REFINING OF SPENT LUBE
USING LOCAL ADSORBENT

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A PROJECT SUBMITTED TO THE
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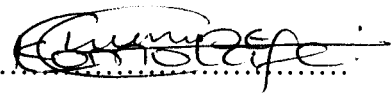## BY

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MINNA.

NOVEMBER 2004.

DECLARATION.

This project was carried out by KOMOLAFE OLUMIDE under the supervisor of ENGINEER M.A OLUTOYE in the department of Chemical Engineering, Federal University Of Technology, Minna.

I declare that this research work is my original work. To the best of my knowledge, it has never been wholly submitted somewhere else before.

....25 /11 /04

KOMOLAFE OLUMIDE   DATE

CERTIFICATION.

This is to certify that the project of title "REFINING OF SPENT LUBE USING LOCAL ABSORBENT " was carried out by KOMOLAFE OLUMIDE under the supervision of ENGINEER M.A OLUTOYE and submitted to the department of Chemical Engineering, School of Engineering And Engineering Technology of the Federal university Of Technology, Minna in partial fulfillment of the requirement for the award of bachelor of Engineering (B.ENG) degree in Chemical Engineering.

....22-11-2004.......

ENGR M.A OLUTOYE   SIGNATURE & DATE
PROJECT SUPERVISOR

....

                                                       SIGNATURE & D

HEAD OF DEPARTMENT

....

EXTERNAL EXAMINER   SIGNATURE & DATE

DEDICATION.

This project is dedicated to GOD ALMIGHTY for he is my source of strength and inspiration and also to my parents Mr. & Mrs. Komolafe for their financial and moral support. My love goes to my siblings and friends.

ACKNOWLEDGEMENT.

atitude goes to my parents Mr. & Mrs. KOMOLAFE for their moral and

.1 the commencement of this project to the present stage and to the entire family,

rs for their support in one way or the other.

I will also like to express my appreciation to my supervisor, ENGINEER M.A OLUTOYE for his moral support throughout the project. He gave advices and encouraged me at all time, believed in me even when I had doubts.

More love goes to my siblings and friends.

ABSTRACT

The project work was centered on the refining of spent lube using local absorbent (clay). The clay was collected from Shelter clay from Pogo local government area of Chanchanga, Niger State. The clay was calcined at a temperature of 500^0C-550^0C and activated using 0.3% concentrated H_2SO_4. The spent was ran on various amount of adsorbent for the duration of 20 minutes. Various parameters are used for analysis which the include: - Viscosity index, Flash point, Kinematic viscosity at 40^0c and 100^0c, Appearance, Specific gravity. The result obtained from the refined spent oil was compared with the similar parameters obtained from the untreated engine oil.

The Kinematic viscosity decreases from 155.99-153.22 m^2s^{-1} at 40^oC and 16.74-16.6 m^2s^{-1} at 100^oC.The flash point increases from 120^oC of the spent engine oil to 136^oC of the refined engine oil. The viscosity index increases from 116-120 while for Total base number is zero.

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CHAPTER ONE

1.0 INTRODUCTION

Nigeria as a developing country has been striving towards an industrial revolution of a Refining process. This research will contribute to the development of local available raw materials for some industrial needs.

Spent lube is defined as any oil that has been refined from crude oil and has been used. The major sources of spent lube includes automotive and industrial. The spent lube oil constitutes one of the major pollutants today, especially in the automotive industries where they are constantly used and subsequently disposed as waste oil. Its predominately contain paraffin, naphthenic and aromatic hydrocarbon. (Aliyu Oyetunde, 1997)

However, this research attempt to explore the application of refining spent lube oil using local adsorbent in order to reduce its disposal and pollution problems, also to check if the Developed adsorbent for the local available materials can adsorb certain constituents of the spent lube oil.

The adsorption is stated as a separation process in which molecules diffuses from the bulk of the fluid to the surface of the solid adsorbent forming a distinct adsorbed phase. It's a Physical-Chemical process, which's developed as a major industrial separation process. Its Involved two phases, which include: - a solid phase and the other liquid or gaseous phase.

Adsorbent is a natural or synthetic material of amorphous structure whose internal pores surface is accessible for selective combination of solid and solute. The two major steps in producing adsorbent include carbonation and activation. (Perry, R.H and Chilton, C.L, 1983)

In this research, the adsorbent used is the activated clay as a local adsorbent. The activated clay is used in the treatment of petroleum fraction, refining of mineral oil.

Clay is defined as a natural earthy, fine-grained material that develops plasticity when mixed with a limited amount of water. (Hiller.S, 1995). The clay are composed essentially of silica, alumina, water and appreciable quantities of iron, alkalis and alkaline. The clay's mineral components provide the essential characteristics and properties of clay. The clay minerals occur in nature, in sedimentary rock of all ages and in continental and marine sediment. (Hiller S, 1995). The major clay resources include kaolinite, halloysite, illites, chlorite, momtmorillonitre (bentonitres), attapulgite, and allophane.e.t.c. (Velder B, 1995).

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However, in this research, the activated clay used was gotten from shelter clay in Chanchanga local government area. The source of the clay used is kaolin.

The clay gotten is been crushed and sieved into a fine powdery form. There are two major process involved in the experimental research which include the calcinations and activation.

The calcinations process involve the heating of the clay at a high temperature of about $500^{\circ}C$ for a duration of six hours inside the furnace while the activation process involve the treatment of the calcined clay with acidified H_2SO_4 (Tetraoxosulphate (vi) acid) as an activated agent. This will increase the adsorptive and porosity of the clay.

1.1 OBJECTIVE

The aim of this project is to regenerate spent lube oil using local developed adsorbent. The local adsorbent used is clay. A comparative study is made on the adsorbent and the retention capacity with the view to improve on the quality of the spent lube oil.

Among others, the project attempts to: -

- Determine the particle size effect of the local adsorbent.

- Analyze the adsorption and retention capacity of the adsorbent.

However, the result will then be compared with the standard products of the Lube Blending company of Nigeria (LUBCON) that conforms to standard requirement as published by the Society of Automotive Engineers (SAE) and American Petroleum Institute (API).

1.2 SCOPE

The project research cover on the characteristics of spent lube oil before and after It has been refined by the developed local adsorbent and also comparing their characteristics, So as to find out if certain impurities had been adsorbed.

1.3   PROJECT MOTIVATION

In the Nigeria market today, it is evident that the availability of refined spent lube oil is more on the minimal side and that even some of the products present in the market are of low quality. It's also to know if the clay can be used as an adsorbent, by adsorbing the impurities containing in the spent lube oil or not.

I have therefore been motivated based on this facts to chose this project topic in other to increase the numbers of refined spent lube manufacturer in the market, that there might be more of the products in the market and more importantly, to ensure that the products is of good quality.

