packing of silica tetrahedron and aluminum octahedron sheets, a layered clay structure is formed.

In addition to the structural arrangement as just discussed, stacking of the layers can also occur by different types of unit layers in a regular or irregular pattern. The structure of these minerals may vary widely since two or more different types of unit layers may be stacked together.

2.5.2 Kaolinite Group

Kaolinite minerals are hydrated aluminosilicates, with a general chemical composition $\text{Al}_2\text{O}_3:\text{Si}_2\text{O}_5:\text{H}_2\text{O} = 1:2:2$, or $2\text{Si}_2\text{O}_5 \cdot \text{Al}_2\text{O}_3 \cdot 2\text{H}_2\text{O}$ per unit cell. Structurally they are 1:1 type phyllosilicates. The crystal is composed of aluminum octahedra sheets stacked above silica tetrahedron sheets. The unit cell is

nonsymmetric with a silica tetrahedra sheet on one side and an aluminium Octahedra sheet on the other. Consequently, the basal plane of Oxygen atoms in one crystal unit opposite the basal plane composed of hydroxyl ions of the next layer.

Kaolinite has a variable or PH-dependent, negative charge. Because of the tightness of the structural bonds, Kaolinite particles are not easily broken down. This is also the cause of low plasticity and shrinkage and swelling properties.

Going by these properties of the group, the negative charge makes it viable for catalytic purposes because since catalysis involve ionic exchange through the active sites.

Similarly, considering the structural description of the group which is crystalline composing of aluminum octahedra sheets.

This makes it play a vital role in catalytic processes that is as a support for catalytic purposes.

2.5.3 *Halloysite*

Halloysite has a general composition $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 4\text{H}_2\text{O}$, and is similar in structure to Kaolinite.

The differences lie in the disordered stacking layers and in the presence of two or more interlayers of water as noted earlier for this mineral.

The water molecules are linked together in an hexagonal pattern. In turn they are bonded to the crystal layers by

hydrogen bonding. Because of the presence of Interlayer water, halloysite exhibits a basal spacing of 10.1 Å, which upon heating can be reduced to 7.2 Å. The dehydrated species is called metahalloysite, Halloysite at 50°C, but the heating reduces the spacing, it does not affect the random stacking of layers.

Halloysite, in general, is tubular in form. This is in contrast to Kaolinite, which is hexagonal.

However, recently sheetlike halloysite, called tubular halloysite, has been detected in some of the soils in Texas. The tubular crystal form is considered to be rolled up sheets.

2.5.4 **Montmorillonite**

Minerals in this group are sometimes called smectite and have a variable composition.

However, the formula is often expressed as $\text{Al}_2\text{O}_3 \cdot 4\text{SiO}_2 \cdot \text{H}_2\text{O} + x\text{H}_2\text{O}$. The name montmorillonite is reserved for the hydrated aluminosilicate species with little substitution.

Many clay deposits in the United States contain large amounts of montmorillonite.

This type of clay is frequently called bentonite, and commercial grade montmorillonite is also often referred to as bentonite.

A wide range of minerals exist within the montmorillonite group, and the principal end members in the dioctahedral sub-

crystalline anhydrous ferric oxides have also been found in soils:- hematite and maghemite.

The composition of these and other hydrous oxide minerals are listed in the following table:

Goethite	α - FeOOH
Lepidocrocite	γ - FeOOH
Hematite	α - Fe ₂ O ₃
Maghemite	γ - Fe ₂ O ₃

2.5.6 *Amorphous clays, allophane, and imogolite.*

With the recent progress in clay mineralogy, it is currently known that many soils also contain amorphous clays (R.L Stanton, 1972).

These clays are non-crystalline and include a wide variety of materials (e.g silica gel, sesquioxide gels, silicates, and phosphates).

They are amorphous to X-ray diffraction pattern. Udo Schwetman (personal communication) is of the opinion that the term amorphous is used erroneously in soil science.

It is perhaps the methods of analysis that are inadequate to detect crystallinity in the so-called amorphous clays, since most of these clays occur as very fine, micro, crystal forms, Wada (1977) prefers the use of the term noncrystalline over the term amorphous.

The most important type of clay in this group is perhaps allophane. It is found especially in volcanic ash soils. The name allophane was first introduced by Stromeyer and Hausmann in 1861 for hydrous aluminosilicates occurring in nature. Since then, the name allophane has found general acceptance for a wide variety of clay material amorphous to X-ray diffraction analysis (Ross and Kerr, 1934).

Allophane was formerly classified as Kaolin clay, since it has a sheet structure similar to Kaolinite. Many definitions for allophane are present today.

1. Ross and Kerr (1934) define allophane as an amorphous material that is commonly associated with halloysite. It has no crystal structure and no definite composition and is a mutual solution of silica, alumina, water and minor amounts of bases.

Currently, allophane associated with halloysite is frequently called halloysitelike allophane with a hypothetical formula of $0.5 \text{ Al}_2\text{O}_3 \cdot \text{SiO}_2 \cdot 1.4\text{H}_2\text{O}$. The aluminum in allophanelike halloysite is in octahedral form.

2. Van Olphen (1971) defines allophane as a series of naturally occurring minerals that are short-range order hydrous aluminium silicates of various chemical compositions characterized by Si-O-Al bonds. They exhibit DTA curves with a strong low-temperature endothermic peak and a high-

temperature exothermic peak, with no intermediate thermal features.

3. Farmer et al. (1985) indicate that allophane is a group name for non-crystalline clay minerals consisting of silica, alumina and water in chemical combination.
4. Wada (1989) reported that allophane is a group name for hydrous aluminosilicates with a composition characterized by a molar S_i/Al ratio of 1:2 to 1:1. The formula is proposed to be:
 $S_iO_2 \cdot Al_2O_3 \cdot 2H_2O$ or $Al_2O_3 \cdot 2S_iO_2 \cdot 2H_2O$

The mineral consists of hollow irregular spherical particles with a diameter of 3.5 – 5nm.

5. Parfit (1990) is of the opinion that allophane is a group name of clay-sized minerals with short-range order. It contains silica, alumina and water in chemical combination.

Both Van Olphen and Parfit suggest that allophane is a clay-sized mineral of short – range disorder, such as glass, X-ray diffraction as well as electron diffraction analysis show no repeat of structural units in any spatial direction. Hence, Parfit believes that the term non-crystalline is more important and appropriate than the term short-range order for allophane.

Another important type of clay in this group is imogolite. This clay mineral was reported for the first time in 1962. It was found in weathered volcanic ash or pumice beds, called Imogo.

Since then it has been detected in many volcanic ash soils in Japan, South America, and in the Islands of the Pacific.

The composition formula of Imogolite has chemical characteristics similar to allophane. Several authors believe that allophane is a precursor of imogolite. However, in contrast with allophane, imogolite has a better-defined crystal shape. Electron microscopy shows evidence of the presence of hairlike or spagettilike crystal forms. The term paracrystalline has been suggested for the structure of Imogolite. The intermediate phase between allophane and imogolite, or imogolitelike allophane is called protoimogolite. This is allophane with a structure close to that of Imogolite, but lacks the crystal order of Imogolite.

