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College of Science
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STUDY OF RECYCLING PROCESS AND PETROLEUM PROPERTIES OF SPENT LUBRICATING OIL

A Thesis

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By

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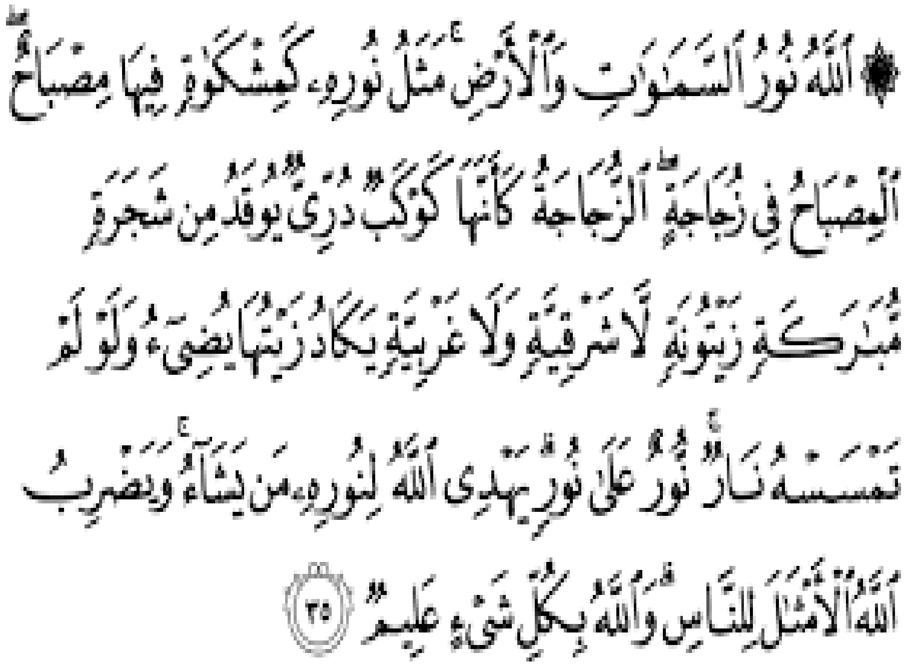
بسم الله الرحمن الرحيم



۞ ٱللَّهُ نُورُ ٱلسَّمَٰوَٰتِ وَٱلْأَرْضِۚ مَثَلُ نُورِهِۦ كَمِشْكَوٰةٖ فِيهَا مِصْبَاحٌۖ
ٱلْمِصْبَاحُ فِي زُجَاجَةٍۖ ٱلزُّجَاجَةُ كَأَنَّهَا كَوْكَبٞ دُرِّيّٞ يُوقَدُ مِن شَجَرَةٖ
مُّبَٰرَكَةٖ زَيْتُونَةٖ لَّا شَرْقِيَّةٖ وَلَا غَرْبِيَّةٖ يَكَادُ زَيْتُهَا يُضِيٓءُ وَلَوْ لَمْ
تَمْسَسْهُ نَارٞۚ نُّورٌ عَلَىٰ نُورٖۚ يَهْدِي ٱللَّهُ لِنُورِهِۦ مَن يَشَآءُۚ وَيَضْرِبُ
ٱللَّهُ ٱلْأَمْثَٰلَ لِلنَّاسِۗ وَٱللَّهُ بِكُلِّ شَيْءٍ عَلِيمٞ ٣٥

صدق الله العظيم

النور

Dedication

To the spirit of the man who raised me and taught me the simplicity of life ....

my dear father

To the kindhearted heart that got me this way ....

my precious mother

To those who sacrificed for the sake of my happiness ....

my dear husband

To candle my life and light my way ....

my kids

To those who taught me the letter and the word ….

my teachers

To all friends and family….

Elaf

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Abstract

Lubricating oil-type Baghdad (15W-40) , in Middle Refineries Company / AL- Daura Refinery and those used for a distance of (1000, 1500 and 2000) km in small saloon car (Mercedes-Benz-Internal Combustion Engine) tested at different operating conditions . The physical properties of new, used and recycled oils were investigated .

Kinematic viscosities at (40 and 100) ˚C of the used and recycled oils were observed to be lower when compared to those of the new one . The viscosity index of lube oil will also change , which decreased in used oils of 1000 km , 1500 km and recycled oil , but at 2000 km it increases, because high decreases of the kinematic viscosity when compared with those of the new oil .

The specific gravity of a used oil can be lower or higher than that of the new lube oils depending on the type of contamination .So , the result for the new oil specific gravity at 15 ˚C was 0.889, while the specific gravity of used oils at (1000 , 1500 and 2000) km were (0.886 ,0.885 and 0.890) respectively. American Petroleum Institute (API˚) gravity of new ,used and recycled oil are between (29>API>8.5) .So the oil used in this study is heavy oil .

Flash point of oil used at 1000 km , 1500 km and 2000 km are (221 ,216 and 187) ˚C respectively and they are lower than those of the new oil which is 224˚C . A high flash point was obtained for the recycled oil, which is 203 ° C, when compared to the result obtained from the oil used for a distance of 2000 km.

The pour point of the new oil is -36 ˚C , while for the oil used at 1000 km and 1500 km is also -36 ˚C and for oil used at 2000 km is -39 ˚C and decrease was observed in the pour point of recycled oil .

The results of color cannot be obtained for the used oils as it were dark, but, after vacuum distillation , the darker color of oil used at 2000 km turned into yellow with the value of 5 ASTM color .

Carbon residue and ash contents for oil used increased when compared with the new oil , but after vacuum distillation it was decreased to 0.47 %, and 0% respectively in the recycled oil . So, we used the residue after distillation to find the ash contents balance values . The metal content was determined by using flame atomic absorption spectrophotometer for contents (Iron , copper , magnesium , zinc and manganese) contained in new , used and recycled oils .

Fourier Transform Infrared Spectroscopy (FTIR) and UV- visible spectrophotometer were measured for new , used and recycled oils . The tests proved that the recycled oil contains all the functional groups which exist in the new oil .

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TERMINOLOGY

Symbol	Definition
API	**American Petroleum Institute**
ASTM	**American Society for Testing and Materials**
SAE	**Society of Automotive Engineers**
VI	**Viscosity Index**
SI	**International System of Units**
cP	**Centipoise**
cSt	**Centistoke**
rpm	**Rotation Per Minute**
ppm	**Part Per Million**
FT-IR	**Fourier Transform Infrared**
UV	**Ultraviolet**
Dil	**Dilution**
mmHg	**Millimetre of Mercury**
km	**Kilometer**
MEK	**Methyl Ethyl Ketone**

Chapter One

Introduction & Previous Studies

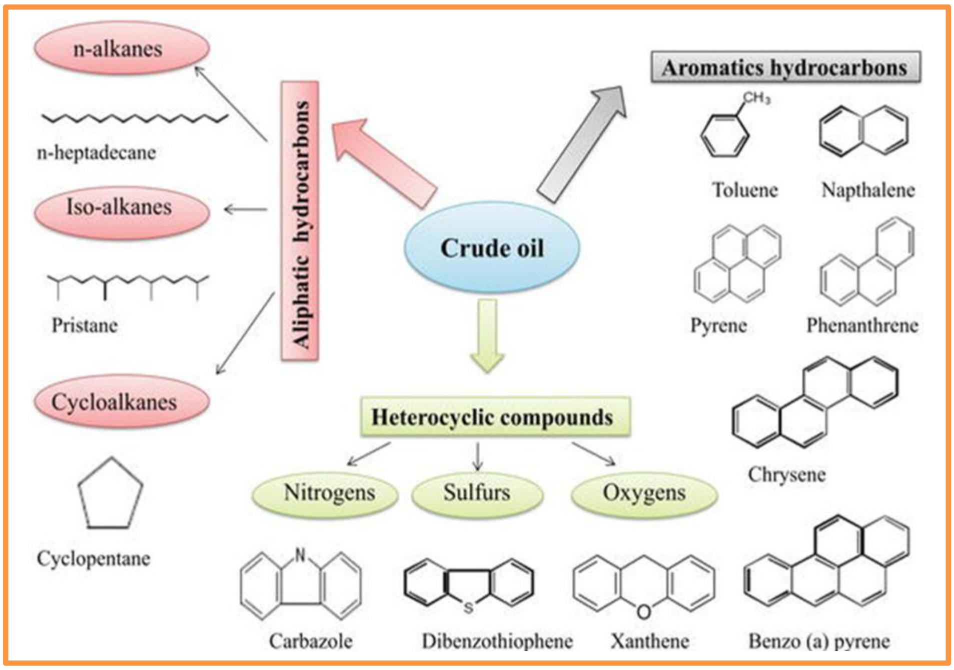
1.1 Introduction

Crude oil is a complex hydrocarbons mixture that varies in composition and color from greenish- yellow to black and also in viscosity, it may be liquid with low viscosity or it may be of high viscosity and becomes non viscous [1] . The compounds in crude oil are essentially hydrocarbons or substituted hydrocarbons in which the major elements are (Carbon at 85%–90%) and (Hydrogen at 10%–14%) , and the rest with non- hydrocarbon elements Sulfur (0.2% – 3%), Nitrogen (< 0.1–2%), Oxygen (1% – 1.5%) ,and organo-metallic compounds of Nickel , Vanadium, Arsenic, Lead, and other metals in traces (in parts per million or parts per billion concentration) , inorganic salts of Magnesium Chloride, Sodium Chlorides, and other mineral salts are also accompanied with crude oil from the well either because of water from formation or water and chemicals injected during drilling and production [2] . Crude oil have a specific gravity at (15.6 ˚C ,60 ˚F) that varies from about 0.75 to 1.00 ,and API 57° to 10° , with the specific gravity of most crude oils falling in the range 0.80 to 0.95 , API 45° to 17° , the boiling range of crude oil varies from about (20 ˚C , 68 ˚F) to above (350 ˚C , 660 ˚F) , above which active decomposition ensues when distillation is attempted . Crude oil can contain from (0 % to 35 %) or more of gasoline, as well as varying proportions of kerosene, gas oil hydrocarbons and higher boiling constituents up to the viscous and nonvolatile compounds present in lubricant oils and in asphalt [3] .

1.2 The Composition of Crude oil

1.2.1 The Chemical Composition of Crude oil

Crude oil is composed of two major groups : compound hydrocarbon group and non-hydrocarbon one ,as shown in Fig (1.1).



Figure(1.1) : Composition of crude oil [4]

1.2.1.1 Hydrocarbon Groups

Compounds that are solely made of carbon and hydrogen are called hydrocarbons, these hydrocarbons are grouped as paraffines, naphthenes, aromatics, and olefins, crude oil contains these hydrocarbons in different proportions ,except olefins, which are produced mainly during processing [2] .

A-Paraffins : Saturated hydrocarbons where all four bonds of a carbon atoms are linked to four separate atoms, such as :. methane, ethane, propane, butane, pentane, hexane ,etc…, with the generic molecular formula of (C_nH_{2n+2}) , where n : is the number of carbon atoms in that compound , the series starts with methane, which has the chemical formula CH_4 . Paraffin are relatively unreactive as compared to aromatics and olefins, at room temperature, paraffins are not affected by concentrated fuming sulfuric acid or powerful oxidising agents. Paraffins are subdivided to n- paraffins and iso -paraffins, n-paraffins are straight chain compounds and iso-paraffins are branched compounds, n-paraffins and iso-paraffins have the same formula (the same number of carbon and hydrogen atoms) , but they differ widely in their physical and chemical properties because of isomerism and the number of isomers of n-paraffins increases with the number of carbon atoms in the paraffin series . For example, paraffins with carbon numbers of five, six , and eight will have iso-paraffins of three, five, and eighteen isomers, respectively. Iso-paraffins are more reactive than n-paraffins, n-paraffins can be converted to iso-paraffins by thermal or catalytic processes, this is known as isomerisation . Catalytic processes in which platinum catalysts is used [2,5] .

B- Naphthenes : A cyclic saturated hydrocarbons with the general formula, like olefins (C_nH_{2n}) , also known as cyclo-alkanes: An example of which are cyclo-propane , cyclo-butane , cyclo-pentane and cyclo-hexane. Naphthenes are an important part of all liquid refining products and form about 50 % of the crude [6,7].

C- Olefins : Those in which the double bond or bonds is present between the two carbon atoms in the chemical formula. The generic formula is (C_nH_{2n}) , and the lowest number of this homologous series is ethylene, C_2H_4 . Olefins are found in some crude oil raw materials at rates of up to 3% of the weight of crude, and are found in some crude oil products due to thermal breakdown during distillation process. Olefins react easily with acids, alkalies, halogens, oxidizing agents, etc, and not usually present in crude oil but they are produced by thermal and catalytic decomposition or dehydrogenation of paraffins. It may be straight chain (normal) or branched chain, and can be determined by the bromine or iodine number by reaction with bromine or iodine .They are readily converted to di- olefins in the presence of oxygen and to form a gum-like substance. Those present in crude oil products can be removed by absorption in sulfuric acid [2,7].

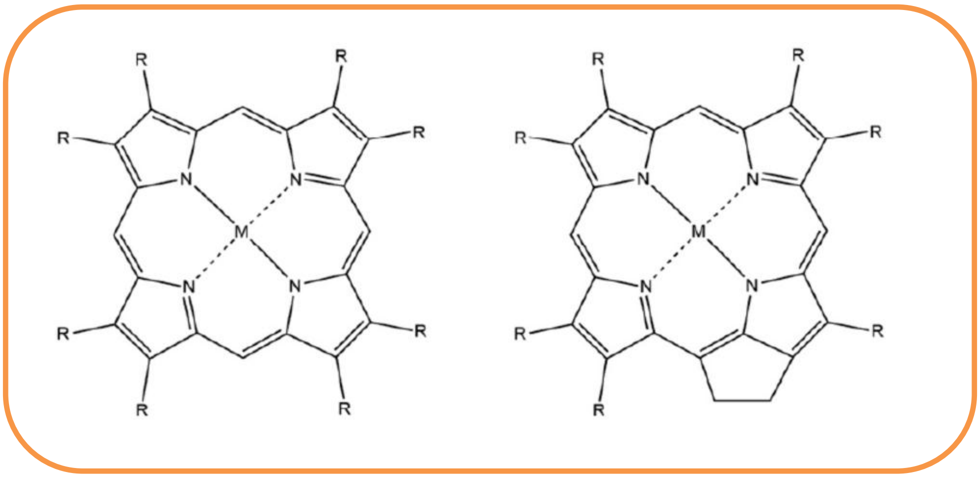
D-Aromatics : Their general formula is (C_nH_{2n-6}) , aromatics are closed unsaturated ring chains, and form a small fraction of all distillates about 15% .Those in hydrocarbons containing one or more aromatic nuclei such as, benzene, naphthalene, and phenanthrene ring systems that may be linked up with (substituted) naphthalene rings or paraffin side-chains . Aromatics crude oil products can be separated by extraction with solvents such as phenol , furfural and diethylene glycol [2,7].

1.2.1.2 Non Hydrocarbon Groups

The non-hydrocarbon components in crude oil fall into two groups :

A- The first group consists of a number of organic compounds. They are in one form or another in crude oil, and are found in all oils at varying levels regardless of the source .Its substances contain sulfur compounds (sulfide H-S-R , di sulfide R-S-S-R, and thiophenes C_2H_4S) and hydrocarbons containing nitrogen such as carbazole compounds [8] . The crude oil that contains large amounts of H_2S is called sour crude. Sulfur present in crude oil fuel products also forms various oxides of sulfur (SO_x) during combustion , which are strong environmental pollutants. H_2S can be removed from gases by absorption in an amine solution . In light distillates sulfur may be present as H_2S, mercaptans, and thiophenes, but in the heavier fractions of crude oil, 80% – 90% of the sulfur is usually present in the complex ring structure of hydrocarbons, sulfur is recovered during refining and sold as a product, sulfur also has a poisoning effect on various catalysts . Nitrogen compounds in hydrocarbons are usually found in the heavier parts of the crude oil, these are responsible for color and color instability and poisoning of certain catalysts. Nitrogen in crude oil fuels causes the generation of oxides of nitrogen (NO_x), which are also strong pollutants of the atmosphere, nitrogen can be eliminated from crude oil products by catalytic hydrogenation, like sulfur . Nitrogen in the heavier parts of crude oil cannot be removed without severe cracking or hydrogenation reactions. Crude oil may contain oxygen containing compounds, such as naphthenic acids, phenols, and cresols, which are responsible for corrosive activities, oxygen also acts as a poison for many catalysts, and it can be removed by catalytic hydrogenation . Excessive oxygen compounds may even lead to explosion [2] .

B- The second group of non-hydrocarbon components consists of small amounts of several elements. Some reports indicate that crude oil contains different elements (Ni ,V , Si , Fe , Al , Ca , Mg , Cu , Pb , Zn , Sb , Hg , Sn and Cr) concentrated in heavy residues in the form of dissolved organic metal derivatives or soap salts or suspensions, and have a detrimental effect on the auxiliary factors used in the liquidation process. It has been found that the presence of small quantities of these elements affect the quality and performance of crude oil [9,10,11] . Vanadium and nickel are found in the form of organo-metallic compounds mostly in the heavier fractions of crude oil where the metal atoms are distributed within the compound in a complex form called porphyrins, as shown in Fig (1.2). Crude oil fuels containing these metallic compounds may damage the burners, lines, and walls of the combustion chambers [2] .



Figure(1.2) : Structures of metal porphyrins [12]

1.2.2 Composition of Crude Oil Distillates According to Boiling Points [13 ,14]

A- Light Distillates Products:

1- **Refinery gases or off gas:** density (0.20-0.38) gm/m³, draw off at temperature of < 65 ˚C **.**

2- **Liquefied crude oil gases (LPG):** density (0.52-0.58) gm/m³, draw off at temperature >65 ˚C .

3- **Naphtha:** density (0.68-0.71) gm/m³, draw off at temperature (100-120) ˚C .

4- **Kerosene:** density (0.76-0.80) gm/m³, draw off at temperature (180-220) ˚C

5- **Diesel fuel:** density (0.82-0.84) gm/m³, draw off at temperature (230-260) ˚C .

B-Middle Distillates:

1. **Light fuel oil:** density (0.92-0.96) gm/m³, draw off at temperature (+265) ˚C .

2-**Heavy fuel oil:** density (0.97-1.00) gm/m³, draw off at temperature (+300) ˚C

C- Heavy Distillates:

1. **Asphalt, carbon black and tar:** density (2-2.36) gm/m³, draw off at temperature (+360) ˚C .
2. **Crude oil coke:** density (1.00 -1.7) gm/m³, draw off at temperature +450 ˚C
3. **Lubricating and transformer oil:** density (0.83-0.89) gm/m³, product of solvent dewaxing and thickening process.
4. **Waxes and greases:** density (1.80-2.00) gm/m³, product from solvent dewaxing process.

