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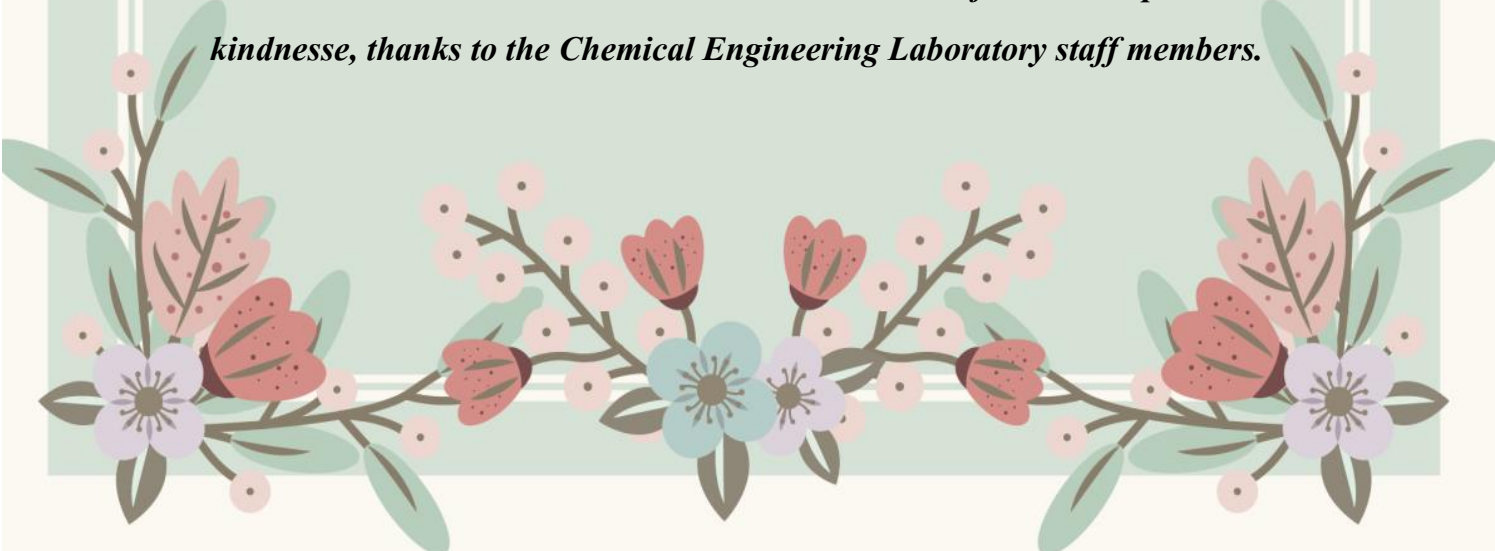
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Dedicat

I would like to dedicate this work to my beloved
Mother and Father
Brother and Sisters
Rahma; Islem; Mariya; Sejda; Belkiss)

Family
Grandfather, Tata: Sonya Hakim, Farok،
Missa Dounya, Midou، Assala، Wadia Insaf Aboudi Souma،
Zinouba Lemiss, Youssef, Yaakoub,
Mouhamed heytoma, noussa,
(Ami: Fayssal, Kheyrou, Feteh, Elhadi)
(Kheli: Sahran, Khidora, Sofyene)
Friends
Souheylla ;Amina ;bessma ;imen ; Sbouha
Hassina, Sara, Khadra, Warda, Yemina, Samira
Zeyneb, Abla, Joumana, Bessma, Wafa, Rayan
Ranya, Feryel, Ikram.

Bouiada Meryem

Dedication

I would like to dedicate this work to my beloved

Mother and Father

Brother and Sister

{kamel and Razika (Adem and Isslam)}

Family

{OUERTH ; HADDAD and BELABED}

Freinds

{3azou dima ;chirif ;isslam ; mehsen ;khirou ;samir ; oussama ;haroun ;bourhan ;aniss ;nabil ;rezek ; ahmed ;mazou ;yassin ;tarek ;rabeh ;brahim ;hakim ;adlan ;farouk ; amer ;taher ;hamza ;nazim }

{badrou ;ali ;mehdi ;sami ;3abdala;saief ;omar ;khaled ;malek bd lakrim ;darwich ;brahim ;louay ;salah ;bassam ;fawzi ;nouri}

Haddad Amar

Abstract

Abstract

The amount of used lubricating oil that is collected or dumped in our country is very large, because of the increase in population of automobiles and industrial facilities. The used lubricating motor oil is a highly pollutant material that requires responsible management. It may cause damage to the environment, if it's being disposed of appropriately.

In this study many tests carried out in the laboratory in order to find out an appropriate process to recycle used lubricating engine oil collected from a car wash station. By applying appropriate treatment: washing, settling, solvent extraction and distillation for solvent recovery, adsorption by bentonites, the base oil was obtained. The refined base oil was then characterized and compared with the virgin oil.

The type of solvent used and the mixing ratio applied for different samples has shown significant effects on the yield of recovered oil. MEK +gasoline solvent with mixing ratio of 6:1 gave the best result. A Maximum yield of 92,1% was obtained using MEK+gasoline binary solvent and mixing ratio of 6:1, minimum recovery yield is 81,62% was obtained using propanol and 6:1 mixing ratio.

A good quantity of The recycled engine oil obtained at the end of the process is ready to be used again or blended with other virgin base oils.

KEY WORDS:OIL, RECYCLING, SOLVENT, BENTONIT, SETTLING.

ملخص

إن كمية زيت التشحيم المستخدمة التي يتم جمعها أو إلقاءها في بلدنا كبيرة جدا، وهذا راجع لزيادة في عدد السيارات والمنشئات الصناعية. زيت التشحيم المستخدم هو مادة ملوثة التي تتطلب إدارة مسؤولة. وقد يتسبب في إلحاق الضرر بالبيئة عندما لا يتم التخلص منها بطريقة مناسبة.

في هذه الدراسة قمنا بالعديد من الاختبارات التي أجريت في المختبر من أجل معرفة عملية مناسبة لتجديد زيت تشحيم المحرك المستخدمة التي تم جمعها من محطة غسيل السيارات. من خلال تطبيق العلاج المناسب: الغسيل، والاستقرار، واستخراج المذيبات والتقطير لاستعادة المذيبات، الامتزاز من قبل البنتونيت، تم الحصول على النفط قاعدة. ثم تم وصف النفط قاعدة المكرر ومقارنتها مع النفط البكر.

وقد أظهر نوع المذيب المستخدم ونسبة الخلط المطبقة لعينات مختلفة تأثيرات كبيرة على محصول الزيت المسترد. ميثيل ايثيل سيتون + البنزين المذيبات مع نسبة خلط 6: 1 أعطى أفضل نتيجة. A تم الحصول على الحد الأقصى لمعدل 92،1٪ باستخدام ميثيل ايثيل سيتون + البنزين ثنائي المذيبات ونسبة الخلط من 6: 1، وحاصل الاسترداد الحد الأدنى هو 81،62٪ تم الحصول عليها باستخدام بروبانول و 6: 1 نسبة الخلط.

كمية جيدة من زيت المحرك المعاد تدويره التي تم الحصول عليها في نهاية العملية جاهزة للاستخدام مرة أخرى أو المخلوطة مع الزيوت الأساسية العذراء الأخرى.

الكلمات المفتاحية : زيت، تدوير، مذيب، بانتونيت، إستقرار.

First part: Bibliography

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Acronyms

ASTM	American Society for Testing and Material
API	American Petroleum Institute
EPA	Environmental Protection Agency
ISO	International Organization for Standardization
KTI	Kinetics Technology International
LPG	Liquid Petroleum Gas
NFPA	National Fire Protection Association
MEK	Methyl Ethyl Ketone
PAH	Polycyclic Aromatic Hydrocarbons
PCB	Poly Chlorinate Biphenyls
SAE	Society of Automotive Engineers
TCT	Thermal Clay Treatment
BTU	British Thermal Unit
VI	Viscosity Index
RCRA	Resource Conservation and Recovery
TDA	Thermal Deasphalting
TAN	Total Acid Number
US	United States
NG	Neglected
Cl	Chlorine
W	Winter
NO$_2$	Nitrogen dioxide
H$_2$S	Hydrogen Sulphide
Sb	Antimony
Cr	Chrome
Ni	Nickel
Cd	Cadmium
Cu	Copper
Pb	Bullets
Zn	Zinc
DIYers	Do-It-Yourselfers
EOR	Evergreen Oil Refinery
Mw	Megawatt

Eqs	Equations
SG	Specific Gravity
VI	Viscosity Index
S	Sulfur
RI	Refraction Index

Introduction

  Lubricant base oils are mixtures that are essentially formed by the fractions of petroleum, they are used primarily for reducing friction between moving parts of various machinery or equipment They get contaminated after few thousands kilometers of driving, they represent high pollutant materials when they are changed and disposed in land, water or even burned as fuel **[1, 2, 3]**.