The suggested chemical formula is: $\text{Al}_2\text{O}_3 \cdot 0.6 - 1.0 \text{SiO}_2 \cdot 2.5 - 3.0 \text{H}_2\text{O}$. It has a morphology of hollow spherules with an outside diameter of 3.5-5.0nm.

The presence of allophane gives the soil unique properties. Allophane has a large variable charge, it also behaves amphoterically and is reported to fix considerable amounts of phosphates.

The presence of allophane has also an important effect on several soil properties. Soils high in allophanic clays are characterized by low bulk density values, high plasticity, although they are non-sticky when wet. The water-holding

1. In a catalyst reaction, the catalyst is unchanged at the conclusion of the reaction; but it may participate in the intermediate steps so as to increase the reaction rate.
2. When more than one mechanism is feasible for a reaction a catalyst may exhibit selectivity favouring one mechanism over others. This situation usually results in a product distribution different from those by other mechanisms. Proper application of catalyst selectivity would enable the desired reactions to be accelerated while the undesirable reactions were retarded.
3. The rate of reaction is generally proportional to the catalyst concentration. For a solid-catalyzed reaction, the surface area of the catalyst and the concentration of the so-called active centres or catalyst sites (Locations of high chemical activity on the surface) become important.
4. In a reversible reaction the catalyst accelerates the reverse reaction as well as the forward one. Thus, the equilibrium composition of the reacting system would be the same as that of the uncatalyzed system.
5. In an autocatalytic reaction where one of the reaction products functions as a catalyst, a small amount of the product must be present to initiate the reaction. A plot of conversion versus time for this type of reaction results in a characteristic S-shaped curve.

capacity appears to be increased substantially by allophane. It is assumed that allophane and imogolite will also undergo interaction processes with soil organic compounds, such as humic and Fulvic acids. The latter reaction is called complex formation or chelation. Most soils containing allophane are known to have black A horizons, extremely high in soil organic matter contents. Formally, these soils were called andosols or andosoils (from the Japanese ando, black).

Identification of these minerals is mostly done by DTA (Differential Thermal Analysis), since X-ray analysis yields featureless diffraction curves.

2.5.7 Identification of Clay Minerals

In the preceding sections some of the methods for the identification of clays have been mentioned briefly without going into details on the techniques and physicochemical reactions involved. For a better comprehension it is perhaps necessary to discuss briefly three of the major methods often used (e.g. differential thermal analysis, X-ray diffraction analysis and infrared spectroscopy).

2.5.7.1 Differential Thermal Analysis

The differential thermal analysis method, commonly referred to as DTA, is a widely used technique and is particularly useful

especially in the identification of amorphous material when X-ray diffraction analysis yields only featureless curves (Tan and Hajek, 1977). It found applicable first in geology and later has been extended to research and analysis in ceramics glass, polymer, cement, plaster industries and so forth.

Differential thermal analysis measures the differences in temperature development between an unknown and a reference sample, as the two are heated side by side at a controlled heating rate from 0 to 1000⁰c. The reference material, also called standard material, is a substance that is thermally inert over the temperature range under investigation. A number of compounds have been used as standard sample (e.g. calcined Al₂O₃ and calcined Kaolinite (heated at 1000⁰c).

The heating must be controlled at a uniform and steady rate through the analysis. Heating rates may vary from 0.1 to 2000⁰c/min. for most purposes a heating rate of 20⁰C/min is used during the heating process, the unknown sample undergoes a thermal reaction and transformation.

The latter is reflected by a difference in temperature between the unknown and reference sample. This difference in temperature is plotted in a graph, usually against the temperature at which the difference occurs as shown in the curve below

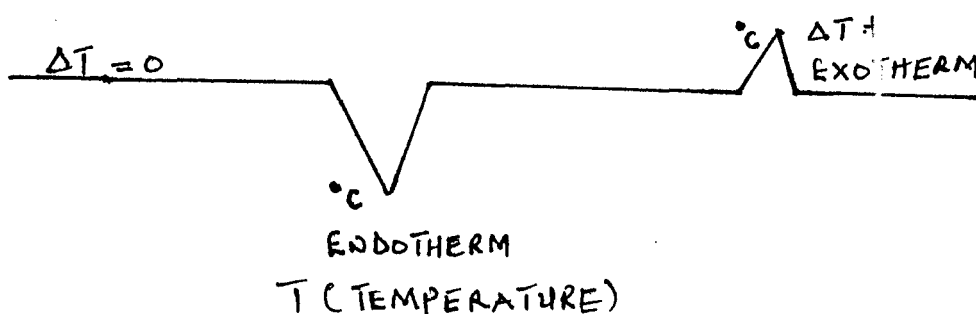


Fig. 5: An Idealized DTA Curve

If the temperature of the unknown sample becomes lower than that of the reference material, ΔT is negative, an endothermic peak is produced. When the temperature of the sample becomes higher than that of the reference material, ΔT is positive, an exothermic peak develops. The portion of the curve for which $\Delta T = 0$ (No difference in temperature between unknown and reference sample) is considered the baseline ideally the baseline is a straight line. Upon analysis by DTA, the mineral may undergo several thermal reactions, culminating in one or a series of endo-and exothermic peaks. The curve with the peaks serves as a fingerprint, and the specific temperatures at which the peaks develop are diagnostic for the identification of the mineral. In addition, the peak height or peak area of the main endothermic reaction can be used for quantitative determination.

Generally DTA can be performed with liquid or solid samples. With soil samples, whole soil, sand, silt, or clay fractions can be used. When whole soils are analyzed, the less than 2mm fraction should be treated first with 30% H_2O_2 to remove organic matter, which may interfere by giving strong exothermic reactions. In general analysis, whole soils give only peaks of low intensity. These same peaks are very large and intense if the clay fractions are analyzed.

Quantitative identification of minerals can be achieved by using the DTA curves as fingerprints and comparing or matching them together.

2.5.7.2 *X-Ray Diffraction Analysis*

The X-ray diffraction method is perhaps the most widely used technique in the identification of clays. It is mainly for quantitative analysis, although frequently semi-quantitative determination of clays has been carried out. X-ray diffraction analysis is a non-destructive method, meaning that the sample is not affected by the analysis, and can be used for other analysis. However, the method is not applicable to analysis of amorphous or non-crystalline materials.

The basis for the use of X-rays in the investigation of soil clays is the systematic arrangement of atoms or ions in crystal planes. Each mineral species is characterized by a specific atomic arrangement, creating characteristic atomic planes that can diffract (reflect) X-rays; X-rays are electromagnetic radiation of short wavelength.

In most crystals, the atomic spacing, or crystal planes, have almost the same dimension as the wavelength of X-rays.