CHAPTER TWO

2.0   LITERATURE REVIEW

2.1   Historical Background on Lubrication

The knowledge of lubrication as catalyst to industrial revolution dates from the early

History in Egypt, Greece and Rome where grease was made of a combination of animal fats and

calcium were used to lubricate the chariot wheels. (Mc Graw hill, 1982)

In the early nineteen-century, vegetable oil from olives, rape seeds, castor seeds and animals

oil from tallow, land and whales were mixed with potassium, sodium, calcium soaps to make grease

use for lubrication of stream cylinders, cutting metals and later an aircraft engines during the World

War 1 (1914-1916). (Engr. Kayode S, 1994)

The breakthrough in the Petroleum distillation and refining technology mid-19th century,

replaced fatty oils with mineral oils for lubrication where some of the viscous by-products were

investigated as better substitute for vegetable and animal oil used for lubrication of engine parts.

The knowledge of lubrication has helped to reduce considerably the problem posed by the loss

of energy due to friction developed in all surfaces of the relative motion.

2.1.1   Lubricant

A lubricant can be defined as an oil products that separates the metal parts of an engine,

Reduce friction and keep it clean. Lubricant deals with the application of lubricating oil to machine.

(Willet A.S, 1968)

The lubrication system of a engine is intended to avoid the increased of wear, overheating and

seizure of rubbing surfaces to reduces the expenditure of indicated power on

overcoming mechanical losses in the engine and also to remove wear products of a machine.

There are four major types of lubricants namely: - liquid, solid, gaseous and plastics Lubricant.

Examples of lubricant include oils, greases, air and graphite. Liquid and Plastics lubricant are the most

commonly used lubricant in industries because they are inexpensive, easily applied and good coolants

while Gaseous and Solid lubricant are recommended only in some special application.

(Willet A.S, 1968)

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In view of the problem encounter, lubricating oil is designed to impact varieties of properties and to protect engine in so many ways. Thus, lubricating oil is highly specialized products carefully developed to perform many essential functions among which are the followings: Permit easy starting of engine, reduced friction, protecting machine against rust and corrosion, lubricating of engine parts.e.t.c. (Williams T.O, 1995)

2.1.2 Sources and Composition of Lubricating oil

Mineral Based oils.

Modern mineral based oils are produced from naturally occurring crude petroleum oils by initial process of vacuum distillation. It's usually followed by additional purification process. Such as solvent extraction, dewaxing, Ferro fining clay treatment, acid refining and neutralizing depending on ultimate analysis. The chemical composition of the mineral oils is produced depending on both the original crude and refining processes. (Cockl V. R, 1988)

TABLE 2.1.2: Composition of Mineral Based oils.

CHARACTERISTIC	100N	150N	250N	500N	BS
Kinetic Viscosity (cst)	39-4.3	4.9-5.5	6.9-7.7	108-12.0	305-325
Viscosity Index	95	95	95	95	95
Flash Point (^{o}C)	193	204	210	221	226
Fire Point (^{0}C)	198	210	217	236	234
Pour Point (^{0}C)	-9	-9	-9	-9	-9
Appearance	Clear and Bright	Clear and Bright	Clear and Bright	Clear and Bright	Clear and Bright
Water content	Nil	Nil	Nil	Nil	Nil

Table above show the physical composition of mineral based oils. (Williams T.O, 1995)

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2.1.3 Recycling and its Mechanism

Recycling is the process of generating used material substances so that it can be used

Again. The recycling of used engine oil has to do with the mechanism and processes involved in

removing the impurities in the used oil and bringing it to the initial state. Engine oil is used

Up, when there's no function of additives again. (Aliyu. O, 1997)

The used oil is dark in colour due to carbon from wears. The acid present maintains the colour

of the engine oil, but in used oil, the acid is weakened by the neutralization of a base.
The most popular physical methods include setting, centrifugation, filtration, distillation and scrubbing

with water to remove water-soluble acids.

The additives are incorporated into the recycled engine oil and sometimes mixed with fresh oil.

(Whitney J.C, 2000)

2.1.4 Additive Technology

Additives are chemical compound added to the refined based stocks to impact some specific

properties to the lubricating oils either by enhancing the inherent properties or adding new but useful

ones into the finished products. (Lukanin V.N, 1990)

A lot of unfortified base oil with additive is being sold as motor oils in our country. So, the base

oil have to consists some constitutes required for lubrication. However, these are not sufficient to meet

the lubrication requirement of modern highly rated engines. Additives must
Therefore be added to the base oil to produce quality lubricants. (Whitney J.C, 2000)

2.1.5 Types of Additives

There are different types of additives used in blending the various classes and grades of

lubricating oils to enable such finished products meet the performance requirement of the engine. The

properties, specification and characteristics of additives enable them to perform one of more specific

functions in achieving the desired level of improvement in the finished

Engine oil. (Whitney J.C, 2000)

The different types of additives commonly in use include as follows: -

a) Detergent and Dispersants.

b) Pour Point Dispersant.

c) Viscosity index improvers.

d) Oxidation inhibitors.

a) Detergent And Dispersants

Detergent is a metallic organic compound of sodium, calcium, magnesium, phenolates, phosphonates and suffocates. They are soluble metallic compounds high molecular weight, Organic acid that have the affinity to dissolve the carbonaceous deposit in ring grooves, Piston surfaces and single deposit in internal combustion engines. (Williams T.O, 1995)

b) Pour point Dispersant

There are polymers of alky-aromatics and polymethacrylate that enable oils to flow easily without any agitation, they are used to parent growth of large wax crystals into interlocked Structures as the oil cool down.

c) Viscosity Index Improvers

These are large chains that have high molecular weight polymers such as polysolutyfenes, Polymethacrylates, olefin copolymers and acryl ate polymers that prevent the decrease in viscosity of oil at high temperature.

d) Oxidation Inhibitors

These are chemical property modifier called anti- oxidants.eg zinc, aromatics amines, Hundred phenols and sulphorized esters. They inhibits oxidation process, acid formation, Oil deterioration to prevent varnish and lacquer deposit and also catalytic effect of metals on the oxidation process at high temperature of air. (Williams T.O, 1995)

However, these additives package listed above are needed in blending process of the Lubricating oil because each has a specifics function it contributes for the better performance Of the oil.

2.1.6 Properties Of Lubricant.

a)   Viscosity

This is a measure of the flow ability of oil. It develops and maintains a certain amount of shearing stress and offer resistance to flow.

b) Viscosity Index

This is an empirical number indicating the rate of change in viscosity of an oil within a given temperature range. The higher the number, the better the oil.

c) Percentage Sulphates Ash

This is left over of solid material when oil has been burn. High ashes content tend to form more sludge and deposit to the engine while low ash tend to promote long valve life.

d) Flash Point

This is the temperature at which oil gives off vapors that can be ignited with a flame held over the oil. The lower the flash points, the greater the tendency for the oil to suffer vaporization.

e) Pour Point

This is 5^0F above the point at which a chilled oil shoes on movement at the surface for 5 seconds when inclined. The measurement is especially important for oil used in winter.

f) Specific Gravity

This is the ratio of mass of a given volume to the mass of an equal volume of water. Specific gravity is dimensionless. It decreases with increased in temperature and decreases slightly as viscosity decreases or similar compositions.

g) Total Base Number (TBN)

This is the quantity of acid expresses in terms of the equivalent number of milligram of potassium hydroxide that is required to neutralize all acidic constituents present in one gram of a sample.

h) Neutralization Number

This is an indication of the acidity or alkalinity of oil. The number is the weight in milligrams of the amount of acid or base express as potassium hydroxide equivalent to neutralized one gram of oil in accordance with an ASTM test method.