  Oil does not wear out it just gets dirty after use, it still has lubricating value and heat value. Following the proper treatment to remove contaminants, used oil can be: Rerefined in to base lubricating oil, Reprocessed as fuel oil, Used as feedstock to produce petroleum based products or other commercially valuable products via different processes.

  The most encouraged option for recycling those oils even by governments is: it does not only save a tremendous amount of time and money but more importantly helps to conserve our natural nonrenewable resources**.** The present work aims to find a complete recycling process wish could all so be industrially feasible by our government, the first part of the work is based on finding a cheap solvent or mixter of solvents witch can reduce sludge concentration in used lubricating oil to a minimum level with higher recovery and the second part deals with adsorbent witch can produce base oil **[4, 5, 6 ,7,8]**.

  This thesis is devised to three chapters, the first chapter contains general information about lubricating oil and its properties, used oil and its properties and the existing treatment technologies in the world. The second chapter is the experimental part which includes the treatment steps of the used motor oil and the conditions of work. The third chapter shows the results of the experiments and the discussion of those results.

  The present work aims at finding a complete recycling processe wish could all so be industrially feasible by our government, the first part of the work is based on finding a cheap solvent or mixter of solvents wich can reduce sludge concentration in used lub oil to a minimum level with higher recovery and the second part deals with adsorbent wich can produce base oil.

I.1. Base Oils from Petroleum

Most of lubricants are made of base oils and variety of chemical additives. The majority of the base oils is produced from the refining of crude oil since large crude oil refining operations can produce base oils with high quality and excellent performance at an economical price. Base oil composition may vary depending on the nature crude oil it is made from. For differen applications, lubricants ar formulated by blending different base oils and additives to meet a series of performance specifications. Hence, formulations and performance specifications for automotive lubricants, industrial lubricants, aviation lubricants, and marine lubricants are greatly different **[9].**

Base oil plants are integrated with the mainstream refinery process. Overall production capacity for lubricant base oils is only about 1 percent of the total refinery production lines. Figure I.1 indicates where a lube plant fits into a typical refinery process scheme and the inter-relationship between each step **[9].** Figure I.2 is a typical process scheme for base oil production in which the numbers roughly indicate the relative amount of the intermediates and final products throughout the manufacturing process **[9].**
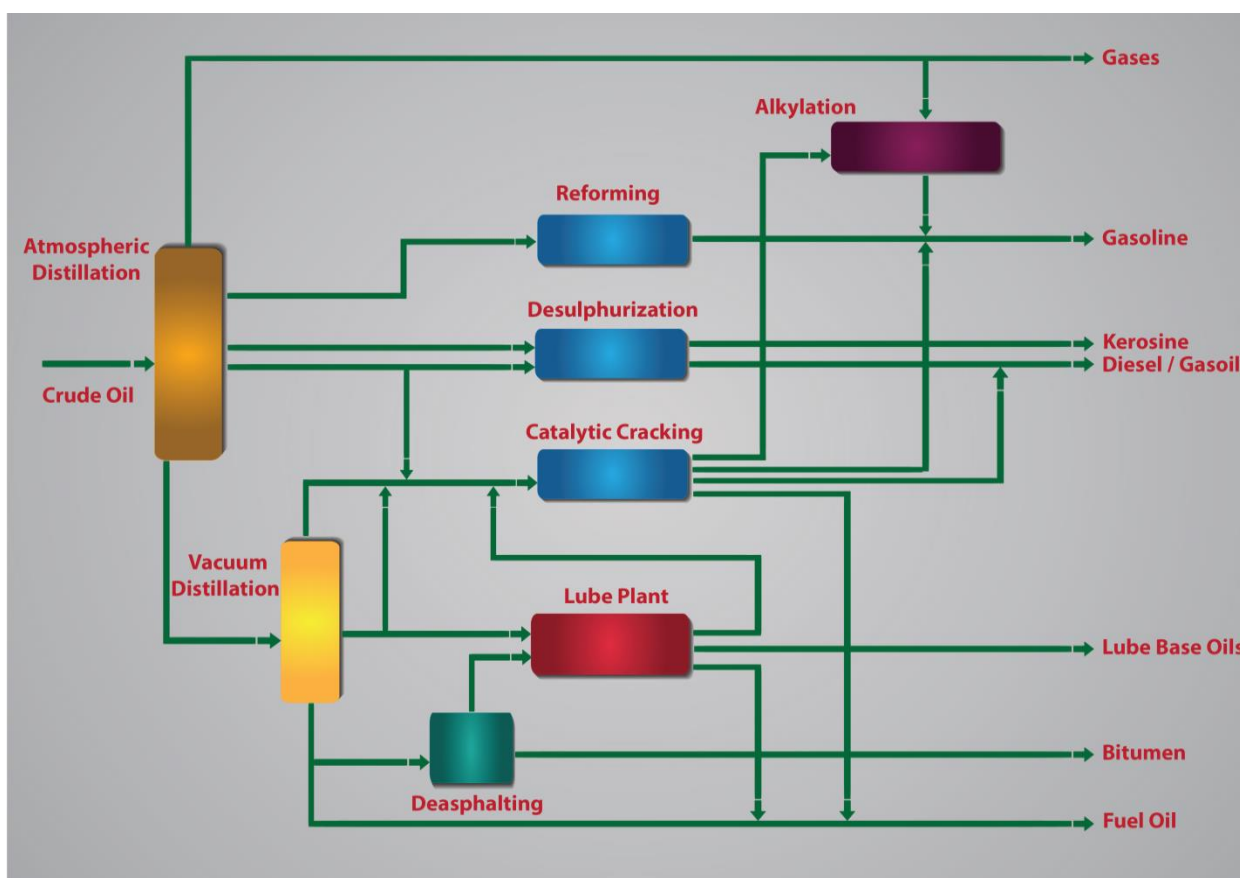
Figure I.1. Indicates where a lube plant fits into a typical refinery process scheme and the inter-relationship between each step
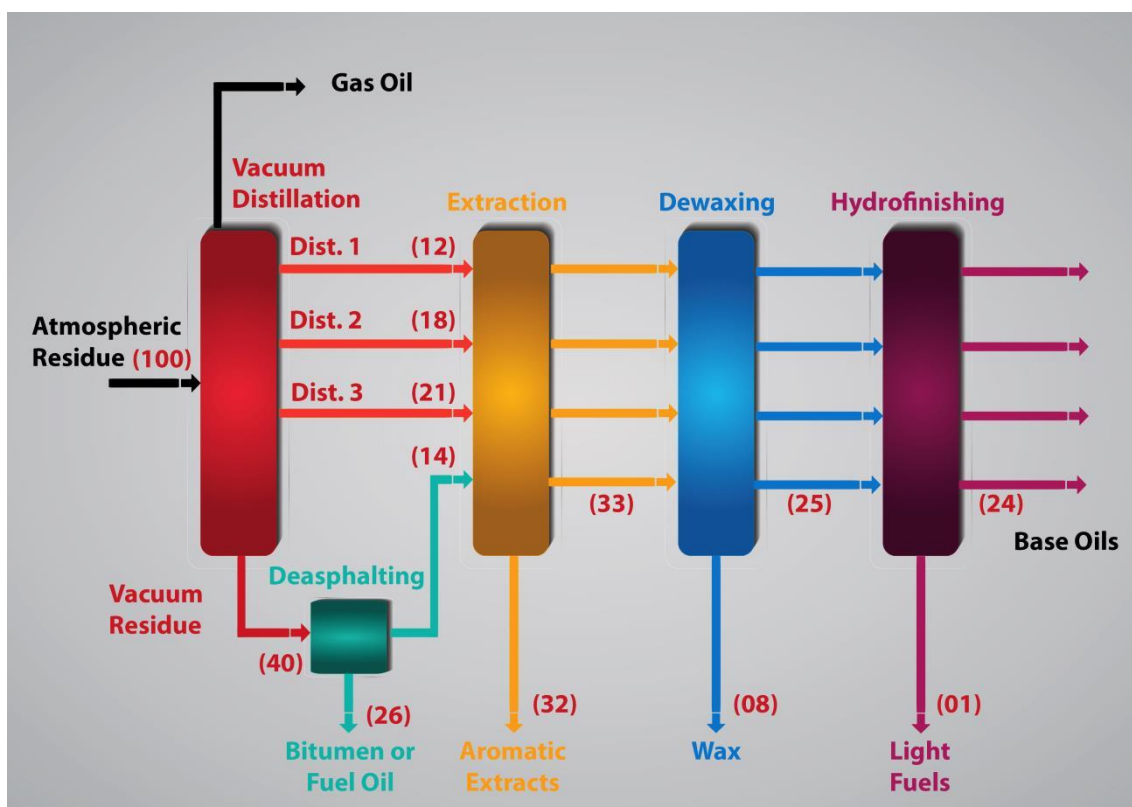
Figure I.2. Typical process scheme for base oil production steps

I.2. Properties of lubricating oils

I.2.1. Density: Density is the ratio of mass of a substance to the volume of the substance **[10].**