Laue was perhaps the first to discover, in 1912, that X-rays can be diffracted by the atoms in a crystal plane, producing characteristic patterns when recorded. This diffraction pattern is used as a fingerprint in the identification of mineral species. X-rays are produced in a X-ray tube by fast-moving electrons hitting a metal target. The excited atoms in

the target emit radiation with a wavelength between 0.01 and 100Å, the wavelength of $K\alpha$ and $K\beta$ radiation.

Most metals emit wide bands of $K\alpha$ and $K\beta$ radiation (e.g. Copper (Cu) target). By using a nickel filter, the Cu $K\beta$ radiation can be blocked or adsorbed and the Cu $K\alpha$ radiation hits a crystal plane of a mineral, the X-rays are scattered by the atoms of the crystal. To have diffraction occurring, reinforcement must take place of the scattered X-rays in a definite direction. Reinforcement of scattered X-rays becomes quantitative only if Bragg's law is obeyed. Bragg's Law is defined as follows: $n\lambda = 2d \sin\theta$.

Where

d = spacing between atomic planes in the crystal

λ = wavelength

θ = glancing angle of diffraction

n = Order of diffraction

Bragg's Law predicts that all planes in a crystal diffract X-rays when the crystal is inclined at certain angles to the incident beam. The angles θ depend on wavelength λ and on d . The sample of X-ray diffraction analysis can be prepared as a random powder sample or as an oriented sample. In a random powder sample, the crystals lie in a random position to each other. With the aid of glycerol and gum tragacanth the clay sample is made into a paste, and rolled into a rod of 0.3 to

0.5mm thickness. Powder samples can also be prepared by pushing the paste into specially designed wedge holders. The random powder samples are usually analyzed by a powder camera X-ray unit. A currently more popular method of mounting samples for X-ray analysis is the preparation of oriented samples on microscopic glass slide or a porous ceramic plates.

2.5.7.3 *Infrared Spectroscopy*

Recently, infrared spectroscopy has found extensive applications in clay mineralogy studies. Amorphous as well as crystalline clays absorb infrared radiation, and the method can be used when X-ray analysis makes identification difficult. The infrared absorption spectrum of a mineral has a characteristic pattern, which not only permits the identification of the minerals, but also reveals the presence of major functional groups within the structure of the particular compound under investigation. Infrared absorption is related to molecular or atomic vibrations and only radiation with a similar frequency as that of the vibration will be absorbed. Atoms and molecules within a compound oscillate or vibrate with frequencies of approximately 10^{13} - 10^{14} Hz. These frequencies correspond to the frequencies of infrared radiation, and infrared radiation can, therefore, be absorbed by molecular vibrations when the

interaction is accompanied by a change in dipole moment, and absorption of infrared radiation is intense on the other hand, a weak vibration of atoms produces a slow change in dipole, and consequently, absorption of infrared radiation is relatively weak. Symmetric molecules will also often not absorb infrared radiation.

Molecular or atomic vibrations cause the inter-atomic distance to change because the atomic movement, called oscillation, subjects the atoms to a periodic displacement relative to one another. The frequency of vibration obeys the law of simple harmonic motion formulated as

$$V = \frac{1}{2\pi c} \sqrt{\frac{K}{M}}$$

Where:

V = Frequency of Vibration in Hz

C = Velocity of light in vacuum.

M = Reduced mass of the vibrating atoms

K = Force constant in N/M

Two types of vibrations are distinguished:

1. Stretching vibrations, or deformation, in which the atoms are oscillating in the direction of the bond axis without changing bond angles; and

2. **Bending Vibrations.** In which the movement of atoms produces a change in bond angles.

The restoring force acting on the stretching vibration is usually greater than that required to restore bending vibrations. Therefore, stretching vibrations occur at higher frequencies than bending vibrations. The highest frequencies observed in minerals are those of the stretching vibrations of hydroxyl groups (OH) that occur between 3700 and 2000 C_m^{-1} . Bending vibrations occur at lower frequencies, from 1630 to 400 C_m^{-1} . Liquid, gas, and solid samples can be used in infrared spectroscopy. Liquid samples are usually pipetted or injected into infrared cells provided with a Sodium Chloride (NaCl) or Potassium bromide (KBr) Crystal Window. Gas samples are also introduced in cells, similar to the aforementioned cells above.

Several methods have been proposed for mounting solid samples in infrared analysis:

1. Mull method
2. KBr pellet techniques, or
3. Clay film

Technique on demountable cells or other support material. The most widely used method is the KBr pellet technique by which a weighed sample (1-10mg) is carefully ground with 100 MgKBr, and pressed into a clear transparent pellet method. Currently, the use of clay films has attracted considerable attention.

In clay film – mounting method, clay or soil samples are made into a suspension by sonification.

They are then pipetted onto Irtan-II window cells. So that $1\text{mg}/\text{cm}^2$ or $5\text{mg}/\text{cm}^2$ are transferred onto the cells. After drying at room temperature, the cells are scanned from 4000 to 600C_m^{-1} or lower.

2.5.7.4 *Surface Chemistry of Soil Clays*

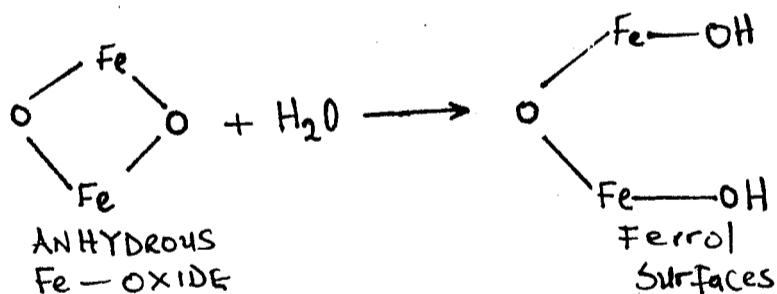
Many, if not all, of the chemistry reactions of soil clays, are surface phenomena (e.g. cation exchange, adsorption of water). From the preceding section on clay mineral structure, it follows that clay surfaces can be divided into at least three categories.

1. Surfaces formed mainly by $\text{S}_i\text{-O-S}_i$ linkages of silica tetrahedrons.
2. Surfaces formed by O-Al-OH linkages of alumina octahedrons
3. Surfaces formed by $\text{S}_i\text{-OH}$ or Al-OH of amorphous compounds.

The first category of surfaces is characterized by surface planes of oxygen atoms, underlaid by silicon atoms of the tetrahedrons. The $\text{S}_i\text{-O-S}_i$ bond is called a siloxane bond by sticher and Bach (1966) and because of this, this type of surface can conveniently be called the siloxane surface is mainly attributed to isomorphous substitution of the underlying silicon atoms of the tetrahedrons. The second type of clay surfaces is characterized by planes of exposed hydroxyl, OH ,

groups, underlaid by Al, Fe, or Mg atoms in the center of the octahedrons. Because of the latter, it can perhaps be called the oxyhydroxide surface. Kaolinite and other 1:1 types of clay usually have siloxane surfaces on one basal plane. The exposed hydroxyl groups are subject to dissociation and, therefore play an important role in the development of negative charges.