1.3 Vacuum Distillation

1.3.1 Introduction

Vacuum distillation is distillation at pressures below one atmosphere, reduced pressure permits vaporization at reduced temperatures, this has two distinct advantages:

1- The energy requirement for both heating and cooling is reduced .

2- Thermal decomposition and degradation of the fluid and its additives are avoided .

Distillation involves heating , vaporization, condensation and cooling of vapors . Vaporization which is the change from a liquid to a vapor state, the change typically requires the addition of heat energy to the liquid, heat can be introduced just prior to distillation , condensation is the change in state from a vapor to a liquid , generally requiring the removal of heat from the vapor in a condenser ,and the condensation is the reverse of vaporization [15] .

1.3.2 Vacuum Distillation Processes

Distillation of crude oil is carried out at atmospheric pressure and under vacuum. Low boiling fractions vaporise at atmospheric pressure up to a temperature (below 400 ˚C) without cracking the hydrocarbon compounds. High boiling fractions are vaporised under vacuum at a temperature much lower than 400 ˚C and cracking is avoided . Crude oil is first washed in desalting to remove salts and metals that could cause corrosive damage or catalyst deactivation in downstream units. After desalting the feed is preheated in a series of heat exchangers and then heated to the process temperature in a furnace. Feed enters the distillation column at 2-5 barG (29-73 psig) and around 350-390 ˚C (660-730 ˚F). Light vapours rise to the top of the column and heavier liquid hydrocarbons fall to the bottom. Hydrocarbon fractions are withdrawn from the tower according to their specific boiling temperatures. Stripping steam at the column bottom improves the separation of lighter boiling components. The vapours are condensed at the overhead cooling and recycled back to the column as reflux. Circulating reflux (pump around) and side stripping with steam improves the separation of different fractions. Heavy residue with boiling points exceeding 400 ˚C (750 ˚F) are sent to a vacuum tower to allow further recovery without cracking the feed. Three types of vacuum towers are used: dry (no steam), wet without stripping and wet with stripping. Vacuum distillation is carried out with an absolute pressure of around 25-40 mmHg and a temperature of around 380-420 ˚C (720-790 ˚F) . The operating pressure is maintained by using steam ejectors and condensers. Atmospheric distillates are the major source of crude oil fuels and vacuum distillates are the source of fuels, wax, lubricating oil base stocks, bitumen, crude oil cokes, etc., mainly produced by secondary treatments like extraction, thermal , and catalytic treatments [2,16] .Fig (1.3) show a diagram for separation .

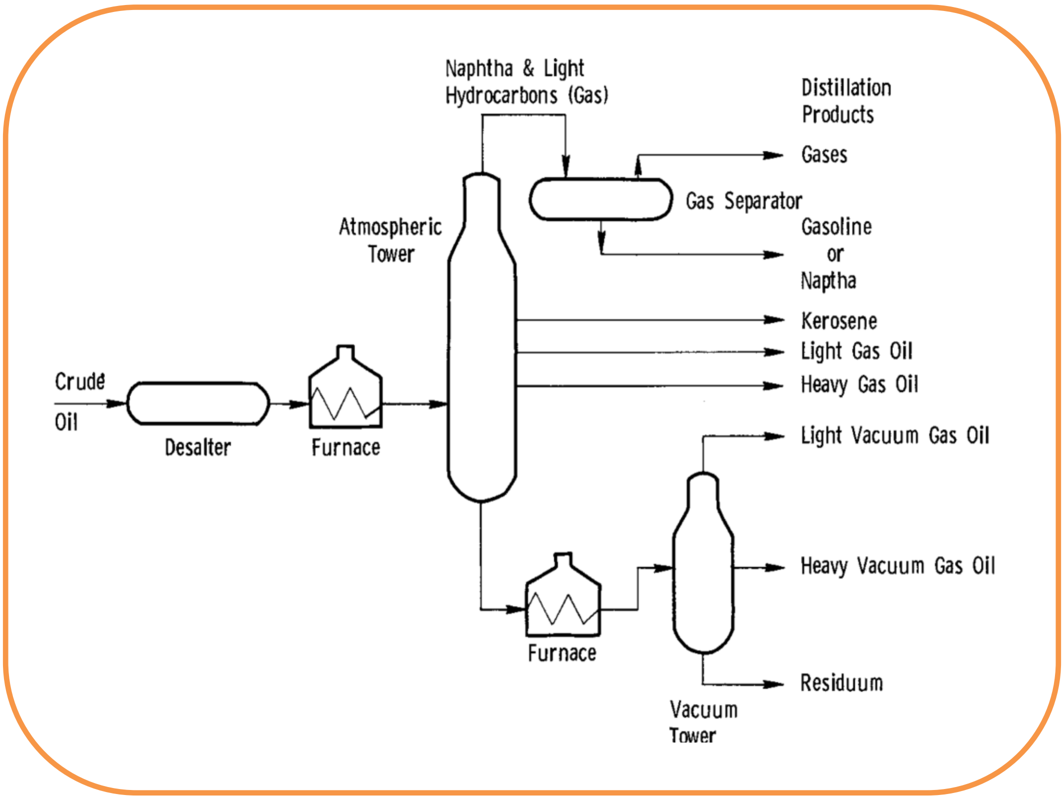


Figure (1.3) : A diagram showing the separation of crude oil components by distillation [17]

1.4 Lubricating Oils

1.4.1 Lubrication Phenomena and Oil Composition

Machinery is very important in our life and oil is said to play an important role in its working life . It can be said that oil is considered a blood for the machine like blood in our bodies . It doesn't only protect the machine but it provides with working condition [18] .The most important crude oil fraction is lubricating oil that is used in almost all vehicles and machines . Lubricating oils are used to decrease rubbing between surfaces in moving parts , after oxidation most impurities are generated in base oil during its application in internal combustion engine . This contamination contains unsaturation , phenolic compound , acidic compound , aldehyde , metals , additive, gums ,varnish and other asphaltic compounds originating from the overlay of bearing surfaces and degradation of the base oil components [19] . A lubricant oil can be defined as an oil product that divide the metal parts of an engine, reduce friction and keep it fresh [20] . Lubricating base oils consist of paraffinic , naphthenic and aromatic molecules with small amounts of sulfur, oxygen and nitrogen containing compounds inter-mixed within the three basic structures. Most molecules are combinations of two or three of the basic hydrocarbon types but are classified by their dominant properties [21] . Paraffins have good resistance to change in viscosity with temperature (high viscosity index) and best oxidation performance of all molecules used to blend lubricants. Naphthene ring structure with long side chains can have high viscosity index and good oxidation performance. Multi-ring structure with short side chains have low (VI) . Single ring aromatics with long side chains can have high viscosity index and good lube properties. Multi-ring and naphtheno-aromatics are generally considered to be poor base oil molecules due to poor oxidation performance and due to that their concentration is minimized during manufacture [22] . In all types of machines, the surfaces of moving or sliding or rolling parts rub against each other, due to the mutual rubbing of one part against another a resistance is offered to their movement. This resistance is known as (friction) . It causes a lot of wear and tear of surfaces of moving parts. So, the main purpose of lubricant is to keep the moving /sliding surfaces apart that friction and consequent destruction of material is minimized by the introduction of lubricants in between them ,the functions of lubricants oil are [23] to:

1) Reduce wear and tear of the surfaces by avoiding direct metal to metal contact between the rubbing surfaces by introducing lubricants between the two surfaces .

2) Decrease expansion of metal due to frictional heat and destruction of material

3) Act as coolant of metal due to heat transfer media .

4) Reduce power loss in internal combustion engines .

5) Lower maintenance cost .

1.4.2 Phenomenon of Blackness of Oils after Use

When the oil is used, its color changes throughout the period of use. The reason is that the improving material has taken its role in cleaning the engine from the carbon materials and keeping it stuck and dispersed in the oil until it is replaced. The blackness of the oil is proof of its quality and efficiency. The oil, which does not change color after use, does not function properly. There are many possible processes to regenerate base oil from used oil, such as , Purification using concentrated sulfuric acid , Purification using propane ,and Purification by hydrogenation [24] **.**

1.4.3 Recycling of Used Lubricating Oils

Used lubricating oil (waste oil) : is any lubricating oil whether refined from crude or synthetic components, which has been contaminated by physical or chemical impurities as a result of use. Lubricating oil loses its effectiveness during operation due to the presence of certain types of contaminants, these contaminants can be divided into: 1- extraneous contaminants 2- products of oil [25,26] . Oil is contaminated from several external sources:

A – Water : As a result of burning fuel consists of water vapor, which comes out of the majority of the exhaust cars, but a part of it condenses on the walls of the pistons and enters the oil portfolio and cause oil pollution.

B - Acids : Resulting from combustion of fuel as sulfur form sulfuric acid.

C- Fuel : (non-burning and incomplete combustion) causing oil pollution [24] .So, the quality of the oils decreases and the acidity, the mineral impurities and the water are increased . They are distinguished by changing their smell which becomes (strong and sharp) and their color change to dark .The oils used should be changed directly, because they do not cover the need for their use, and increase the appearance of impurities and corrosion in the engines and machinery , which hinders work and affects the machines [27]. Recycling of used lubricating oil deals with subjecting the oils to a series of processes that are able to eliminate most contaminants, including water, oxidation products, additives and thereby allowing the initial characteristics of the base oil to be re-established [28,29] . The unused oil contain (71-96 wt. %) base oil and the rest were additives [30] . There are many types of additives some of them contain metal and others are high molecular weight hydrocarbons. During automobile operation, some of the chemicals will be broken down or cracked to smaller molecules. It is essential to remove most sludge , carbon residue, ash content, and improve base oil color and increase viscosity index . Economics plays a great role in deciding the type of the recycling processes used and the operation condition of it . So, the process may vary from country to country. It depends on the type of origin base oil and type of oil required [31,32,33] .

1.5 Physical Properties of Lubricating Oils

Crude oil exhibits a wide range of physical properties and several relationships can be made between various physical properties [34] . For lubricating oil, the important one are :.

1- **Kinematic Viscosity**
2- **Viscosity Index**
3- **Specific Gravity**
4- **Pour Point**
5- **Flash Point**
6- **Color**
7- **Carbon Residue**
8- **Ash Content**
9- **Metal Content**

1.5.1 Kinematic Viscosity

Viscosity is defined as the property of a fluid that causes it to resist . It may be visualized as a result of physical interaction of molecules when subjected to flow [35]. It can be considered as one of the most important tests performed on the lubricating oil . The increase in viscosity value gives the indication of the presence of contaminant (deterioration additives and oxidation products) , while the presence of fuel contaminants leads to reduce the value of viscosity [25,36] . Lubricating oils have long chain hydrocarbon structures, and viscosity increases with chain length [35] **.** Viscosity is a state function of pressure, temperature and specific gravity . There is an inverse relationship between viscosity and temperature. When the temperature of the engine oil decreases the viscosity increases and vice versa . Oxidation of motor oils during use in an engine environment produces corrosive oxidized products and deposits which lead to an increase in the viscosity [37] . In the centimetre-gram-second system (**cgs**) unit , it is expressed in terms of (gm cm/sec or poise) .Usually it is expressed in centipoise (cP), where : (1 poise = 100 cP) ,in SI ,unit viscosity is expressed as (pascal. sec), while the term (centistoke) is more popularly used as the measure of viscosity, which in fact is the kinematic viscosity [2] .

1.5.2 Viscosity Index (VI)

Viscosity Index is strictly an empirical number and indicates the effect of change in temperature on viscosity. High viscosity index indicates a small change in viscosity with temperature , which also means better protection of an engine that operates under vast temperature variations. Viscosity index improvers are among the common additives that improve the efficiency of the oil . However, engine oil with a high addition level of viscosity index improvers tends to degrade more rapidly. High viscosity index is due to the absence of aromatic and volatile compounds, it also means good thermal stability and low temperature flow behaviors [38]. The VI test is usually done using viscosity .The accuracy of this method is dependent on the accuracy of the kinematic viscosity value determination at 40 ˚C and 100 ˚C [39] **.** So , if the viscosity of lubricating oil falls rapidly as the temperature is raised, it has a low viscosity index . While the viscosity of lubricating oil is only slightly affected on raising the temperature, its viscosity index is high [23] .

1.5.3 Specific Gravity and API°

Specific gravity is the ratio of the density of the material to density of the equal volume of water. The temperature at which the specific gravity is been measured must be known for specific gravity changes as temperature changes[40]. The other defined for specific gravity (the ratio of the mass of a given volume of liquid at 15 °C to the mass of an equal volume of pure water at the same temperature) and relative density (same as specific gravity) , are important properties of crude oil products as they are a part of product sales specifications although they only play a minor role in studies of product composition . while , density (the mass of liquid per unit volume at 15°C) . Usually a pycnometer , hydrometer or digital density meter is used for the determination in all these standards [14]. Specific gravity is influenced by the chemical composition of the oil . An increase in the amount of aromatic compounds in the oil results in an increase in the specific gravity, while an increase in the saturated compounds results in a decrease in the specific gravity [41] . The specific gravity of mineral oils also varies from (0.86 to 0.98). Specific gravity of water is 1.00 at 15.6 ˚C ,while specific gravity decreases with increased temperature and decreases slightly as viscosity decreases for similar compositions [42] . API° is defined as the most common measurement performed on crude oil products. Specific gravity is expressed in terms of API° gravity. This measurement determines the weight of a crude oil per unit volume at 60 °F, and it is calculated from specific gravity using the following equation :

$$\textbf{API gravity (degrees)} = (141.5/\text{sp gr } 60/60\ ^\circ\text{F}) - 131.5 \qquad \text{......(1-1)}$$

,and is also a critical measure for reflecting the quality of crude oil . So, the higher the specific gravity lower is the API° value [43] .

1.5.4 Pour Point

Pour point is defined as the lowest temperature at which the oil will just flow under standard test conditions [25] . And for of the lubricating oil it is usually low (below 0 ˚C) . This feature is very important because it keeps the oil from ablution or coagulation when used in cold operating conditions [44] . Most engine base oils contain waxes and paraffins that solidify at cold temperatures. Engine oils with high wax and paraffin content will have a higher pour point. The pour point is highly affected by an oil’s viscosity and engine oils with high viscosity are characterized by having high pour points. The pour point of an engine oil is an important variable, especially , when starting the engine in cold weather. So, the oil must have the ability to flow into the oil pump and then be pumped to the various part of the engine , even at low temperatures [45] .

1.5.5 Flash Point

Flash point is the lowest temperature to which the oil must be heated under specified conditions to give off sufficient vapor to form a mixture with air that can be ignited spontaneously by a specified flame . The flash point of engine oil is an indication of the oil’s contamination. A substantially low flash point of an engine oil is a reliable indicator that the oil has become contaminated with volatile products such as gasoline . The flash point is also an aid in establishing the identity of a particular crude oil product. The flash point increases with increasing molecular mass of the oil . Oxidation would result in formation of volatile components which leads to decrease the flash point [46,47, 48] . Flash point is used to indicate fire hazard of crude oil products and evaporation loses under high temperature loses. It gives us the idea about the maximum temperature below which the oil can be used . It is used as the means of identification of specific lubricating oil for detection of contamination in the given lubricating oil [49] .

1.5.6 Color

Different types of engine oil have different types of color . Color is used to estimate the engine oil quality . The color of used engine oil will change after certain time [50] . Determination of the color of crude oil products is used mainly for manufacturing control purposes and is an important quality characteristic. In some cases, the color may serve as an indication of the degree of refinement of the material [14] .The color of the lubricating oil indicates the uniformity of a particular grade or brand [25] .

1.5.7 Carbon Residue

Carbonaceous residue remains after combustion and measures used oil or fuel oil . During service , lubricating oil produces carbon residue precursors in the form of polynuclear aromatic systems that were not present in the original lubricating oil . However, these polynuclear aromatic systems cannot be detected and typically remain soluble in the used oil. Depending on the character of the fuel oil to be blended with the used lubricating oil , the polynuclear aromatic systems may separate out as a solid phase with the potential of interfering with the performance of the injector nozzles. As a component of a high carbon residue, the polynuclear aromatic systems can be part of the cause of rapid carbon buildup and nozzle fouling [25]. Carbonaceous residue remaining after thermal decomposition of engine oil in a limited amount of air is also called coke or carbon forming tendency. Also, the test for carbon residue can be used at the same time to evaluate the carbonaceous depositing characteristics of engine oils used in internal combustion engines. The carbon residue value of engine oil is regarded as indicative of the amount of carbonaceous deposits engine oil would form in the combustion chamber of an engine and now considered to be of doubtful significance due to the presence of additives in many oils. For example, an ash forming detergent additive can increase the carbon residue value of engine oil yet will generally reduce its tendency to form deposits [51] .

1.5.8 Ash Content

Combustion ash is the inorganic residue that remains after the combustion of oil in air at a specified, high temperature , lubricating oil do not contain ash but they do contain ash precursors. Ash forming materials found in heavy fuel oil are normally derived from the metallic salts and organometallic compounds found in crude oils , on the other hand used lubricating oil typically contains metals and other ash forming constituents produced in the oil during service . Unless such materials are removed , ash formation will ensue during combustion and depending on the use of the fuel oil and used lubricating oil . Ash composition can have a considerable bearing on whether detrimental effects will occur [52,53,54,55] .

1.5.9 Metals Content

Many oil constituents contain metallic elements that have been added to enhance the oil’s efficiency. In general, metals in engine oils are regarded as contaminants that should be removed completely in order to produce suitable base oil for producing new virgin oil [56] . Metals are regarded as heteroatoms found in engine oil mixtures . The amounts of metals are in range of a few hundred to thousands of ppm and their amounts increase with an increase in the boiling points or decrease in the API° gravity of the engine oil , where metallic constituents are associated with heavy compounds and they mainly appear in the residues . Base engine oils have very little metal content, which indicates their purity. Some metals present in virgin oils in high concentrations are in the form of various additives which improve the performance of the engine oil ,many others are introduced in to the oils after using due to depletion of various additives and dilution of the engine oil with fuel containing metal additives [57]. Lubricating oils consist of a base of mineral or synthetic oil and several substances added in order to enhance different properties of the product [58] .

1.6 SAE Viscosity Grading System

Lubricating oils are identified by Society of Automotive Engineers (SAE) number, the SAE viscosity numbers are used by most automotive equipment manufacturers to describe the viscosity of the oil they recommend for use in their products [59] . (SAE) had established a viscosity grading system for engine oils according to the SAE viscosity grading system all engine oils are classified into monograde or multigrade ; Monograde engine oils are designated by one number (20, 30, 40, etc). The number indicates a level of the oil viscosity at a particular temperature. The grade numbering is directly proportional to the oil viscosity, viscosity of engine oils designated with a number only without the letter (W) (SAE 20, SAE 30, etc) is specified at the temperature (100°C) 212°F [60] . Viscosity of such engine oils is specified at both low and high temperature called multigrade oils, they are designated by two numbers and the letter (W) (SAE 5W-30, SAE 15W-30, SAE 20W-50, etc). The first number of the designation specifies the oil viscosity at cold temperature ,while the second number specifies the oil viscosity at high temperature. For example, SAE 15W-30 oil has a low temperature viscosity similar to that of SAE 15W, but it has a high temperature viscosity similar to that of SAE 30, this gives multigrade oils wide temperature range [61,62] .