I.2.2. Specific gravity: is the ratio of density of substance to the density of water determined at the same temperature. The level of impurities in the used oil is indicated by the density and specific gravity. The specific gravity increases with the increase of aromatic content in the oil. Used engine oil has higher density and specific gravity due to the presence of contaminants in it**[10].**

I.2.3. Viscosity: Viscosity is the resistance offered to the flow of fluid. Viscosity testing of oil indicates the presence of contaminants in it. The viscosity of the lubricating oil decreases due to the addition of fuel, water and other contaminants added to it during its time inside the engine. Viscosity of the oil plays a cardinal role in reducing friction and it should be high **[10].**

I.2.4. Viscosity index: Viscosity index is a number that indicates the change in viscosity at different temperatures. If the viscosity index is high, the viscosity change with the temperature is less. This means that the oil has higher thermal stability and provides good engine protection **[10].**

I.2.5. Flash point: Flash point is the minimum temperature at which the vapor produced by heating the oil produces a momentary flame when introduced to an ignition source. A low value

of flash point indicates the addition of volatile products to the lubricating oil and the presence of contaminants **[10].**

I.2.6. Fire point: Fire point is the minimum temperature at which the vapor produced by heating the oil continues to burn when introduced to an ignition source. The fire point of oil indicates the maximum temperature up to which the lubricating oil is efficacious. With the continued usage of oil in the engine, the oil gets contaminate with the impurities and leads to a decrease in the fire point. This is undesirable and the oil needs to be replaced when the fire point temperature reaches a lowest value **[10].**

I.2.7. Pour point: The pour point is the minimum temperature at which the oil can flow freely inside the engine. The lubricating oil contains waxes and paraffins, which causes the oil to solidify at lower temperatures. The oil should have higher pour point or else the flow of oil into the oil pump and into the various engine parts is affected at low temperatures **[10].**

I.2.8. Cloud point: Cloud point temperature indicates the presence of wax in the lubricating oil, which forms a cloudy appearance. The presence of high volume of wax stymies the flow of oil inside the engine and blocks the fuel injectors at cold temperatures. The cloud point temperature is determined by cooling the oil sample and noting the temperature at which a cloudy appearance is formed **[10].**

I.2.9. Aniline point: The aniline point refers to a temperature at which equal amount of aniline and the lubricating oil mixes completely. The aniline point is a measure of aromatic content present in the oil **[10].**

I.2.10. Total acid number: The Total Acid Number (TAN) indicates the amount of acid content present in the oil. The TAN is the amount of potassium hydroxide required to neutralize the acid present in one gram of the oil. The engine oil undergoes oxidation at elevated temperatures producing carbonyl products and carboxylic acid. Higher TAN indicates the higher acid content and the oil needs replacement **[10].**

I.3. Automotive Lube Oil

For special performance specifications, automotive lube oil has unique characteristics different from industrial lube oils. The most important responsibility of the automotive lube oil is to form a layer between metal surfaces of various engine parts so that friction and wear is minimized. It also serves as a sealant to fill the microscopic ridges and valleys in any metal surfaces to increase the engine's efficiency **[7].**

Lube oil also acts as the coolant in the automatic transmission and engine. It helps to take away the heat from the combustion of fuel as well as from friction. In addition, it serves as a cleaning agent to carry away dirt or other debris that may damage the bearings or other parts that are operated in tight tolerance. Debris is removed through the engine oil filter or the transmission filter. The detergent additives in the auto lube oil can combat the combustion by-products. Burning gasoline or diesel fuel produces acids, moisture, soda, ash and other contaminants. Hence, detergent would fight these by-products and inhibit their buildup as sludge or varnishes **[7].**

In order to meet the performance specification as mentioned above, a specific additive package is required to prolong the oil's life and improve oil's protective qualities. The figure I.3 represents the constituents of lub oil **[7]**. The major additives in automotive lube oils are listed in the table I.1 **[7].**

Table I.1: The major additives in automotive lube oils

Additives	Performance
Detergents	To keep high temperature engine parts clean and free from deposits.
Dispersants	To suspend and disperse sludge- and varnishes-forming materials and prevent them to clog the engine.
Anti-wear	To add film strength to prevent wear of heavily loaded surfaces.
Friction modifiers	To reduce the friction losses throughout the engine.
Corrosion inhibitors	To fight the rust and wear caused by acids and moisture.
Oxidation inhibitors	To prevent oil and oxygen combination (produces damaging materials) and to reduce thickening of the oil and sludge formation.
Foam inhibitors	To limit the growth of bubbles and break them up quickly in the oil, allow the oil pump to only circulate oil, not oil and air throughout the engine.
Viscosity Index **improver**	To fight viscosity change with temperature variations.
Pour point depressant	To improve the oil's ability in winter to flow at very low temperature**.**
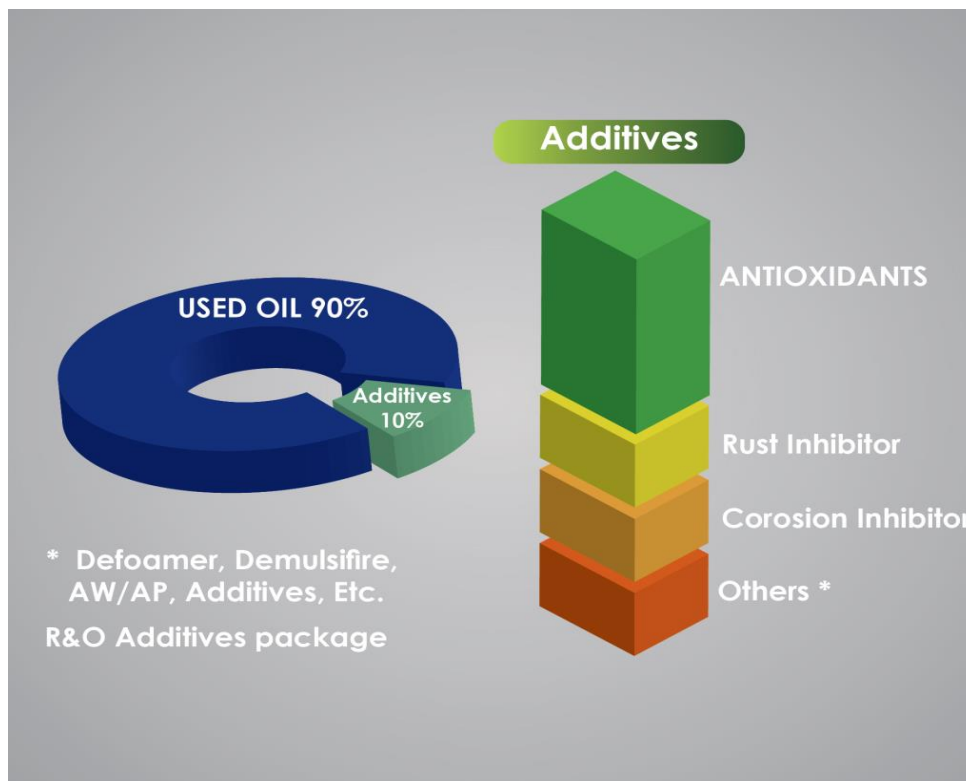
Figure I.3. Constituents of lubricating oil

1.4. Used Lube Oil and its properties

1.4.1. Difinition of used oil

The term "Used Oil" applies to any oil that is no longer useful to the original purchaser as a consequence of extended storage or contamination with hazerdous non-hazardous impurities such as dirt and water **[9].**

- Additive packages blended into base oils range from 10 to 20 percent, by volume, of the finished lubricants. Performance of the lubricants deteriorates over time as the additives are chemically changed and the oil becomes contaminated.

Contamination sources could be **[9]**:

- The breakdown of the additives and their subsequent reaction.
- Dirt and dust, metal particles from engine wear.
- Residual gasoline or diesel fuels from incomplete combustion.
- Water from combustion, blowby vapors, and rain water/salt water ingress.
- The mixing or dumping of other materials (chemicals or other oil types) into used oil.

Some industrial lubricants, such as transformer oils and hydraulic fluids, can be treated relatively easily and recycled. They can be readily collected and segregated without potential

cross contamination. These oils may be regenerated to a recognized standard and returned to the original source while automotive engine oils often require a more sophisticated treatment to regenerate usable base fluids to acceptable quality **[9]**.

