

REFINING OF SPENTLUBE OIL USING LOCAL ADSORBENT

BY

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SUBMITTED TO

THE DEPARTMENT OF CHEMICAL ENGINEERING FEDERAL

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**DEPARTMENT OF CHEMICAL ENGINEERING
SCHOOL OF ENGINEERING AND ENGINEERING TECHNOLOGY,
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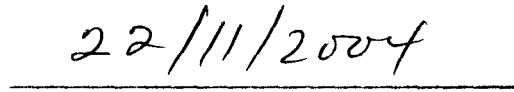
DECLARATION

Illah Martins Enedunyi declare that this project presented for the award of Bachelor of Engineering in Chemical Engineering Department has not been presented either wholly or partially for any other degree elsewhere.



STUDENT

;



DATE

CERTIFICATION

This is certify that the project “Refining of Spent Lube Oil using Local Adsorbent” is the work of Illah Martins Enedunyi.

Thus it has been duly supervised and approved for submission to the Department of Chemical Engineering School of Engineering and Engineering Technology, Federal university of Technology, Minna

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Engr. M.A. Olutoye
(SUPERVISOR)

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DATE

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DR F.ABERUAGBA
HEAD OF DEPARTMENT

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DATE

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EXTERNAL SUPERVISOR

.....

DATE

DEDICATION

This work is dedicated to my parents, Mr. Raymond Illah and Mrs Daria Raymond Illah my beloved Brothers and Sisters. (Iyoma, Charles, Vincent, Boniface, Catherine, Beatrice, Barnabas, Philip, Omaye, Anna). They believed in me in my difficult moments. Above all to almighty God for his mercy and love.

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My greatest appreciation is to almighty God who has given me the grace by keeping me alive and the enablement to successfully completion of this work.

My profound gratitude then goes to my Supervisor Engr Moses Olutoye who has been a father and never grew wearing on the work but took time to encourage me, supervise and review the work we have today. My special appreciation also goes to Mr Emmanuel, Oando lube blending plant, Kaduna. My H.O.D Dr F. Aberuagba, Mr Alhassan and entire Staff of Chemical Engineering, may god bless you all (Amen).

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Lastly, to the entirely final year Students of Chemical Engineering Department, I thank you all.

ABSTRACT

Refining of spent lube (used Automobile engine oil) has been undertaken using Acid treated Calcined Clay. The Clay was obtained from Chanchaga Shelter Clay. Minna. The same quantity of the used oil was refined with different quantity of the Clay sample, i.e. at 50g, 100g, 150g, 200g and 250g. The following parameters, such as viscosity at 40°C and at 100°C, Flash point, Viscosity index, Specific gravity and Appearance were analyzed. Better results were obtained from refined used lube oil with 250g of acid treated Calcined Clay when compared with similar parameters result obtained from the untreated used engine oil sample. The Flash point increased, from 120°C of the spent lube to 126°C of the refined oil at 250g Clay, Viscosity decreased from 17.07 to 16.70 at 100°C and 156.94 to 153.04 at 40°C, Viscosity index increases from 116 to 118. The appearance has no significance change. Total Base Number is zero respectively. This result for the refined lube oil were compared with the fresh engine oil standard values with similar parameters, 245°C-285 °C for the flash point, 5.0 – 8.0 mgKOH/g for the TBN, 0.880 – 0.895 for the specific gravity 250-280cst and 19-21cst for 40 °C and 100 °C respectively. This values obtained from refined oil are closer to the standard values but when compared with untreated values of the spent lube oil showed slight improvement

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CHAPTER ONE

1.0 INTRODUCTION

A lubricant can be defined as an oil product that separates the metal parts of an engine to reduce friction and wearing.

Spent lube refers to lubricants that have through their intended use cycle and must be disposed or treated for use. Spent lube constitutes one of major pollutants today.

While automobile engine is running, the motor oil collects heavy metals (lead, calcium, Zinc and barium), Iron steel particles and copper. Several of these contaminants are toxic and harmful to the environment.

The major characteristic of spent lube is the oil degradation and oxidation. Oil degradation implies the physical and chemical change during the period of services by the exposure to heat, oxygen and formation of acids and catalytic metals. While oxidation due to high temperature and extended services will tend to darken the colour of the oil, increase its viscosity and make it taken can odour and insoluble refine be formed and become deposits.

An adsorbent is a natural or synthetic material or amorphous or microcrystalline structure whose internal pore surfaces are accessible for selective combination of solid and solute.

An adsorbent will be developed from locally available materials (clay). This clay is sourced from Shelter Clay Product Chanchaga.

Two major steps will be considered, these are calcinations and activation. Calcinations involve heating of clay at a higher temperature of 55⁰c for five or six hours and activation process involves acid treatment of the calcined clay with H₂SO₄, as activating agent. These will increase the absorptive sites and porosity.

The method adopted here in the refining is the contact method. This involves mixing the acid treated calcined clay with the spent lube.

The economic benefit of this research work is that:

- I. The waste lube may be converted to a useful product
- ii. Since it constitute the major pollutants to the environment, it may be converted to a less harmful substance.
- iii. The materials are locally sourced. It is hoped that this work will provide a source of useful knowledge to the reader.

1.1 OBJECTIVE

The objective is to refine spent lube from locally sourced material, which will serve as adsorbent.

1.2 SCOPE OF WORK

The scope of this work covers the characteristics of spent lube and its analysis after it has been refined by the developed adsorbent. This will be done by comparing their physical and chemical characteristics before and after refining. And also find out if it can be upgraded for use.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.10 LUBRICANT

Lubricant is a gas, liquid or solid used to prevent contacts of parts in relative motion. And as result reduces friction and wear.

2.11 HISTORICAL BACKGROUND

From the early history in Egypt, Greece, Rome and Persia where grease made of a

Combination of animals fats and calcium where used as lubricants on chariots wheels.

In the nineteen century vegetable oils from olives, grape seeds, castor seeds and animal oil from fallow land and were mixed with potassium sodium and calcium soaps to make grease use for lubricating of steams cylinders, cutting metals and during the first world war (1914 – 1916) are: used in an aircraft engine, further researches in the inherent properties of the base stocks obtained from petroleum refining revealed the inadequacies and limitations in modern engine lubrication. Hence the introduction of chemical additive in the early 20th century to improve their performance.

The additives are chemical compound added to the refined base stock to improve some specific properties of the lubricating oil either by enhancing the inherent properties. The understanding of lubricant has helped to reduce considerably, the problem posed by loss of energy due to friction developed in relative motion. Motor oil are intended for lubricating the motor engine against friction and wear.

SOURCES

2.21 MINERAL BASE OILS

Modern mineral base oils are prepared from naturally occurring crude petroleum oil by initial process of vacuum distillation. The chemical composition of mineral oils produced depends both on the original crude and on the refining processes. The crude oils are usually defined as Naphthenic or paraffinic depending on the hydrocarbon type present (1).

FUNCTIONS OF LUBRICANT

LUBRICATE AND PREVENT WEAR

Once an engine is started the oil must concentrate promptly and lubricate moving surfaces to prevent the metal-to-metal contact that would result in scoring, scoring or seizure engine part. Oil films on bearing and cylinder walls are sensitive to movement pressure and oil supply. This film must be continually replenished by adequate flow and proper oil distribution. This is called full film lubrication. (2)

3.2 REDUCING FRICTION

Under full film lubrication conditions, thick films of oil prevent metal-to-metal contact between engine part. Relative movements of this lubricant part require enough force to overcome fluid friction of the lubricant. The viscosity should be high enough to maintain an unbroken film but must not be higher than necessary in order to permit easy starting. (2)

AGAINST RUST AND CORROSION

2.33 PF

From carbon dioxide and water under ideal condition. Gasoline for most of the time does not burn all the fuel completely. As a result of incomplete combustion and Divided fuel, there is a formation of soot and Carbon. The soot result

and the Carbon escape through the rings and into the Crank case and combine with to form sludge and deposits on critical engine parts, other Corrosive Combustion gases also get pass the rings and are condensed or dissolve into the Crankcase oil. The water present attacks the metal parts to form rust (2)

2.34 KEEPING ENGINE PARTS CLEAN AND ALSO COOLS.

This is available with the use of Chemical additives (detergent and dispersant) blended into the modern lubricant for necessary cooling.