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2.1.7 Function Of Lubricant

Lubricants comprises of many functions, which include the following: -

a) Lubricate and prevent wear.

b) Reducing friction.

c) Protection against rust and corrosion.

d) Permition of easy starting.

e) Keeping engine parts clean.

a) <u>Reducing Friction</u>

Under full-film lubrication condition, a thick film of oil prevents metal-to-metal contact between moving engine parts. Relative movement of these lubricated parts requires enough force to overcome the fluid friction of the lubricant. The viscosity of the oil should be high enough to maintain an unbroken film but must not be higher than the necessary in order to permit easy starting. (Willet A.S, 1968)

b) <u>Keeping Engine Parts Clean</u>

Lubricants are generally formulated not only to keep engine parts clean, but also to prevent sludge deposit from interfering with proper engine operations. (Willet A.S, 1968)

2.2 Spent Lube Oil

Spent or used lubricating oil constitutes one of the major pollutants today, especially in the automotive industries where they are constantly used and subsequently disposed as waste oil. (Aliyu O, 1997)

Spent oil is defined as a lubricating oil that have passed through their intended used cycle and must be either disposed or treated and re-used. Generally, the term embraces spent automotive lubricating oil and spent industrial oil including those used for lubrication, Refrigeration and process application. (Aliyu O, 1997)

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Petroleum base lubricating oil contain predominantly paraffin, naphthenic and Aromatic hydrocarbons. The spent however contain insoluble degradation products such as polymers, resins and organic acid. Such degradation products include polar organic matters, which contain the presence of strong oxidizing acid, which may be induced to precipitate as adherent lacquer, varnish and asphalt resins. (Guthric V.B, 1987)

2.2.1 Characterization of spent lube oil.

Engine and industrial oils are composed of an organic base stock and additive packages which have been developed for specific lubricating applications in order to significantly increase the performance and life of the oil. (Whitney J.C, 2000)

The lubricating base stock comprises of hundred of thousand of organics constituents, the majority of which are polynuclear aromatic (PNAS). The additive comprises up to15% of the oil by volume, typically contain inorganic constituents such as Sulphur, nitrogen and trace metals.

In recent years, studies have documented the presence of additional compound such as chlorinated solvents in samples of waste oil. Some these compounds are present in refined products, while others are illegally blended with the used oil during storage. The followings are the characteristics of used oil, which include :-(Aliyu.O, 1997)

a) Polynuclear aromatic.

b) Oil degradation.

c) Oxidation.

d) Halogenated organics.

a) Polynuclear Aromatic

Polynuclear aromatics hydrocarbon are present in the petroleum base stock and can be produced during the use of the oil. Several of these compounds, primarily four, five and six ring structures are known as carcinogens and mutagens. Benzo (a) pyrene, B (a) P, is a prime. Example of a PNA that exhibits carcinogenic effects. (Aliyu. O, 1997)

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b) Oil Degradation

Oil in service is chemically and physically contaminated depending on the length of service and the conditions imposed on its by its application. A mineral oil lubricants in service will chemically change during that period of service by its exposure to heat oxygen, the formation of acids, and the catalytic metals it come in contact with.

2.2.2   Tests and Chemical Analysis of Spent Engine oil.

The various engine oil producing comprises of different chemical tests carried out on engine oil. Below are the common ones: -

a) Flash Point And Free Point

The flash point of oil is the temperature at which sufficient vapor is given off from it surface to cause ignition in the pressure of a small flame. A high flash point may indicate well-refined oil with freedom from components of low volality. Low volality components can result in high oil consumption in the case of engine oils. The result of the test may be used as an element of a five-risk assessment of the professed of the product identification.

(Institute of Petroleum, 1987)

b) Pour Point

It is the measure of an ability of an oil to pour out or flow at low temperature from containers or feed in some lubricating system (Institute of Petroleum, 1987). It is the lowest temperature at which it's possible for the oil to flow.

c) Carbon Residue

This is the amount of deposit left when oil is evaporated under certain conditions which can prevent its burning when heated to high temperature. It's considered as an indicator of the amount of deposit, which oil would leave in the combustion chambers in engine, final character; mixture ratio and operation conditions are of great importance.

d) Oxidation Tests

The general deterioration of lubricating oils is due to the formation of oxidation products, therefore any information supplied by a test or series of tests regarding rate of oxidation could prove useful in forecasting behaviour of an oil under working condition. A number of oxidation tests have been proposed, each based on the rate of change of some fundamental property of the oil e.g. viscosity, carbon residue, acid value etc.

e) Total base number.

(TBN And Total Acid Number)

Petroleum products may contain basic and acidic constituents such as an additive in new oils or as combustion by products in used oils. By titration with acid or bases, the relative amount of basic and acid substance in the lubricants can be determined. This relative amount is referred to as acid or base numbers. Acid and basic numbers can be used as standard in the quantity control of lubricating oil formulations. It could be used as a measure of lubricant degradation in services though with caution of implicating results is obtained. (Institute O .P, 1987)

2.2.3 **Organisation Involved In Testing Of Engine Oil.**

Various organizations have helped in establishing methods of measuring some properties of spent engine oil. This is for refinery operation control and for the purpose of identification. A few examples include: -

a) ASTM [America Society Of Testing Materials].

The ASTM established some of the method of measuring the properties of engine oil and also analysis the engine oil for quality control purpose. (Swoboda P.A.T, 1988)

b) SAE [Society Of Automotive Engineers.]

This organization guides in the grade of viscosity number. It denotes the viscosity index thickness or thinness of the oils at 100°C. An SAE 50 which has a grade of 50 [viscosity range of 18-22] is from more viscous to less viscous. While SAE 140 variety is very thick. The SAE is the most widely used. (Swoboda P.A.T, 1988)

c) API [AMERICA PETROLEUM INSTITUTE].

This is a guide to the blend of colour for lubricating oil. It guide in the grading of engine oil. The letters SG to SJ on the API rating signifies it. It increases alphabetically, for example, SECC, which is a guide by API for using additives to get the grade required.

2.3 Adsorbent

An adsorbent is a natural or synthetic material of amorphous structure whose internal pore surface are accessible for selective combination of solid and solute. (Perry R.H, 1983). Adsorbent have two controlling phenomena, which include the physical and chemical, but for this work, it's based on the physical phenomenon and the characterization will based on the physical properties of an adsorbent.