1.7 Iraqi Lubricating Oil Produced by Middle Refineries Company

The Iraqi lubricating oils produced in middle refineries company AL-Daura refinery, and the one produced now are [24,63] :

1- Rasheed Oil : This oil is produced according to the America Petroleum Institute specifications and requirements of Mercedes Benz , are high-quality oils containing the appropriate proportion of detergents and improved materials that prevent oxidation and rust, these oils are used in gasoline engines and for a distance of 8,000 km. They are divided into :

A- Mono - Grade Rasheed motor oil : includes species (10W , 20W , 30W , 40W, 50W).

B- Multi- Grade Rasheed motor oil: includes species (15W- 40) .

2- Baghdad Oil (Multi- Grade Baghdad Motor Oil) : Includes type (15W-40 , 20W-50 **),** this oil is produced according to the American Petroleum Institute specifications and requirements of Mercedes Benz and European market specifications, is a high quality oils containing improved materials that prevent oxidation and rust and also contain a high proportion of detergents and dispersants that carry carbon atoms and make it stuck in oil and prevent the deposition on the parts of the machine, these oils use in gasoline engines and for a distance of 12,000 km .

1.8 Previous Studies

Hussian (2006) studied the thermal expansion of three types of Iraqi base lube oil -Stocks (namely 40 Stock , 60 Stock , 150 Stock and also lubricating oil with additives from Al -Dura refinery). The investigation also covers the effect of additives on the thermal expansion coefficient of a lubricating oil . Measurements were made on the density variation with temperature of the base oil and then blending the basic oil with 4% of poly isoprene and measure the density variation with temperature . Also, this study investigated the thermal expansion coefficient of base oil and additives currently used in Al- Dura refinery and compares that of the (base oil +4% poly isoprene). Finally, good correlation for calculating the thermal expansion coefficient of three base oil at different temperatures are obtained [64] .

Rassoul and Mahmmoud (2010) studied recycled waste oil by using vacuum distillation under moderate pressure and then extraction by using liquid solvents, namely n-butanol and n-hexane .They are used recovered base oil by using n-butanol solvent gives (93.73 %) oil recovery , (95 %) solvent recovery, (88.67 %) reduction in carbon residue, (75.93 %) reduction in ash content and (100.62) viscosity index, at (5:1) solvent to used oil ratio and 40 ˚C extraction temperature. While using n-hexane solvent gives (89.06%) oil recovery, (94.78 %) solvent recovery ,(60.25 %) reduction in carbon residue , (76.54 %) reduction in ash content and (100.3) viscosity index, at (6:1) solvent to used oil ratio and (50 ˚C) extraction temperature [65] .

Udonne (2011) studied four methods of recycling used lubrication oils being acid /clay treatment, acid treatment, distillation/clay and activated charcoal /clay treatment methods. The tests carried out on the recycled lubrication oil include: viscosity, specific gravity ,pour point, flash point and metal contents. The results from the tests showed that, viscosity increased from (25.5 cSt) for used lube oil to (86.2 cSt) for distillation , (89.10 cSt) for acid/clay treatment and (80.5 cSt) is for activated/clay treatment. This is compared with (92.8 cSt) for fresh lube oil and other results from the different tests showed varied degrees of improvement with the best results obtained using the acid/clay treatment [40] .

Udonne and Bakare (2013) investigated the effect of three different acids, hydrochloric acid , sulphuric acid and nitric acid , on three different clay deposit samples . The resulting oil was slightly dark in colour. Tests carried out were flash point , pour point and viscosity index , the results show increase in the flash point and viscosity index of the oil compared to the fresh lube oil [66] .

Sterpu et al. (2013) carried out re-refining a sample of (15W-40) used oil collected from one automobile. The process consists of dehydration, solvent extraction, solvent stripping and vacuum distillation. The process of solvent extraction an alcohol - ketone mixture as a pre- treatment step was followed by vacuum distillation at 5 mmHg. The primary step was conducted before the solvent extraction that involves dehydration to remove water and fuel contaminants from the used oil by vacuum distillation . The solvent extraction and vacuum distillation steps were used to remove higher molecular weight contaminants . The investigated solvent to oil ratios was 2, 3, 4, 5 and 6 . The solvent composition is 25% , 2-propanol , 50% ,1-butanol and 25% ,butanone or methyl ethyl ketone. The percentage oil recovery for the solvent to oil ratio of (6:1) is further improved, but for the ratio values higher than 6:1. So, the operation was considered economically not feasible [67] .

Hamawand et al . (2013) studied recycling of waste engine oils treated with acetic acid . A recycling process was developed which eventually led to comparable results with some of the conventional methods. The advantage of using acetic acid is that it does not react or only reacts slightly with base oils, and the recycling process can takes place at room temperature. It has been shown that base oils and oils additives are slightly affected by the acetic acid . Upon adding 0.8 vol. % of acetic acid to the used oil , two layers were separated , a transparent dark red colored oil and a black dark sludge at the bottom of the container .So, the comparison showed that the recycled oil produced by acetic acid treatment is comparable to those recycled by the other conventional methods [36] .

Isah et al .(2013) presented the result on the regeneration of used engine oil using industrial bleaching earth and activated carbon as the bleaching agents. The used oil undergoes acidification and bleaching steps to remove the aromatic content and to improve the color and quality of the oil. Analysis carried out showed that when the formulated (two method) grades were compared with standard grade (fresh oil). First method showed a better quality of regenerated oil amongst the different grades formulated with a viscosity of (53.16 cP) at 40 ˚C , specific gravity of 0.932 gm/cm^3 and total acid number of 0.54 using industrial bleaching earth. On the other hand , second method gave a fair quality using activated carbon with a viscosity of (40.41 cP) at 40 ˚C , specific gravity of 0.883 gm/cm^3 and total acid number of 0.59 [29] .

Danane et al .(2014) in their work on an experimental basis consists of an analysis and treatment of used motor oil collected by Naftal stored at the port of Algiers. The results of these analysis allowed them to identify the different stages of the process of regeneration including pre-treatment, metal removal by a chemical agent, a finishing treatment by passage through bentonite and then filtration . This process has enabled them to eliminate most of the impurities and to optimize different parameters, the engine oil obtained at the end of the process is a basic oil ready to be used again [68].

Daham (2015) studied recovery of used oil by solvent extraction and clay treatment where many affecting variables have been examined in the solvent extraction process, such as, solvent /used oil ratio, mixing speed ,temperature, and the solvent type. A statistical method central composite design was used in the solvent extraction process in order to study the effect of each variable on the amount of percent sludge removal (Sludge%) and to describe the effect of interaction between these variables. The optimum condition in extraction process were: solvent/ used oil ratio of 2.4 and 3.12 vol / vol , temperatures of (54 and 18) ˚C and mixing speeds of 569 and 739 rpm for 1-butanol and methyl ethyl ketone respectively. In the clay adsorption process, the variables that have been investigated were clay / oil ratio, temperature, contact time and the type of clay. The best conditions in the adsorption process were:. Clay/ oil ratio being 15 wt/vol % , temperature, 120 ˚C and contact time ,90 and 120 minutes for bentonite and attapulgite respectively. Finally tests were measured for recovering base oil to compare it with the Iraqi standard base oil [69] .

Abu-Elella et al . (2015) investigated the treatment of waste motor oils using four different acids (sulphuric acid, acetic acid , phosphoric acid, and formic acid) followed by treatment with clay. The effect of these treatments on the properties of the treated used oil was studied .Tests include flash point , specific gravity and kinematic viscosity. The results showed that the formic acid-clay treatment improve the flash point of the used motor oil and made it comparable with fresh motor oil and the sulphuric acid, acetic acid and formic acid followed by clay treatment improved the kinematic viscosity of the oil while the treatment with phosphoric acid-clay has no improvement action on the used oil flash point and kinematic viscosity [70]

Aremu et al . (2015) studied three solvents:. 1-butanol , 2-propanol and mixtures of 1-butanol- ethanol to segregate impurities from spent lube oils in the form of sludge at different extraction factors. The performances of the solvents were investigated by determination of percent sludge removal and percent oil loss. The investigation revealed that 1-butanol produced the best extraction performance [71] .

Yash (2015) studied used engine oil which contains a number of additives and is contaminated by impurities and residues resulting from the combustion process. Some of them are poisonous and carcinogenic like lead and poly aromatic hydrocarbons, also the oils used in transformers contains poly chlorinated biphenyls, which are highly carcinogenic as well. From various sources such kind of used lubricating oils are generated and are disposed improperly. The burning of used oil in kilns and in generator produces lots of ash and carcinogens causing environmental pollution. Re-refining Process which involved dehydration, vacuum distillation , solvent extraction and final atmospheric distillation , the objective of re-refining is to remove the degraded additives and contaminants and to restore the properties of the oil identical to the standards provided [72] .

Uthman (2017) studied ten samples of spent lubricating oil obtained from different road side in Minna / Nigeria. Five samples were collected from both premium motor spirit and automotive gas oil engines respectively. Samples were analysed using atomic absorption spectrophotometer (AAS) . Metals content average concentration (ppm) , were as follows:. (1.1 and 1.43 Lead) ,(4.78 and 10.10 Iron) ,(0.39 and 0.16 copper) ,(10.50 and 17.43 zinc) ,(2.40 and 3.83 chromium), (0.633 and 0.60 magnesium) and (135.39 and 128.06 calcium). Results showed that copper and magnesium were the least concentration while calcium has the highest one . These spent lubricating oil was recycled using locally sourced soap (surfactant) and tetra oxo sulphate acid , H_2SO_4, producing cleaner spent lubricating oil suitable for combustion in industrial burner. Recycled spent lubricating oil was found to have a specific gravity of 0.8972 gm/cm^3 , carbon residue of 3.89 % , water content of 10 % wt and a flash point of 127 ˚C . It can be inferred from various analyses conducted that the method employed is efficient and suitable for spent lubricating oil treatment [73] .

Hegazi et al. (2017) studied recycling of waste engine oils treated by (acetic acid and formic acid),the advantage of using the (acetic acid or formic acid) is that it does not react or only reacts slightly with base oils, the recycling process takes place at room temperature. It has been shown that base oils and oils' additives are slightly affected by the acetic acid. Upon adding (acetic acid or formic acid) to the used oil , two layers were separated , a transparent dark red colored oil and a black dark sludge at the bottom of the container ,the recycled oil produced by acetic acid and formic acid treatment showed excellent results compared to fresh oil. Using volumetric ratio of 10:1 oil to acetic acid result in density of 0.87 g/cm^3 and this is close to those of fresh oil mean while using ratio of (10:1) oil to formic acid result in viscosity value being (30 cP) which is also equivalent to value of fresh oil [74] .

Osman et al. (2018) examined the refining of lubricating oils from waste lubricating oil utilizing a novel blend of solvent extraction and activated alumina adsorbent. The activity of these solvent extraction blends: (toluene, butanol and methanol (A)) , (toluene, butanol and ethanol (B)) and (toluene, butanol and isopropanol (C)) was evaluated experimentally, oil to solvent proportions from 1:1 to 1:3 were analyzed for mixture blend (C). The results confirm that solvent mixture (A) gave good efficiency with the highest percent sludge removal , the maximum percent of sludge removal improves with the increase of solvent to oil ratios . The results show the change in the properties of recycling oil with good efficiency [75] .

Oladimeji et al. (2018) studied three different types of solvents ,two single solvents and a composite one .The single solvents used were methyl ethyl ketone and propan-2-ol while the composite solvent comprised of 75% MEK and 25% propan-2-ol . The performance of the three solvents chosen were studied and compared based on certain parameters . It was observed that (MEK) had the best performance because it gave the highest sludge removal and closest properties to the fresh lubricating base stock. It was also determined that increase in temperature improved the quality of oil obtained up till 50 ˚C ,above this temperature poorer quality of oil was observed . They concluded that solvent to oil ratio has a greater effect on the quality of oil produced after treatment [39] .

Yousef and Ali (2018) studied extraction of aromatics from multi-component hydrocarbon mixtures by using high density, high polar, and high boiling point solvents such as, dimethyl sulfoxide ,N- methyl pyrrolidone , N-formyl morpholine , glycols, and sulfolane .They studied operating conditions are:. extraction temperature of 70 ˚C, settling time of 30 min , mixing time of 15 min , solvent to oil ratio range from 1:1 to 2:1 (wt./wt.) and co-solvent concentration from 0% to 20%. The results of the investigation show the best co solvent used is formamide: N-methyl pyrrolidone co-solvent with 15% concentration, since it gives acceptable refractive index of 1.4546 and acceptable sulfur content of 0.51wt.% [76] .

Hatami et al. (2018) studied **t**he effects of possible factors such as temperature , solvent to clay ratio, time, aggregation size and rotation speed of the stirrer (degree of mixing) on the efficiency of extracting lubricating oil by solvent extraction .They concluded that the most important factor in obtaining the appropriate output was solvent to clay ratio .The tests conducted to investigate the effect of grain size on the efficiency indicated that agglomerates size did not have a positive effect on efficiency. The solvent to clay ratios ranging from 2.48- 9.53 ml /g and a time period ranging from (5 to 40) minutes and the best efficiency was obtained at the highest level of solvent to clay ratio of 9.53 ml/g and at the time of 22.5 minutes that led to 88.60 % oil extraction from the clay and the accuracy of the model output was estimated to be 96% [77] .

1.9 Aims of Study

1) The Evaluation of the physical properties of used lubricating oil (type 15W-40 Baghdad) ,which is produced by the Middle Refineries Company / AL- Daura Refinery , for a distance of (1000, 1500 and 2000) km. The used lubricating oils are collected directly from small saloon car, (Mercedes-Benz-Internal Combustion Engine) at different operating conditions. The study was conducted during november and december of 2018 .
2) The Evaluation of the physical properties of new oil , used oil , and recycled oil , including kinematic viscosity , VI , specific gravity , API°, pour point , flash point , color , carbon residue, ash content , and analysis of some metals for all oil samples .
3) Recycling the oil used for a distance of 2000 km by using vacuum distillation and extraction processes by using different solvents such (hexane , methanol ,2- propanol) .

Chapter Two

Materials & Methods

2.1 The Material Used

A- The chemical materials used are given in table (2.1) below :

Table (2.1): Chemical materials used

	Materials	**Origin**	**Function**	**Purity%**
1	**Hydrochloric acid (HCl)**	**India**	**Solvent**	**36**
2	**Nitric acid (HNO_3)**	**India**	**Solvent**	**72**
3	**2-Propanol (C_3H_7OH)**	**United Kingdom**	**Solvent**	**99.7**
4	**Methanol (CH_3OH)**	**Belgium**	**Solvent**	**99.8**
5	**n-hexane (C_6H_{14})**	**Belgium**	**Solvent**	**95**

B- Oils used

Virgin Baghdad oil type (15 W – 40) produced by the middle refineries company / Daura refinery and those from the same oil used for a distance of (1000 , 1500 and 2000) km were used in the study .

2.2 The Instruments Used

The instruments used are shown in table (2.2) .

Table (2.2): Instruments used

Instrument Name	The Company and Model	Location
Viscometer	**German origin . HVU 481**	**The ministry of science and technology – oil and chemicals directorate**
Hydrometer	**USA origin. Fisher brand , CAT.NO.11-5838**	**College of Science, University of Diyala**
Pour point	**ISL CPP5Gs , instrument able to determine pour point for all sample types at ultra-low temperatures below -90 ˚C (-130 ˚F)**	**Ministry of oil –Oil research and development center**
Cleveland open cup apparatus	**USA origin. Koehler instrument company , Model K13990 , Volt/Hz 230V/50-60Hz**	**College of Engineering University of Diyala**
Petroleum Color	**USA origin . Koehler instrument company , Petroleum AF650**	**College of Engineering , University of Diyala**
Bunsen Burner	**Korea origin . JEFLEX AIR HOSE 10 mm W/P 20BAR B/P 6OBAR ISO-2398**	**College of Science, University of Diyala**

Furnace	**German origin . 30-3000 ˚C**	**College of Science, University of Diyala**
Hot plate	**Korea origin . model LMS-1003**	**College of Science, University of Diyala**
Electronic Balance	**KERN & Sohn GmbH, Type ACS 120-4, NO. WB12AE0308 ,CAPACITY 120g, READABILITY 0.1mg.**	**College of Science, University of Diyala**
Atomic Absorption spectrophotometer	**AA-7000 SHIMADZU**	**College of Education for Pure Science / Ibn Al-Haitham University of Baghdad**
FTIR	**Japan origin . Affinity-1, SHIMADZU .**	**College of Education for Pure Science , University of Diyala**
UV/VIS Spectrophotometer	**Japan Origin . SERIAL NO.B 093961150 V-650**	**College of Science, University of Diyala**
Vacuum distillation unit	**Local assembly**	**The ministry of science and technology – oil and chemicals directorate**

2.3 Characterization of Oil Samples

There are a series of tests which were done for the new ,used and recycled lubricating oils . The aim of the analysis was to evaluate the properties of them .

2.3.1 Determination of Kinematic Viscosity, ASTM D-445

Viscometer was used to determine the kinematic viscosity of new, used and recycled oils .The viscometer tube was charged with the required amount of oil sample (10 ml) and then placed in the oil bath and was left to attain the desired temperature (40 ˚C and 100 ˚C) , and then the kinematic viscosity was calculated by measuring the time needed for 10 ml lubricant oil to flow under gravity through the capillary tube of the viscometer. The viscosity was then calculated using the viscometer constant which is 0.5 cSt /sec as in ASTM D-445 [78] , Fig (2.1) show the device of the kinematic viscosity measurement .

Kinematic viscosity = c × t ……….(2-1)

Where:

c = Viscometer constant (0.5 cSt /sec)

t = Time (sec)

Figure (2.1) : Device of the kinematic viscosity measurement

2.3.2 Viscosity Index calculation, ASTM D-2270

For calculating the viscosity index (VI) of lubricating oils from their kinematic viscosities at (40 ˚C and 100 ˚C) , as in ASTM D-2270 [79] .

If the kinematic viscosity of the oils at 100 ˚C is less than or equal to 70 mm^2/s (cSt), extract the corresponding values for L and H, and the viscosity index is calculated as below :

A- If U > H , calculate the viscosity index of the oil as follows:

$$VI = [(L - U) / (L - H)] \times 100 \quad \dots\dots\dots (2\text{-}2)$$

L = Is kinematic viscosity at 40 ˚C of an oil of 0 viscosity index having the same kinematic viscosity at 100 ˚C as the oil whose viscosity index is to be calculated, mm^2/s (cSt) .