2.40 TYPES OF ADDITIVES.

The basic functions of additives can be summarized as protection of metal surfaces (rings, bearing, gears e.t.c.) extending range of lubricant applicability and extending lubricant life. There are different types of additives used in blending the various classes and grades of lubricating oils to enable such finished product meets the performance requirement for the engine. (3)

2.41 CORROSION INHIBITORS.

These are Zinc dithiophosphate either Sulphur and Phosphorus containing materials, they are highly alkaline materials. They neutralize corrosive acids that develop in the oil and those induced by contaminants in the oils by chemically bonded on the engine parts to form a protective film to reduce corrosion and wear.

2.42 RUST INHIBITORS. (3)

These are compounds with higher polar attraction towards metal surfaces such as alkylated phenoxyl, alkaline oxides, alkenyl succinic acid, alkaline earth sulphonateds and other derivatives of fatty and organic acids that help to prevent water in oil from attacking metal by forming continuous films on such surfaces.

2.43 ANTI-WEAR AGENTS. (3)

These are organic phosphate ester such as alkaline detergent, tri-aryl phosphate, zinc dithiophosphates and dithiocarbonates that helps to reduce friction, wear during friction. In some application, at higher temperatures and under heavy loads, sulphurized olefins, chlorinated waxes, phosphorous and Sulphur compound are used as extreme pressure (EP) anti-wear additives by forming film of metallic salts, which acts as a solid lubricants to prevent sever surface damage.

2.44 DETERGENT AND DISPERSANTS. (3)

These are metallic organic compounds of Sodium, Calcium and Magnesium phosphates, Phosphates and Sulphonates. These are soluble metallic Compounds of higher molecular weight organic acid that have the affinity to dissolve the Carbonaceous deposit in the ring grooves, piston surfaces and single deposit in internal Combustion engine.

2.45 POUR POINT DEPERSANT.

There are polymers of alky-aromatic and polymethacrylate that enable oils to flow easily without any agitation; they are used to prevent the growth of large wax crystals into the inter-locked structures as the oils cool down. (3)

2.46 VISCOSITY INDEX IMPROVERS.

These are large chain and high molecular weight polymers such as polymethacrylates, polysolutyfenes, olefins copolymers, ethylene propylene copolymers and acrylate polymers that prevent the decrease in viscosity of oils at high temperature.

The viscosity temperature characteristics of oils show that viscosity index (VI no.) shows a small decrease in viscosity with increasing temperature. (3)

2.47 OXIDATION INHIBITORS.

2.51 These are Chemical property modifiers called anti-oxidants. E.g. Zinc dithiophosphates, hindered phenols, aromatic amines, sulphurized esters. These prevent oxidation process at high temperatures of air (3)

2.50 OIL CLASSIFICATION.

Oil viscosity and the lubricant service requirement for a particular vehicle must be considered before selecting.

Two bodies namely, Society of Engineers (SAE) and American Petroleum Institute (API) has classified motor oil accordingly based on the viscosity and quality (service requirement) respectively.

2.51 SAE CRANKCASE OIL VISCOSITY CLASSIFICATION SYSTEM.

The Society of Automobile Engineers (SAE) developed a classification system based on Viscosity. This system, established distinct motor oil classification of grade. These include among others SAE5W, SAE10W, SAE15W, SAE30, SAE40 and SAE50. W stands for winter and indicates that oil is suitable for use in cold temperature region. Those classifications as SAE30, SAE40 and SAE50 do not include W and defined grades for use at higher temperatures.

2.52 MOTOR OIL COMPOSITION.

The motor oil is composed of mainly the base oils and the Chemical additives blended together in pre-determined proportions and under specific conditions. The types of base stocks and choice.

Base oils are highly viscous index distillate of crude oil existing in five grades: 100 Neutral; 150N; 250N, 500N and the bright stocks (BS). (3)

2.60 DEFINITION OF LUBRICANT CHARACTERISTICS.

VISCOSITY: Viscosity is the resistance to flow or thickness of a liquid, viscosity of oils decrease with increase in temperature, hence the temperature at which viscosity is measured must always be specified. The Kinematics L^2/T , where L is the length and T is time. The cgs unit is one centimeter squared per second (one Stokes), while the SI unit is one squared meter per second and is equivalent to 10^4 (3)

VISCOSITY INDEX: Is a number on a Conventional scale used to characterize the temperature. A high viscosity index indicates a relatively small change of viscosity with temperature and vice-versa.

POUR POINT: Lowest temperature at which a liquid petroleum product will flow when it is cooled under the condition of the standard test method. (3)

NEUTRALIZATION NUMBER: An indication of the acidity or alkalinity of an oil, the number is the weight in milligrams of the amount of acid or base expressed as potassium hydroxide (KOH) equivalent required to neutralize 1 gram of oil in accordance with an ASTM test method. (3)

TOTAL BASE NUMBER (TBN): The quality of acid expresses in terms of the equivalent number of milligrams of KOH that require to neutralize all basic constituents present in 1 g of sample. (3)

TOTAL ACID NUMBER: The quality of base expressed in milligrams of KOH that is required to neutralize all acidic constituents present in 1g sample (3)

FLASH POINT: The lowest temperature under closely specified conditions at which a combustible will give off sufficient vapour to form a flammable mixture with air in a standardized vessel. Flash point tests are used to access the volatilities of petroleum products. (3)

FIRE POINT: This is the lowest temperature at which ignited sample under flash point test sustained burning for a period of 5 seconds. (3)

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2.60 BASE OIL

These are high viscosity index distillate of crude oil existing in five grades. 100 Neutral 150N, 250N, 500N and the bright stock Bs) the table blow shows the physical properties of the base oils (3)

Characteristic	100N	150N	250N	500N	BS
Kinetic viscosity (CST)	39.43	4.3-5.5	6.9-7.7	108-12.0	305-325
Viscosity index	95	95	95	95	95
Flash pat (°c)	193	204	210	221	226
Fire pat (°c)	198	210	217	236	234
Pour point (°c)	-9	-9	-9	-9	-9
Appearance	Clear & bright	"	"	"	"
Water content	Nil	Nil	Nil	Nil	Nil

2.70 USED ENGINE OIL

This refers to lubricating oil that have been through their intended cycle and must be either disposed or treated for reused.

2.71 CHARACTERISTICS OF SPENT OIL

(i) Oil Degradation

Oil in services is chemically and physically contaminated depending on the length of service and the condition imposed on it by its application. Although they are linked together in many cases and one factor may influence another factor. I will try to separate them for case of understanding.

A mineral oil lubricant in services will chemically change during that period of services by its exposure to heat oxygen. The formation of acids and the catalytic metals it comes in contact with in the application. (15)

(ii) Oxidation

The most important type of chemical reaction affecting oils in services is oxidation, which very often determines the services life. The effect of oxidation is to introduce oxygen atoms into the base oil molecules, converting the hydrocarbon molecules into aldehydes and then into acids. Because these products are less stable than the original hydrocarbons molecules, they tend to be further attacked and the final products may be highly oxidized gums and lacquers, or even solid residues such as carbonized materials. Oxidation due to high temperature and extended services will tend to darken the colour of oil, increase its viscosity and make it take an odour. Insoluble resins will ultimately be formed and becomes deposit forming in the application.

The rate of oxidation depends on the oxidation resistance of the oil and on the operating temperature. Certain metals present such as Iron, steel, aluminium, also catalyze oxidation or copper surface are been worn.

The presence of anti oxidants, natural or additives, will reduce the rate of oxidation but when used up, rate of oxidation will increase rapidly. (15)

(iii) Contamination

Contamination from the application contributes majorly to oil degradation both physically and chemically. Build up dirt in a system will cause accelerated wear. Wear metals can act as a catalyst in the oxidation process as can the acid formed by the oxidation of the products of oxidation will act as a catalyst for further oxidation. (15)

(iv) Evaporation

Lubricants are not generally volatile and it is only with lower viscosity oils at higher temperature or in vacuum application that evaporation is likely to be significant. Its effects is to produce an increase in viscosity. (15)

(v) Polynuclear Aromatics

Polynuclear aromatic hydrocarbons are present in the petroleum base stocks and can be produced during the use of the oil. Several of these compounds, primarily 4,5 and 6 rings structures are known carcinogens and mutagens. Benzo (a) pyrene B (a) P is a purine example of a PNA, which exhibits carcinogenic effects. (15)

2.72 RECYCLING AND ITS MECHANISM

Recycling is the process of regenerating used materials substances so that it can be used again. 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 its initial state. Engine oil is used up when there are no function of additives again. The used oil is dark in colour due to carbon from wear. The acid present maintains the colour of the engine oil, but in used oil, the acid is weakened by the neutralization by a base.