The third type of surface is formed is S_1 -OH called silanol surfaces, Al-OH, called aluminol surfaces and Fe-OH, called ferrol surfaces. These surfaces are typically present in soils containing large amounts of silica gel, amorphous Al and Fe oxides, or allophane. The anhydrous Al or Fe-oxide surfaces become hydrated upon contact with moisture, and aluminol or ferrol surfaces will be developed. The reaction can be illustrated by the following reactions:-



The behaviour of these surfaces is expected to be quite different from other two aforementioned types usually, the compounds with silanol, aluminol, and Ferrol Surface have a very large surface and variable charges, and all the hydroxyl groups are easily accessible.

2.5.7.5 *Surface Areas*

In connection with the surfaces of clay minerals is the problem of surface areas, needed for quantitative interpretation of surface properties in relation to soils and clay behaviours. Rate of adsorption and cation exchange are proportional to surface areas. The surface area generally increased with decreased particles size. It can be measured by several methods (e.g, calculation, adsorption analysis, and other procedures). By using the calculation method, the surface area can be measured in terms of

1. Total surface area, or
2. Specific Surface

Total surface: Assume that a cubical container, with side (width) = L , is filled with spherical particles. If the dimension of each sphere = d , N = number spheres in the container, and A = total surface area of all spheres, the:

$$N = (L/d)^3$$

Since surface area of one sphere = πd^2 , hence:

$$A = N\pi d^2 \text{ or } A = (L/d)^3 \pi d^2$$

The total surface area, A , = $\pi L^3/d$ square units.

Specific surface: The specific surface area, or specific surface, particles. If the surface area of a sphere = πd^2 , and the volume of the same sphere with diameter d equal $1/6 \pi d^3$, then by definition.

Specific surface, $S = \frac{\pi d^2}{(1/6\pi d^3)}$ or

$S = 6/d$ square units/cubic unit.

If we assume that clay particles are sphere with $d = 0.002\text{mm}$, then the specific surface of clay is $S = 6/0.002 = 3000\text{mm}^2/\text{mm}^3$

Various types of colloids start to display colloidal properties at different specific surface values.

Spangler and Handy (1982) believe that soil constituents begin to exhibit colloidal characteristics when their specific surface reaches values of $6000 - 10,000\text{mm}^2/\text{mm}^3$. However, the value of specific surface of $3000\text{mm}^2/\text{mm}^3$ for the lowest limits at which the colloidal behaviour starts to appear, conforms better with the definition of clays (soil constituent with a diameter less than 0.002mm).

PREPARATION OF CATALYST USING CLAY-ALUMINA/SILICA-ALUMINA AS SUPPORT

2.6.0 CATALYST

Generally, a catalyst is a substance that changes the rate of a reaction but does not influence the equilibrium of the reaction.

It follows that a catalyst must alter the rate in the forward and reverse directions by the same factor, so as to preserve, unchanged the ratio of rate coefficients, the equilibrium constant, we therefore have

$$K = \frac{K_f}{K_r} = \frac{K_f^1}{K_r^1}$$

Where the unprimed and primed rate coefficients are those in the absence and the presence of a catalyst respectively.

Thus, the addition of a catalyst does not affect microscopic reversibility. The question to be addressed is "How does a catalyst work?" This is usually answered in a simple way.

A catalyst works by providing an alternative reaction mechanism that changes the rate usually by altering energy for the thermal decomposition of $(\text{CH}_3)_3\text{COH} - (\text{CH}_3)_2\text{C} = \text{CH}_2 + \text{H}_2\text{O}$ is 274 KJ/Mol whereas the presence of HBr as a catalyst reduces the activation energy to 127 KJ/Mol. Although the activation energy is not identical with the barrier height along the reaction coordinate, we know that the two quantities are closely related. A catalyst is thus a substance that, by interacting with one or more of the reactants enlarges the number of coordinates describing the potentials surface and thereby permits a path from reactants to products with a different barrier height than that of the uncatalyzed reaction (a lower barrier height for positive rate increasing catalysis). For example just cited, a possible but not confirmed, mechanism is the direct addition of HBr to $(\text{CH}_3)_3\text{COH}$ to form the intermediate (Berry, R.S 1984).

However, the mechanism of catalyst depends strongly on the type of reaction involved as well as on the nature of the catalyst.

2.6.1 *Catalysis*

Catalysis generally refers to chemical process in which chemical reaction rates are subjected to the influence of substances which may or may not change chemically during the reaction. Such a substance is termed a catalyst.

Basically, a catalyst is considered to form an intermediate compound with some of the reactants which in turn interacts with other reactants to form desired products and to regenerate the catalyst. In this way, a catalyst enables a reaction to proceed at a faster rate by a mechanism that requires a lower activation energy than that for the uncatalyzed one.

Catalysis is customarily classified into two major categories, namely homogenous and heterogeneous catalytic reactions.

In the former the catalyst forms a homogenous phase with a phase distinct from the reaction mixture.

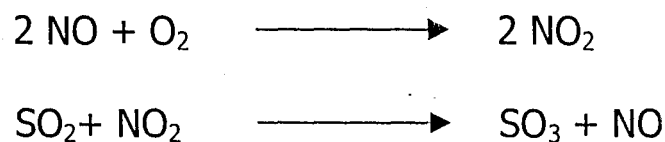
2.6.2 *Basic Characteristics of Catalysis*

These may be summarized as follows:

2.6.3 **Homogeneous Catalysis**

Homogeneous catalytic reactions have been observed to take place either in the gas phase or in the liquid phase. A great number of catalysis in this category have been found to follow rate equations which are first order with respect to the catalyst concentration.

2.6.3.1 **Gas-phase catalysis:-** A well known example of homogeneous catalysis: A well – known example of homogeneous catalysis in the gas phase is the oxidation of SO_2 to SO_3 , catalyzed by nitric oxide in the lead chamber Sulfuric acid process. The presence of Nitric Oxide promotes the normally very slow oxidation process by the following mechanism.



Additional examples can be found in the molecular Iodine – catalyzed pyrolytic decomposition of such organic compounds as acetaldehyde, formaldehyde, methyl alcohol, ethylene oxide, and several aliphatic ethers.

2.6.3.2 **Liquid-phase catalysis:** most homogenous catalysis occurs in catalytic reaction in the liquid-phase. Acid-base catalysis is the type of liquid-phase catalytic reaction that has been most extensively studied. Examples of

important organic reactions the rate of which are controlled by acid-base catalysis includes:

1. Inversion of sugars
2. Hydrolysis of esters and amides
3. Halogenation of acetone and nitroparaffins
4. Mutarotation of glucose
5. Esterification of alcohols
6. Enolization of aldehyde and Ketones.

For general acid-or general base-catalyzed reactions, the relationship between the catalyst effectiveness (in terms of the rate constant) and the strength of the acid or base (ionization constant) has been frequently expressed by the Bronsted equation, $K = Ck^a$, where K represents the rate constant (or catalytic constant), k is the ionization constant of either acid or base, and c and a are empirical constants governed by the type of reaction, the temperature and the solvent. The observed values of a lie in the range 0.3 to 0.9. the Bronsted equation has been found to hold rather well for many acid-base catalyzed reactions.