H = Is kinematic viscosity at 40 ˚C of an oil of 100 viscosity index having the same kinematic viscosity at 100 ˚C as the oil whose viscosity index is to be calculated mm^2/s (cSt) .

U = Is kinematic viscosity at 40 ˚C of the oil whose viscosity index is to be calculated mm^2/s (cSt) .

B- If U < H , calculate the viscosity index of the oil as follows:

$$VI = [((\text{antilog } N) - 1) / 0.00715] + 100 \quad \dots\dots (2\text{-}3)$$

where:

$$N = (\log H - \log U) / \log Y \quad \dots\dots (2\text{-}4)$$

or

$$Y^N = H / U \quad \dots\dots (2\text{-}5)$$

Where: **Y** = Is kinematic viscosity at 100 ˚C of the oil whose kinematic viscosity is to be calculated, mm^2/s (cSt) .

N= power required to raise the kinematic viscosity of the oil at 100 ˚C to equal the ratio of the H and U kinematic viscosities at 40 ˚C .

C- If U= H , the viscosity index of the oil equals 100 .

2.3.3 Determination of Specific Gravity, and API˚ Gravity , ASTM D- 1298

A calibrated glass hydrometer with specific gravity range of (0.800-0.910) and glass cylinder 500 ml was used. The cylinder is partially filled with the oil sample and then hydrometer is immersed into the oil and allowed to settle vertically and the specific gravity is measured from the graduations of the hydrometer to surface of the oil. The temperature of the oil is set at 15 ˚C (60 ˚F) [80] .The API˚ gravity can be calculated according to the equation mentioned in the chapter one , section (1.5.3) . Fig (2.2) show the setup of specific gravity and API˚ gravity hydrometer experiment **.**

Figure (2.2) : Hydrometer for specific gravity measurements

2.3.4 Determination of Pour Point, ASTM D-97

For pour point determination, (50) ml of the lubricating oil sample was placed into a container and then electrode fixed the inside sample and cooled , until lube oil stop to flow where the temperature is then recorded as a pour point according to ASTM D-97 [81] .

2.3.5 Determination of Flash Point, ASTM D-92

A sample of 70 ml of lubricating oil was put into a test cup of Cleveland open cup apparatus. The temperature of the test sample is increased rapidly at first and then at a slower heating rate until the flash point is approached. The lowest temperature of the ignition caused the vapor above the surface of the liquid to ignite was noted and recorded as the flash point [82, 83]. Fig (2.3) shown device of the flash point measurement used .

Figure (2.3) : Device of the flash point measurement

2.3.6 Determination of Color , ASTM D-1500

There are three cylindrical glass sample jars with an internal diameter of 33 mm .The central jar is half filled with the oil sample and the outer ones are filled with distilled water. The sample is viewed through a prism which brings the sample and the standards into adjoining fields of view. The two discs containing the color standards are rotated by turning the control knobs on the front of the comparator until the color of the oil sample falls between two standards which are 0.5 apart, or until it exactly matches one of the standards. The reading is given directly as ASTM color is then taken from the scale on the disks. [84], as follows :

1- If the color of the sample, the designation of the glass producing a matching color ,for example: 4 ASTM color .

2- If the color of the sample is between those of two standard glasses{the sample is less than the darker glass designation} , so ,report the designation of preceded by the letter (L) ,for example: L 5-5.5 ASTM Color .

3- If the sample is darker than 8 color, one may either stop the test and report (D8 ASTM Color) or continue the test and mix 15 volumes of sample into 85 volumes of solvent kerosine or alternative material and observe the color of the mixture ,and report the color of the mixture followed by the dilution (Dil) ,for example: L 5 Dil ASTM Color. Fig (2.4) show device of the color.

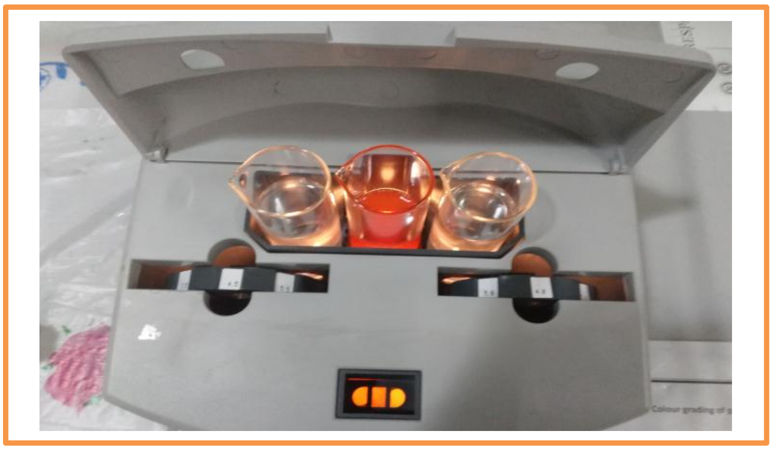


Figure (2.4) : Device of the color measurement

2.3.7 Determination of Carbon Residue and Ash Content of Crude Oil Products, ASTM D- 482

2 gm of lubricant oil sample was weighed in a suitable crucible and carefully heated and ignited by flame until leaving a carbonaceous material only. Then , the carbon residue was calculated [85].Fig (2.5) shows oil sample in crucible before heating , and Fig (2.6) shows the carbon residue after being heated by Bunsen burner **.**

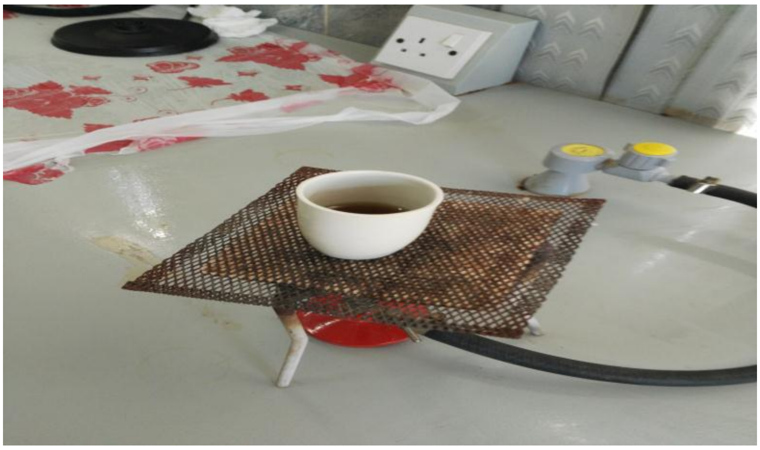


Figure (2.5) : Oil sample in crucible before heating

After carbon residue obtained , it was then heated in a furnace at 775±25 ˚C until the carbonaceous material disappeared . The crucible was removed from the furnace and left to cool at room temperature ,and the ash content was calculated [86] .

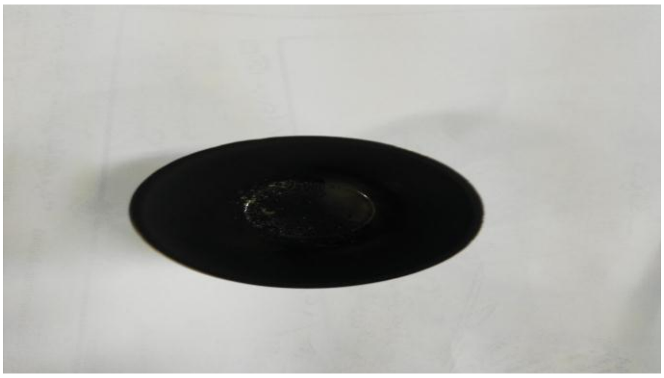


Figure (2.6) : The carbon residue after being heated by Bunsen burner

2.3.8 Determination of Metals Content in Lubricating Oil

Flame atomic absorption (AA-7000 SHIMADZU) was used for the determination of metals content in lubricating oil . The ash inside crucible thus obtained in section 2.3.7 is dissolved in aqua regia solution which consists of 1 volume HNO_3 and 3 volume HCl , 50 ml was put on hot plate and heated until the volume of solution is reduced to half, then distilled water was added and the processes repeated 3 times and finally the solution was stored to 250 ml volumetric flask and completed to the mark with distilled waters .Then , the determination of metals content (Iron , copper , magnesium , zinc and manganese) in new , used and recycle lubricating oil was done by using flame atomic absorption spectrophotometer and then convert the percentage of metals in these solution to those in oils according to the following equation :

$$\textbf{Metal content of oil, ppm} = \frac{\textbf{250 gm} \times \textbf{metal concentration in solution(ppm)}}{\textbf{2 gm}} \quad \textbf{.....(2.6)}$$

Where:
250 gm = 250 ml , because the density of water = 1
2 gm = weight of lubricant oil sample

2.4 Fourier Transform Infrared Spectroscopy (FTIR)

Infrared spectra of prepared compounds and complexes were recorded in (KBr) disc by using PERKIN ELMER SPEACTRUM -65/ Germany in the range (400 – 4000) cm^{-1} . The IR spectra was measured for samples of new , used and recycled oils .

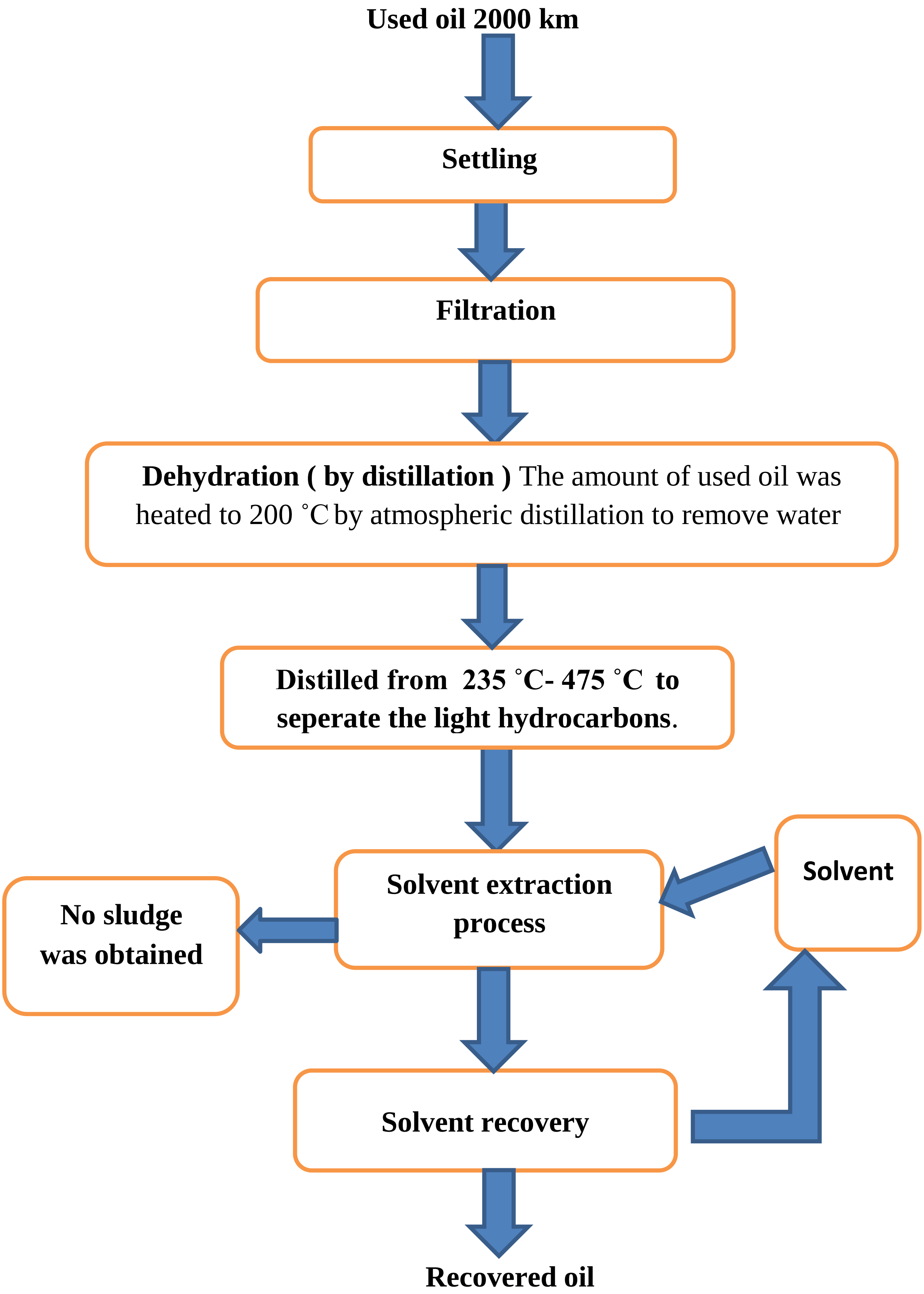
2.5 UV Visible Spectrophotometer

UV spectra were measured for new , used and recycled oils with 1 cm quartz cell ,using n-hexane as standard reference solvent .

2.6 Recycling the Lubricating Oil Used

2.6.1 Re-Refining the Used Oil (2000 km) Experimental Procedure

The used lubricating oil was recycled as in the following stages:.



Dehydration (by distillation) The amount of used oil was heated to 200 ˚C by atmospheric distillation to remove water

Distilled from 235 ˚C- 475 ˚C to seperate the light hydrocarbons.

2.6.2 Settling

The first step in the recycle process of used lubricating oil is settling by gravity to remove solid contaminants .

2.6.3 Filtration

The second step in the recycling process of used lubricating oils was filtration . It was carried out by the filter paper to remove some impurities and additives that formed as a result of exposure to high temperatures .

2.6.4 Dehydration (Vacuum Distillation)

1250 ml of used lubricating oil at 2000 km was put in vacuum distillation unit to remove water and light fractions, and we obtained (600 ml) recycled oil and (650 ml) residue of distillation. The light component in the used lubricating oil distilled off because their presence changes the lubricating oil properties. The amount of used oil was heated at atmospheric pressure [87] to remove water and distilled up to 200 ˚C and then under vacuum pressure to eliminate the light hydrocarbon [88] . Fig (2.7) show samples of used lubricating oil and oil obtained after distillation.

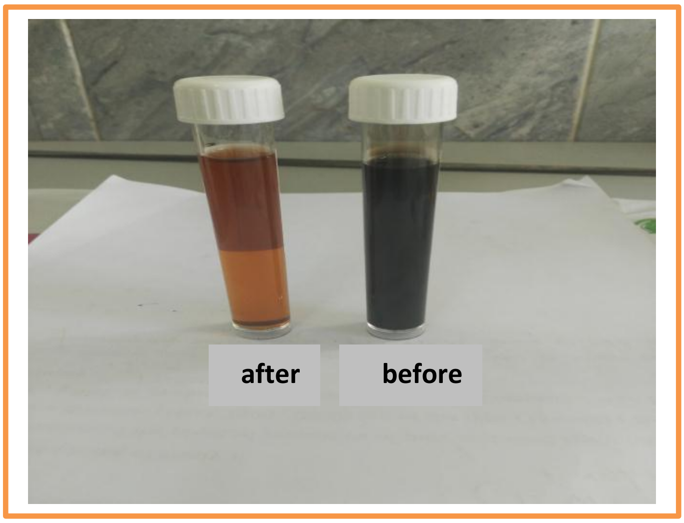


Figure (2.7) : The used oil and oil after distillation samples

2.6.5 Solvent Extraction Process

Solvent extraction: solvent such as methanol and 2-Propanol are polar solvents and n-hexane are non –polar solvent used in the solvent extraction process for recycle used lubricating oil from vacuum distillation . The extraction method was used to remove the remaining contaminants if present in oils obtained from vacuum distillation process. There are two methods used to extract impunities present in vacuum distilled oil.

1- The first method [39,89,90]

A- First experiment : 5 ml of oil sample was mixed with 10 ml (n- hexane) at ratio (1:2), the oil and the solvent are then stirred for 30 min to ensure that the mixture is adequately mixed using an electric hot plate at a speed of 500 rpm . Then, The mixture was allowed to settle at a constant temperature for (48 hour) in a separating funnel **.**

B- Second experiment :. 5 ml of oil sample was mixed with 10 ml (2- propanol) at ratio (1:2) , the oil and the solvent are then stirred for (30 min) to ensure that the mixture is adequately mixed using an electric hot plate at a speed of 500 rpm . Then, The mixture was allowed to settle at a constant temperature for (48 hour) in a separating funnel .

C- Third experiment :. 5 ml of oil sample was mixed with 10 ml (75% 2- propanol + 25% methanol) at ratio (1:2) , the oil and the solvent are then stirred for (30 min) to ensure that the mixture is adequately mixed using an electric hot plate at a speed of 500 rpm . Then, The mixture was allowed to settle at a constant temperature for (48 hour) in a separating funnel, as shown in Fig (2.8) .

Figure (2.8) : The oil after distillation with solvents

2- **The second method** : In this method we use increasing ratio of the solvent to used oil and at temperature of 40 ˚C , because the results from previous studies indicated that at (40 ˚C) gave best oil recovery , and increasing the solvent to used oil ratio will increase the percent of oil recovery **[65,91]** , as well .

A-First experiment : 5 ml of oil sample was mixed with 30 ml (n- hexane) at ratio (1:6), the oil and the solvent are then stirred for (1 hour) to ensure that the mixture is adequately mixed using an electric hot plate at a speed of 500 rpm and at temperature (40˚C) . Then, The mixture was allowed to settle at a constant temperature for (48 hour) in a separating funnel .

B- Second experiment :. 5 ml of oil sample was mixed with 30 ml (2- propanol) at ratio (1:6) , the oil and the solvent are then stirred for (1 hour) to ensure that the mixture is adequately mixed using an electric hot plate at a speed of 500 rpm and at temperature (40 ˚C) . Then, The mixture was allowed to settle at a constant temperature for (48 hour) in a separating funnel .

C- Third experiment :. 5 ml of oil sample was mixed with 25 ml (75% 2- propanol + 25% methanol) at ratio (1:5) , the oil and the solvent are then stirred for (1 hour) to ensure that the mixture is adequately mixed using an electric hot plate at a speed of 500 rpm and at temperature (40 ˚C) . Then, The mixture was allowed to settle at a constant temperature for (48 hour) in a separating funnel, as shown in Fig (2.9) .

Figure (2. 9) : The oil after distillation with solvents

2.6.6 Solvent Recovery

Solvent recovery and type are an important factors that must be consider to select the best economic solvent extraction. The solvent recovery was done by distillation unit and taking into consideration the boiling point of all solvents used .

Chapter Three

Results & Discussion

This chapter contains the results obtained from the analysis of samples for the new oil, used and recycled oils, for Baghdad oil of the type (15W-40) , which is produced in the middle refineries company / Daura refinery . Evaluations of the results are also made by comparing the measured values with other researchers, and according to ASTM specification .