The choice of method of regenerating base oils is determined by the nature of the constituents. Some oil requires only purification from mechanical impurities while others require deep purification with the use of chemical and physiochemical methods (4).

The physical methods of regeneration include settling, centrifugation, filtration, distillation, and snubbing water to remove water-soluble acids.

Of the physiochemical methods adsorption treatment is employed as an independent method or sulphuric acid treatment (5). Recycling will only bring back

the base oil without additives. Additives are incorporated into the recycled engine oil and sometimes mixed with fresh oil.

2.80 Clay

Clay may be defined as a natural, earthly fine-grained material that develops

plasticity when mixed with a limited amount of water. The term is also used to designate the smallest particles in sedimentary rocks and soils. The maximum size of particles in the clay size grade is commonly considered to be two microns (0.002 millimeter). Clays are composed essentially of silica, alumina, water and appreciable quantities of iron, alkalis and alkaline earths are frequently present.

Clay has wide variety of physical characteristics such as plasticity, refractory, colour and colloidal properties that make them suitable for a wide variety of industrial purposes (6).

2.81 TYPES OF CLAYS

1. KAOLINITE MINERALS:

These are of composition $Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$ and are the most prevalent minerals group in natural clays although hydrous micas or illite is also common. The minerals, Kaolinite is not of frequent occurrence and may be restricted to those clays, which have been formed under hypogenic action i.e. sub-surface reaction at high temperatures and pressures, and to the direct sediments of those clays (6).

2. MONTMORILLONITE MINERALS:

These are more common than kaolins but numerous examples are known. They are of variable chemical composition, containing aluminium, silicon,

magnesium, Iron and other elements. The general name given to clays, which contains appreciable amount of montmorillonite minerals, is bentonite (6)

3. ALTAPULGITE:

The mineral is not of common occurrence but in a pure state, it is particularly valuable in for drilling fluids and as a filler. It is essentially a hydrated magnesium silicate, but other elements may be present. (6)

4. CHLORITES:

These are group of hydrous alumino-silicates with Iron and magnesium as essential ingredients. They occur as greenish flakes resembling micas. They have a hardness of 1.5 and a specific gravity of 2.5 - 2.8. Chlorites are the most usual alteration products in recent clays derived from minerals rich in Iron and magnesium and they are common constituents in many sedimentary clays. (6)

5. VERMICULITES

They are hydrated magnesium silicates but other elements, chiefly iron are frequently associated. They owe their name to their property of exfoliating on heating. They occur as plate-like flakes, are softer than micas and are usually red, brown or black. (6)

2.82 PROPERTIES OF CLAY

1. Clay water relationship

Clay materials contain water in several forms; the water may be held in pores and may be removed by drying under ambient conditions. Water may be adsorbed on the surface of the clay minerals structure in attapulgites, the water may occur in interlayer positions or within the structural channels. Clay mineral structures contain hydroxyls that are lost as water at elevated temperature. (6)

2. High Temperature Reactions

When hydroxyls are lost by heating, the lattice structure may be destroyed or simply modified depending on the composition and structure of the clay minerals in presence of flux such as iron or potassium fusion may follow dehydration very quickly. In the absence of such component particularly for aluminous dioctahedral minerals, a succession of new phases may form at increasing temperature, prior to fusion. (6)

(3) Size and shape

The size and shape of the clay minerals have been determined by electron micrographs, allophone, the amorphous clay mineral, show no definite morphology. Electron microscope shows that attapulgite occurs in single and bundles of elongated laths. The individual lath is many microns in length and 50-100Å° in width. (6)

(4) Solubility of Clay minerals.

Solubility of the clay minerals in acids varies with the nature of the acids, the acid concentration, the acid to clay ratio, the temperature and the duration of treatment. It also varies as a function of heating. The clay minerals and the firing temperature prior to the acid attack. The acid first attacks the absorbed cations and then the component of the octahedral part of the clay mineral structure. The silica sheets are not attacked and morphology of the clay minerals may not be retained after solutions of all components except silica. (6)

(5) Iron Exchange

Clay minerals are able to absorb certain positively and negatively charged particles (cations and anions) and retain them around the outside of the structural unit in an exchangeable state.

The exchange differs from simple sorption because it is stoichiometric (involving equal amounts). It does not generally affect the silica alumina structure. The

exchange capacity varies with particle sizes, perfection of crystallinity and the nature of the absorbed ion. Cation exchange capacity results from broken bonds around the edges of the structural units, which rise to unsatisfied charges.

Anions exchange capacity may be due to replacement of hydroxyl ions at the edges of the lattice structures and adsorption because of the geometry of the anion in relation to the geometry of the clay-minerals structural units. (6)

2.83 ACTIVATION OF CLAY

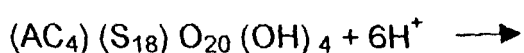
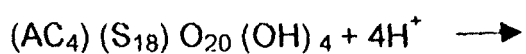
Clays to be used as recycling agents are usually subjected to various physical and chemical treatments to enhance their refining capacity. It gives them certain desired properties with respect to their application. The process involved in making clay as a recycling agent is called Activation.

2.84 ACID ACTIVATION

In acid activation, exchangeable ions like potassium, sodium and calcium are replaced by hydrogen ions in the inter-molecular space. Apart of aluminium, iron and magnesium ions are leached out from the lattice structure, thereby rendering the clay physically more porous and electrically more active. (7)

Sulphuric acid or hydrochloric acid is commonly used as the activating agent. The base components of the montmorillonite are attacked, magnesium, aluminium, and ions proceed from the octahedral position to exchange site and then go into solution. The removal of aluminium and other ions is not affected though unbroken silica-oxygen layers sandwich both sides of the basic lattices constituent. The acid penetration, this into the interior of the structures from the edges leaving a framework processing a large area (5).

The change taking place on an idealized montmorillonite is expressed as (8).



Half of the aluminium atoms are removed from the structure together with 1000 hydroxyl groups the remaining aluminium atoms tetrahedrally coordinated with the remaining oxygen atoms. The change from octahedral to tetrahedral coordination leaves the crystal lattice with negative charge balanced by a hydrogen ion (13)

This is thus, the source of the acidity, which is considered to be related to activity for refining oil.

2.85 CALCINATION

Calcination is a heat treatment given to materials. It involves heating the material to temperatures high enough to drive away water and burn off any organic components in the material in the presence of air

The production of new physical and chemical states when clays are heated, often results in materials with very different chemical reactivity from that of the different temperatures, the reactivity of the product when it is cooled to room temperature shows many unusual characteristics. Investigation has been done on the extent of which kaolinite and other clays can be leached by acids following calcinations. After treatment at various temperatures, decompositions reactions took place which liberated alumina from its normal fixed lattice position enable it to be taken up freely into acid solutions. (9)

2.86 THEORY OF ADSORPTIVE REFINING

The refining process of using earthen material (clay) is based on the operation of absorption. Absorption is a process in which a substance or substances become attached to the surface of a solid. The substances absorbed on the solid surface may be gases, liquid or solids. The amount of material absorbed by a given mass of adsorbent depends on the physical condition of the materials to be adsorbed. Most material is adsorbed when the solid is porous or

finely divided which is in accordance with the theory that adsorption is a surface phenomenon. (11)

Adsorption is the selective collection and concentration into solid surface of particular types of molecules contained in a liquid or gas. When the solid fluid contacting operation of adsorption is used to treat a fluid stream on an industrial scale, one of the following characteristics has usually been responsible for its selection as the most economic method. High selectivity of the adsorbent related to the selectivity, chemical instability of the adsorbate (solute) restricting it to temperature unsuited for other separations e.tc. (16)