There are two major steps involved in the production of adsorbents and these include carbonization and activation. In carbonization, the carbonaceous material is decomposed at a high temperature at about 800^0C in the absence of oxygen to give the primary carbon while in activation process, it's involved the treatment of primary carbon obtained from carbonization with activating Agents such as steam hydrochloric acid; zinc chloric. These increase the adsorptive sites and porosity. (Bugaje I.M, 1980)

In the thirteenth century, Macro Polo found that the ashes of a certain wood, un-gven were used to refine sugar. (Mantell O.L, 1951)

Priestley unknowingly produced a partially activated carbon in his effort to increase the conducting power of the carbon. (Priestley, 1770)

According to Venlipman ill and Trommsdorf purified smell wires with wood charcoal In 1788. (Mantell O.L, 1951)

In the year 1971,Fuller, Sohatsbery and Nagy developed an adsorbents from wood-dust and straw, used as floating adsorbents for oil removal in USA. (Olaofe.O, 1977)

However in 1980, Bugaje produced an activated carbon from bagasse with a yield of 24% As a mean of converting industrial products to useable raw materials. (Bugaje I.M, 1980)

Recently, in the year 1988, HyMore and Iyayi conducted the production of activated carbon from coconut shells and activated clays from Benin. (Hymore F.K, 1988)

2.3.1 Nature Of Adsorbents

Adsorbents are available as irregular granules, extruded pellet and formed spheres. The size will reflect the need to pack as much surface area as possible into a given volume of bed at same time, minimizing the pressure drop for flow through the bed. Sizes up to about 6mm are common.

To be attractive commercially, an adsorbent should embody a number of features, which include: -

- It should have a large internal surface area.
- The area should be accessible through pores, big enough to admit the molecules adsorbed. It is a bonus if the pores are also small enough to exclude molecules desired not to adsorb.
- The adsorbent should be capable of being regenerated easily.
- It should not age rapidly that is losing its adsorptive capacity through continuous recycling.
- It should be mechanically strong enough to withstand the bulk handling and vibrate that are part of any industrial unit.

2.3.2 Characteristics Of Adsorbents

Adsorbents are specific in their nature and properties, which gives explanation why some adsorbents are used for one operation. The general properties of adsorbents include the following: - (George S.B, 1986)

a) Shape of particles: - they could be cylindrical, pellets, granular powder, spherical.

b) Size range: - Based upon by US standard mesh.

c) Internal porosity (x%): - It's a measure of the uptake of water and will vary with the ion held.

d) Bulk density, pb (kg/m^3): - This is the weigh of dry material per unit bulk volume as packed in a column.

e) They attract the substance molecules via adhesion.

f) Granular adsorbents are employed in filter beds, but powdered adsorbents are stirred into the liquid and are usually more effectives.

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g) It should have a large internal surface area.

h) The adsorbent should be capable of being regenerated easily.

i) It should be mechanically strong enough to withstand the bulk handling and vibrate that part of any industrial unit.

2.3.3 Theory Of Adsorption

A system, which consists of physically homogenous part, is referred to as phase. A system consisting of a single phase is called homogenous while those consisting of two or more phased called heterogeneous system. When we are dealing with heterogeneous system, the boundary between the phases is termed as an interface. This system may be equilibrium or at non-equilibrium. In non-equilibrium system, the composition or thermodynamic parameter changes with time. (Philip A.B)

Adsorption may be defined by a chemical engineer, as a separation process and in common with other such processes; it involved two phases between which components become differentially distributed. The adsorption process is always exothermic regardless of the type of forces involved.

Adsorption differs from absorption by the degree of homogeneity that exists at the equilibrium in that place to which molecules are transferred. (Coulson, 1983)

a) Unit Operation Of Adsorption

Adsorption concern itself with the concentration as the result of surface forces existing on a solid, gases, vapors, liquids or solutes (i.e. solids dissolved in a solvent). The extent of adsorption is large or small depending on the nature and character of the adsorbent and adsorbente, i.e. material concentrated, localized, fixed or separated. (Mantell O.L, 1951)

b) Adsorption And Absorption

From the experimental angle, some confusion is met in differentiating between adsorption and absorption. At ordinary temperatures, adsorption is usually caused by intermolecular forces rather than the formation of a new chemical bonds, this is referred to as Physical adsorption or Physiosorption

At a high temperature, Chemical bonds are broken; this is referred to as Chemisorptions or activated adsorption. In absorption, there's a phase change and chemical reactions is permanent.

(Perry R.H, 1983)

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c) Gibbs Isotherm

An entirely different approach to equilibrium adsorption is to assume that adsorbed layers behave like liquid films, the adsorbed molecules being free to move over the surface. It's then possible to apply the equations of classical thermodynamic. The properties, which determine the free energy of the film, are its pressure and temperature, the number of molecules it contain and the area available to the film. The Gibbs free energy, G, can be written as: - (Robert H.P)

$$G = F[P, T, I_s, As] \quad\text{...(1.)}$$

Hence,

$$\partial G = \left[\frac{\partial G}{\partial P}\right]T, I_s, As \ \partial P + \left[\frac{\partial G}{\partial T}\right]p, I_s, As \ \partial T + \left[\frac{\partial G}{\partial I_s}\right]T, P, As \ \partial I_s + \left[\frac{\partial G}{\partial As}\right]T, P, I_s$$

$$\partial As \quad\text{...(2.)}$$

At constant temperature and pressure, this becomes: -

$$\partial G = \left[\frac{\partial G}{\partial I_s}\right]\partial I_s + \left[\frac{\partial G}{\partial As}\right]\partial As \quad\text{...................................(3.)}$$

$$= \mu_s \partial I_s - \Gamma \partial As \quad\text{...(4.)}$$

Where,

μ_s = Free energy per Mol or chemical potential of the film,

Γ = Two-dimensional or spreading pressure.

The total Gibbs free energy can be written: -

$$G = \mu_s I_s - \Gamma A_s \quad\text{..(5.)}$$

So that,

$$\partial G = \mu_s \partial I_s + I_s \partial \mu_s - \Gamma \partial A_s - A_s \partial \Gamma \quad\text{.............................(6.)}$$

A comparison of equations (4) and (6) shows: -

$$\partial \Gamma = \left[\frac{I_s}{A_s}\right]\partial \mu_s \quad\text{..(7.)}$$

If the gas phase is ideal and equilibrium exist between it and the sorbed phase then, by definition: -

$$\partial \mu_s = \partial \mu_\theta = RT\partial[LnP] \quad\text{.................................(8.)}$$

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Where,

$\mu\theta$ = Chemical potential of the gas.

$\therefore$ Substituting for $\partial\mu$s

$$\partial\Gamma = \frac{ns}{As}RT\partial[\ln P]$$ ....(9.)

Equation (9) has the form of an adsorption isotherm because it relates the amount adsorbed to the corresponding pressure. It's known, as Gibbs Adsorption Isotherm. An expression is needed for Γ. Assuming an analogy between adsorbed and liquid films, HARKINS and JURA proposed: - (Robert H.P)

$$\Gamma = \alpha_1 ---------- \beta_1 \, am$$ ....(10.)

Where,
α_1, β_1 = contants.

$a_m = \dfrac{A_s}{N\mathrm{I}_s}$, the area per molecules of adsorb ate.

N = Avogadro number.