3.1 Characterization of Lubricating Oil Sample.

The new, used and recycle oils were subjected to a series of tests, especially with regards to the performance and its characteristics**,** as follow:

3.1.1 The kinematic viscosity

The kinematic viscosity at (40 ˚C and 100 ˚C) of the used oils was observed to be lower if compared to those of the new oil because the viscosity of the used oil decrease with oil use in the engine, which results in impurities and the formation of light hydrocarbon as a result of high temperature [74]. Viscosity of the recycle oil was less than the used oils, as shown in table (3.1) at both temperatures , this decrease in viscosity values is due to removal of the non-metallic polymeric materials which was added as viscosity index improvers and other contaminants presents [69] , which means that the method are effective in removing the oxidized products, deposits, and varnishes from the used oil [37]. This may also mean removing the additives which have been usually added to enhance the viscosity [36] . The viscosity index of lube oil will also change as a result of temperature changes and thermal degradation leading to chain scission and dilution with fuel amongst other [66] .The viscosity index of new oil (134.58) decrease in used oils of 1000 km and 1500 km to (123.63 and 118.88) respectively , and becomes 133.70 at 2000 km use resulted from a high decreases of the kinematic viscosity of oil use at 2000 km . as shown in Fig (3.1) .

Table (3.1): Kinematic viscosity according to ASTM D-445 and viscosity index according to ASTM D-2270

Type of oil	Kinematic Viscosity at 40 ˚C (cSt)	Kinematic Viscosity at 100 ˚C (cSt)	Viscosity index (VI)
New	110.06	14.536	134.58
Used 1000 km	109.51	13.73	123.63
Used 1500 km	101.98	12.792	118.88
Used 2000 km	82.59	11.789	133.70
Recycled oil	21.27	4.335	108.25

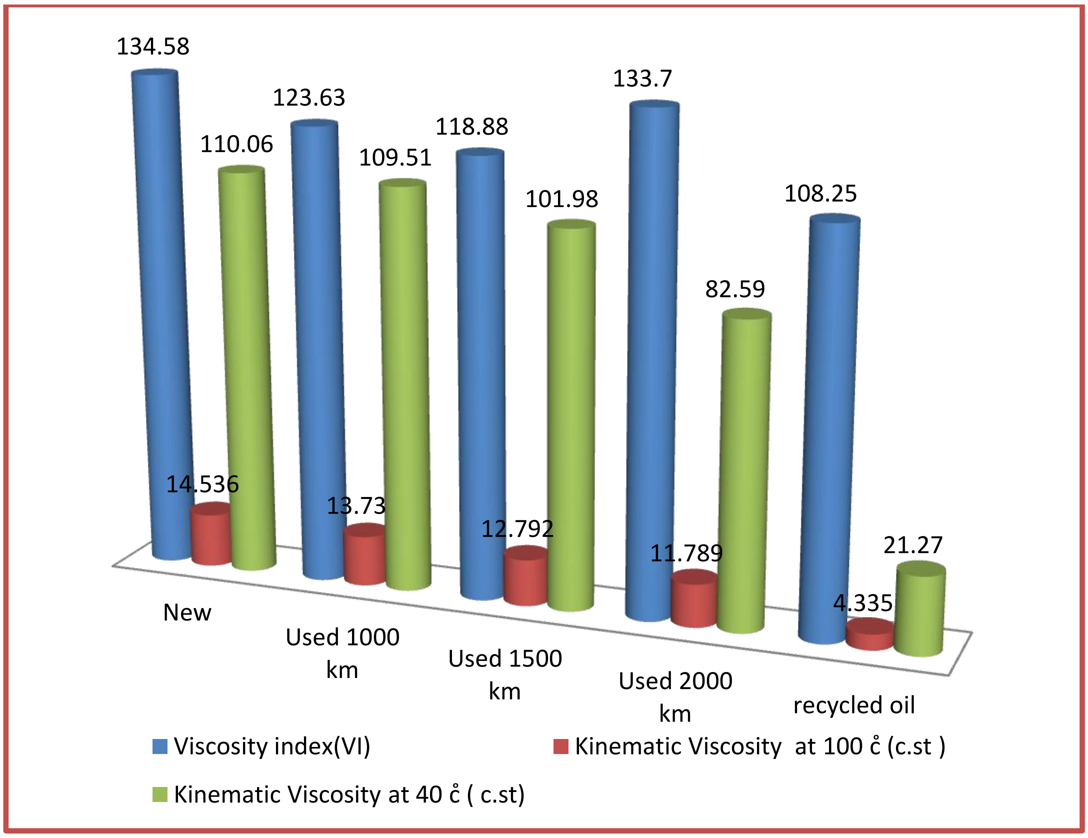


Figure (3.1): Kinematic viscosity at (40 ˚C and 100 ˚C) and viscosity index of different oils

3.1.2 Specific Gravity of Oils

The specific gravity of a used oil can be lower or higher than that of the new lube oils depending on the type of contamination . If the used oil was contaminated due to fuel dilution and/or water originating from fuel combustion in the engine and accidental contamination by rain, its specific gravity will be lower than that of its new lube oil or the re-refined one . The other hand when the specific gravity of used oil is greater than that of the new lube oil , then the lube oil may have contaminated with component of a higher chain carbon atoms .So , the result for the new oil specific gravity at 15 ˚C was 0.889,while the specific gravity of used oil at (1000, 1500 and 2000) km were (0.886 ,0.885 and 0.890) respectively [92] .

API˚ gravity is common unit of classifying oil so : for{ Light oil (API >38) , Medium oil (38> API >29) , Heavy oil (29>API>8.5) ,Very heavy oil (API<8.5) } [93] .The result of new ,used and recycled oil is between (29>API>8.5) ,So the oil used in present research is heavy oil , as shown in table (3.2) . API˚ of the recycle oil is greater than API˚ of used oil at 2000 km because the API˚ gravity of oil increases as the paraffin content rises. and the greater the specific gravity, the lower the API˚ gravity [2] .So , the specific gravity of oil at 15 ˚C after recycle was 0.881,while the specific gravity of used oil at 2000 km was 0.890 , as shown in Fig (3.2) .

Table (3.2) : Specific gravity and API° gravity ,according to ASTM D-1298 of oils

Type of oil	Specific gravity at 20 °C	Specific gravity at 15 °C (60 °F)	API° gravity at 15 °C
New	0.883	0.889	27.66
Used at 1000 km	0.879	0.886	28.20
Used at 1500 km	0.877	0.885	28.38
Used at 2000 km	0.883	0.890	27.48
Recycled oil	0.873	0.881	29.11

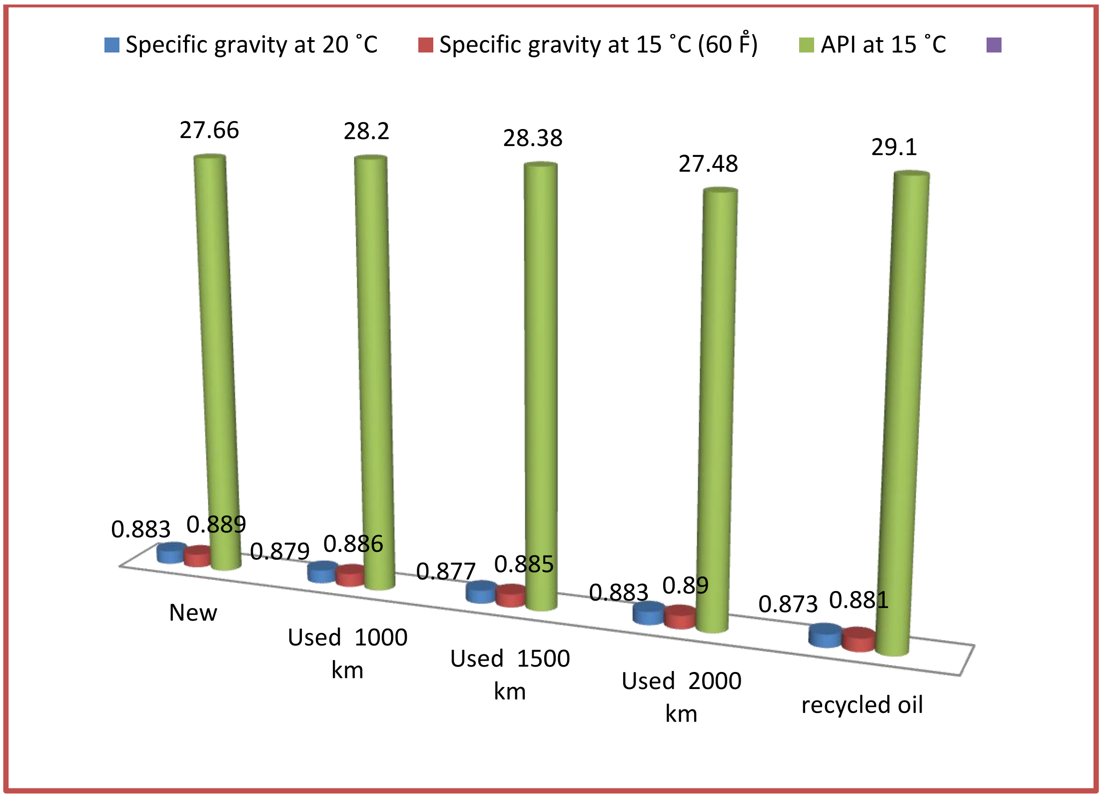


Figure (3.2) : Specific gravity and API° gravity for different oils

3.1.3 Pour Point Results

From table (3.3) , the pour point of the new oil is -36 ˚C . while for the oil used at 1000 km and 1500 km is also -36 ˚C .However , the pour point of oil used at 2000 km is -39 ˚C , this is because of the degradation of additive in the lube oil. Pour point especially is of interest when an oil used under relatively cold condition. Pour point will vary widely depending on the base , the source of the lube oil and the method of refining, especially if dewaxing has been done [92, 94]. In addition, the lower the pour point of lube oil the more aromatic it is, and the higher the pour point the more paraffinic it is. So, the pour point of new lubricating oil more paraffinic compounds compared with the recycled oil that is more aromatic components [43], as shown in Fig (3.3).

Table (3.3) : Results of pour point according to ASTM D-97

Type of oil	Pour point ˚C
New	-36
Used at 1000 km	-36
Used at 1500 km	-36
Used at 2000 km	-39
Recycled oil	-18

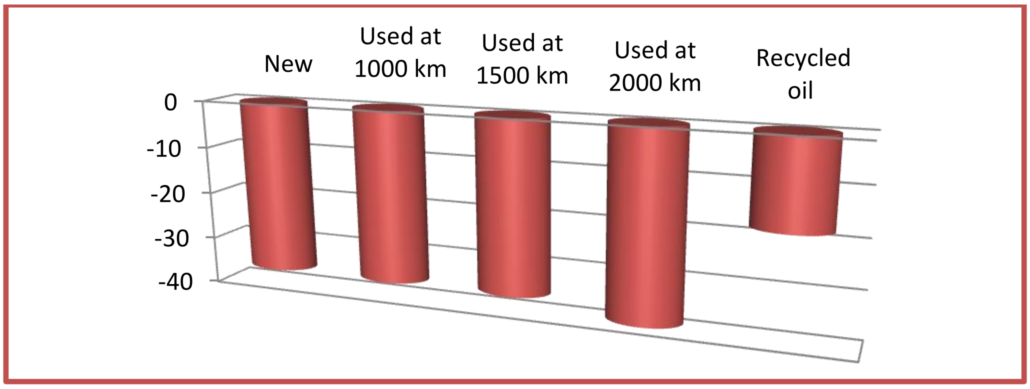


Figure (3.3) : Pour points for different oil samples

3.1.4 Flash Point Results

Flash point values of oil used at 1000 km , 1500 km and 2000 km are (221,216 and 187) ˚C respectively , being lower than those of the new oil being 224 ˚C. This is attributed to the formation of light product as a result of lubricant oil oxidation or deterioration of a flash point improver. It is well known that the flash point increases with increase of the molecular weight of oil component, which gives an indication of lower molecular weight of used oil compared with new oil [17,25, 36], as shown in table (3.4) **.**

Also the flash point of the new oil is higher because it contains many different additives which contribute to improve it [70] . A high flash point of 203˚C was obtained for the recycle oil. This is good and gives an indication that the lube oil produced was free of light hydrocarbon , especially when compared with the typical value for the new oil , as shown in Fig (3.4).

Table (3.4): Results of flash points according to ASTM D-92

Type of oil	**Flash point** ˚C
New	224
Used at 1000 km	221
Used at 1500 km	216
Used at 2000 km	187
Recycled oil	203

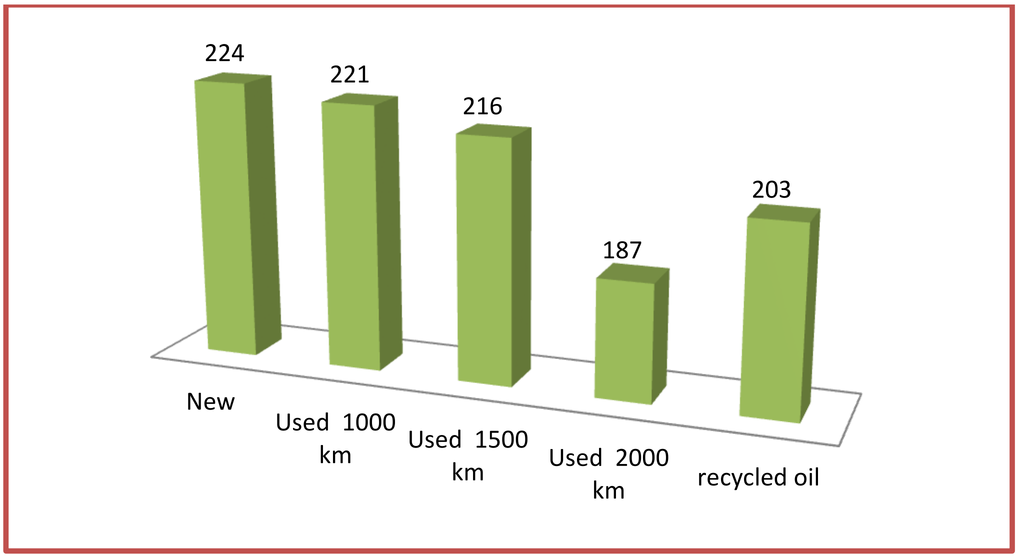


Figure (3.4) : Flash points for different oil samples

3.1.5 The Color Results

The results of color cannot be obtained for the used oils as it were dark. Therefore, the oils were diluted with kerosene by mixing 15 volumes of sample with 85 volumes of kerosene because the kerosene have a color value less than +21 saybolt color [84] .The results of the oil used for a distance of 1000 km was 5-5.5 degree. While ,the oils used for a distance of 1500 km and 2000 km did not give any result when even diluted with kerosene . The dark color of used oil, when compared with new oil, can be attributed to the presence of impurities, these impurities result either from using the oil in the engine or as a result of oxidizing the oil when exposed to high temperature leading to formation of polyolefin and carbon [95], as shown in table (3.5). After vacuum distillation , the darker color of oil used at 2000 km turned into a yellow one with the value of 5 degree , that means good purity recycle oil was obtained, and that the presence of impurities have decreased and even removed significantly after vacuum distillation .

Table (3.5): Results of color done according to ASTM D-1500

Type of oil	Color
New	4 ASTM color
Used at 1000 km	L 5–5.5 Dil ASTM color
Used at 1500 km	D8 ASTM color
Used at 2000 km	D8 ASTM color
Recycled oil	5 ASTM color

3.1.6 Carbon Residue and Ash Content

The carbon residue of new oil is 0.825 % , as for, carbon content after vacuum distillation it was decreased to 0.47 % . This is close to the Iraqi specifications required for oils and the significant decline of the carbon content gives an indication of the efficiency and effectiveness of this process to recover oil from used oil .

The ash content is considered an important test in evaluating the purity of oils. It is known that the percentage of ash represents the residues of the mineral substances in the oil and that the low percentage of it represent evidence of a high purity processed oil. The results of ash content for new oil is 0.567% was reduced to 0% after vacuum distillation . Lubricating oil contains organic mineral enhancers. Therefore, from table (3.6), it was found that the ash content of the new oil was 0.567 % . When the oil was used for distances 1000,1500 and 2000 km , the ash contents increased to 1.230 ,1.256 and 1.282 % respectively . This is due to the internal pollutants resulting from the damage of the additives as well as the external pollutants from the dust and the engine friction products [86] with the results shown in Fig (3.5) .

Ash can results also from extraneous solids such as dirt and rust [96]. In Fig (3.5) it found that the ash content reduced from 1.282 % in the oil used at 2000 km to 0 % in the recycle oil . So, we used the residue after distillation to find the ash contents balance values .

Table (3.6): Results of carbon residue and ash content done according to ASTM D-482

Type of oil	Carbon wt. %	Ash wt. %
New	0.825	0.567
Used at 1000 km	1.59	1.230
Used at 1500 km	1.902	1.256
Used at 2000 km	4.628	1.282
Recycled oil	0.47	0
Residue of distillation	4.055	2.23

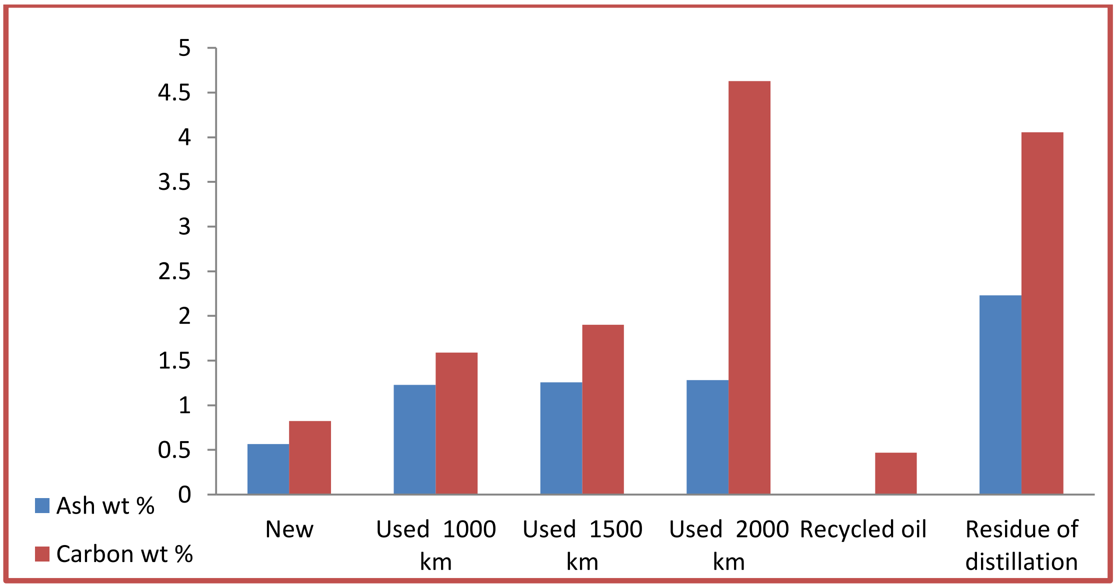


Figure (3.5) : Carbon residue and ash content values for different oil samples

3.1.7 Elements Analysis in Oils

Table (3.7) shows the elements analysis of used lubricant oil. The most common wear metal in a car's engine that is introduced into the engine oil after a period of use is iron (Fe). Iron comes from many various places in the engine such as liners, camshafts and crank shaft, pistons, gears, rings, and oil pump. Iron concentration in engine oil depends on the bearing conditions inside the engine. If a bearing fails, iron concentrations in used engine oil increases. In the engine, the wear rises at a faster rate during the starting of the engine [97]. Copper (Cu) is introduced to engine oils after use from bearings, wearing and valve guides. Engine oil coolers can also be contributing to copper content along with some oil additives [98]. Magnesium (Mg) is regarded as the most common wear metals in used engine oil and is present in virgin oil in the form of magnesium phenates and magnesium salicylates that behave as antioxidants at high temperatures [99]. The element analysis is shown in Fig (3.6) .