Adsorption from the liquid phase has long been used for removal of contaminants present at low concentrations in process stream. In some instances the objective is removal of specific components while in others, the contaminants are not well defined and the objective is the improvement of general properties such as colour, taste, odour and storage stability. (13)

Adsorbent fall into two general classes. The polar and non polar, the polar or hydrophilic types are employed when the materials to be improved are more polar or hydrophobic type are employed to remove ten polar back stream. (13)

2.87 ADSORPTION AND ITS MECHANISMS

The adsorption treatment of oil is based on the ability of absorber to selectively extract resinous and Sulphur containing compounds and organic acid residues from oils. (10)

Natural clay (bleaching earth) and artificial aluminosilicate are used as adsorbents in the treatment of oils. There are basically two treatment methods: contact treatment method and percolation of filtration through a bed of granulated adsorbent. (15)

1) CONTACT TREATMENT

For this treatment the oils are mixed with finely comminuted bleaching earth. Very finely ground clay is used because the activity of the adsorbent depends on the degree of its comminution. (10)

The oil is heated to lower the viscosity of the oil and improve its penetration into adsorbent pores. The process temperature depends on the quality of the stock, the nature of adsorbent and the required degree of product purification

Most often, light distillate oils are subjected to contacting at 80°C - 120°C (5)

The efficiency of the process is affected by the duration of contact of oil with clay.

This time generally is 20-25 minutes.

(2) PERCOLATION METHOD

Filtration is performed through a layer of granulated adsorbent when the latter is stationary. The oil is filtered through a fixed bed of clay (10).

The filter is a vertical allow cylindrical apparatus packed with the adsorbents, which is the clay, the first portion of oil, are purified. As the adsorbent becomes saturated with the substances extracted from the oil the extent of purification drops.

2.88 KINETICS OF ADSORPTION

Physical adsorption surface is extremely rapid and the kinetic are invariably controlled by mass or heat transfer rather than by intrinsic rate surface process.

(11)

2.89 FORCES OF ADSORPTION

Most adsorption, separation bleaching processes depends on physiosorption rather than chemisorption. The force of physical adsorption consist of normal dispersion-repulsion force (Vander Waal's forces) which are fundamental

properties of all matters and electrostatic forces (polarization field, gradient dipole field gradient e.t.c.) which are significant polar adsorbents such as activated clay is essentially non-polar although oxidation may impart some polarity to the surface (12)

2.90 FACTORS FOR RATE ADSORPTION

The factors that affect the rate at which substances is adsorbed by adsorbent include:

- (1) Time of contact of the substances to be adsorbed with the adsorbent.
- (2) Temperature of substances to be recycled.
- (3) Concentration of adsorbent
- (4) Nature of the adsorbent.

2.91 ADSORPTION ISOTHERMS

In 1915, Langmuir proposed a theory based on the believe that the adsorbed layer was unimolecular. langmuir assumes elementary spaces or point of residual valency exist on the surface of a crystal. The adsorptive forces are concentrated at these points and adsorption consists in the fixing of the adsorbed atoms in the elementary spaces for a certain time. He assume that these spaces or points of residual valiancy can hold only one atom of molecule therefore, the adsorbed of layer can be only one molecule thick, thus, the force between the atoms of the adsorbed substance decreased rapidly with the distances between the atoms. (13, 12.)

Freundlich logarithms though it's empirical has found a vary wile use and application the extent of adsorption decreases with increase in temp and increase, with increase in concentration. In order to represent the variation of extent of adsorption with concentration, Freundlich suggested an empirical isotherm similar to the equation below.

$$\frac{X}{M} = Kc^{\frac{1}{n}} - 2.1$$

X & M are masses of the substances adsorbed and of adsorbent respectively, c is the equilibrium concentration of the adsorbed substances in the solution, k and n are empirical constants

Taking logarithms

$$\log \frac{x}{m} = \log k + \frac{1}{n} \log c - 2.2$$

This implies that a plot of $\log \frac{x}{m}$ against $\log c$ should be a straight line

With slope $1/n$ and intercept k (13)

CHAPTER III

3.00 EXPERIMENTAL PROCEDURE AND ANALYSIS.

3.10 MATERIALS AND EQUIPMENTS

1. Vibrating Shaker and Sieve Vest (500 μm and 250 μm and receiving pan).
2. Weighing balance.
3. Beaker.
4. Electric hot plate.
5. Oven.
6. Furnace.
7. Mortar and Pestle.
8. Electric Stirrer.
9. Burette (500ml).
10. Standard Volumetric Flask.
11. Wash bottle.
12. Distilled water.
13. Clay samples.
14. Automobile waste lube sample.
15. H_2SO_4 sample.

3.20 PREPARATION OF MATERIALS.

3.21 CALCINED CLAY PREPARATION.

The raw Clay sample gotten from the Chanchaga Shelter Clay product was broken down into loose particles in a mortar with a pestle. The required mesh size, which was 250 μm , were gotten with the aid of the Vibrating shaker and sieves nest. The required mesh size was then heated at Temperature of 550^oC for 6 hours and then cooled.

3.22 CLAY ACID TREATMENT

0.1% H₂SO₄ Solution with distilled water was made by measuring 1ml of concentrated H₂SO₄ into the Volumetric Flask containing little distilled water and then made up to 1000ml with the distilled water. This Solution of 0.1% of H₂SO₄ made was well mixed with the Calcined Clay and then oven dried at the Temperature of 42⁰ C After which the Clay was homogenized.

3.23 REFINING METHOD.

The method adopted here was the Contact method of adsorption mechanism. The procedures are as follows:

1. 50g of Acid treated Calcined Clay was measured and mixed with 400ml of used engine oil heated to a Temperature between 50 – 55⁰C.
2. The mixture was stirred with Electric Stirrer for 20 minutes and allowed to settle.
3. This mixture was then filtered with cloth sieve. The Refined engine oil is the filtrate.
4. This process was repeated for 100g, 150g, 200g and 250g respectively and 400ml used oil was kept constant.
5. Analysis was then taken to compare the filtrate with the used engine oil

3.30 ANALYSIS OF SAMPLES.

The analysis was carried out at Oando tube blending plant Kaduna. The following tests were carried out: that is, Appearance, Specific gravity, and Kinematic Viscosity at 40⁰C and at 100⁰C, Viscosity index.

3.31 TEST FOR SPECIFIC GRAVITY AT 15⁰C.

APPARATUS: Hydrometer, Hydrometer Cylinder, Log table
(Hydrometer/Temperature ⁰C).

METHOD:

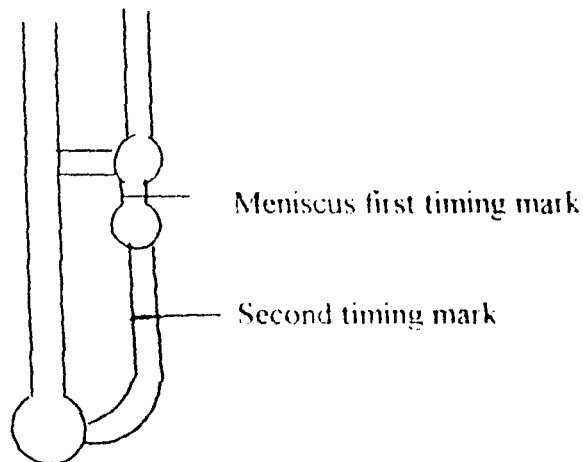
1. The sample was transferred into a clean Hydrometer Cylinder
2. The Hydrometer was depressed into the sample in the Cylinder and then released.
3. Sufficient time was allowed for the Hydrometer to come to rest, floating freely away from the walls of the Cylinder.
4. Then the Hydrometer reading was taken and recorded.
5. After the sample in the Cylinder was stirred with a thermometer until a steady reading was obtained. This steady sample Temperature was recorded
6. The Log table for Hydrometer/Temperature was then used to get the specific gravity. (See Appendix iii and iv for the Log).

3.32 TEST FOR KINEMATIC VISCOSITY

APPARATUS: ASTM Capillary Viscometer, Suction pump with tube,

Viscometer holding, Constant Temperature bath

DIAGRAM.