Automotive used oils tend to have high concentrations of potentially hazardous heavy metals. Industrial oils tend to have high levels of chlorinated hydrocarbons and PCBs. The contaminants typically found in used oils are listed in table I.2 .It should be noted that most of these contaminants are known to be carcinogenic and listed as priority pollutants by the U.S. EPA **[9].**

Table I.2: The contaminants typically found in used oils

Categories components	Automotive Used Oil Concentration(ppm)	Industrial Used Oil Concentration(ppm)
Metals		
Arsenic	5-25 ppm	NG
Barium	50-500 ppm	NG
Cadmium	2 ppm	NG
Chromium	3-30 ppm	NG
Lead	100-1200 ppm	NG
Zinc	100-1200 ppm	NG
Chlorinated Hydrocarbons		
Dichlorodifluoromethane		NG
Trichlorotrifluoroethane		NG
Tetrachloroethylene	⇒ 1000-4000 ppm	
1,1,1-Trichloroethane		⇒ 1000-6000 ppm
Trichloroethylene		
Total chlorine	1000-4000 ppm	1000-6000 ppm
Other Organic Compounds		
Benzene	100-300 ppm	100-300 ppm
Toluene	500-5000 ppm	500-5000 ppm
Xylene	500-5000 ppm	500-5000 ppm
Benzo(a)anthracene	10-50 ppm	NG
Benzo(a)pyrene	5-20 ppm	NG
Naphthalene	100-1400 ppm	NG
PCBs	NG*-20ppm	100 -1000 ppm

I.5. Sources of waste lubricating oils

The largest source for used oil in developing countries is lubricating oils from motor vehicles, combustion engines and gear boxes. Apart from that, minor amounts are generated from hydraulic systems, transformers and other diverse industrial applications. Due to increase of the automotive traffic in developing countries, the amount of used oil from motor vehicles increased steadily in the past. The majority of used engine oil is generated in small quantities at a great number of places, e.g. garages, small workshops and private premises. There are few major generator of waste oil like railways, large truck fleet operators and large industries **[9].**

Contaminated lubricating oil must be changed and removed from the automobile after a few thousand kilometres of driving because of stress from serious deterioration in service. The amount of lubricating oils that is collected annually in Europe and USA is very large, approximately 1.7 to 3.5 million tons. This large amount of waste engine oils has a significant impact on both economic and environmental aspects **[9].** Figure I.4 represents the sources and percentage of contribution of waste oils **[11].**
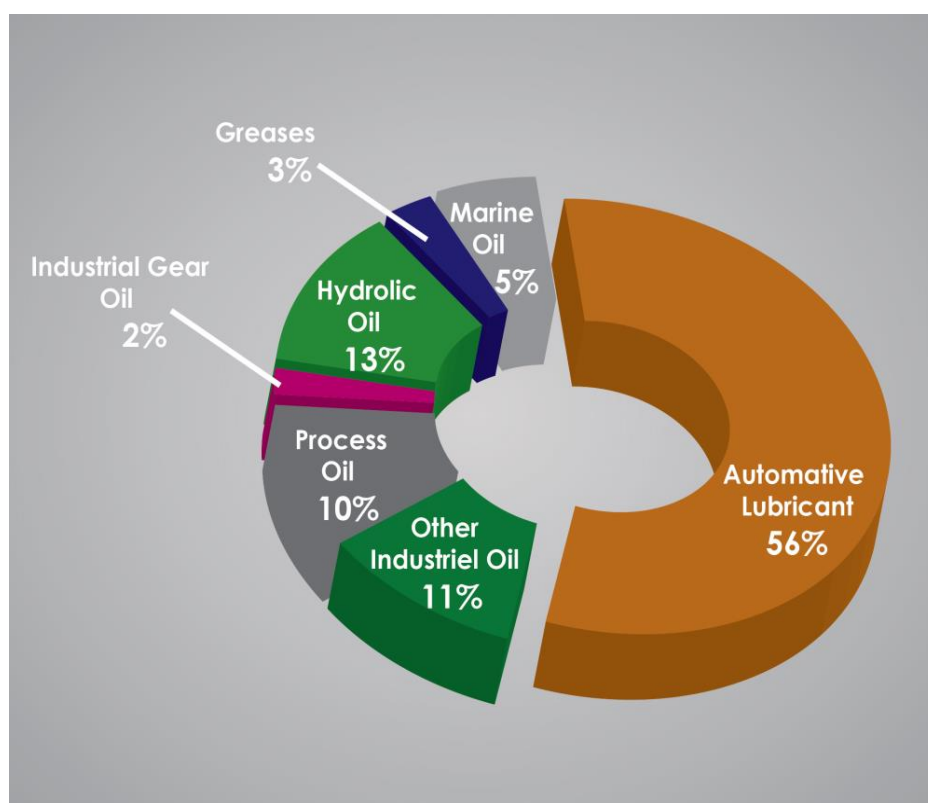
Figure I.4. The sources and percentage of contribution of waste oils

I.6. The impact of used oil on humen helth and envirement:

Used mineral oils are classified according to current European regulations as hazardous waste due to the effects that they can have both on health and environment.

I.6.1. The direct effects on humen health:

Includes the following **[9]**:

- Irritation of lung tissue due to the presence of gases that contain aldehydes, ketone, aromatic compounds,… etc.
- The presence of chemical elements such as Cl (chlorine), NO_2 (nitrogen dioxide), H_2S (hydrogen sulphide), Sb (antimony), Cr (chrome), Ni (nickel), Cd (cadmium), and Cu (copper) that affect the upper respiratory tract and lung tissue.
- They produce asphyxiating effects that prevent oxygen transportation due to their content of carbon monoxide, halide solvents, hydrogen sulphide, etc.
- Carcinogenic effects on the prostate and lungs due to the presence of metals such as lead, cadmium, manganese,… etc.

I.6.2. The direct effects on the environment

Includes the following **[9]**:

- According to EPA, just one quart of used oil is able to make 1 million gallons of water undrinkable.
- When used oil enters surface water, oil films will block sunlight, impair photosynthesis, and prevent the replenishment of dissolved oxygen, which lead to the death of aquatic plants and animals.
- Used oil filters are not regulated as hazardous waste for most states and are allowed to be disposed in municipal landfills. Serious problems for the groundwater supplies surrounding the landfills are caused by residual oils from the filters that leach into the ground.
- Used oil that is dumped into soil can be washed into surface water by rain or snow, or it can seep through the soil into groundwater to contaminate our water sources.
- Used oil in the soil can also evaporate into the air. The contaminants in used oil that enter the air through evaporation or improper burning can then settle, or be washed by rain or melting snow, into surface water or into the soil.

I.7. The Importance of Recycling and Reusing Used Oils

Used oil is definitely a vital source of energy. Oil does not wear out, it just gets dirty after use.Used oil can be re-refined into base lube oil, reprocessed as fuel oil, or used to as feedstock to produce petroleum-based products or other commercially valuable products via

different methods. There are some facts about conserving resources by proper handling of used oil. Re-refining used oil takes about 1/3 of the energy needed to refine crude oil to lubricant quality, and one gallon of used lube oil that is re-refined produces the same 2.5 quarts of lubricating oil as 42 gallons of crude oil does (API, 1996). Collecting and recycling used oil, therefore, not only protects our environment from used oil contamination, but also conserves a valuable non-renewable resource **[9].**

From the standpoint of energy conservation, recycling used oil efficiently saves a precious, nonrenewable resource. Only one out of 70 barrels of crude oil is processed into virgin lube stock. That's why many oil companies spend billions of dollars on exploring, recovering, and refining crude oil into quality lube oil. If the used oil generated by consumers can be recycled back to useful products such as pure lube oil again and again, tremendous time and money can be saved. More importantly, our natural resources will be conserved **[9].**

I.8. Management systems for used oils

I.8.1. Burning for Energy Recovery

Used oil can be thermally destroyed by incineration or combusted for energy utilization. Due to the economic benefit of recycling used oil, only a very small quantity of used oil is currently incinerated. The used oil that is incinerated generally has high concentrations of toxic contaminants that make recycling impractical or unsafe. Burning used oil is to utilize oil's heating value and reduces the consumption of the non-renewable fossil fuels. It can create significant environmental problems however, by releasing major and trace elements and organic contaminants to the atmosphere. Proper flue gas pollution control equipment should be equipped with the cement kilns or industrial/utility boilers to minimize this problem. Otherwise, only the used oil that meets specified standards for maximum contaminant levels and minimum heating values can be burned. All used oil collectors for the purpose of resale as fuel must document whether or not the waste meets the standards **[9].**

EPA has to regulate used oil burning because toxic contaminants can end up in the smoke and ashes. A service station, county garage or other small site user does not need a regulatory permit for burning used oil if all the following conditions are met **[9]**:

- 🌐 The heater is designed to have a maximum capacity of not more than 500,000 BTUs per hour.
- 🌐 The heater burns only used oil that the owner or operator generates or that is received from DIYers who changed their oil at home.
- 🌐 Absolutely nothing has been added to the used motor oil no antifreeze, no solvents, and no other liquids.

🌑 The heater is vented to the outdoors.