2.6.4 Heterogeneous Catalysis

The catalytic reaction system under this classification involves two or more of gas, liquid and solid phases. The catalyst is present in a phase different from those of the reactants.

The solid-catalyzed flood-phase different from those of the reactants are by far the most important and commonly encountered types in industrial processes.

(Robert H. Perry, 1982).

2.6.5 Factors Affecting Catalytic Reactions

They include the following:

1. Fluid dynamic factors e.g. mass velocity
2. Catalyst properties e.g. particles size, porosity and pore dimensions, and surface characteristics.
3. Diffusion characteristics of fluid reactants and products
4. Activation energy requirements for adsorption and desorption of fluid reactants and products.
5. Activation energy of the surface reaction.
6. Thermal factors e.g. temperature, heat-transport characteristics

2.7 Support Chemistry

The support chosen for a catalyst has a critical impact on catalyst activity, selectivity, and ease of catalyst recycling. The support can impart an acidic or basic environment for the active catalyst component. Each support chemistry has different tendencies towards impurities (which can poison the desired reaction or enhance a competing reaction).

In addition, each support chemistry has a unique range of available pore size distributions and stability to thermal, hydrothermal or acidic conditions. The ability to maintain surface area can minimize sintering of metals on the support surface. Davison catalyst has a wide variety of supports. These customized products offer a wide range of chemical and structural properties based on highly porous silica, Alumina. Mixed oxides and zeolites for heterogeneous catalysis and biotechnology applications. Final specifications of these are a result of extensive testing and feed back in close cooperation.

2.7.1 *Preparation of catalyst sample using clay/alumina as support*

Alumina supports have a wide range of surface areas and pore volumes. The supports be treated for excellent stability at high temperatures to avoid agglomeration sintering of surface metals. Appropriate for intermediate PH.

2.7.2 *Preparation of a catalyst samples using clay/silica-alumina as support*

Silica/Alumina supports are designed to have a broad range of surface areas. Coupled with an acid function. The acid site concentration is determined by the S_i-Al ratio of the support.

2.7.3 ***Clay Supports***

Clay supports are inexpensive and can provide some catalytic functionality at higher temperatures.

2.7.4 ***Alumina/Titania Support***

Alumina/Titania have the advantages of alumina with the additional functionality of Titania.

2.7.5 ***Alumina/Zirconia Support***

Alumina/Zirconia supports have the pore structure of alumina with the additional functionality of zirconia.

2.7.6 ***Carbon Hybrids Support***

Carbon hybrid supports are a new class of catalyst support that combine the features of carbon and solid oxide supports to provide catalytic surface with unique properties.

2.7.7 ***Ceria/Zirconia Support***

Ceria/zirconia supports are designed for applications that require a large oxygen storage capacity coupled with a high rate of oxygen release appropriate for high PH conditions.

2.7.8 Silica Support

Silica supports are designed to have the broadest range of surface areas and pore volumes. Silica can be surface treated to minimize additional catalysis effects. These materials can be used over a wide range of PH conditions.

2.7.9 Silica -Titania Support

Silica/Titania supports have the pore structure of silica with an additional acid functions.

2.8 Zeolites Support

Zeolite supports are designed for reactions that can take advantage of the shape-selectivity or size exclusion of the zeolite pore. Typical pore sizes are between 4 and 8 Angstrous.

(Jason, M.M1993).

CHAPTER THREE

D EXPERIMENTALS

These includes:

- a. calcination
- b. Formation of Pellets for X-ray Analysis
- c. Loss of Ignition test (L.O.I)
- d. Preparation of slides.

3.1 *Calcination*

Generally, calcination involves heating above 500⁰C.

A 500g each of the four samples were taken and calcined at a temperature of 600⁰C using a furnace.

The samples were grounded using a mortar, and a sieve of mesh 250 μ m was used to collect the fine particles of the samples.

The sieved samples collected were bottled and labeled accordingly for the next phase of the evaluation.

3.2 *Formation of pellets for x-ray analysis*

This is usually the preparation of good quality pellets so as to avoid inconsistent result from the X-ray. It takes an average of five minutes to prepare a pellet. The samples were placed on a piece of paper and was mixed thoroughly using a spatula, a 20.00g of the samples were weighed and 0.4g of stearic acid

also weighed. The samples and the stearic acid were slightly mixed.

A weight of 1.0g of the stearic acid was taken and put into the aluminum pellet cup where a heap of the sample was added into the aluminum cup. The surface aluminum cup containing the mixture was leveled using a spatula, and the Herzog press was made ready with the swirling cross head in the open position and the ram sufficiently low to enable cup insertion, that is adjustable with the level controller.

The base of the cup was cleaned and inserted into the press where it was turned on using the forward/reverse lever to the left with the function of the Herzog machine; then the swirling cross head was closed and tightened.

The lever was normally operated until a pressure of 200KN was reached and the pressure was allowed for one minute before the forward/reverse lever was turned to the center position gently. The swiveling cross head was opened and the forward/reverse lever was turned to the right position where the manual lever was operated to raise the sample to the unloading position. The pellets were removed and labeled accordingly.

Inspection of the pellets was done for any sign of cracks or contamination in especially for silvers of aluminum near the centre of the pellet; if there were any cracks or contamination,

the process would have been replaced with more of the grind sample.

The sample were identified before it was placed into the X-ray machine for analysis, were the result was a basic/systematic arrangement of atoms or ions in crystal planes of each samples.

3.3 ***Loss of Ignition Test (L.O.I)***

This involves the determination of the loss of ignition of the product.

A platinum crucible was washed and ignited in the muffle furnace at $950 \pm 250^\circ\text{C}$ for about 3 minutes, then it was removed and placed in a dessicator for about five minutes to cool in order to cool the crucible.

The empty crucibles were weighed, and 1.00g of sample was weighed into it, and the crucibles containing the samples were put into the furnace and ignited at $950 \pm 25^\circ\text{C}$ for about 30 minutes. The crucibles were removed and allowed to cool in a dessicator for about five minutes, then weighed; the weighed of the content's of the crucibles were determined by difference and recorded as W.