$\therefore$ Substituting for $\partial\Gamma$ in equation (9) and integrating at constant As from some condition P_1, $\cap s_1$ at which the adsorbed film becomes mobile to an arbitrary coverage $\cap$s at pressure P, gives:-

$$In\frac{P}{P_1} = \frac{1}{RT}\frac{\beta_1 A_s^2}{2N}\left[\frac{1}{\mathrm{I}_{s_1}^2} - \frac{1}{\mathrm{I}_s^2}\right]$$ ---(11)

Equation (11) can be rewritten as: -

$$In\frac{P}{P^0} = L^1 - \frac{M^1}{V^2}$$ ---(12)

Where,

V=Volume occupied in the gas phase by $\cap$s mole of sorbate at a temperature, T and pressure, P

$$L^1 = In\frac{P_1}{P^0} + \frac{1}{RT}\frac{\beta_1 As^2}{2N}\frac{1}{\mathrm{I}_{s_1}^2}$$
---(13)
$$M^1 = \frac{1}{RT}\frac{\beta_1 As^2}{2N}\frac{1}{\rho^2 g}$$

Where,

ρg = molar density of gas phase.

Equation (12) is the Harkins-Jura (H-J) equation. It can be used to correlate adsorption data and to obtain an estimate of the surface area of an absorbent

d) Langmuir Isotherm

At higher gas phase concentrations, the number of molecules adsorbed soon, increases to the point at which adsorption is hindered by lack of space on the adsorbed surface. The rate of adsorption then becomes proportional to the empty surface available as well as to the fluid concentration. At a fixed temperature the rates of desorption will be proportional to the surface area occupied adsorbate. When the rate of adsorption and desorption are equal, a dynamic equilibrium exist. For adsorption, which is confined to monomolecular layers, the equilibrium can be written as:

$$K_0 a_0 C = K_0 [1 - a_1] C = K_1 a_1$$

$$a_1 = \frac{B_0 C}{1 + B_0 C} \text{--(1)}$$

Where,

a_o = Fraction of empty surface.

a_1 = Fraction of surface occupied by a monolayer of adsorbed molecules.

$$B_0 = \frac{K_0}{K_1}$$

Ko = velocity constant for adsorption onto empty surface

K_1 = velocity constant for desorption from a monolayer

Since equation (1) has been developed for adsorption from the gas phase, it is convenient to write it in terms of partial pressures. Hence,

$$\frac{C_s}{C_{sm}} = \frac{B_1 P}{1 + B_1 P} \text{--(2)}$$

Where,

Cs = Concentration of the adsorbed phase

Csm = Concentration of the adsorbed phase when the monolayer is complete.

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$$B_1 = \frac{B_0}{RT}$$

P = Partial pressure of the adsorbate in the gas phase

Equation (**1**) and (**2**) have the form of the LANGMUIR equation

developed in 1916 to descried the adsorption of gases onto plane surface of glass, mica and platinum.

As well as been limited to monolayer adsorption, the Langmuir equation assumes:

- No interaction between adjacent molecules on the surface

- The energy of the adsorption is the same all over the surface.

- Molecules adsorb at fixed sides and do not migrate over the surface.

When B_1, $P << 1$, equation (**2**) can be rewritten in linear form: -

$$\frac{P}{C_s} = \frac{P}{C_{sm}} + \frac{1}{B_1 C_{sm}} \text{---(3)}$$

So that a plot of $\frac{P}{C_s}$ Vs P will be a straight line when applied o a system that behaves in accordance

with the Langmuir isotherm.

e) <u>Heat Of Adsorption</u>

Since adsorption in variably decreases with rise in temperature. It's to be expected that it should

take place with the evolution of heat.

The differential heat of adsorption (-H) is defined as the heat liberated at constant temperature

when a unit quantity of vapour is adsorbed upon a large quantity of solid already containing

adsorbente, concentration is unchanged.

From Othmer and Sawyer, the slope is $\frac{\partial lnP^*}{\partial lnP} = \frac{(-A)M}{\lambda r Mr}$ from the isotere.

Where,

$\overline{H}$ = Energy per mass of adsorbed vapor.

λr = Latent heat of vaporization of reference substances

M and Mr = Molecular weights of vapor and reference substance respectively

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If $\bar{H}$ is compacted at constant temperature for each isothere, the integral heat of adsorption, at this temperature can be stated below, thus: $\Delta H^1 A = \int_0^x H \partial x$

Where,

$\Delta H^1 A$ = Energy per mass of adsorb ate from solid. In the liquid state adsorbed substance is given as thus: $-\Delta HA = \Delta H^1 A + \lambda X$, all $\Delta H^1 S$ are negative, if heat is evolved during the adsorption.

2.3.4 Adsorption And Its Mechanism

The adsorption treatments of oil is based on the ability of adsorbed, to selectively extract resinous and sulphur containing compounds unsaturated and polycyclic areness and also organic acids residues of sulphuric acid and solvents from oils. (Swoboda P.A.T, 1988)

Natural clay and artificial aluminuosilicates are used as adsorbents in the treatment of oil. Clay that has been activated and dried is used. There are basically two methods, which include contact treatment with a finely comminuted adsorbent and percolation or filtration through a bed of granulated adsorbent. (Labones J.A, 1990)

2.3.5 Adsorption-Desorption.

The individual components of a mixture can be desorbed on some or different solvent, selectivity, consecutively and this will be separated from their mixture during adsorption, (Mantell O.L, 1951) their component is separated in an unchanged state in the form of separate fractions and can be analyze separately. Some terms to note are as follows: -

- Adsorbent: - It's the solid (activated clay) or liquid, which adsorbs.

- Adsorb ate: - It's the solid (liquid example is engine oil), which is adsorbed as atoms.

- Physical adsorption (physiorption): - this is reversible adsorption by weak interaction only no covalent bond is formed between the adsorbent and adsorb ate.

- Chemical adsorption (Chemisorptions): - This involved strong interaction between adsorbent usually accompanied by the rearrangement of atoms within or between adsorb ate.

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2.3.6 Factors Affecting Rate of Adsorption.

The factors affecting the rate at which substances are adsorbed by adsorbent include: -

(Mantell O.L, 1951)

- The time of contract of the substances to be adsorbed with the adsorbent.

- Temperature of substance to be recycled.

- Concentration of adsorbent.

- Nature of the adsorbent.

2.4 History Of Clay.

Clay is a secondary minerals that was formed through the weathering and hydrous alteration of certain parent rocks and their constituent minerals. Common igneous and metamorphic rocks such as granites, gneisses abndschists contain minerals such as feldspars, hornblendes, micas and other complex alumina are necessary precursors to the formation of clay minerals. (Hiller S, 1995)

Long periods of physical weathering breaks down the rocks into finer, grain size and allows an accelerated rate of chemical weathering of mineral ore to decompose. In some cases, the decomposition takes place in-situ and the alteration products may accumulates at the site of the origin or carried away by water and wind.

The clay is formed at the site of the parent's rock called Primary clay even though they are secondary in origin after other minerals. These as a result of transportation and redeposit ion of rocks and minerals. Fragments or transported clay from the original site of the origin is called Secondary clay.

Secondary minerals of clay are common occurrence, which include: - kaolin, illites, chlorites, Momtmorillonitre, Vermiculites and other less common minerals. Usually, secondary clay are also mixed with other minerals such as micas, quartz which are more resistant to weathering.