Table (3.7) : Analysis results of elements content of different oil samples

Metal	Oil type	Concentration (ppm)
Iron (Fe)	New	12667.837
	Used at 1000 km	10732.5
	Used at 1500 km	11775.275
	Used at 2000 km	12693.962
	Residue of distillation	24411.466
Copper (Cu)	New	9.837
	Used at 1000 km	9.487
	Used at 1500 km	10.562
	Used at 2000 km	12.362
	Residue of distillation	23.733
Magnesium (Mg)	New	348.237
	Used at 1000 km	888.187
	Used at1500 km	361.612
	Used at 2000 km	227.862
	Residue of distillation	438.192

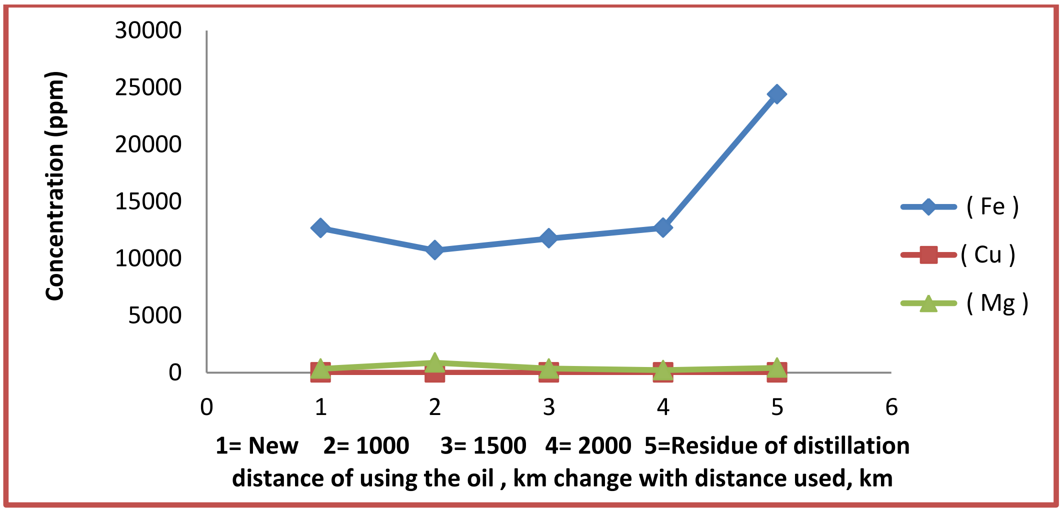


Figure (3.6) : Show the elements content for different oil samples

Oils contain a lot of additives, some of which work to clean the engine or the dispersion of oil so as not to accumulate and block, including oxidation inhibitors. The most important additives are zinc. Zinc is introduced to base oil in the form of additives package as anti-oxidant, corrosion inhibitor, anti-wear, detergent and extreme pressure tolerance [100]. The result for Manganese (Mn) was zero ,this indicates that the oil does not contain manganese as additives , with the results shown in table (3.8) .

Table (3.8): Analysis results of metal content (additives) of different oil samples

Metal	Oil type	Concentration (ppm)
Zinc (Zn)	New	1618.15
	Used at 1000 km	672.562
	Used at 1500 km	750.287
	Used at 2000 km	1533.450
	Residue of distillation	2948.942
Manganese (Mn)	New	0.00
	Used at 1000 km	0.00
	Used at 1500 km	0.00
	Used at 2000 km	0.00
	Residue of distillation	0.00

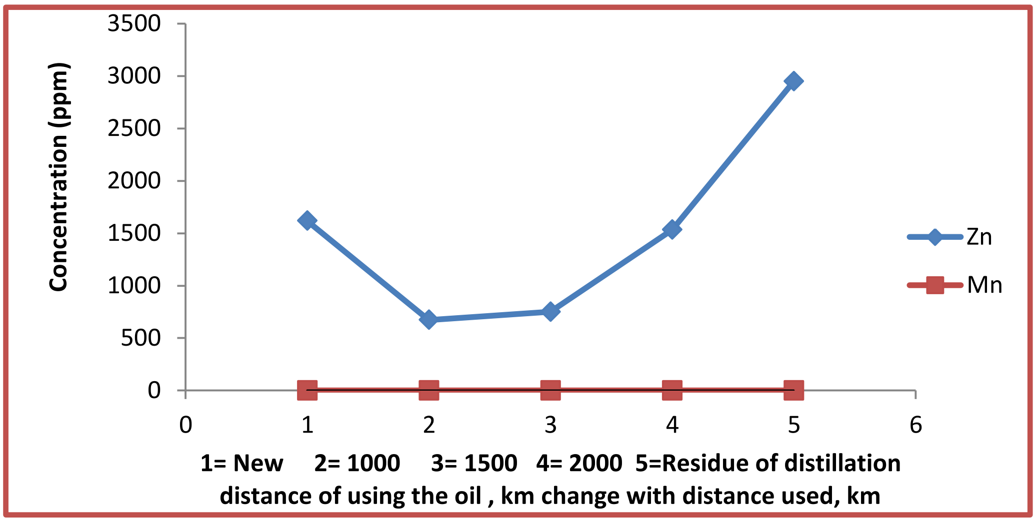


Figure (3.7) : Additives content for different oil samples

3.2 Recycling of the Used Lubricating Oil by Vacuum Distillation

The recycled the oil included the steps which are the first step is the settling and filtration to remove large contaminants in the used oil which indicated that there is no solid contaminants materials present .Then the solvent extraction with (hexane, benzene, methanol and 2-propanol) and another adsorption experiments with activated alumina and bentonite followed by adsorption chromatography to separate the spent oil to fractions seem also to be not useful, which enforced us to use vacuum distillation route .

Before vacuum distillation we used settling and filtration as mentioned in chapter 2 ,section (2.6.2) and (2.6.3) . This was applied in the purification of waste lubricating oils and to improve the physical properties of it and compare it with new one . It is clear that vacuum distillation was good technique for the removal of contaminants from used oil which enhances the desired properties and quality of lubricating oils. Table (3.9) shows that a significant decrease in the value of kinematic viscosity ,specific gravity , pour point and carbon residue, and also complete removal of ash. On the other hand it is observed that there is a marked increase in the flash point , API˚ gravity and color as well .

After vacuum distillation we used different extraction processes by using different solvents such (hexane , methanol ,2- propanol) as mentioned in chapter 2 ,section (2.6.5) to see if there are any contaminant or solid material that exist and this indicated that these materials was not present .

Table (3.9): Compared the Physical properties between the used oil at 2000 km and oil obtained from vacuum distillation

Test	Used 2000 km	Oil produced from vacuum distillation
Kinematic Viscosity at 40 ˚C (cSt)	82.59	21.27
Kinematic Viscosity at 100 ˚C (cSt)	11.789	4.335
Viscosity index (VI)	133.70	108.25
Specific gravity at 15 ˚C (60 ˚F)	0.890	0.881
API˚ gravity at 15 ˚C	27.48	29.11
Flash point (˚C)	187	203
Pour point (˚C)	-39	-18
Color	D8 ASTM color	5 ASTM color
Carbon residue wt. %	4.628	0.47
Ash content wt. %	1.282	0

3.3 FTIR Spectra for New ,Used and Recycled Oil

The FTIR results for new ,used and recycled oil are showed with the frequencies in wave number of the main functional groups peaks given in table (3.10) and the Figs (3.8) - (3.12) respectively . The main peaks observed in all types of oils show in above figures are associated with major functional groups present in this type of engine oils. Peaks at (2947.23 , 2954.95, 2922.16 , 2926.01 , 2949.16) cm^{-1}, and also (2852.72 , 2856.58 ,2854.65, 2852.72) cm^{-1} correspond to C-H stretching of saturated n-alkyl groups which appeared in all types of oil . IR peaks at (1460.11 ,1462.04 ,1305.81) cm^{-1} show C-H deformation of CH_2 groups, vibrations at (1367.53,1375.25) cm^{-1} correspond to C-H deformation of CH_3 of n-alkanes [101 , 102] . Infrared peaks for used oil 2000 km at (1732.08 and 1703.14) cm^{-1} correspond to C=O due to oxidation process occurs for compounds in the oil because of the fact that (C=O) bond strongly absorbs at this frequencies, as well as in the recycle oil the peak at 1722.43 cm^{-1} correspond to C=O, so the oxidation process occurs but at a lower rate . Also the peaks in oil used at 2000 km at (1002.98 , 1157.29, 1226.73) cm^{-1} and the peak in recycled oil at 1209.37 cm^{-1} correspond to C-O , the peak at 3468.01 cm^{-1} correspond to $-NH_2$ asymmetric and 3635.82 cm^{-1} correspond to $-NH_2$ symmetric (nitrogen compounds) . The only difference between the oil used at 2000 km and recycled one, is that the peaks at (2727.35 and 2681.05) cm^{-1} correspond to H-C=O present in oil used at 2000 km , is the aldehyde components resulted from oxidation . Those vibrations in IR spectrum corresponding to out of plain aromatic C-H stretching exhibit stronger intensity and well-separated peak from 437.84 cm^{-1} to 975.98 cm^{-1} . Finally C=C peak due to olefins and polyolefin present in spent oil in addition to those related to $–NH_2$ groups [103,104] .

Table (3.10): Results of FTIR analysis for new, used and recycled oils

Group	New oil (cm^{-1})	Used oil at 1000 km (cm^{-1})	Used oil at 1500 km (cm^{-1})	Used oil at 2000 km (cm^{-1})	recycled oil (cm^{-1})
C-H Stretch Alkane	2947.23 , 2852.72	2954.95 , 2922.16 ,2856.58	2954.95 , 2926.01 , 2854.65	2949.16 , 2852.72	2954.95 , 2926.01 , 2854.65
C-H bending Alkane $-CH_2$	1460.11	1462.04	1460.11	1460.11, 1305.81	1460.11, 1305.81
C-H bending $-CH_3$	1367.53	1375.25	1375.25	1375.25	1375.25
C=C Aromatic	/	/	/	1602.85	1639.49 , 1604.77
$-NH_2$ Asymmetric	/	/	/	3468.01	/
$-NH_2$ Symmetric	/	/	/	/	3635.82
C-O	/	/	/	1002.98 , 1157.29, 1226.73	1209.37
C=O Stretch	/	/	/	1732.08 , 1703.14	1722.43
H-C=O Stretch	/	/	/	2727.35, 2681.05	/
C-H bending Aromatic Out of plan	/	721.38	723.31	725.23, 659.66 , 650.01 , 599.86, 561.29 , 509.21 , 447.49 ,437.84 975.98 , 923.90 , 862.18 , 810.10	725.23 , 565.14 812.03