3.4 *Preparation of Slides*

This involves the process of getting the structure of the clay samples. Basically, the apparatus used in this experiment/preparations are as follows;

1. Glass Slides
2. Cover slips

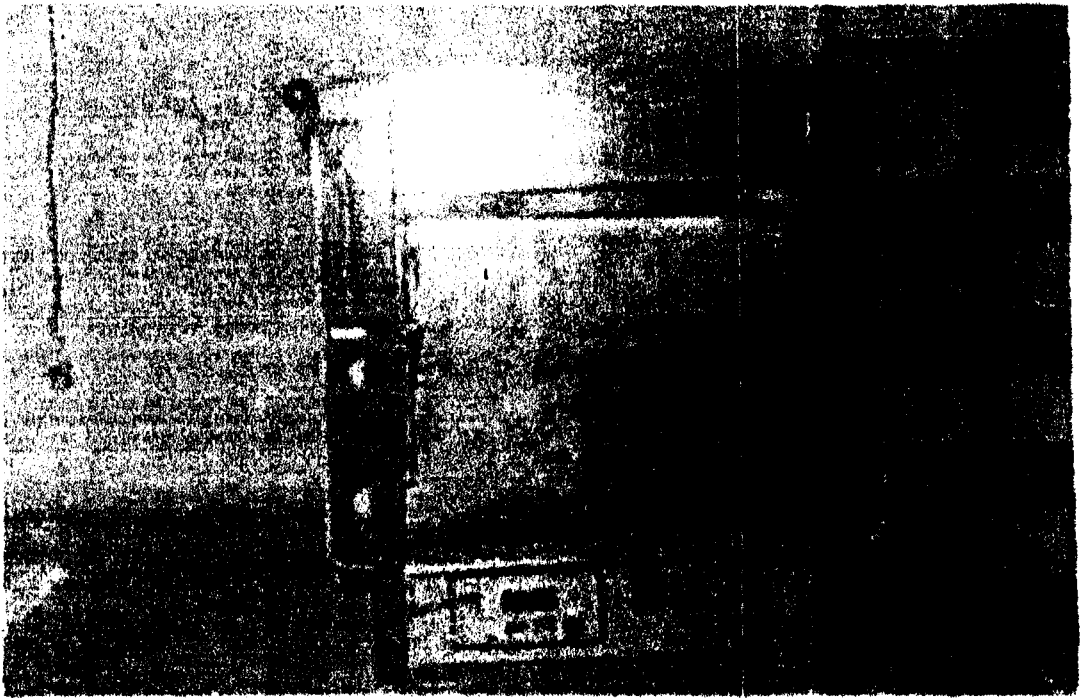
The preparation is of two methods, the surface and the vacuum; but the surface was employed in this case. The samples were first impregnated with binding epoxy-resin (Arladite and hardener), and when the clay particles was introduced into proper portion of the arladite and hardener it was mounted on a hot plate for solidification, after which the temperature was regulated to a desired state; then it was switched off and allowed to cool for 24 hours.

The item was removed as a compact material and was grounded, this process of grinding is called lapping and it is for the reduction of the samples into a desired thin section of $30\mu\text{m}$ (micrometer) or 0.03mm , with the silicon crucible having various grades of C₆120, C6220, to F₁₀₀₀ being the finest and C₆120 being the coarse.

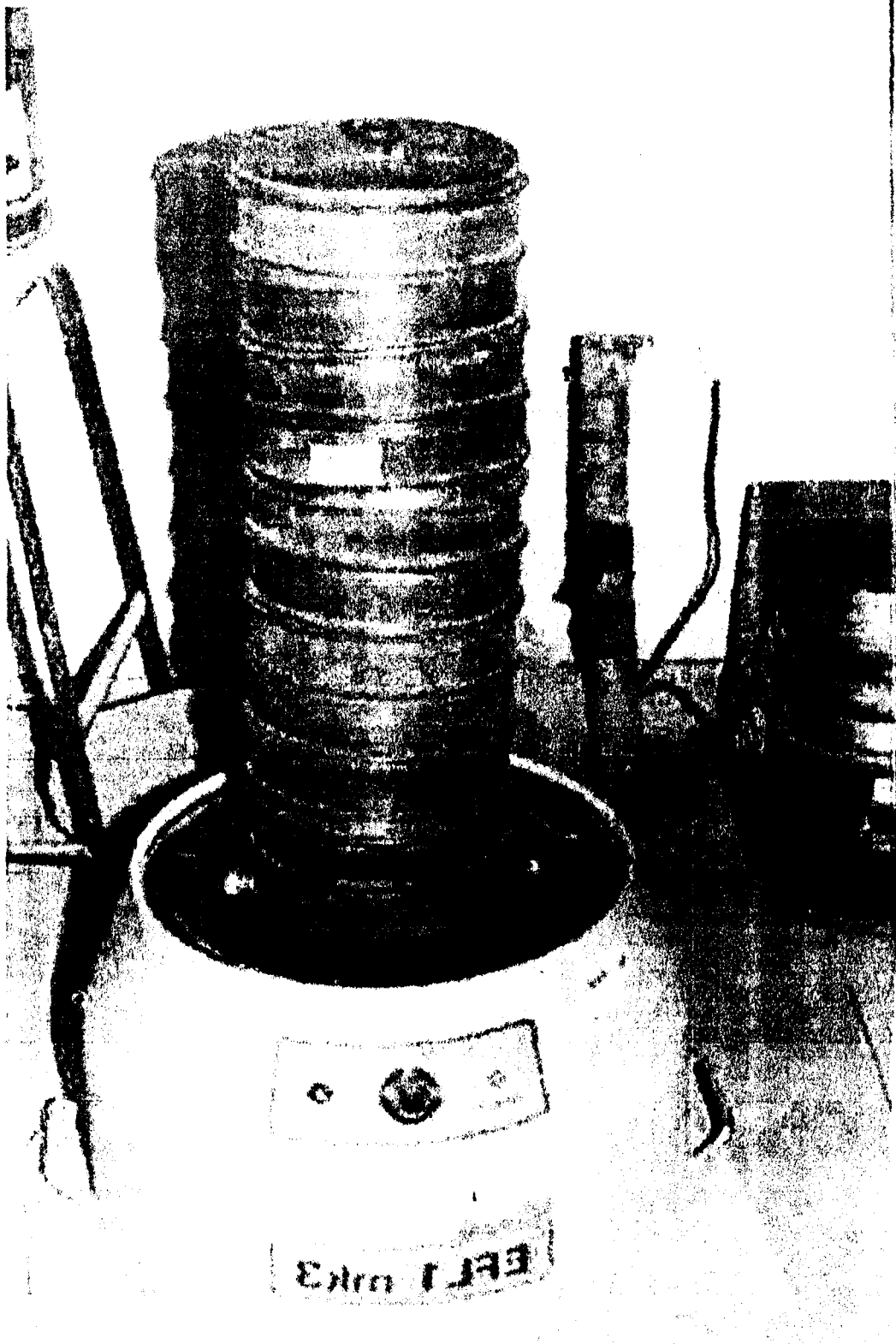
As the particle size progresses, it was thoroughly washed to avoid contamination; and at the $30\mu\text{m}$ stage, it was observed under the petrological microscope for the desired thickness.