ASTM VISCOMETER

1. The Viscometer mark was inverted and suction applied 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.
2. The Viscometer was then turned to its normal position and then fixed in the Viscometer holder.
3. The Viscometer assembly was then mounted into the Constant Temperature bath depending on what Temperature to be used (40°C or 100°C) the Viscometer was mounted in a vertical position
4. The charge was allowed to remain in the bath for 30 minutes
5. The suction was then used on the thinner end of the tube to adjust the head level of the test sample to about 5mm above the first timing mark. With the sample flowing freely, the time required for the meniscus to pass from the first timing mark to the second was taken and recorded.

6. Viscosity was calculated by the formula stated below

$$V = C \times t$$

V → Kinetic Viscosity

t → time in Seconds

C → Calibration constant of the viscometer

(See appendix vii for picture diagram)

3.33 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 a fume Cupboard).

METHOD:

1. The cup was filled so that the top of the meniscus is exactly at filling line.
2. The test flame lighted was adjusted in diameter to about 3.2 -- 4.8mm.
3. Then, heat was applied on the hotplate.
4. As the flash point was observed, the Temperature on the Thermometer was then taken and recorded. This Thermometer reading indicates the flash point. (see appendix vi for picture diagram)

3.34 TEST FOR TBN.

REAGENTS: Chlorobenzene, Glacial acetic acid, Perchloric acid (HClO₄)

APPARATUS:

Potentograph (Automatic).

METHOD:

1. 120ml of titration solvent of ratio 2:1 by vol of Chlorobenzene and Glacial acetic Acid was weighed into the sample.
2. The beaker was placed on the titration stand and stirred until the solution dissolved.
3. The burette was filled with the titration (Perchloric acid) solution (HClO_4).
4. Then the titration was determined by potentiograph (See graph in appendix). From the equation below TBN can be calculated

$$\text{BN, mg KOH/g} = ([E - F] \text{NA.56.1})/S.$$

E → Vol. of HClO_4 (ml) used to titrate the sample to the end point

F → Vol. corresponding to E for blank titration.

M → Molarity of HClO_4 solution.

S → Sample in grams (see appendix vi)

3.35 VISCOSITY INDEX.

APPARATUS:

Log table of Viscosity at 40°C and against 100°C

METHOD:

1. When the viscosity at 40°C and 100°C was used on the Log table to determine the value of V.I.
2. The value of VI was traced at a point on the graph when the viscosity at 100°C and 40°C meets.
3. See Appendix for the Log table. (See appendix i and ii)

CHAPTER FOUR.

4.10

RESULTS AND DISCUSION OF RESULTS.

4.11

RESULTS.

TEST	50g clay	100g clay	150g clay	200g clay	250g clay	Used Engine Oil	Fresh Engine Oil
Appearance	Dark	Dark	Dark	Dark	Dark	Dark	Clear
S.G@ 15 ⁰ C	0.8964	0.8965	0.8969	0.8970	0.8976	0.8962	0.880-0.895
K.Viscosity @ 40 ⁰ C	155.94	154.70	153.51	153.06	153.04	156.94	250-280CST
K.Viscosity @ 100 ⁰ C	16.86	16.86	16.71	16.70	16.70	17.07	19-21CST
Viscosity Index V.I	166	117	118	118	118	116	95-100
Flash Point	122 ⁰ C	122 ⁰ C	126 ⁰ C	126 ⁰ C	126 ⁰ C	120 ⁰ C	240-285 ⁰ C
TBN	0	0	0	0	0	0	5.0-8.0mg KOH/g

4.12

DISCUSSION OF RESULTS.

1. Viscosity index is an empirical number indicating the rate of change in viscosity of oil within a given Temperature range. Higher numbers indicate a low change; lower numbers indicate a relatively large change. The higher the number the better the oil, therefore, from the table. Viscosity index increases with increase in activated Calcined clay sample used from 50g – 100g. but remain constant from 150g – 250g (clay). The viscosity index for used oil was 116 and increased to 118 when the used oil was refined with 250g acid treated Calcined clay. This result shows a little improvement in the quality of the spent oil.

2. Kinematic Viscosity is a measure of the flowability of oil. The Kinematic viscosity at 40°C and at 100°C decreases with increase in the quantity of acid treated Calcined clay used, at 40°C , it decreases from 155.94 at 50g of clay to 153.04 at 250g clay and from 16.86 to 16.70 at 100°C respectively. Also, when these values were compared with the used engine oil value as shown on the table of Results, which is 17.07 at 100°C and 156.94 at 40°C . These values are higher compared to those of the refined oil. The values further show that the acid treated Calcined clay had adsorbed certain quantity of impurities from the used oil, because the higher the impurities the more viscous the sample will be.
3. Flash point is the Temperature at which oil gives off vapours that can be ignited with a flame held over the oil. The lower the flash points, the greater tendency for the oil to suffer vaporization loss at high temperature and to burn off on hot cylinder walls and pistons. Flash point is lowered by the extent of impurities. From the result gotten, the spent lube flash point was 120°C and increased to 126°C , when the oil was treated with the acid treated Calcined clay, at 150g, 200g and 250g. This implies that the clay from the original sample has adsorbed some impurities.
4. Total Base Number (TBN) is a measure of the amount of basic substances in the oil. This because lubricants product contains basic constituents that present as additives. The nature of graph shown in the appendix for TBN indicates that its value is zero, since the graph is not sinusoidal in shape. It

also shows that the additives have been used up and what it contains is just the base oil.

5. There was no significant change in the appearance of the samples.
6. Specific gravity is the ratio of the mass of a given volume to the mass of equal volume of water. The specific gravity of mineral oils vary from 0.86 – 0.98 since the viscosity of water is 1 at 15⁰C. Specific gravity decreases with increase in temperature and decreases slightly as viscosity decreases for similar composition. API. From the table, the specific gravity increases as the quantity of clay increases from 0.8964 -0.8962. since viscosity decreases as shown on the table. The value is not in line with the fact mentioned above. This error may result from experimental error.
7. comparing the values of the treated spent lube oil with the fresh engine oil standard values on the table, the appearance of the treated oil is dark this is because of the carbon deposits and that of the fresh is bright due to the absence of impurities. The S.G at 15⁰C of the treated oil is within the range of the fresh engine oil standard values; the kinematics viscosity at 40 and 100⁰C are below the range of that of the fresh engine oil values with similar parameters. These is because there are still deposit in the oil refined. although there were slight improvement when compared with the untreated spent oil values with similar parameters; the viscosity index of the treated oil is above the range of that of the fresh engine oil; the flash point values are below the range of the fresh engine oil, but showed improvement when compared with the untreated spent lube oil; the TBN is zero compared to that of the fresh

engine oil which is between 5.0-8.0 mgKOH/g, this is because all the additives in the spent oil refined had been used up

CHAPTER FIVE

5.00 RECOMMENDATIONS AND CONCLUSION.

5.10 CONCLUSION.

This work was successfully carried out and the following conclusions were drawn. It was found out that better results were given when 250g of acid treated Calcined clay was mixed with the spent tube, when compared with the spent engine oil results.

The viscosity decreases from 17.07 at 100⁰C of used engine oil to 16.70 of oil refined with 250g of acid treated Calcined clay and also 156.94 at 40⁰C to 153.04 respectively. This indicates that the clay adsorbent from the used engine oil sample has adsorbed some impurities.

The flash point also increases from 120⁰C of used engine oil to 126⁰C of oil treated with 150g, 200g and 250g of acid treated Calcined clay. This indicates improvement in the quality of the oil after it was refined.

The Total Based Number (TBN) result, which was zero, indicates that the refined oil contains just the based oil without additives.

The viscosity index also increases from the table of results, this also shows the used oil has improved slightly in quality. However the values of the treated oil is not too close to the fresh engine oil, standard values but it showed a little improvement in quality due to adsorption of some impurities by the acid treated calcined clay when compared to the untreated spent lube oil. Effective filtration and addition of additives will improve more on the quality of the treated spent lube oil.

5.11 RECOMMENDATION.

- 1. The activation should be performed using different acids such as HCL, H₂SO₄, HNO₃, and e.g., to authenticate the best for activation.**
- 2. Percolation method of adsorption should be tried using a bed of different mesh size of clay, if better results could be obtained, it may also improve the appearance of the used oil.**
- 3. Chanchaga Shelter clay sample should be studied and analyzed to investigate the type of clay based on its structure and its composition.**
- 4. Filter paper or cloth sieve cannot do effective filtration, since effective filtration further improves the quality of oil by removing the impurities present. The pressure vacuum filter is recommended.**
- 5. The oil refined from 150g to 250g should be refined again in the same manner; this might further improve the flash point and some other parameters.**

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16.80 to 16.98 cSt at 100C

50 to 100 V. L.