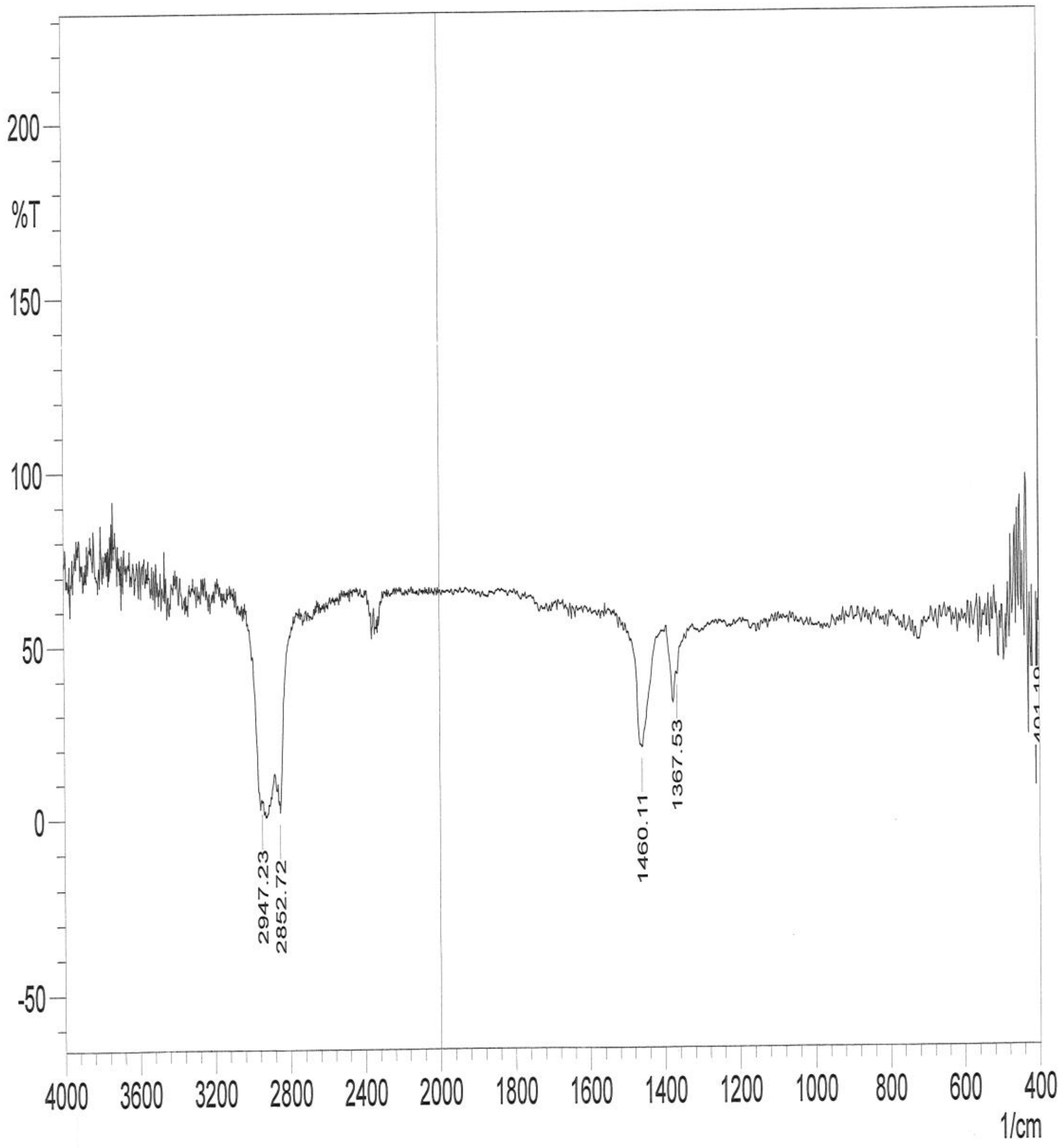


Figure (3.8): The FTIR spectrum of the new oil

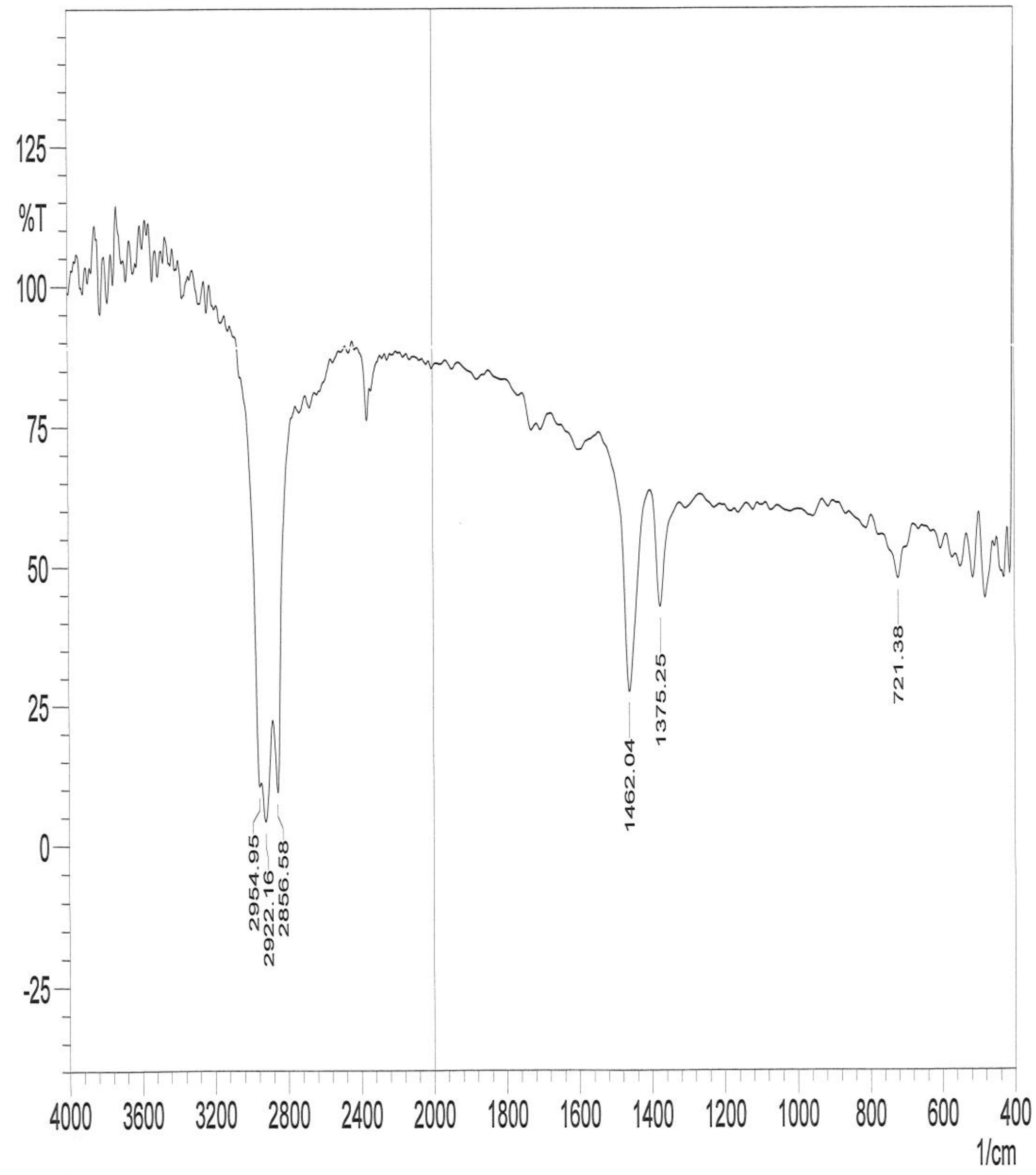


Figure (3.9): The FTIR spectrum of used oil at 1000 km

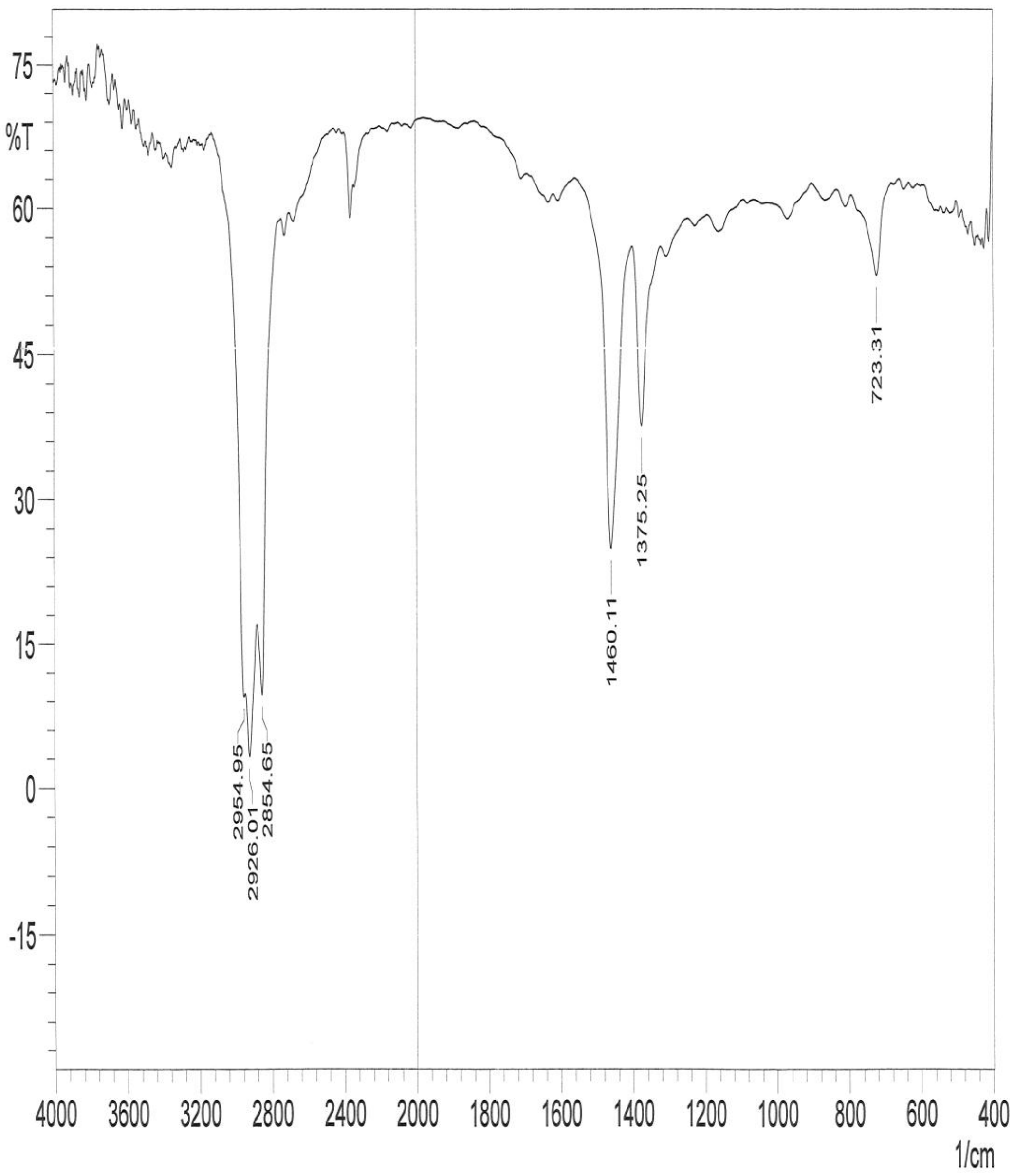


Figure (3.10): The FTIR spectrum of used oil at 1500 km

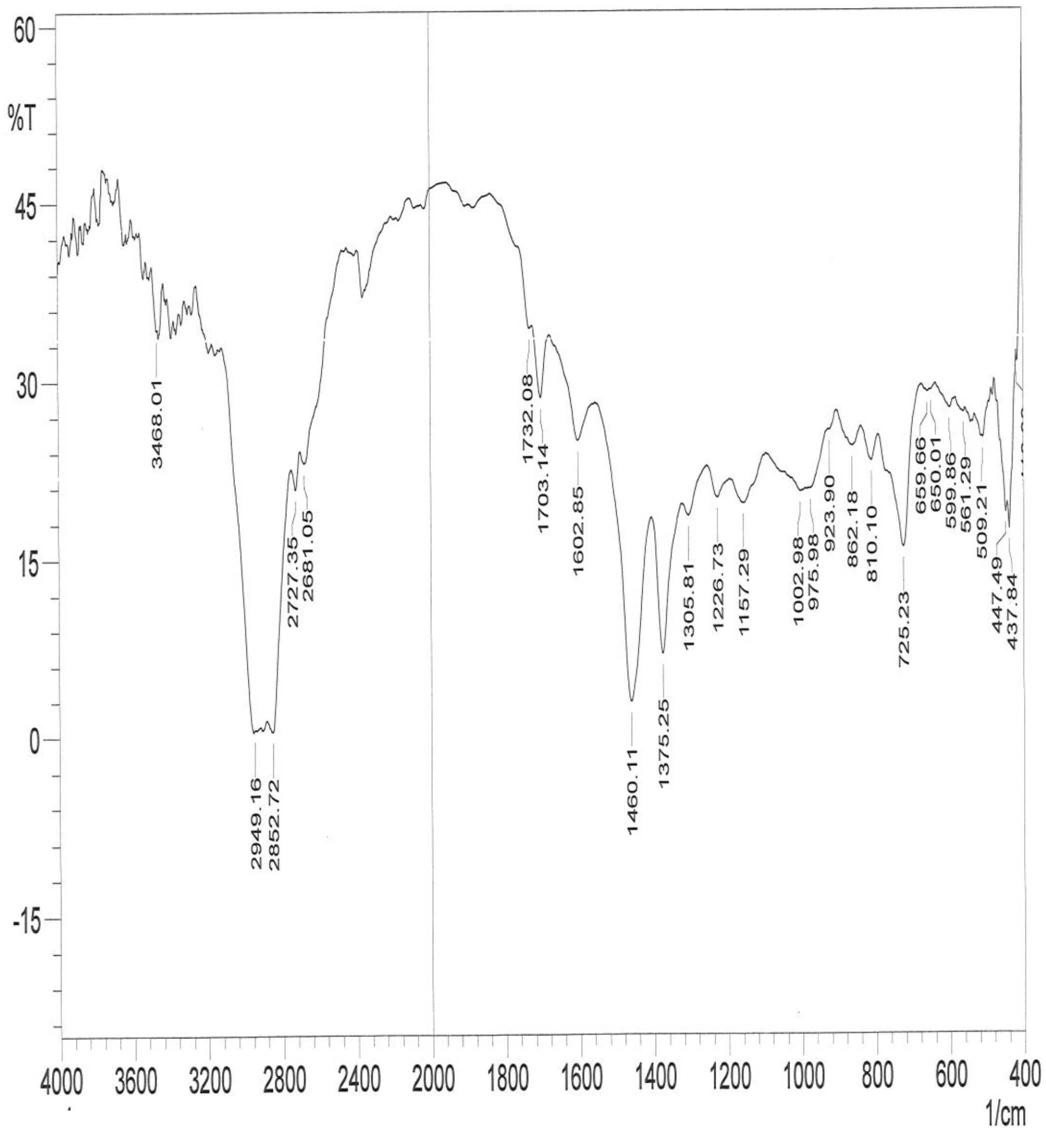


Figure (3.11): **The FTIR spectrum of used oil at 2000 km**

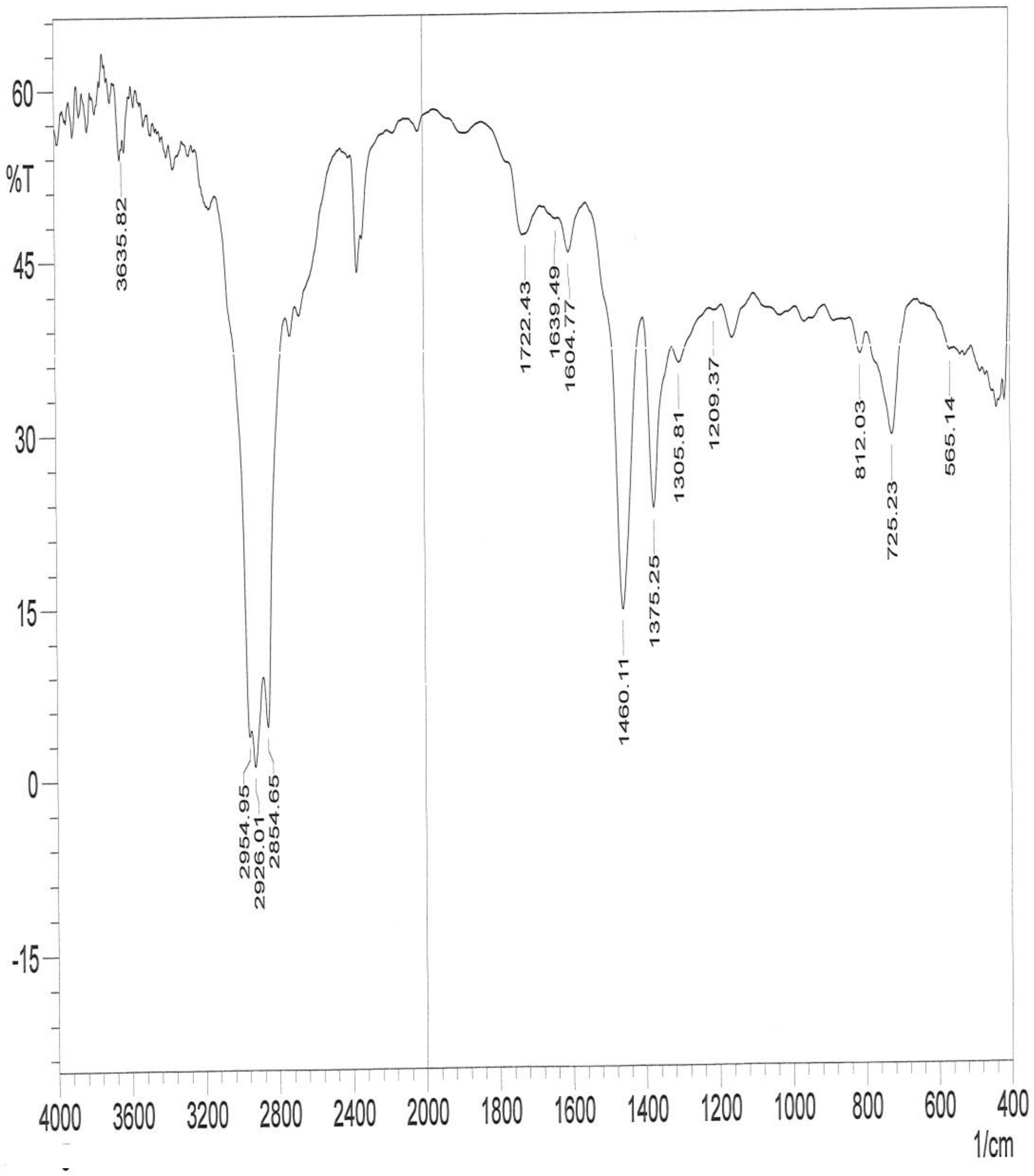


Figure (3.12): **The FTIR spectrum of recycled oil**

3.4 UV Visible Spectrophotometer Analysis for New ,Used and Recycled Oils

Ultra violet spectra were measured for samples of new , used and oil after recycling. Table (3.11) shows the absorbance and the wavelengths of maximum absorption . The transitions of all type of oils used are of type $\pi \rightarrow \pi *$. The absorption transition wavelength indicated a different types of π bonds C=C and /or C=O , for molecules that possess π bonds like alkenes, alkynes, aromatics and acryl compounds [105] ,and this is shown in Figs (3.13)- (3.17) .

Table (3.11): Results of UV spectra of the new, used and recycled oils

Type of oil	Absorbance	λ_{max} (nm)	Transition
New oil	1.44589	278.2	π -π^*
Used oil at 1000 km	1.57896	285.6	π -π^*
Used oil at 1500 km	1.52834	279.8	π -π^*
Used oil at 2000 km	1.58971	285.4	π -π^*
Recycled oil	0.0719291	200	π -π^*