The excess arladite was removed and covered with cover slips

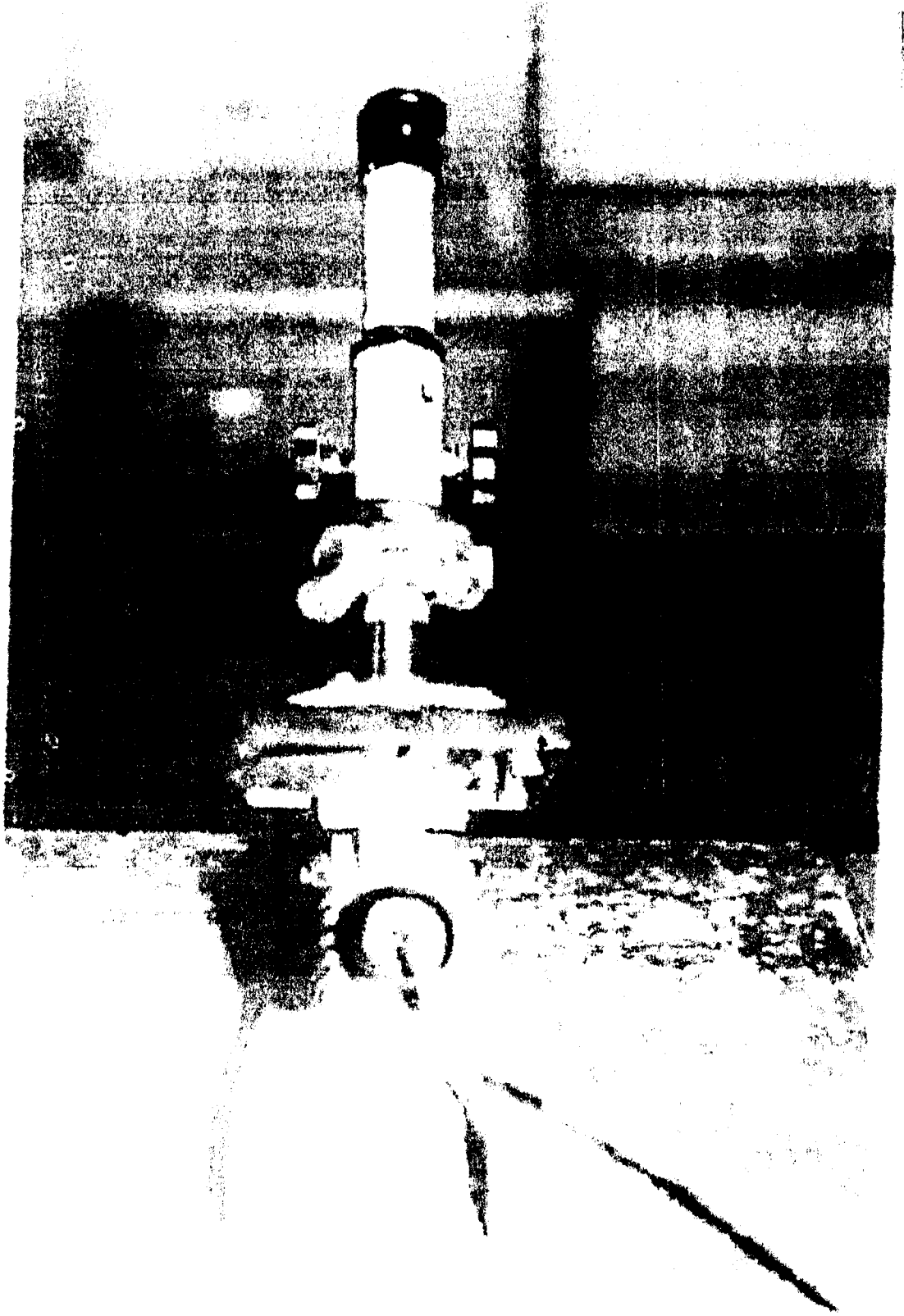
to prevent contamination then the inscriptions were transferred.



FURNANCE



GRINDING MACHINE USE FOR THE PREPARATION
OF SLIDES



PETROLOGICAL MICROSCOPE

CHAPTER FOUR

4.0 RESULTS

Calcination

A weighed Mass of 500g of the samples were taken and calcined. These were the results gotten after the calcination with respect to the weight.

TABLE 4.1: Showing the weight after calcination.

Clay type (area)	Weight before calcining (g)	Weight after calcining (g)
TOMBIA	500	192.4
OLODI-APAPA	500	96.1
MAIKUNKELE	500	113.3
PAIKO	500	110.2
TOTAL	2000	512

TABLE 4.2: Showing the loss of ignition test of the clay samples.

SAMPLES	TOMBIA CLAY	OLODI-APAPA CLAY	MAIKUNKELE CLAY	PAIKO CLAY
WEIGHT OF CRUCIBLE + CONTENTS BEFORE IGNITION TEST	55.4755	54.8269	15.2919	55.5925
WEIGHT OF CRUCIBLE + CONTENTS AFTER IGNITION	55.4620	54.7737	15.2828	55.5728
LOSS OF IGNITION	0.0135	0.0532	0.0091	0.0197
PERCENTAGE LOSS OF IGNITION	1.35	5.32	0.91	1.97

TABLE 4.3: Shows the X-ray Analysis of Tombia Sample. This analysis shows the composition of the clay sample.

TOMBIA CLAY

CONSTITUENTS	CONCENTRATIONS IN PERCENT
SiO ₂	81.13
Al ₂ O ₃	10.42
Fe ₂ O ₃	4.05
CaO	0.06
MgO	0.25
K ₂ O	1.33
Na ₂ O	0.06
SO ₃	0.08
CaCO ₃	0.11
MgCO ₃	0.52
SR	5.61
AR	2.57
LSF	0.03
TOTAL	97.37%

TABLE 4.4: Shows the constituents of Olodi Apapa Clay**CONSTITUENTS OF OLODI-APAPA CLAY**

CONSTITUENTS	CONCENTRATIONS IN PERCENT
Al ₂ O ₃	51.79
SiO ₂	29.78
Fe ₂ O ₃	6.99
CaO	-0.21
MgO	-0.39
K ₂ O	0.24
Na ₂ O	0.10
SO ₃	0.09
Cl	0.061
CaCO ₃	-0.38
MgCO ₃	-0.81
SR	1.41
AR	4.26
TOTAL	88.44%

TABLE 4.5: Shows the composition of Maikunkele clay**MAIKUNKELE CLAY**

CONSTITUENTS	CONCENTRATIONS IN PERCENT
SiO ₂	73.55
Al ₂ O ₃	18.14
Fe ₂ O ₃	3.14
CaO	-0.08
MgO	-0.47
K ₂ O	1.28
Na ₂ O	0.10
SO ₃	0.08
Cl	0.049
CaCO ₃	-0.14
MgCO ₃	-0.98
SR	3.46
AR	5.77
TOTAL	95.80%

TABLE 4.6: This shows the composition of Paiko clay**CONSTITUENT ELEMENTS OF PAIKO CLAY**