I.8.2. Reprocessing

One of the economically attractive ways to manage used oil is to burn it as fuel. Thus, most used oil being reprocessed is utilized as fuel. Compared to direct burning, the major advantage from reprocessing used oil is that it improves the burning quality of used oil by removing/reducing some contaminants. A typical used oil reprocessing system is simplified as shown in Figure I.5. Processes such as adding chemicals, heating, filtration, and centrifugation or combinations of the above can separate the undesirable constituents from valuable portions. Odor produced from nitrogen and sulfur species is the greatest concern in operating these facilities. Severe corrosion may occur in the equipment when acidic gases from additive elements and water vapors combine during the process. In reality, processing used oil is a widespread industry. Reprocessors and re-refiners often have to compete with each other to acquire used oil stock from independent transporters. Reprocessing, however, is a much more readily available recycling option than re-refining **[9].**
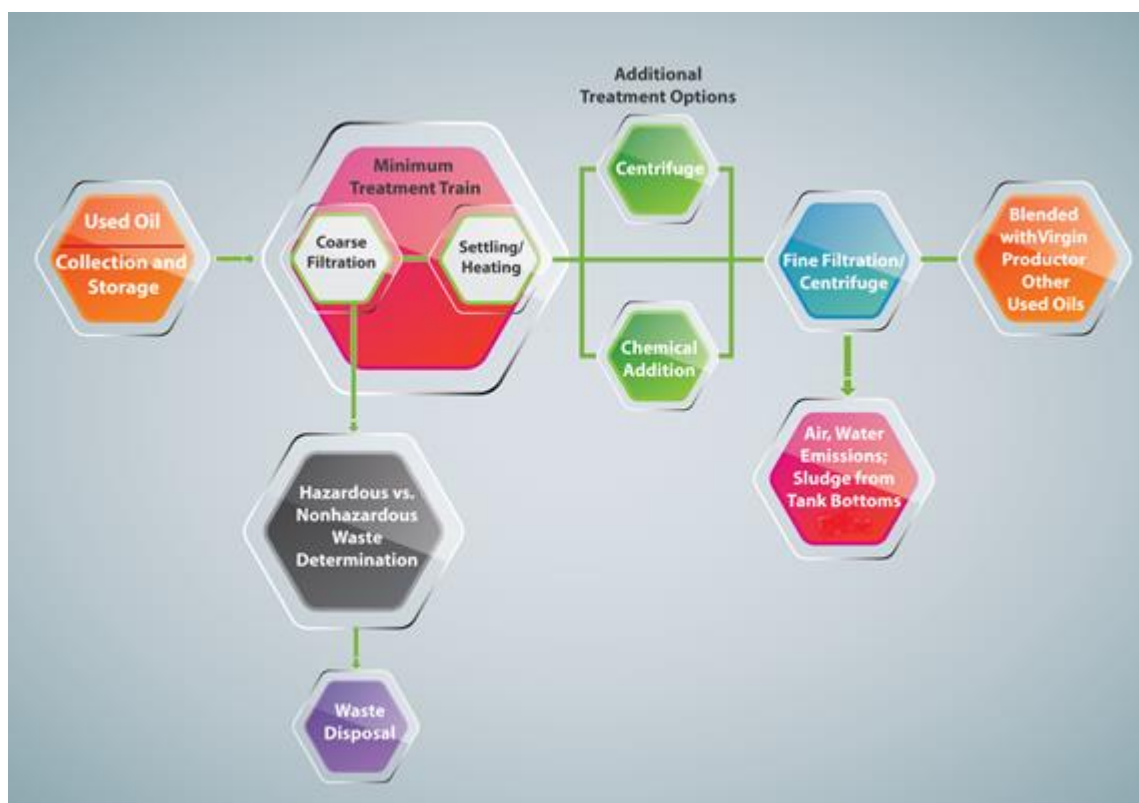

Figure I.5. Simplified used oil reprocessing system

I.8.3. Re-refining

Used oil can be re-refined into base lube oil. Lube oil is a premium substance that can be re-refined and reused again and again. In general, water and dissolved low boiling point organic are removed by atmospheric or moderate vacuum distillation. Lube oil is then recovered

and fractionated by distillation. Light ends by-products are commonly used for plant combustion fuels. Diesel fraction and gas oil fractions can be recovered as high quality by-products after further advanced treatment. Residual streams from distillation can be used by asphalt industry as an asphalt flux to produce roofing asphalt, paving asphalt, insulating materials, and other asphalt based products **[9].**

The major differences among all the processes are in how additives, impurities and sludges are removed. Considering technical feasibility, economical profitability, and operational easiness, re-refining processes are narrowed down to combinations of solvent treatment, distillation, hydrotreating, vacuum distillation, clay polishing, chemical treatment, and demetallization. Marketability of products and by-products will affect the combination choices of the processes. Processes, which do not generate hazardous wastes that are difficult to handle, will be preferred in the future. Another problem is that the retail acceptance of recycled refined base oils in their blends, not because of quality issues, but mainly because of public acceptance. The public needs to learn that re-refining of used motor oil is a very sophisticated process that produces high quality base lube oil. Re-refined oil carrying the API logo meets the same high quality standards as lube oil made from crude oils **[9].**

Although there are many companies devoting their efforts to developing technologies for re-refining used oils, profitable used oil re-refining has not been so widespread due to the high capital and operating cost **[9].**

The most recent and advanced technologies use distillation/hydrotreatment. The process is as shown in figure I.6 .Not all available used oil streams are selected to proceed through this process. The better feedstocks include automotive and railroad crankcase oils, hydraulic and transmission fluids, and other high quality used oil streams. Potential feedstocks with high water, excessive contamination, or animal fats are processed as fuel. After pretreatment and thin-film distillation, the base oil fraction is then hydrotreated under moderate conditions and yields a range of base oil streams with different viscosities. By-products from this process are low boiling distillates, gas oils, and non-hazardous asphaltic residues. Metals are removed in the distillation step while higher boiling halogenates and polar compounds are removed in hydrotreating step, thereby reducing the acid number **[9].**
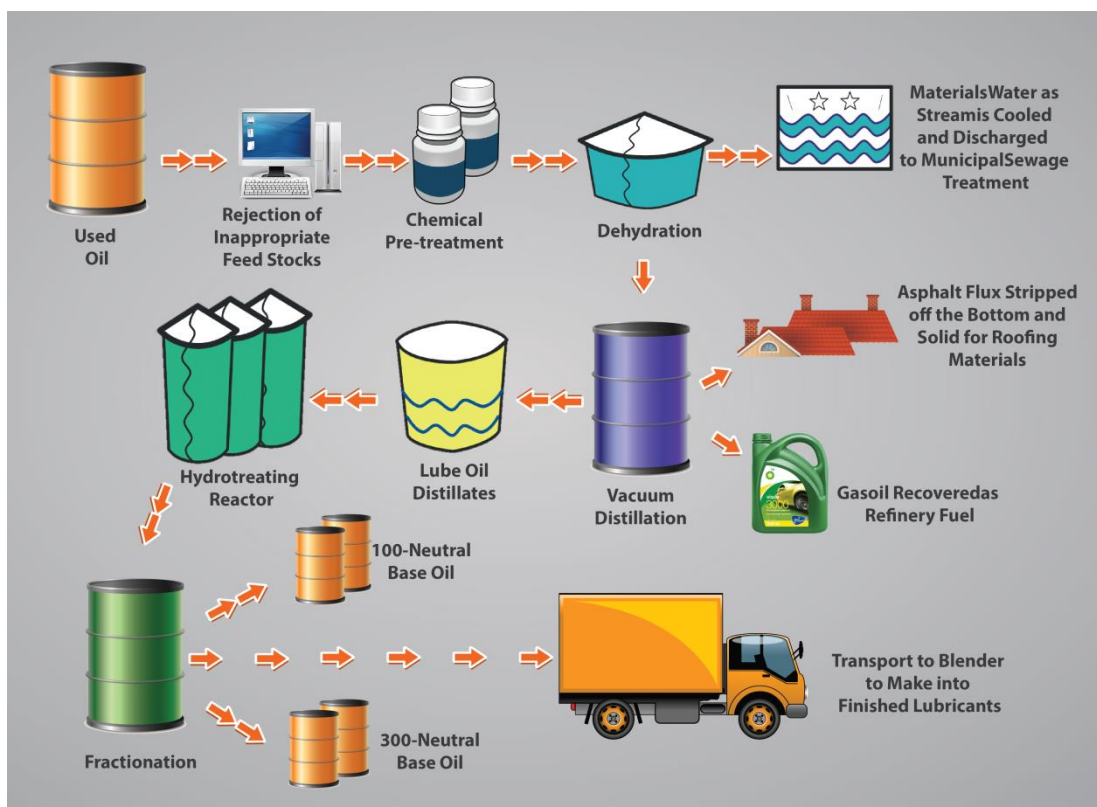
Figure I.6. Simplified schematic diagram of a re-refining process for used oil

Re-refined lube oil has to meet strict standards and must be approved by API. It has to contain at least 40% by volume re-refined oil in the base stock and contain limited amounts of certain toxic chemicals in base stock **[9]**:

- Organic halide < 5ppm total.
- PCBs < 1 ppm total.
- RCRA solvent waste < 1 ppm.
- Metals (Cd, As, Cr, Pb, Ba, Zn < 10 ppm, individual < 2 ppm).
- Benzo(a)pyrene and benzo(a)anthracene < 5 ppm each.