Table with columns for V.L. and Kinematic Viscosity at 40C and 100C. The table shows a grid of viscosity values for various V.L. ranges from 50 to 150.

Table with columns for V.L. and Kinematic Viscosity at 40C and 100C. This table continues the grid from the first table, covering V.L. values from 150 to 200.

Log Table for determining viscosity Index (II)

Appendix iv

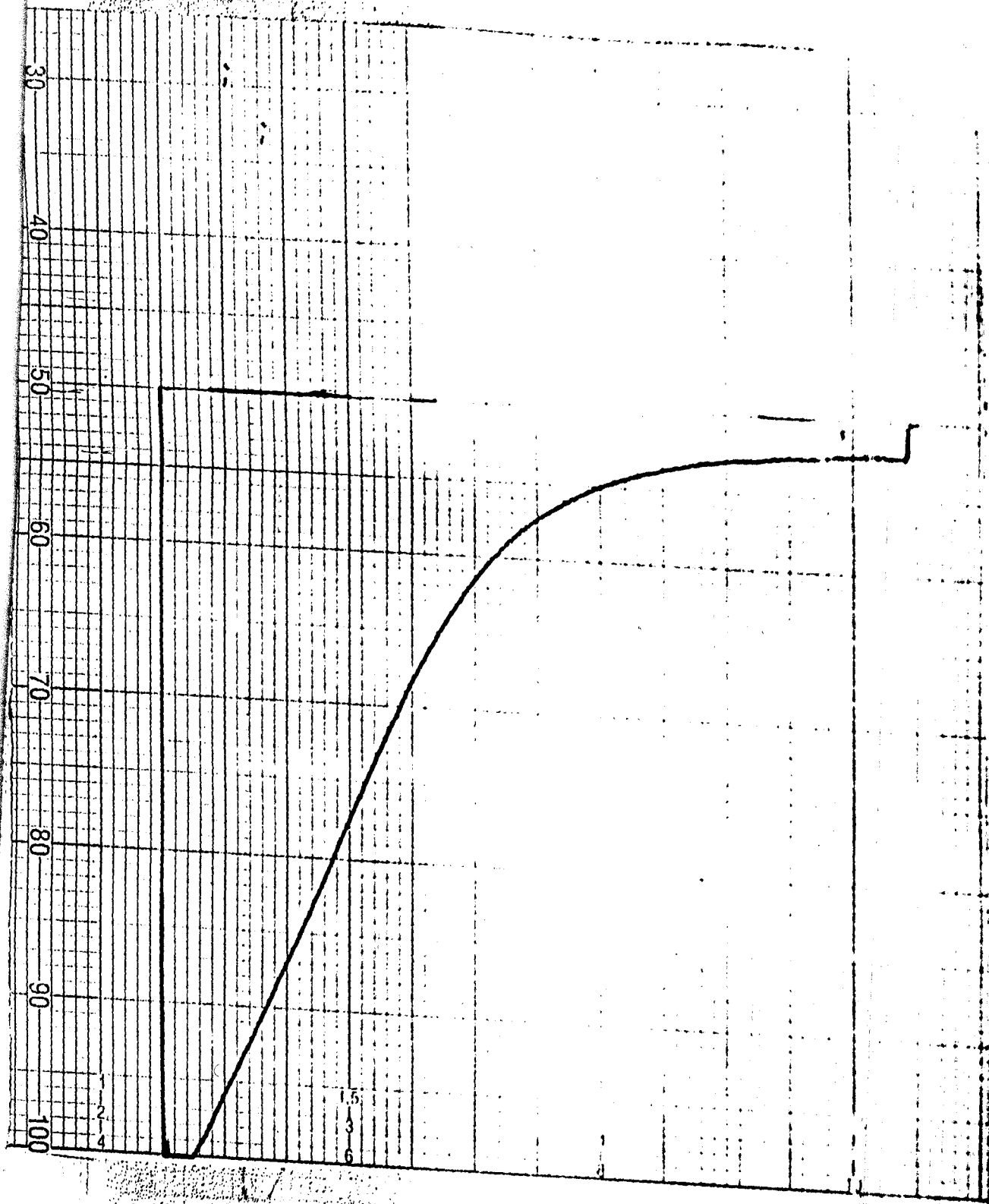
CONVERSIONE DELLE DENSITA' A 15 °C.