Sometime, as in the case of upper beds of White mud formation, where the stream and wave separate the clay from unweathered grains, breaks the latter down to finer grain sizes and carry the clay away to be red posited in a distinct beds. Such process is believed to have given rise to the deposit of plastics ceramics clays in Southern Saskatchewan.

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2.4.1 DEFINITION OF CLAY

This is a particular kind of natural earth when subdivided and mixed with a proportion of water, it becomes coherent, sticking and mould able, under pressure shows little or no tendency to adhered to the fingers. (Hiller S, 1995)

The precise definition of clay is as varied as clay users. The perception of which clay is seen, understood and defined by chemist is different from that of a potter as much as it's also different from a metallurgist. The term "Clay" has different connotation depending on the users.
(Velder B, 1995)

Ordinarily, Clay can be said to be the basis raw materials used in the manufacture of bricks, tiles and other ceramics products, the chemist describe clay as a naturally occuring earthy, fine grained material which develops plasticity when mixed with a limited amount of water.

However, clay can be defined as an earthy or strong mineral aggregate consisting essentially of hydrous silences of alumina, plastic when sufficiently pulverized and wetted. (Murray H, 1960)

Table 2.41a

Types Of Clay And Their Composition

ALL IN PERCENTAGE , %

Types of clay	SILICA	ALUMINIUM	POTASSIUM	MAGNESIUM	IRON	SODIUM	CALCIUM
White	48.30	36.10	1.45	0.22	0.09	0.04	---------
Green	---------	-------------	-------------	----------------	-------	----------	------------
Pink	2/3 White + 1/3 Red clay = Pink clay						
Red	63.15	---------------	0.36	0.05	0.54	-----------	0,01
Yellow	48.00	---------------	1.10	0.22	1.84	-----------	0.26

Table 2.41a; above show the types of clay and their composition. (Velder B, 1995)

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The clay minerals occur in nature, in sedimentary rocks of all ages, in continental and marine sediment accumulating at the present time. They are formed as a result of weathering process or hydrothermal activity and are associated with volcanic activity and metallic ore deposits. (Hiller, S, 1995). There are various clay minerals and impurities, which include: - sodium, lithium, potassium and also alkaline earths such as magnesium, calcium, barium that are often present in the molecular structure of clays and have a significant effect in the physical and chemical properties of the clay. (Velder B, 1995)

However, the major clay resources include: - kaolinite, halloysite, illite, chlorite, allophane, vermiculites, momtmorillonitre etc. (Brown G, 1961)

a) Kaolinite

The minerals are formed due to the condensation process of both silica and gibbsite layers. The Silica layer is made up of silicon and oxygen while that of gibbsite layer is made up of aluminum atom and hydrous groups. It's also known as china clay, which's fine white clay. It constituents a structure of $AL_2Si_2O_5(OH)_4$, which is used as an adsorbent. (Brown G, 1961)

b) Momtmorillonite

It's found in Montana as a soft porous rock composed of clayey minerals in various colors. It contains elements such as Al, Si, Mg, Fe and others. It formed a series of minerals that have SiO_2, Al_2O_3, MgO, Fe_2O_3 and H_2O as their principal components. It has a formula of $RMgAl_5Si_{12}O_{30}(OH_6.NH_2O)$ (Brown G, 1961)

c) halloysite

One of the two forms of halloysite has the composition of $(OH)_2SiAl_4O_{10}$ and the other has the composition of $(OH)_2Si_{10}Al_4O_{10}.4H_2O$. The latter form a irreversibly dehydration (losses four water molecules) to the former at a relative low temperature of 60^0C.

d) chlorite

The structure of the minerals consists of alternate mica like and brucitelike layers. The composition of mica layer include $(OH)_4(SiAl)(Mg_2fe)_4O_{10}$ and that of the brucitelike layer include $(Mg_2Al)_6(OH)_{10}$.

Table 2.41b; Properties Of Some Clay Minerals

PROPERTIES	MOMTMORILLONITE	ILLITE	KAOLINITE
SIZE (MM)	0.01-1	0.1-2.0	0.1-5.0
SURFACE AREA (M2/g)	700-800	100-200	5-20
CEC (Meq/100g)	80-100	15-40	3-15

Table 2.41b; above show the properties of some clay minerals. (Ht.tp./www.waterloocal/)

2.4.2 Solubility Of Clay Minerals

The solubility of clay minerals in acid varies with the nature of the acid, the acid concentration, the acid-clay ratio, the temperature and duration of treatment and duration of treatment. In general, the acid attacks the adsorbed the cations and then the composition of the octahedral part of the clay mineral structure. (Velder. B, 1995)

2.4.3 Properties Of Clay Minerals

a) Ion Exchange

Clay minerals are able to adsorb certain positively and negatively charged particle (cations and anions) and retain them around the outside of the structural unit in an exchangeable state.the range of cat ion and anion exchange capacities of the clay mineral is given in the table below: -

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Table 2.4.3

EXCHANGE CAPACITIES OF CLAY MINERALS.			
(Mill equivalents per 100g)			
CATION--- EXCHANGE CAPACITY		ANION- EXCHANGE CAPACITY (APPROXIMATION)	
Kaolinite	3-15	Smectile	23
Halloysite (2H2O)	5-10	Nontronite	20
Halloysite (4H2O)	40-50	Saponite	21
Smectile	80-150	Vermiculites	4
Illite	10-0	Kaolinite	13.3
Verniculites	100-150		
Chlorite	10-0		
Sapiollite	3-15		
Attapulgite			
Palygorskite			

Table 2.4.3b; above show the exchange capacities of clay minerals. (Brown G, 1961)

The ion-exchange properties of the clay minerals are extremely important because the properties determine their physical characteristics and economics use.

b) Clay---Water Relations

Clay materials contain water in several forms. The water may be in held in pores and removed by drying under ambient temperature conditions. The water may be adsorbed on the surface of clay mineral structures and in smectites, verniculites and attapulgites, the water occurs in interlayer position. Finally, the clay mineral structure contains hydroxyls that are lost as water at elevated temperature.

2.4.3 Activation Of Clay

Clays to be used as recycling agents are usually subjected to various physical and chemical treatments to enhance their refining capacity. It gives them certain desired properties with respect to their applications. The process involved in making clay as a recycling agent is called Activation.

2.4.4 Industrial uses of Clay Minerals.

a) Ceramics

Clays composed of kaolinite, which are required for the manufacture of porcelain, white ware and refractories. Absence of iron in clay minerals give it a white burning colour, the absence of alkalis and alkaline earth gives a high fusion temperature. Illites are used in the manufacture of bricks, tiles, stoneware and glazed products. (Brown G, 1961)

b) Oil Industry

Bentonite clays composed of smectiles, which are used primarily in the preparation of muds for drilling oil wells. This type of clay swells to several times its original volume in water, it provides desirable colloidal and wall building properties. Attapulgite clay is used because of their resistance to flocculation under high salinity condition. Certain clays such as kaolinite, smectile and halloysite types are used as catalyst in the manufactured of most gasoline. (Brown G, 1961)

c) Paper

Large tonnages of kaolinite clays are used to fill and coat paper. The coating clays are washed to free them from grit and processed by physical and chemical techniques to improve their whiteness and viscosity. Attapulgite clays are used in the preparation of no carbon required paper because of the colour they develop during reactions with certain colourless organic compound.

d) Other Uses

Clays have tremendous number of uses. For examples, attapulgite and smectile clays used as a carrier for insecticides and pesticides. Smectile clays are used, as plastic zing agents and kaolinite

clay are use as extenders and fillers in large number of organic and inorganic bodies. Smectile and kaolinite clays are used in a variety of Pharmaceutical and medicine preparations.