The re-refining of used motor oil is a very efficient process. According to EOR, 1.41 gallons of wet used motor oil can yield 1 gallon of re-refined motor oil. Comparing this value to crude oil refining, it would take about 84 gallons of crude to yield 1 gallon of motor oil (data from American Petroleum Industry based on 1995 average yields for U.S. refiners). Of course, we cannot simply compare those ratios and conclude that refining from crude oil is inefficient. Refining crude oil obtains a large amount of fuels of various types. Below is the comparison of refining from used motor oil and refining from crude oil **[9].**

Table I.3: Comparison of refining from used motor oil and refining from crude oil

Refining one unit of crude oil yields:	Re-refining one unit of used motor oil yields:
84% fuels (46% Gasoline, 38% others)	5% fuels
9% gases	14% asphalt
4% coke	10% water
3% asphalt and road oil	71% lube oil
3% petrochemical feedstocks	
1% lube oil	

The table below chows a Comparaison of waste oil management techniques **[11].**

Table I.4: Comparaison of waste oil management techniques

Technique	Advantage	Disadvantage
Re-refining	• Environmentally sound long-term solution. • Created jobs. • Reduces the amount of imported lubricant oil.	• Require a well-developed collection system. • Re-refined lube oil requires well-developed market. • Requires extensive capital investment. • The re-refining option requires a reputable recycling company to ensure the marketability of the product. • Proper disposal of end-waste residues are costly.
Reprocessing	• Good substitute for second grade fuels. • Limits the negative effects of the practice of uncontrolled burning of waste. • The quality control of the re-processed fuel oil is monitored by the purchaser.	• Requires a well-developed collection system. • Requires extensive capital investment. • Proper disposal of end-waste residues are costly.
Incineration	• Economically feasible at lower processing volumes. • Cement factories are willing to procure the waste oil. • Less capital intensive than the previous options • Concentrates waste oil disposal to limited sites that can be more easily reglated and controlled.	• Air emissions, although minimal, still need to be addressed. • Opposition by regulatory and government institutions.

I.9. Treatment technologies

The different processes that exist are described below and are grouped according to the basic technologies being used **[12]**:

· Technologies based on acid / earth treatment.
· Technologies based on vacuum distillation and hydrogenation.
· Technologies based on vacuum distillation and earth treatment.

I.9.1.1. Acid / earth treatment processes

These are processes that are nowadays obsolete. These processes are basically as follows:
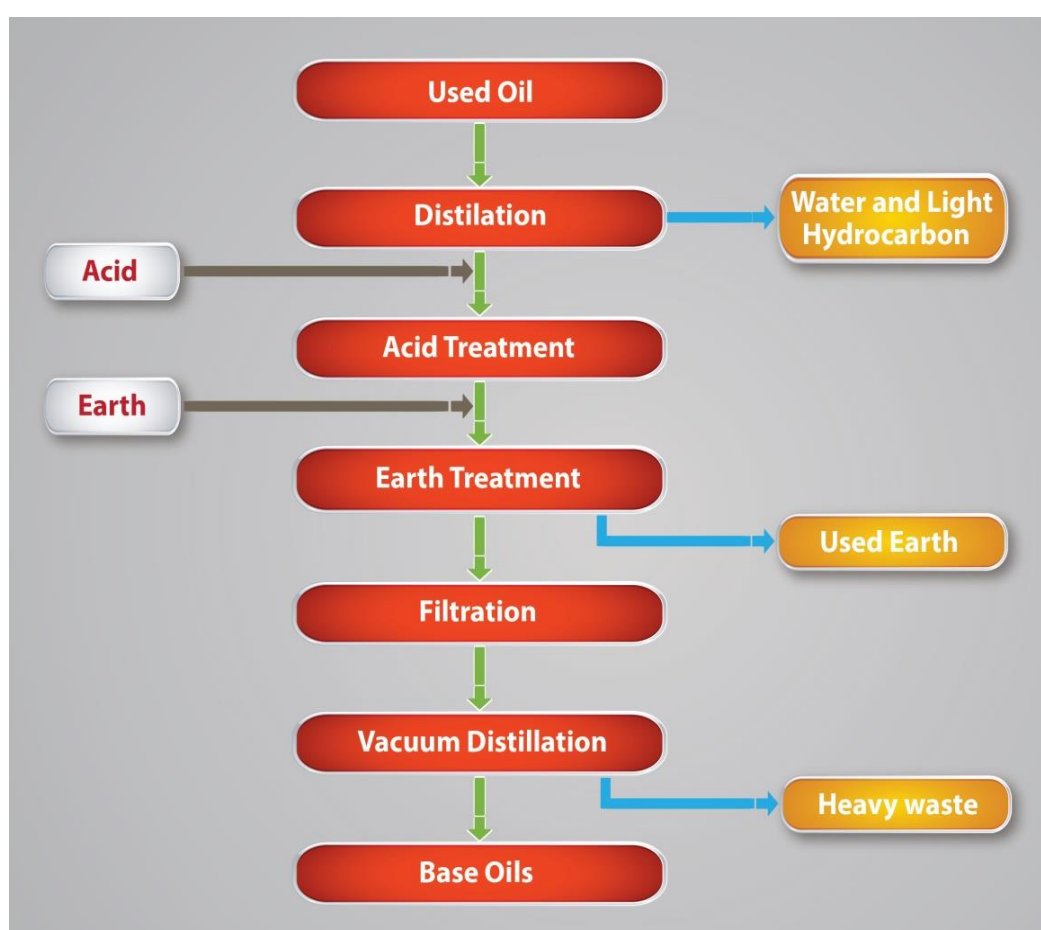
Figure I.7. Basic outline of acid/earth treatment technologies

Acid/earth technologies based on treating the substrate with sulphuric acid to eliminate the polluting substances and then treatment it with earth to neutralise the resultant product. In this way, treatment with earth gives the required colour and odour. It does raise the problem of how the acid waste that is generated going to be used and applied, which in many cases is toxic and hazardous with the resulting problems of disposal **[12].**

I.9.1.2. Meinken technology

Meinken technology is no longer used for economic reasons and because of the problems that it generates with the treatment of acid earth. It also involves problems of internal corrosion and disposal **[12].**

There are some refineries that work with modified Meinken technology at the present time. The inclusion of thin film and contact distillation techniques enables them to reduce the quantity of sulphuric acid to 3% and of earth to 3.5%. Some of them also include hydrogenation.

The main advantages of this process are the low investment and maintenance costs, the possibility of treating low quality used oils, and the flexibility and ease of handling the process itself **[12].**

I.9.2. Vacuum distillation and hydrogenation processes

These types of process are the ones that are most used in Italy, where 93% of collected oil is treated by these technologies.

The following diagram gives a general outline of technologies based on vacuum distillation and hydrogenation **[12].**
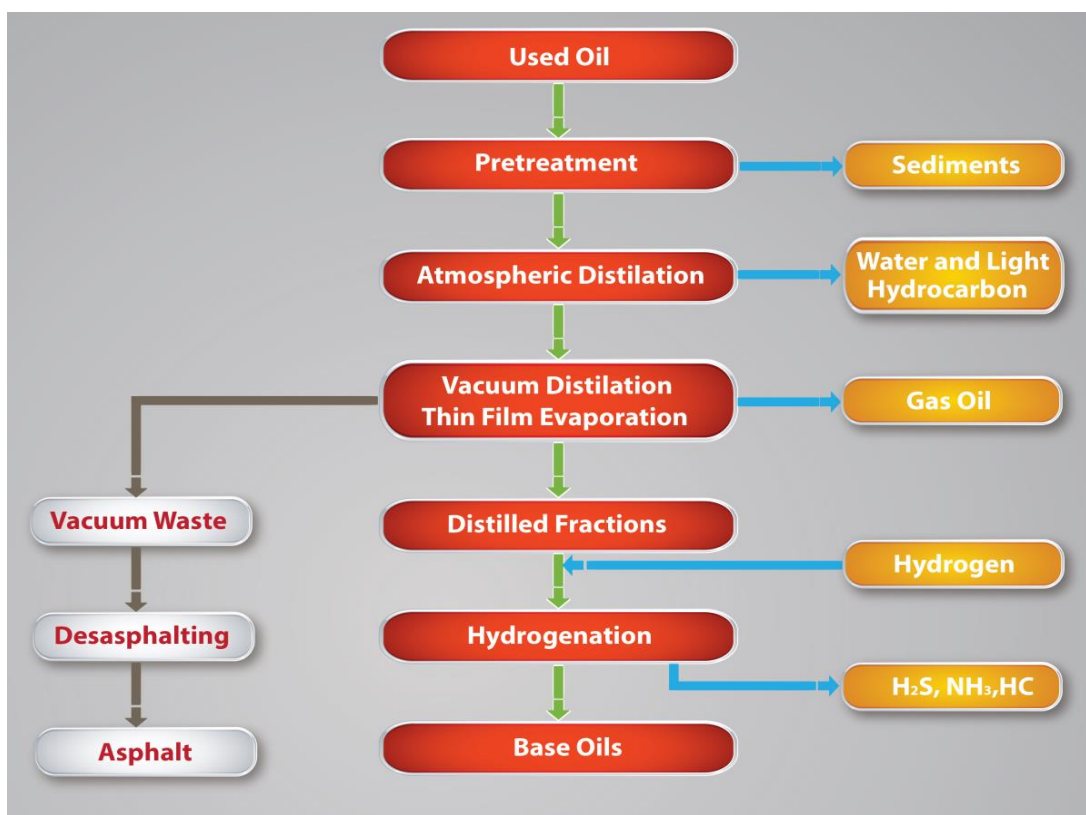
Figure I.8. Diagram of vacuum distillation and hydrogenation processes