Temperature °C.	Densità alle temperature indicate											Temper. °C.
	00.0	01.0	02.0	03.0	04.0	05.0	06.0	07.0	08.0	09.0	10.0	
Densità corrispondente a 15 °C.												
-3.0	067.9	069.9	071.0	073.9	075.9	077.9	079.9	081.9	083.9	085.9	087.9	0.1
-4.0	068.2	070.2	072.2	074.2	076.2	078.2	080.2	082.2	084.2	086.2	088.2	-0.1
-4.5	068.5	070.5	072.5	074.5	076.5	078.5	080.5	082.5	084.5	086.5	088.5	-0.2
-5.0	068.8	070.8	072.8	074.8	076.8	078.8	080.8	082.8	084.8	086.8	088.8	-0.3
-5.5	069.1	071.1	073.1	075.1	077.1	079.1	081.1	083.1	085.1	087.1	089.1	-0.3
-2.0	069.4	071.4	073.4	075.4	077.4	079.4	081.4	083.4	085.4	087.4	089.4	-0.2
-3.0	069.7	071.7	073.7	075.7	077.7	079.7	081.7	083.7	085.7	087.7	089.7	-0.1
-1.0	070.0	072.0	074.0	076.0	078.0	080.0	082.0	084.0	086.0	088.0	090.0	-0.2
-1.5	070.3	072.3	074.3	076.3	078.3	080.3	082.3	084.3	086.3	088.3	090.3	-0.3
-0.5	070.6	072.6	074.6	076.6	078.6	080.6	082.6	084.6	086.6	088.6	090.6	-0.3
0.0	070.9	072.9	074.9	076.9	078.9	080.9	082.9	084.9	086.9	088.9	090.9	0.0
0.5	071.2	073.2	075.2	077.2	079.2	081.2	083.2	085.2	087.2	089.2	091.2	0.0
1.0	071.5	073.5	075.5	077.5	079.5	081.5	083.5	085.5	087.5	089.5	091.5	0.1
1.5	071.8	073.8	075.8	077.8	079.8	081.8	083.8	085.8	087.8	089.8	091.8	0.1
2.0	072.1	074.1	076.1	078.1	080.1	082.1	084.1	086.1	088.1	090.1	092.1	0.1
2.5	072.4	074.4	076.4	078.4	080.4	082.4	084.4	086.4	088.4	090.4	092.4	0.2
3.0	072.7	074.7	076.7	078.7	080.7	082.7	084.7	086.7	088.7	090.7	092.7	0.2
3.5	073.0	075.0	077.0	079.0	081.0	083.0	085.0	087.0	089.0	091.0	093.0	0.2
4.0	073.3	075.3	077.3	079.3	081.3	083.3	085.3	087.3	089.3	091.3	093.3	0.2
4.5	073.6	075.6	077.6	079.6	081.6	083.6	085.6	087.6	089.6	091.6	093.6	0.2
5.0	073.9	075.9	077.9	079.9	081.9	083.9	085.9	087.9	089.9	091.9	093.9	0.2
5.5	074.2	076.2	078.2	080.2	082.2	084.2	086.2	088.2	090.2	092.2	094.2	0.2
6.0	074.5	076.5	078.5	080.5	082.5	084.5	086.5	088.5	090.5	092.5	094.5	0.2
6.5	074.8	076.8	078.8	080.8	082.8	084.8	086.8	088.8	090.8	092.8	094.8	0.2
7.0	075.1	077.1	079.1	081.1	083.1	085.1	087.1	089.1	091.1	093.1	095.1	0.2
7.5	075.4	077.4	079.4	081.4	083.4	085.4	087.4	089.4	091.4	093.4	095.4	0.2
8.0	075.6	077.6	079.6	081.6	083.6	085.6	087.6	089.6	091.6	093.6	095.6	0.2
8.5	075.9	077.9	079.9	081.9	083.9	085.9	087.9	089.9	091.9	093.9	095.9	0.2
9.0	076.1	078.1	080.1	082.1	084.1	086.1	088.1	090.1	092.1	094.1	096.1	0.2
9.5	076.4	078.4	080.4	082.4	084.4	086.4	088.4	090.4	092.4	094.4	096.4	0.2
10.0	076.7	078.7	080.7	082.7	084.7	086.7	088.7	090.7	092.7	094.7	096.7	0.2
10.5	077.0	079.0	081.0	083.0	085.0	087.0	089.0	091.0	093.0	095.0	097.0	0.2
11.0	077.3	079.3	081.3	083.3	085.3	087.3	089.3	091.3	093.3	095.3	097.3	0.2
11.5	077.6	079.6	081.6	083.6	085.6	087.6	089.6	091.6	093.6	095.6	097.6	0.2
12.0	077.9	079.9	081.9	083.9	085.9	087.9	089.9	091.9	093.9	095.9	097.9	0.2
12.5	078.2	080.2	082.2	084.2	086.2	088.2	090.2	092.2	094.2	096.2	098.2	0.2
13.0	078.5	080.5	082.5	084.5	086.5	088.5	090.5	092.5	094.5	096.5	098.5	0.2
13.5	078.8	080.8	082.8	084.8	086.8	088.8	090.8	092.8	094.8	096.8	098.8	0.2
14.0	079.1	081.1	083.1	085.1	087.1	089.1	091.1	093.1	095.1	097.1	099.1	0.2
14.5	079.4	081.4	083.4	085.4	087.4	089.4	091.4	093.4	095.4	097.4	099.4	0.2
15.0	079.7	081.7	083.7	085.7	087.7	089.7	091.7	093.7	095.7	097.7	099.7	0.2
15.5	080.0	082.0	084.0	086.0	088.0	090.0	092.0	094.0	096.0	098.0	100.0	0.2
16.0	080.3	082.3	084.3	086.3	088.3	090.3	092.3	094.3	096.3	098.3	100.3	0.2
16.5	080.6	082.6	084.6	086.6	088.6	090.6	092.6	094.6	096.6	098.6	100.6	0.2
17.0	080.9	082.9	084.9	086.9	088.9	090.9	092.9	094.9	096.9	098.9	100.9	0.2
17.5	081.2	083.2	085.2	087.2	089.2	091.2	093.2	095.2	097.2	099.2	101.2	0.2
18.0	081.5	083.5	085.5	087.5	089.5	091.5	093.5	095.5	097.5	099.5	101.5	0.2
18.5	081.8	083.8	085.8	087.8	089.8	091.8	093.8	095.8	097.8	099.8	101.8	0.2
19.0	082.1	084.1	086.1	088.1	090.1	092.1	094.1	096.1	098.1	100.1	102.1	0.2
19.5	082.4	084.4	086.4	088.4	090.4	092.4	094.4	096.4	098.4	100.4	102.4	0.2
20.0	082.7	084.7	086.7	088.7	090.7	092.7	094.7	096.7	098.7	100.7	102.7	0.2
20.5	083.0	085.0	087.0	089.0	091.0	093.0	095.0	097.0	099.0	101.0	103.0	0.2
21.0	083.3	085.3	087.3	089.3	091.3	093.3	095.3	097.3	099.3	101.3	103.3	0.2
21.5	083.6	085.6	087.6	089.6	091.6	093.6	095.6	097.6	099.6	101.6	103.6	0.2
22.0	083.9	085.9	087.9	089.9	091.9	093.9	095.9	097.9	099.9	101.9	103.9	0.2
22.5	084.2	086.2	088.2	090.2	092.2	094.2	096.2	098.2	100.2	102.2	104.2	0.2
23.0	084.5	086.5	088.5	090.5	092.5	094.5	096.5	098.5	100.5	102.5	104.5	0.2
23.5	084.8	086.8	088.8	090.8	092.8	094.8	096.8	098.8	100.8	102.8	104.8	0.2
24.0	085.1	087.1	089.1	091.1	093.1	095.1	097.1	099.1	101.1	103.1	105.1	0.2
24.5	085.4	087.4	089.4	091.4	093.4	095.4	097.4	099.4	101.4	103.4	105.4	0.2
25.0	085.7	087.7	089.7	091.7	093.7	095.7	097.7	099.7	101.7	103.7	105.7	0.2

Densità corrispondente a 15 °C.

Log Table for determining specific gravity (II)

Appendix v



Grape For Total Base Number (T.B.N)