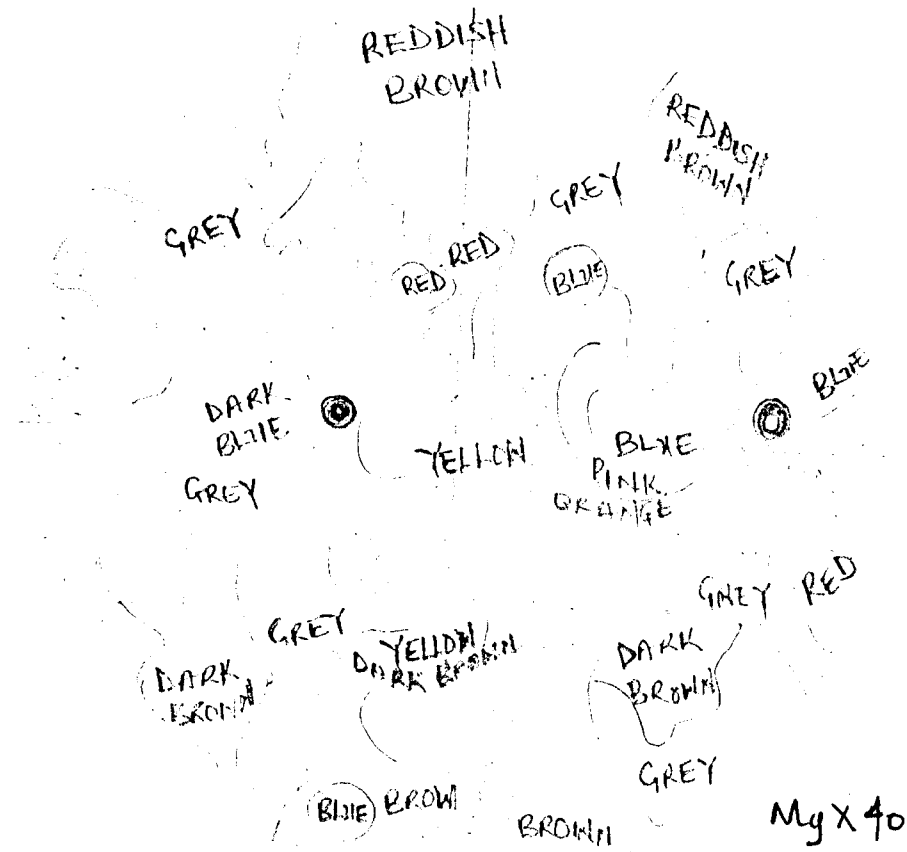
CONSTITUENTS	CONCENTRATIONS IN PERCENT
SiO ₂	62.31
Al ₂ O ₃	20.64
Fe ₂ O ₃	4.28
CaO	1.17
MgO	-0.44
K ₂ O	1.79
Na ₂ O	0.10
SO ₃	0.08
Cl	0.056
CaCO ₃	2.10
MgCO ₃	-0.93
SR	2.50
AR	4.83
TOTAL	89.98%

MICROSCOPIC STRUCTURE OF TOMBIA CLAY



M_gX40.

MICROSCOPIC STRUCTURE OF MAIKUNKELE CLAY



MICROSCOPIC STRUCTURE OF OKODI-APAPA CLAY

CHAPTER FIVE

5.0 DISCUSSION OF RESULTS

From table 4.1: Each of the samples were reweighed after calcining, it was discovered that there was a loss of weight of each sample with an initial weight of 500g.

Tombia clay was noted to have the highest weight after calcining with 192.4g and Olodi-Apapa having the least weight. The weights of Maikunkele and Paiko were 113.3g and 110.2g respectively showing a close range of their losses.

Generally, these loss of weights observed can be due to bonding of the particles of the clays with the Tombia clay having a more compacted particles with high moisture content thereby resulting to a lower loss of weight.

When compared, the Olodi-Apapa clay has the most weight loss, this can be due to the fact that it has a weaker bonds and its moisture content is of low value.

Similarly, the loss of ignition test in table 4.2, shows that Olodi-Apapa clay has the highest percent of the test with Maikunkele clay having the least, this may be due to the fact that the aluminum ratio (AR) of the Maikunkele clay sample is of higher value when compared with the others.

However, the X-ray analysis of the clay samples shows some discrepancies and differences in their constituents. For instance, it was observed that Tombia clay has the highest

Silicon Oxide Concentrations, this may be due to the fact that it is sedimentary in nature, that is its formation was as a result of decomposed marine and aquatic matters. Also from table 4.3, it was observed that sodium oxide (Na_2O) and Calcium Oxide (CaO) has the least concentration as a constituent of the Tombia clay sample.

In addition, it was observed that 97.37 percent was the total percentage concentration of the constituents with the silicon ratio (SR) being very high at 5.61, this is due to the high amount of concentrations in silicon oxide SiO_2 of the clay sample.

Also from table 4.4, which shows the constituents and its concentrations, it was observed that the aluminum concentration was quite high when compared with the Tombia clay thereby having a high Aluminium ratio (AR) of 4.26 percent, but on observation also, the calcium oxide (CaO), magnesium oxide (MgO), and their carbonates has a negligible percentage concentration with a negative value with the calcium and magnesium oxides having -0.21 and -0.39 respectively, and their carbonates with -0.38 and -0.81 percents. Due to the high level of the Aluminum and Iron oxides, the silicon concentration is quite low when compared with Tombia clay as a result low silicon ratio (SR) is observed.

Maikunkele clay from table 4.5 shows a high concentration of silicon oxide with 73.55 percent and the Aluminum ratio (AR) being on the high side of 5.77 percent. This may be due to the low level of concentrations of the Aluminum (Al_2O_3) and Iron Fe_2O_3 oxides with 18.14 and 3.14 respectively.

The oxides of calcium (CaO), magnesium (MgO) and their carbonates were observed to carry a negative value depicting a negligible value of concentrations of these constituents.

Going through the composition of Paiko clay in table 4.6, the Aluminum Oxide concentrations is quite high when compared with Maikunkele clay with 20.64 percent, thereby having a higher Aluminum ratio (AR) of 4.83 percent. The magnesium oxide and carbonates of this clay shows a negligible concentration of -0.44 and -0.93 respectively.

In effect, these characteristics exhibited by the clay samples gives a viability as a catalyst; for instance the loss of weights indicates the finest of the clay particles, hereby increasing the surface area for catalytic processes.

Also going by the loss of ignition observed, which gives a loss of weight that is associated with the ionic charges of the clay samples thereby making it suitable for use as a catalyst.

CHAPTER SIX

6.0 CONCLUSIONS

Going by the property of a catalyst possessing an active sites that is highly concentrated, and are structurally arranged, the same can be said of the clay minerals, which have a well structured constituents, such as the Tombia clay with high silicon level of concentration, the silicon oxide forms an hexagonal structure, this structural bonds gives room for catalytic process.

Therefore, the various clay samples analyzed has a good property for catalytic reactions.

6.1 RECOMMENDATIONS

1. I recommend that other analysis such as the spectroscopic analyst should be carried out on clay samples.
2. Since only secondary clays were analyzed, there is the need of analyzing the primary form of clay.
3. Viewing the slides should be done with the aid of a stronger magnifying microscope.
4. A photographic microscope should be used for the pictorial viewing of the clay structures.

APPENDIX

This formula was used for the calculation of Loss of ignition on the clay samples.

.; and the percentage loss of ignition was calculated using the formula below.

$$\% \text{ L.O.I.} = (1-W) \times 100$$

Where W is the weight of the contents of the crucibles.

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