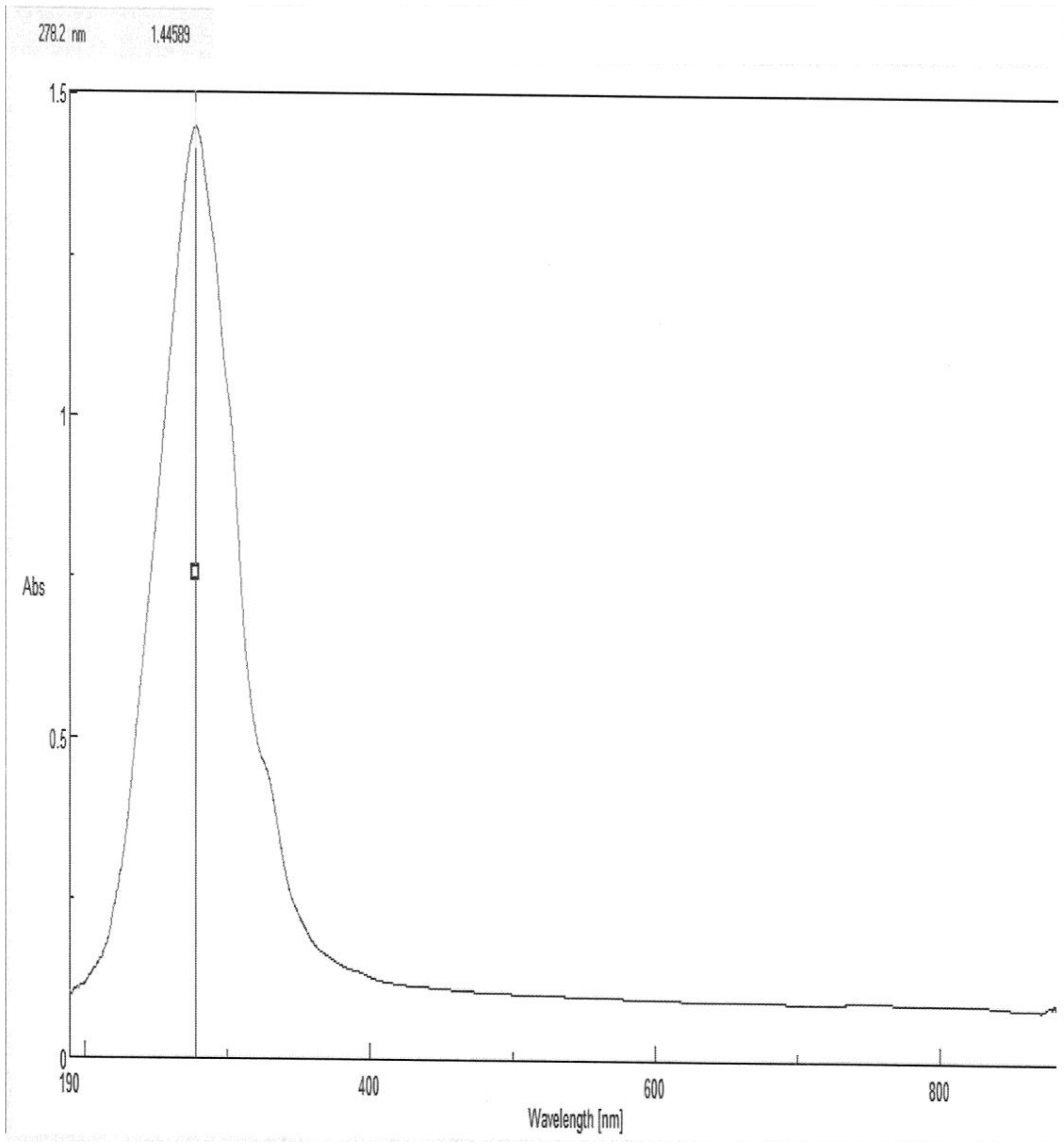


Figure (3.13): UV spectrum of the new oil

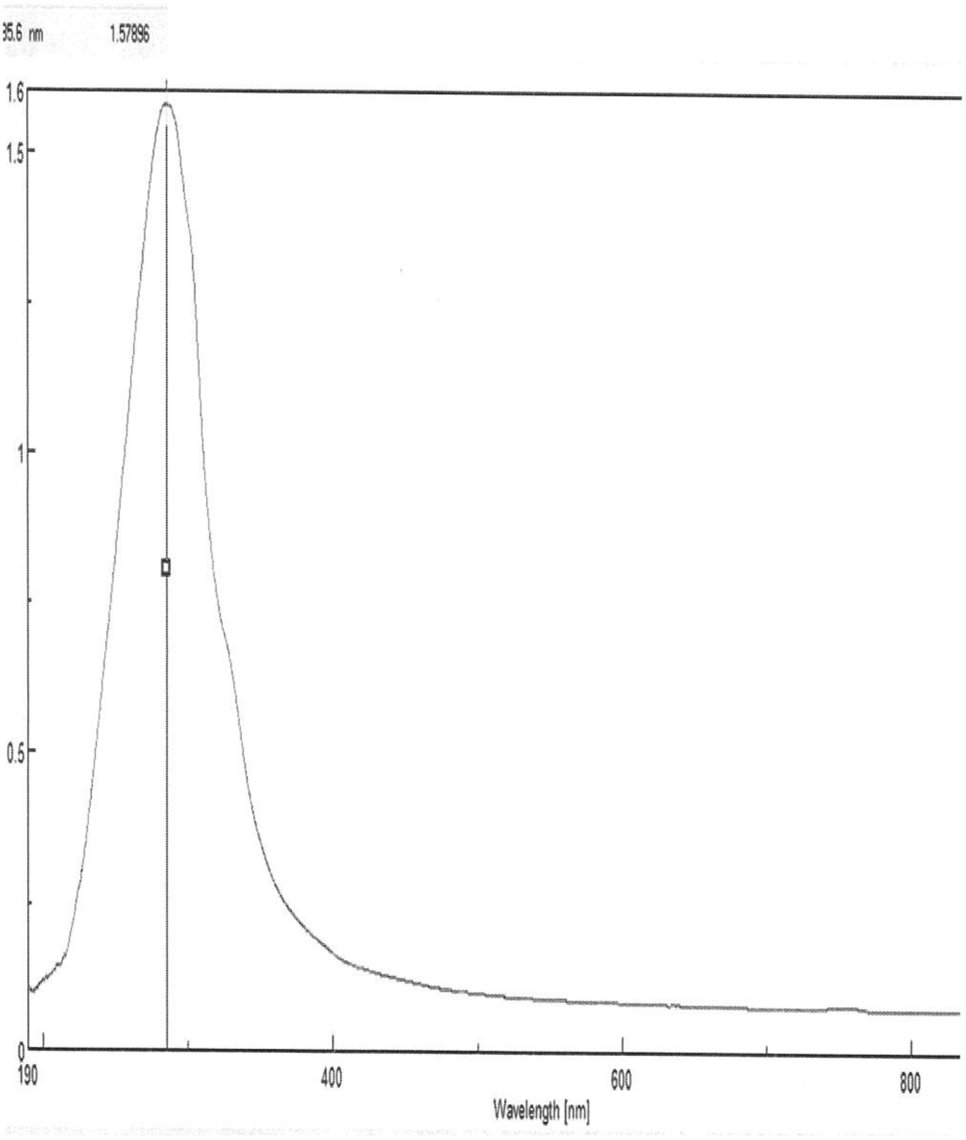


Figure (3.14): UV spectrum of used oil at 1000 km

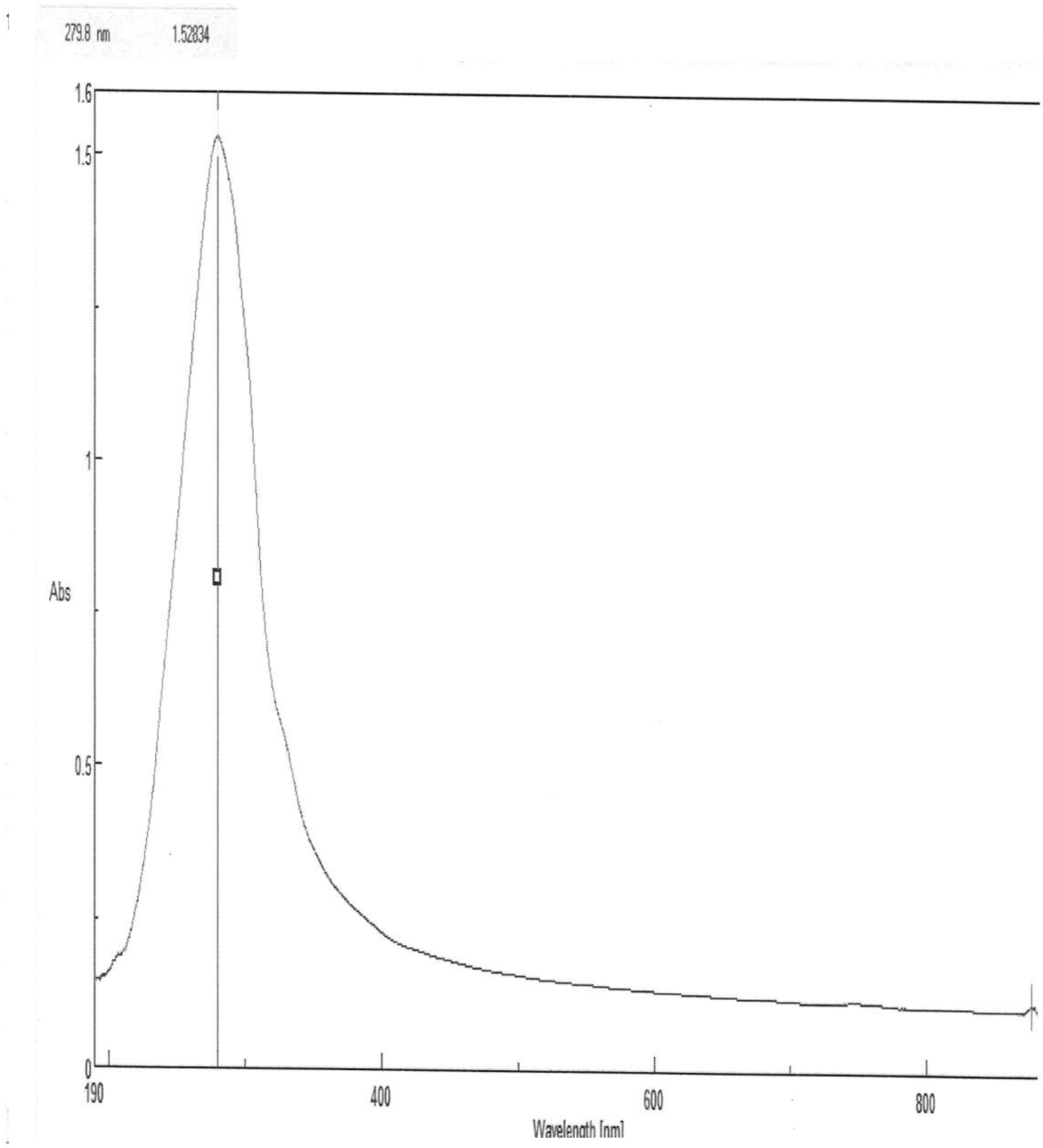


Figure (3.15): UV spectrum of used oil at 1500 km

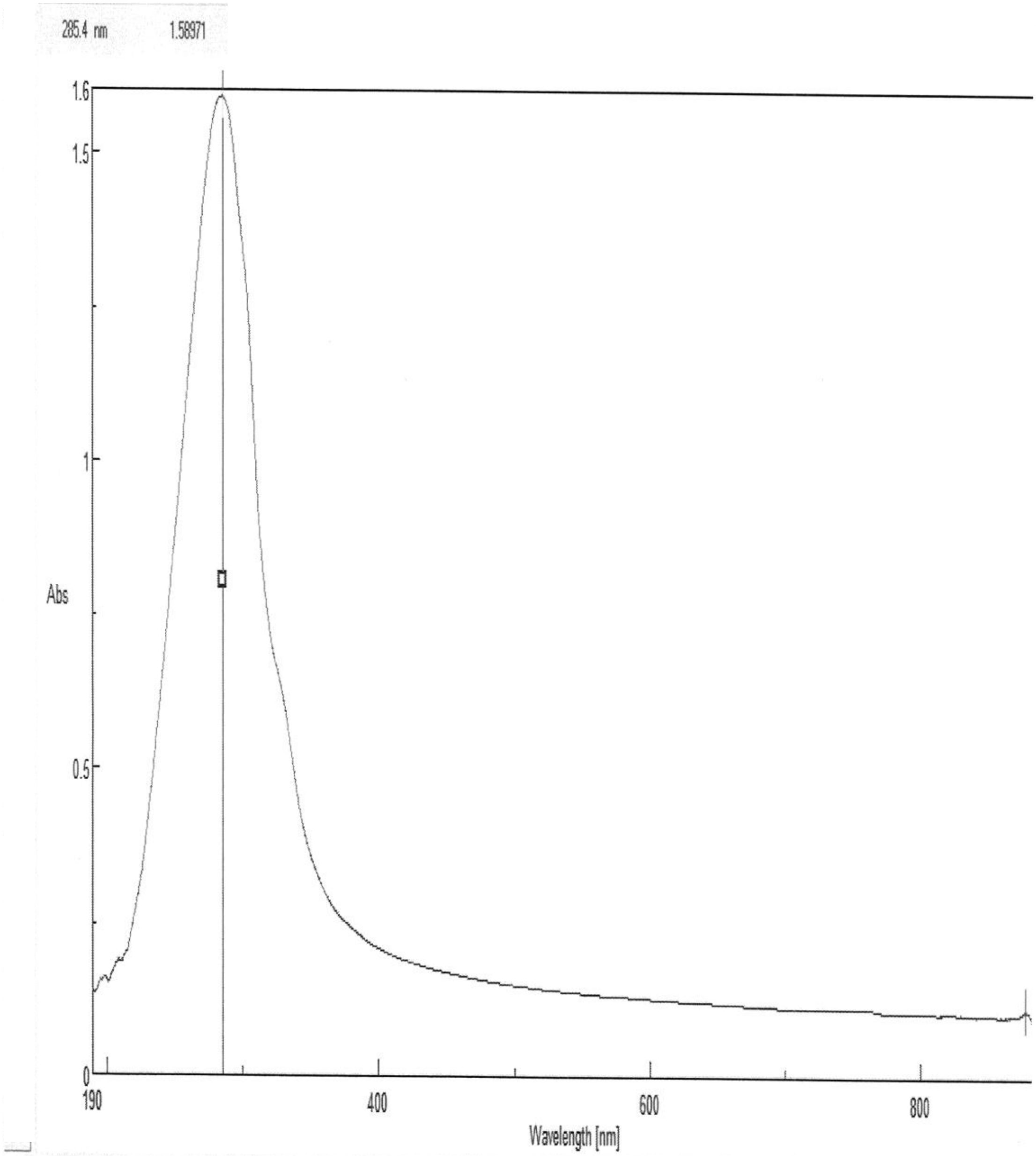


Figure (3.16): UV spectrum of used oil at 2000 km

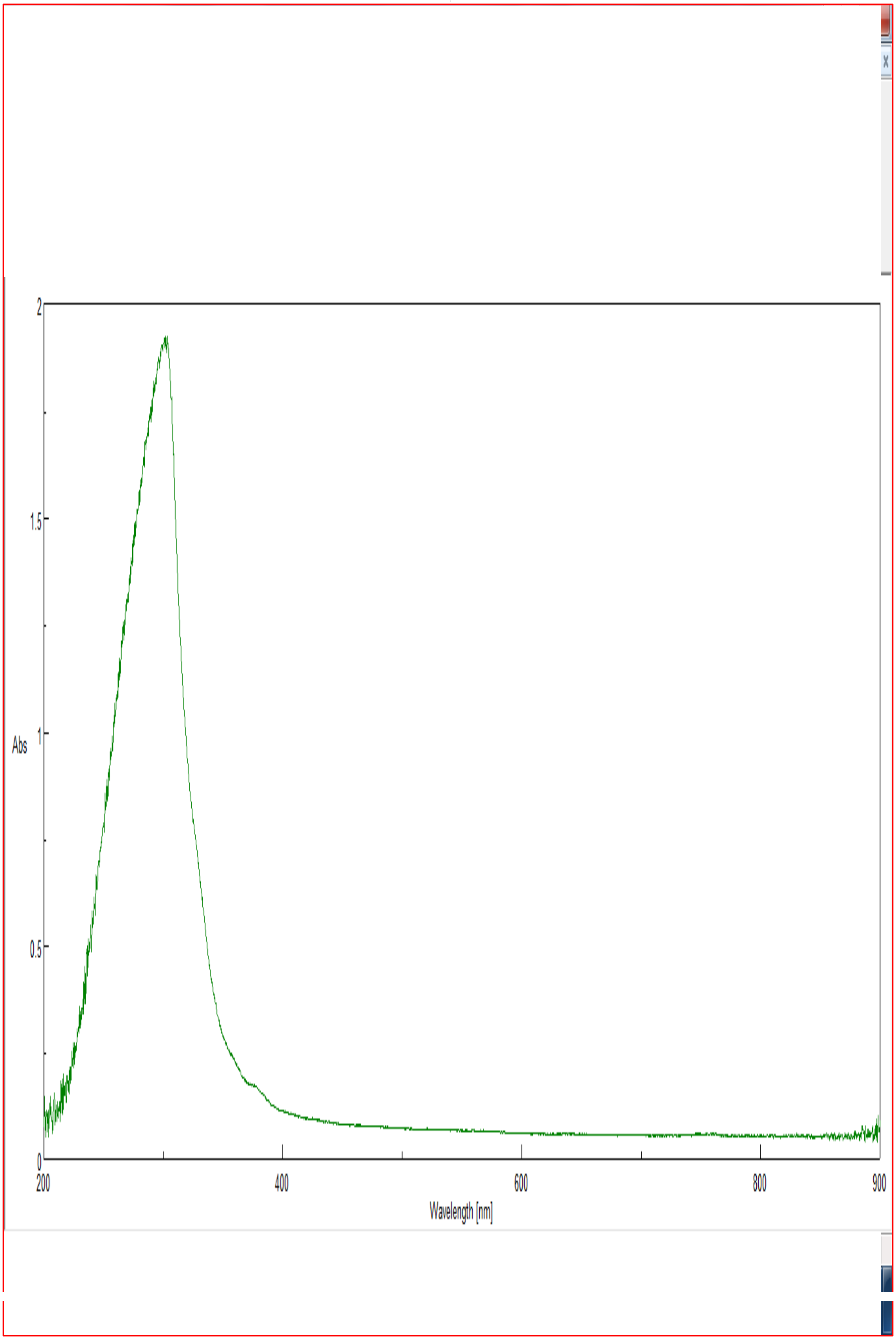


Figure (3.17): UV spectrum of recycled oil

3.5 Conclusions

The following points can be concluded:

1- Vacuum distilled lube oil fraction (235 ° C to 475 ° C) obtained from vacuum distillation of spent oil is sufficient to obtain a pure oil free of contaminants without need for extraction process .

2- Vacuum distillation was economic and can be operated industrially to give lube oil with good color (deep yellow), increase flash point from 187 ˚C in used oil at 2000 km to 203˚C in recycled oil , carbon residue reduction (0.47 %) and ash content reduction to zero .

3- The oil used is heavy one , because the result obtained of API gravity for the new ,used and recycled oil are between (29>API>8.5) .

4- Other results obtained were a decrease in the kinematic viscosity at (40 and 100)˚C for the used oil (1000, 1500 and 2000) km and also recycled oil.

5- There is no solid contaminants materials present in the used oil when we used the filtration step, because the process of collecting the oil was in clean containers.

6- Spectroscopic FTIR analysis showed that the recycled oil contains all functional groups which exist in the new oil such as (2947.23 , 2852.72) cm^{-1} peaks in new oil and (2954.95 , 2926.01 , 2854.65) cm^{-1} peaks in recycled oil .

7- Ultra violet spectra showed the transitions for samples of new , used and oil after recycling are of type $\pi \rightarrow \pi^*$.

3.6 Recommendations

The following points are suggested for future work:

1- Conducting other purification processes on this oil, or the same purification on other oils , such as clay adsorption solvent extraction …

2-The use of another type of oil and those used with more traveling distances.

3-The measurement of other tests such as, boiling point, total acid number and total base number, refractive index, HPLC , NMR ….

4-Using spent oil from spent oil collection centers.

5-Studying some adsorbents such as bentonite, zeolites, raw metal oxide and activated carbon.

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الخلاصـــة

تم فحص الخواص الفيزيائية لزيوت التزييت نوع بغداد (15W-40) ، المنتجة في شركة مصافي التكرير الوسطى / مصفى الدورة وتلك المستخدمة لمسافة 1000 و 1500 و 2000 كيلومتر في سيارة صالون صغيرة (Mercedes-Benz-Internal Combustion Engine) في ظروف تشغيل مختلفة .

لوحظ أن اللزوجة الحركية عند (40 و 100) درجة مئوية للزيوت المستعملة والمعادة كانت منخفضة إذا ما قورنت بالزيت الجديد . كذلك لوحظ ان هنالك تغيير في معامل اللزوجة لزيت التزييت ، حيث انخفض في الزيوت المستخدمة (1000 و 1500) كم وزيت المعاد تدويره (المسترجع) ، ولكن في 2000 كم يزداد ، بسبب الانخفاض الشديد في اللزوجة الحركية مقارنة بتلك الموجودة في الزيت الجديد.

كذلك تبين أن تكون الكثافة النوعية للزيوت المستخدمة أقل أو أعلى من زيوت التزييت الجديدة اعتمادًا على نوع الملوث. لذا ، فإن نتيجة الكثافة النوعية للزيت الجديد عند 15 درجة مئوية كانت 0.889 ، في حين كانت الكثافة النوعية للزيت المستخدم عند (1000 ، 1500 و 2000) كم هي (0.886 ، 0.885 و 0.890) على التوالي . كذلك فأن معهد البترول الامريكي (API°) للزيوت الجديدة والمستعملة والمعاد تدويره بين (29>API>8.5) ، وبالتالي فإن الزيت المستخدم في العمل هو زيت ثقيل.

أما نقطة الوميض للزيوت المستخدمة في (1000 و 1500 و 2000) كم فقد كانت أقل وهي (221 و 216 و 187) درجة مئوية على التوالي مقارنة بتلك الموجودة في الزيت الجديد عند 224 درجة مئوية ، وقد تم الحصول على نقطة وميض عالية للزيت المعاد تدويره وهي 203 درجة مئوية اذا ما قورنت بالنتيجة التي تم الحصول عليها من الزيت المستخدم لمسافة 2000 كم .

نقطة انسكاب الزيت الجديد كانت 36- درجة مئوية. في حين تكون للزيت المستخدم لمسافة 1000 كم و 1500 كم هو أيضًا 36- درجة مئوية ، ولكن نقطة انسكاب الزيت المستخدم لمسافة 2000 كم هي39 - درجة مئوية في حين لوحظ ان هناك انخفاض في نقطة انسكاب الزيت المسترجع .

لا يمكن الحصول على نتائج اللون للزيوت المستخدمة لأنها كانت معتمه ، لكن بعد التقطير الفراغي ، تحول اللون الغامق للزيت المستخدم لمسافة 2000 كم إلى لون أصفر و بقيمة 5 ASTM color .

نتائج الكاربون المتبقي ومحتوى الرماد في الزيت المستخدم مقارنة بالزيت الجديد بينت ان هناك زيادة فيها ولكن بعد التقطير الفراغي انخفضت القيم إلى 0.47 ٪ ، و 0 ٪ على التوالي في الزيت المعاد تدويره . لذلك ، استخدمنا المتبقي من التقطير الفراغي للعثور على قيم توازن محتويات الرماد. وتحديد محتوى المعادن باستخدام مطياف الامتصاص الذري لمحتوى (الحديد والنحاس والمغنيسيوم والزنك والمنغنيز) الموجود في زيوت جديدة ومستعملة والمعاد تدويره .

تم قياس مطياف فورييه لتحويل الأشعة تحت الحمراء (FTIR) ومقياس الطيف المرئي UV للزيوت الجديدة والمستعملة والمعاد تدويره ، وأثبتت الاختبارات أن الزيت المعاد تدويره يحتوي على جميع المجموعات الوظيفية الموجودة في الزيت الجديد .

وزارة التعليم العالي والبحث العلمي
جامعة ديالى
كلية العلوم
قسم الكيمياء

دراسة عملية اعادة التدوير والخصائص النفطية لزيت التزييت المستهلك

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وهي جزء من متطلبات نيل درجة الماجستير في علوم الكيمياء

من قبل

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