(Brown G, 1961)

CHAPTER THREE

3.0   **METHODLOGY**

3.1   **List Of Materials And Equipments**

The basic materials and equipments needed for the experiments include: -

- Sample of waste engine oil collected from a vehicle whose engine oil was to be replaced by new ones.
- Clay sample is gotten from unprocessed type to be used for burnt brick production at Shelter Clay Company, Pogo, Niger states.
- H_2SO_4 sample.
- Distilled water.
- Sieving machine (sieve shaker; 250μm, 500μm and receiving pan).
- Furnace.
- Stainless bowl and plate.
- Digital Weighing balance.
- Electric oven.
- Mortar and pestle.
- Wash bottle.
- Pipette.
- Burette (50ml).
- Standard volumetric flask.
- Beaker.
- Thermometer.
- Stainless spoon.
- Container.

3.2   Preparation Of Materials

3.2.1   Calcining Clay Preparation

During the experiment, the clay sample was collected from the site of chanchanga Shelter clay product, Pogo in Paikoro local government area of Niger State. The collected clay sample was broken down into loose sizes using a mortar with a pestle. The required mesh size, which was 250 μm, was gotten with the aid of vibrating shaker. The required mesh size was then heated in a furnace at the temperature of 550^0C for the duration of 6 hours and allows it to cool. This process is called calcinations.

3.2.2   Acidified Clay Preparation

0.3% of concentration H_2SO_4 solution (Hydrogen Tetraoxosulphate (vi) acid) was measured using a pipette. 100ml of distilled water was also measured respectively.

During the experiment, the required solution of the acid was prepared and 3ml was pipette into the Standard volumetric flask containing little amount of the distilled water. The distilled water was then conveyed into the Standard volumetric flask by a wash bottle to the requirement measurement of 1000ml. The prepared solution of 0.3% concentration H_2SO_4 was shake vigorously so as to obtain a uniform dissolution of the concentrated H_2SO_4 in the distilled water. The prepared solution was then mixed with the calcined clay; there was slight change in the colour. The acidified clay was oven dry at a temperature of 40^0C and then it was homogenized. This process is called the activation process.

3.2.3   Refining Method

The method employed in this process is called the Contact method of adsorptions. The procedures involved in refining method are as follows: -

I.   50g of the acidified clay was measured and mixed with 400ml of the used engine oil, heated to a temperature of $50-55^0C$.

II.   The mixture was stirred with electric stirrer for 20minutes and allowed to settle down.

III.   The mixture was then filter with a cloth sieve. The refined engine oil is then the filter ate.

IV.   The process was repeated for 100g, 150g, 200g and 250g respectively and the 400ml of used engine oil was kept constant.

V.   Analysis was taken to compare the filtrates with the initial used engine oil.

3.2.4   Analysis Of Samples

The analysis were carried out at Oando Lube blending plant laboratory in kaduna. The following tests were carried out, such as the appearance, kinematics viscosity at 40^0C and 100^0C, viscosity index, specific gravity, flash point, total base number (TBN)

a)   Test For Kinematics Viscosity

Apparatus: - ASTM Capillary viscometer, suction pump with tube, viscometer holder,

Constant temperature bath.

Methods: - i. The viscometer was inverted and suction was apply to the larger end of

the viscometer tube while the thinner end was immersed into the sample to

be analyzed . The sample required was drawn.

ii. The viscometer was then turned to normal position and then fixed in the

Viscometer holder.

iii. The viscometer assemblywasthen mounted into the constant temperature both

depending on what temperature to be used (40^0C-100^0C), the Viscometer was

mounted in a vertical position.

Iv The charge was allowed to remain in the bath for 30 minutes.

v. The suction was then used on the thinner end of the tube to adjust the head

Level of test sample to about 5mm above the first timing. With the sample

Flowing freeing, the time required for the meniscus to pass from the first

timing to the second was taken and recorded.

III.   Viscosity was calculated by the formula stated below: -

$$V = C \times T \text{---------------------------- (!)}$$

Where,

V= Kinematics viscosity.

T= time in seconds

C= calibration constant of the viscometer.

b) Test For Viscosity Index

Apparatus : -   Log table of viscosity at 40°C against 100^{0}C.

Method :

I.   When the viscosity at 400C and 1000C was used on the log table

to determined the valve of viscosity index.

II.   The valve of viscosity was traced at a point on the graph when the viscosity

At 100^{0}C and 40^{0}C meets.

III.   See the appendix for the log table.

c) Specific Gravity

Apparatus :-   hydrometer, Log table (hydrometer and temperature ^{0}C), hydrometer cylinder.

Method: -

I.   The sample was transferred into a clean hydrometer cylinder.

II.   The hydrometer was depressed into the sample in the cylinder and then

released.

III.   Sufficient time were allowed for the hydrometer to come to rest,

Floating freely away from the walls of the cylinder.

IV   The hydrometer was read and recorded.

V.   After the sample in the cylinder was stirred with a thermometer until

a Steady reading was obtained. This steady sample temperature was

recorded.

d) Test For Flash Point

Apparatus :-   Cleveland open, cup tester (COC), heating plate (Bunsen burner), test flame

Applicator, thermometer, holder, fume cupboard.

(Procedure was carried out in the fume cupboard)

Method :-

I.   The cup was filled so that the top of the meniscus is exactly at filling line.

II.   The test flame light was adjusted in diameter to adjusted in diameter to about

3.2-4.8mm.

III.   The heat was applied on the heating plate.

IV.   As the flashed point was observed, the temperature on the thermometer was

then taken and recorded. The thermometer's readings indicate the flash
point.

e) <u>Test For Total Base Number (TBN)</u>

<u>Reagents</u>: - Chlorobenzene, glacial acetic acid, perchloric acid ($HCLO_4$).

<u>Apparatus: -</u> Potentograph.

<u>Methods</u>: -   I. 120ml of titration solvent of ratio 2:1 by volume of chlorobenzene and glacial
Acetic acid was weighed into the sample.

II. The beaker was placed on the titration stand and stirred until the solution
dissolved.

III. The burette was filled with the titration (perchloric acid), dilution ($HCLO_4$).

IV. Then the titration is determined by the potentograph.

(See the graph on the appendix)

CHAPTER FOUR

4.0 RESULTS

0.3% ACIDIFICATION

Table 4.0; SHOWING THE ANALYSIS OF REFINED ACTIVATED CLAY.