I.9.2.1 KTI technology

The KTI process (Kinetics Technology International), also known as KTI Relube Technology**.**

The first re-refinery based on this technology was established in Greece in 1992. Plants of this type also exist in Tunisia and California **[12].**

The basic steps of the process are as follows in the diagram appears below:
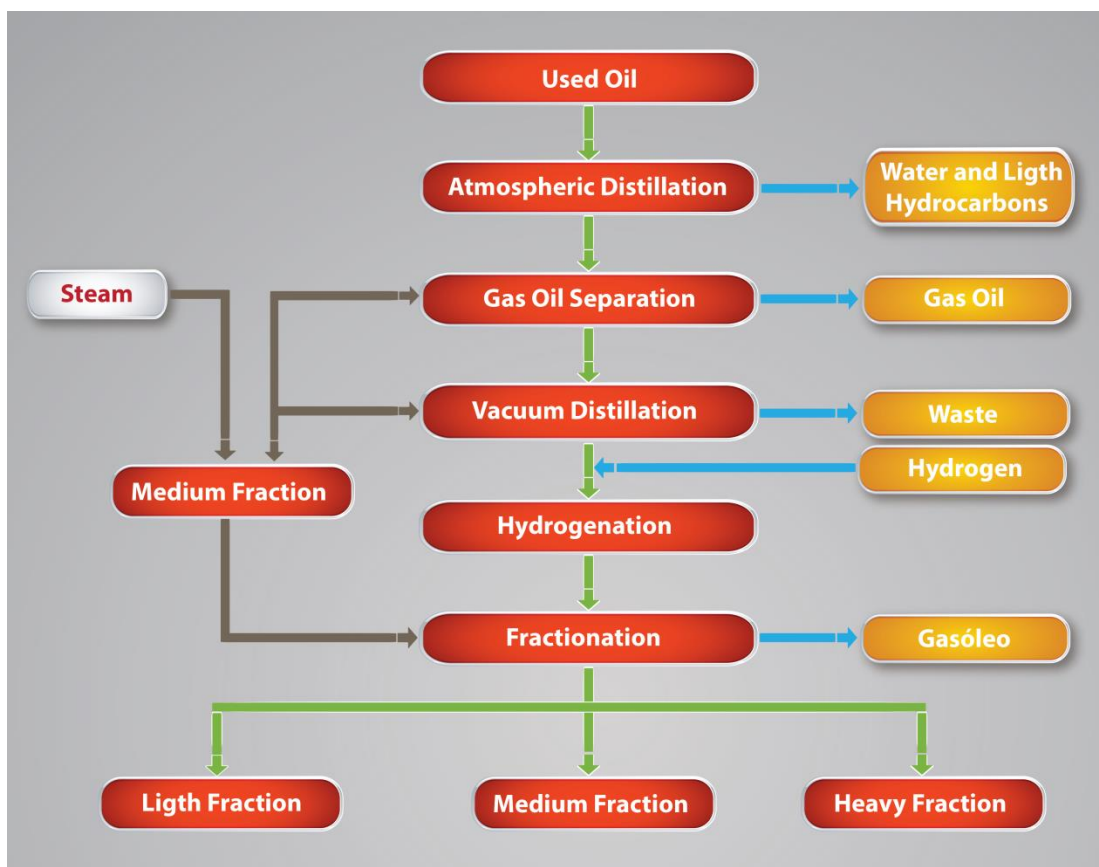
Figure 1.9: Diagram of KTI technology

I.9.3. Vacuum distillation and earth treatment processes

I.9.3.1. Viscolube technology

Viscolube technology, also known as TDA is based on the use of propane, followed by vacuum distillation and an end earth treatment.

The first plant to use this technology was put into operation in Pieve Fissiraga (Milan, Italy) in 1992, and two others have been set up since then in Poland (1994) and Italy (1995) **[12].**

The basic steps of the process are as follows in the diagram appears below:
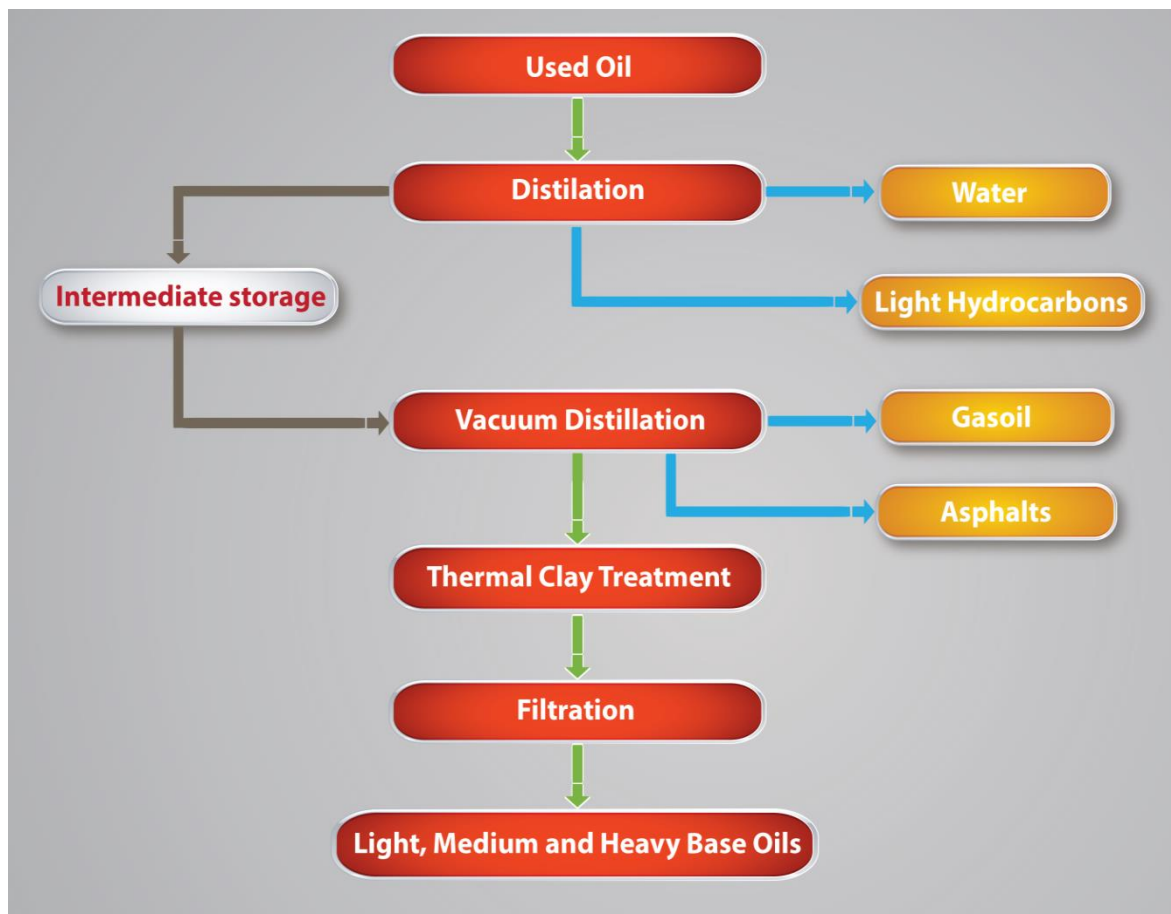
Figure I.10. Diagram of viscoluble technology

Chapter 2: Experimental Part

II.1. Materials and Equipment

In this experimental part, the equipments used are presented in **table II.1** and the chemical substences are presented in **table II.2** . oil samples were taken from "TOYOTA SOCIETY" in Ouargla were the used oil is changed after 5000 km of driving. That sample contains ; any types of rejected oils, from a variety number of cars.

Most of equipment and chemical products provided by KASDI MERBAH LABORATORY of chemical engineering, some of them were purchased from privet chemical supplies. Gasoline provided from the atmospheric distillation unit of Hassi Messaoud.

Table II.1: Materials and Equipment

Materials and Equipment Pictures		
Plastic bottle	Beakers	Test tube
Test tube holder	Pitri dish	Balance
Funnel	Pinch clamp	Ring clamp stand

Dropper	**Magnet**	**Filter paper**
Stopper	**Spatula**	**Separator funnel**
Buchner funnel	**Round bottom flack**	**Filtering flack**
Vacuum pump	**Heating ball**	**Thermocouple**

Mixer	**Oven**	**Condenser**
Rota vapor	**Refrigerant**	**Centrifuge**

The different kinds of used chemicals are listed in the table below with their pictures:

Table II.2: List of chemical products

	NFPA 704	Flash point	Boiling point	Density
Sulfuric acid		Non-flammable	337 °C	1.84 g/cm^3
Ethyl methyl ketone		-9 ° C	79.64 °C	0.8050 g/mL
Petroleum ether		< 0 °C	42–62 °C	0.653 g/mL
Acetic acid		40 °C	118 to 119 °C	1.049 g cm^{-3}
Toluene acid		6 °C	111 °C	0.87 g/mL
Propanol		22 °C	97 to 98 °C	0.803 g/Ml
Butanol		28.9 °C	117.7°C	0.81g.cm^{-3}

Note: NFPA 704: Standard System for the Identification of the Hazards of Materials for Emergency Response.
Red : Flammability
Bleu : Health
Yellow : Instability/Reactivity
White : Special Notice

II.2 Procedure of treatment

In order to treat and remove the impurities from the used lubricating oil many random experiments were carried out.