Appendix Vi

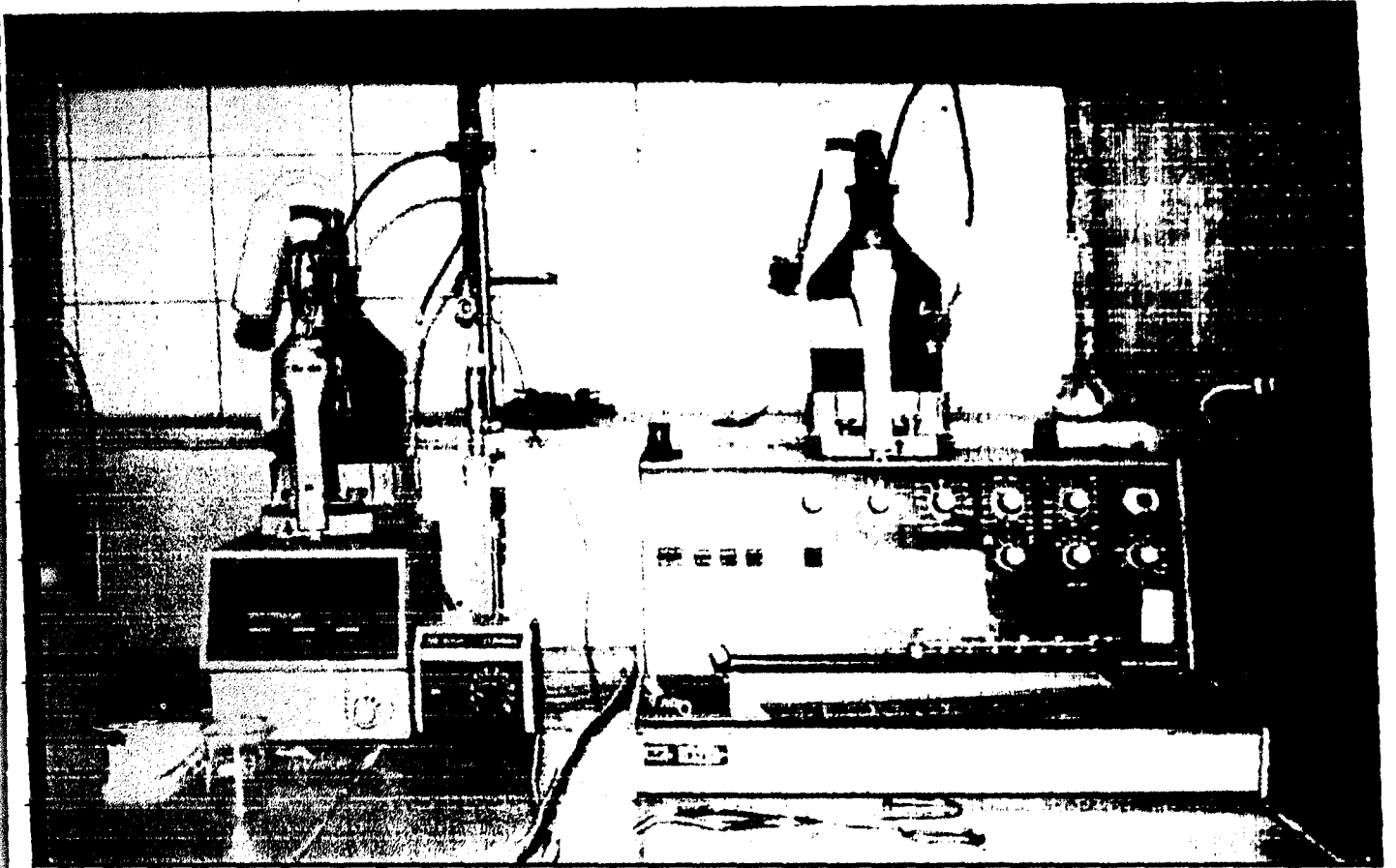


Plate 1 - Potentiograph for TBN analysis

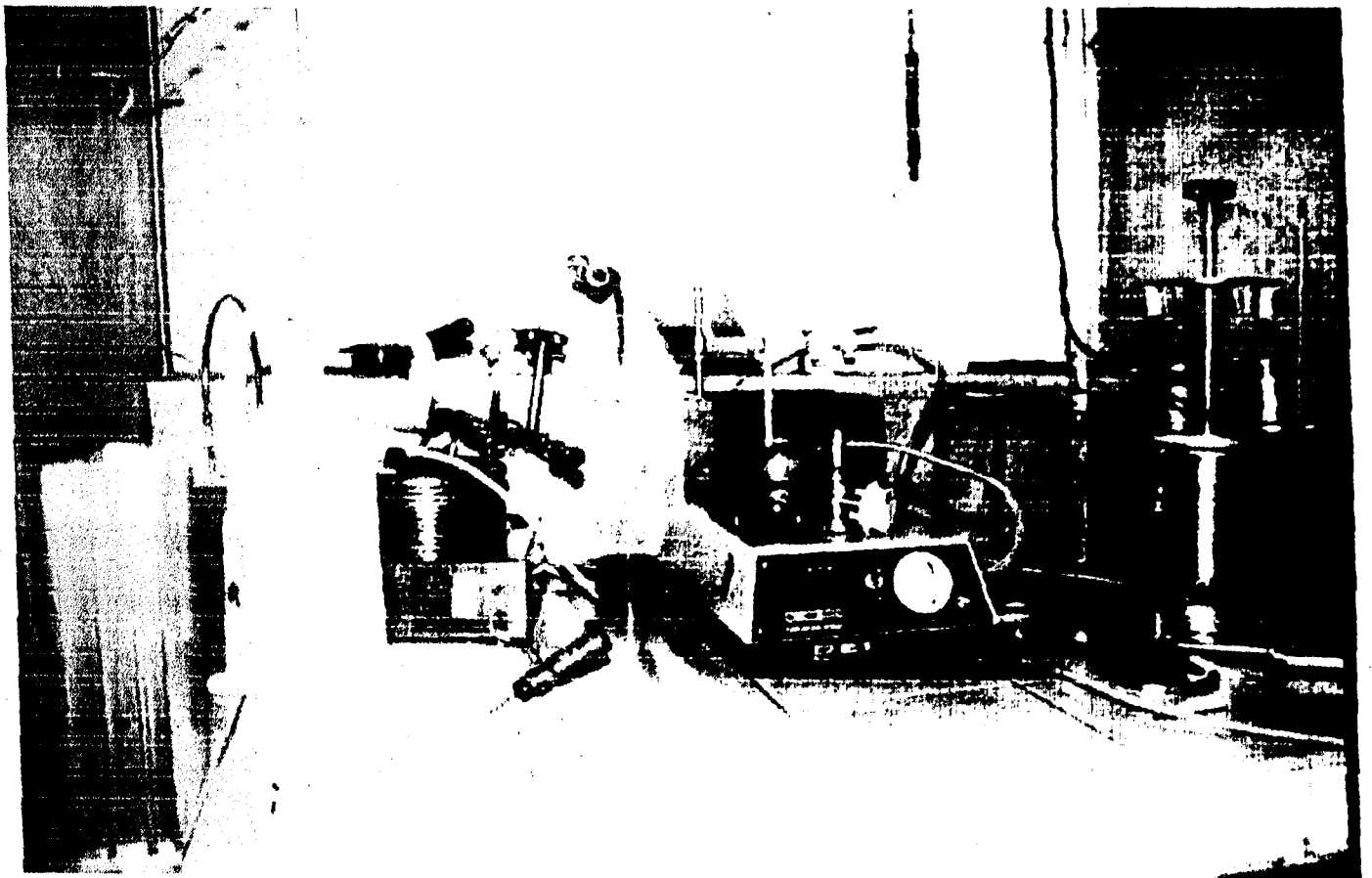


Plate 2 - Flash point analysis

Appendix vii

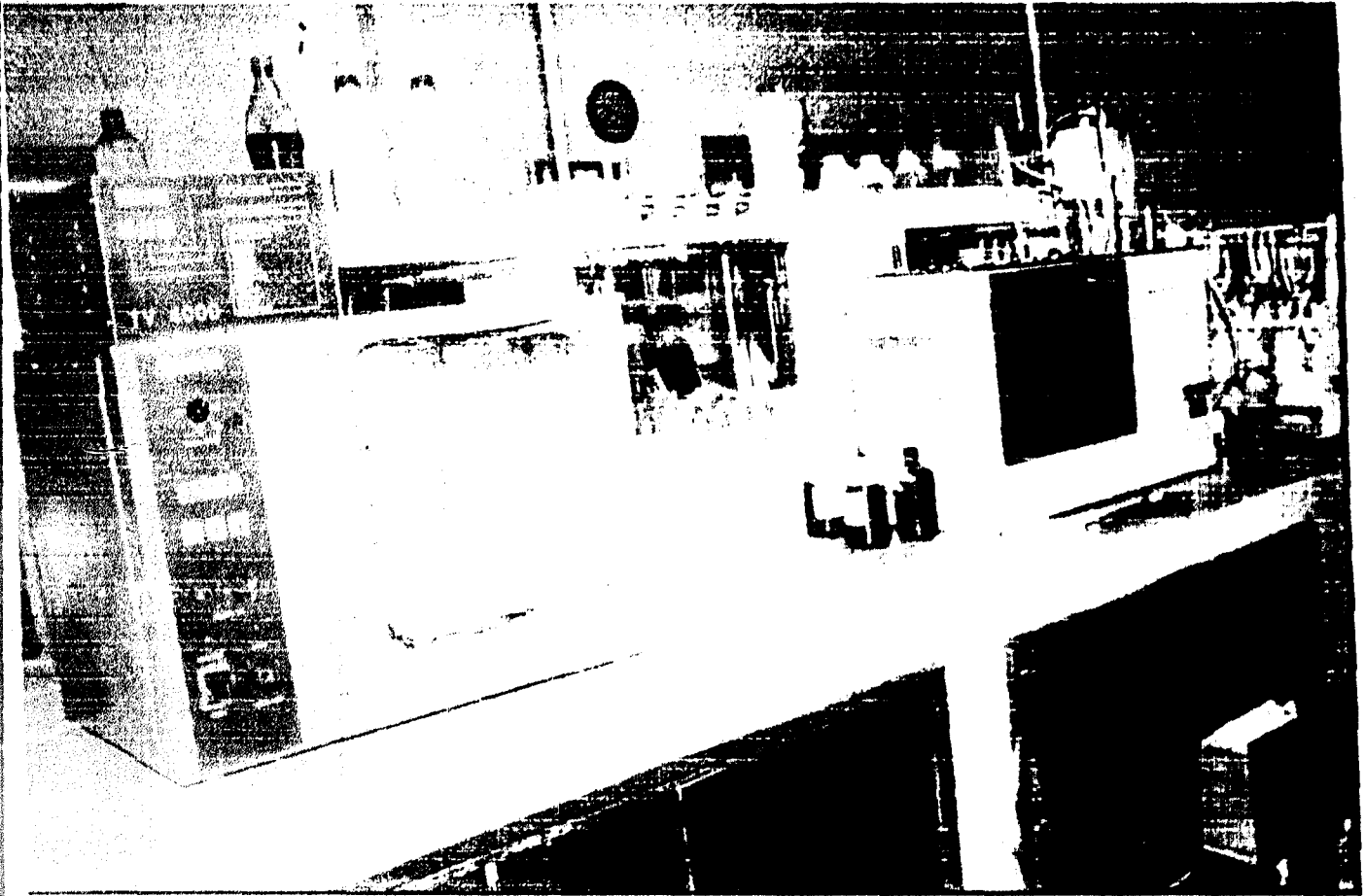


Plate 3 - for Viscosity analysis