TEST	50g CLAY	100g CLAY	150g CLAY	200g CLAY	250g CLAY	USED ENGINE OIL
APPEARANCE	DARK	DARK	DARK	DARK	DARK	DARK
SPECIFIC GRAVITY AT 15^{0}C	0.8967	0.8967	0.8969	0.8972	0.8998	0.8962
KINEMATIC VISCOSITY AT 40^{0}C	155.99	155.95	154.74	153.80	153.22	156.94
KINEMATIC VISCOSITY AT 100^{0}C	16.74	16.71	16.70	16.68	16.61	17.04
VISCOSITY INDEX	116	116	120	115	115	116
TOTAL BASE NUMBER [TBN]	0	0	0	0	0	0
FLASH POINT	126^{0}C	126^{0}C	128^{0}C	130^{0}C	136^{0}C	120^{0}C

Table 4.0: showing analysis of the used engine oil and the refined used engine oil using the Activated clay as adsorbent.

From the result shown above, the following can be said about the properties of used engine oil: -

i.   There is no Total Base number because there are no additives present. The additives have been used up.

ii. The flash point is lowered by the impurities in the oil.

iii. The colour of the used engine oil is dark because of the carbon from wear on engine.

CHAPTER FIVE

5.0   DISCUSSION OF RESULT

From the result of analysis shown in the table 4.0, it was observed that the activated clay contribute to improved the characteristics of the spent lube when compared with the untreated lube oil.

Viscosity is a measure of flow ability of oil. From the table, it can be observed that the Kinematic viscosity decrease with increase in activated clay. The kinematics viscosity at $40^{o}C$ shows that the values decrease from $155.99 m^2 s^{-1}$-$153.22 m^2 s^{-1}$ as the quantity increases from 50g-250g and at $100^{o}C$ the values decreases from 16.74-$16.61 m^2 s^{-1}$. Since viscosity increases with increase in impurities present, it indicates that the adsorption of certain impurities of activated clay had taken place. This leads to the improvement in the quality of the spent engine oil compare with the value of $156.94 m^2 s^{-1}$ used engine oil before refining.

Viscosity index is an empirical number indicating the rate of change in viscosity of oil within a given temperature. Higher number indicates a low change; lower number indicates a relatively large change. The higher the number, the better the oil. From the table, it was observed that the viscosity Index is higher at 150g of activated clay, which are 120.

Flash point is the temperature at which oil give off vapors that can be ignited with flame held over the oil. The lower the flash points, the greater tendency for oil to suffer vapourization. from the table, it is observed the flash point increases with increasing quantity of activated clay used. The flash point is

higher at 250g. Since the impurities reduces he flash point, when compared with the used engine oil at 120°C, it can be deduced that the activated clay had adsorbed more of the impurities.

Total base number (TBN) is a measure of amount of basic substance in oil because lubricants products contain basic constituent present as additives. From the table it's observed that the additives had been used up.

Specific gravity is the ratio of mass of a given volume to the mass of an equal volume of water. From the table, it was observed that it increases from 100g-250g of the activated clay at the valves of 0.8967-0.8978. When compared with the used engine oil, it seen to follow the same tend.

CHAPTER SIX

6.0   CONCLUSIONS AND RECOMMENDATION

6.1   Conclusion

The work was carried out successfully and the following investigations were made. From the analysis carried out on the initial used engine oil and the refined used oil using the local adsorbent, it was shows that the oil has improve slightly in its quality.

The flash point for the used engine oil was 120°C and it increase to 136°C when refined with the activated clay, the kinematics viscosity at 40°C and 100°C for used engine oil was 156.94 –17.04 m^2s^{-1} and decreases to 153.22-16.61 m^2s^{-1} refined oil. This shows that adsorption had taken place and the quality of oil had been improved.

6.2 Recommendation

i.   It should be noted that effective filtrations methods should be provided to improve the quality of the oil by removing impurities present in the oil. The filter paper or the cloth piece cannot do it effectively. So the Vacuum filtration is recommended for filtration.

ii.   Additives should be used to test the quality of lube further. Different types of acids such as HCL, HNO_3 etc should used be in activating clay to know which will give us the best activation.

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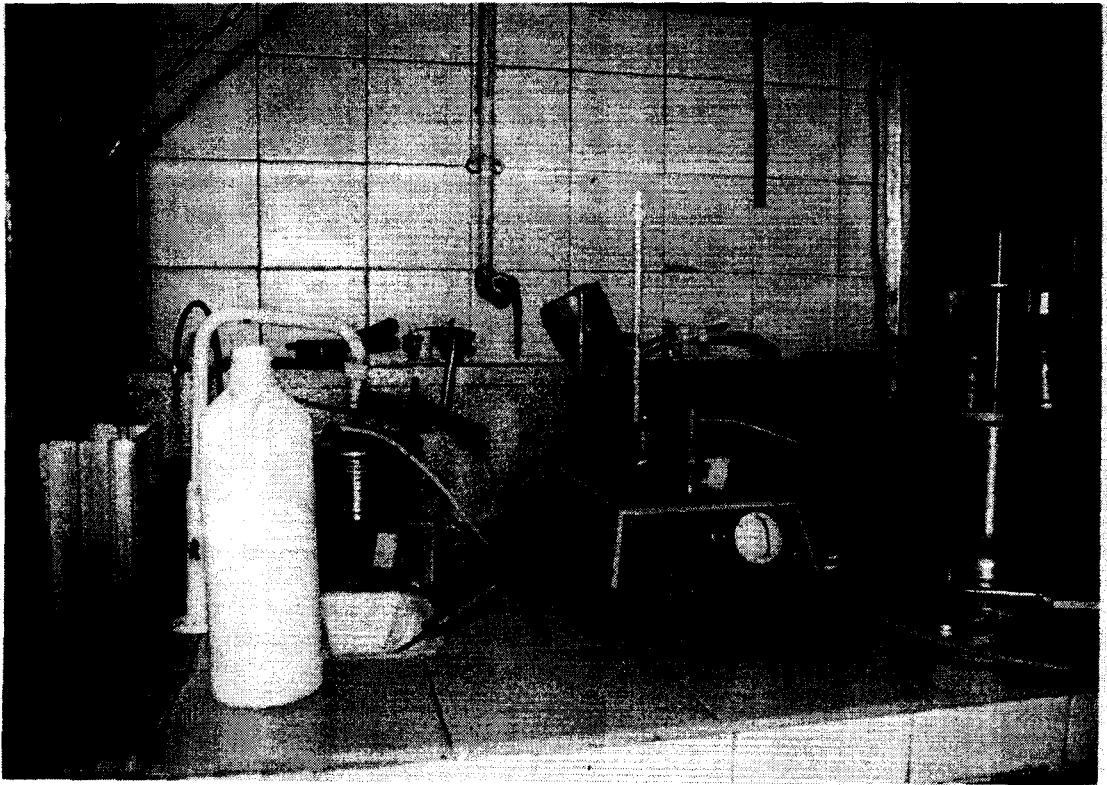
CONVERSIONE DELLE DENSITA' A 15 °C.

Densità alla temperatura osservata

Densità corrispondente a 15 °C.