According to the variety of experiences and reaches done before by the scientists we picked out some methods to use it as a base to start our experimental work. We had proposed a scheme which combines the different steps of our experiences (scheme II.1).

Figure II.1. Scheme which combines the different steps of the treatment procedure

The work and the experiments we had done all the way are:

❖ First to prove the correctness of our proposed steps.

❖ Second and more importantly is to obtain fresh base oil with high quality and yield at the lowest cost possible.

The sample left to settle for 15 days before to start our work, in order to give the heaviest compounds in the used oil the adequate time to settle at the bottom of plastic bottle.

Figure II.2. Settling

II.2.1. Step one: Washing of used oil

The used motor oil contains different types of solids and heavy materials, so first they must be removed .In this step 5 liters of the used oil was washed by heated distilled water at 80ºc, in order to eliminate the heavy impurities .The washed oil left to settle for 48 hours in separators funnel, 3 phases were created, the top is the washed oil the medium is water and the last one is sludge.

Figure II.3. Washing and Settling

The washed oil was collected and used as a base for the different experiment.

Figure II.4. Washed oil

II.2.2. The first group of tests

Variety of tests had been done using different types of solvents. In order to find out which ones are the best for the extraction without the need for centrifugation. For that reason we used 10 ml of the used washed oil and treat it with (petroleum ether, sulfuric acid, gasoline, acetic acid and toluene, butanol, propanol,gasoline mixed with butanol, gasoline mixed with propanol and gasoline mixed with methyl ethyl ketone).

The tests with positive results are used as a base to start the second group of tests.

❖ **Selection of reagents used in the treatment process**.

- Environmentally friendly
- Cost effective
- High efficiency in used lubricant purification and hydrocarbon compounds recovery
- Recyclable and reusable
- Low operating temperature and pressure
- No harmful byproducts

❖ **Evaluation of Binary Solvent Process**

First group of tests confirms the result of binary solvent process as expected. Binary solvent process is capable to purifying used lubricants and recovering hydrocarbon compounds simultaneously under atmospheric pressure and at room temperature. Used organic solvents can be recycled and reused infinitely.

II.2.3. Second group of tests

In this part of the work, five experiments were done using different types of solvents, either one solvent or mixer of tow solvents. The used solvents are listed in the Table II.3 with their ratios. Test number one shows the steps of the rest of the process using the propanol for the extraction; the same procedure is followed for the rest of the four samples.

Table II.3: The ratios of solvents and charge for the experiments

Solvents	Ratio : Solvent / bisolvent	Ratio : Solvent / charge
Propanol, Gasoline	6:6	6:6:1
Propanol	-	6:1
Butanol, Gasoline	6:6	6:6:1
Butanol	-	6:1
MEK, Gasoline	4:2	4:2:1

II.2.3.1. Step two: Extraction by solvent

Solvents such as propanol ,butanol , methyl ethyl keton and gasoline are selective aromatic solvents employed in the solvent extraction by preparing five plastic bottles of 2000 ml.The oil and propanol were mixed to 1:6 ratio for 45 min as shown in Figure II.5 . The mixture (oil + solvent) left to settle for 48 hours .Then followed by filtration with filter paper as shown in Figure II.6.

Figure II.5. Mixing

Figure II.6. Filtration

II.2.3.2. Step three: Recovery of the solvent

The mixture of solvent and the oil was distilled to recover the solvent, at the boiling point of the different solvents, the distillation process was performed and condensed solvent was collected by the flask. The final product, which is the base oil, is obtained finally and left to cool at room temperature. The final products were left to be treated by bentonite.

Figure II.7. Solvent recovery

II.2.3.3. Step four: Adsorption by bentonite

The extracted and filtered oil was decolorized by passing through absorbent, types and particle size of adsorbent, adsorbent to oil ratio effects the efficiency of adsorption. The extracted oil mixed with 45% of activated clay for 30 min and heated on the same time at 40ºc as shown in Figure II.8. The mixture left to settle for 24 hours followed by separation of the oil from the contaminated clay as shown in Figure II.9.

Figure II.8: Mixing and heating of clay and used oil

Figure II.9. Settling and separation

II.2.2.1. Step five: Centrifugation

The oil was vacuum filtered in order to eliminate the particles of bentonite in the oil. Then followed by centrifugation for 4200 round /min in order to remove the suspended particles in the oil, they are so small contaminants particles as shown in Figure II.10.

Figure II.10. Centrifugation

The final regenerated oil is gathered and sends for characterization.

II.3. Analysis Methods (Properties and characterization of the products obtained (raffinat and extract))

The resulting products were physically characterized according to the American Society for Testing and Materials standard methods (ASTM, 2003). These standards are pour point (ASTM D-97), kinematic viscosity (ASTM D-445), refractive index (ASTM D-1747),density(ASTM D-1418), mean molecular weight (ASTM D-2502), color (ASTM D-1500), n-d-M analysis (ASTM D-3238) and sulfur content by using X-ray fluorescence sulfur meter (ASTM D-4294).

II.3.1.1. Determination of relative density according to ASTM-D 1217-93

For the density measurement a pycnometer with capacity of 25,138 ml is used, which is specified for petroleum fractions **[13]**.

The density of an oil is measured at 20 ° C. with respect to the water at 20 ° C. The pycnometer is weighed by a precision balance with its clean, dry cap. The weighing accuracy is 0.0002 g.

- The mass of the empty pycnometer is "a".
- The pycnometer is then filled with distilled water. The pycnometer is closed with a plug and immersed thoroughly in a water bath set at 20 ° C. for thirty minutes.
- Now remove the pycnometer from the water bath, wipe it with a clean dry cloth and weigh it with the same precision.
- The mass of the pycnometer with water is b
- The mass of water is equal to: d = b − a
- To determine the mass of the petroleum product in the same volume of the pycnometer, the same operations are carried out as to determine the mass of the water. Either the mass of the petroleum product is "c"

- The density of this product is given by : $$\rho_{20}^{20} = \frac{c-a}{b-a} = \frac{c-a}{d}$$ (II.1)

- c: mass of pycnometer with raffinat or output
- a: the empty pycnometer mass
- b: mass of pycnometer with water

Water bath

Thermometer

Pycnometer

Figure II.11. Measurement of the density

II.3.1.2. Determination of the refractive index according to ASTM-D 1218

The purpose of ASTM D 1218 **[13]** is to determine the refractive index and the refractive dispersion of light hydrocarbons with an ASTM color index of not more than 4. It is limited to the determination Of refractive indices between 1.33 and 1.50 for test temperatures of 20 to 30 ° C. The refractometer used and type of Abbe.

II.3.1.3. Determination of kinematic viscosity at (37.78 and 98.89 ° C) and at (40 and 100 °C) according to ASTM-D 445

The ASTM standard in D445 **[13]** describes a kinematic viscosity method for light and opaque liquid petroleum products.

It consists in measuring the time taken by a given volume of liquid to flow under standard conditions, by a calibrated capillary at a fixed temperature. The kinematic viscosity is calculated from the flow time by the formula:

C = viscometer constant which depends on the capillary size of the viscometer used.

T = flow time in seconds.

II.3.1.3.a Equipment

- A Ubblohde viscometer.
- A thermostat with pump.
- A water bath and an oil bath for a viscometer.
- A stopwatch.

The kinematic viscosity measurement at 40 ° C. is carried out in a water bath and at 100 ° C. in an oil bath. As the picture below shows.

II.3.1.3.b. Procedure

The method consists in measuring the time required for the flow by a calibrated capillary of a determined volume of the oil contained in the tank of a glass viscometer under an oil load at an exactly controlled temperature.

Before the test, the viscometer was washed with gasoline and acetone and then dried thoroughly.

The sample to be tested must be freed from water and filtered; The temperature required for the water bath (the variation should not exceed ± 0.3 ° C) should be established and maintained with careful circulation of the water through the thermostat pump. To determine the viscosity at 100 ° C., the oil bath (of glycerine) was used.

The tank of the selected viscometer is filled with 8 to 10 ml of sample of oil to be tested.

The viscometer is filled by suction of the oil from the small vessel closing a tube of the viscometer with the finger.

The viscometer is placed in the water bath, using a fixing device. The viscometer must be in the vertical position, see Figures II.12 and II.13. The liquid is sucked into the upper reservoir of the viscometer by means of a pair simultaneously closing the tube of the viscometer with the finger hanging for 10 minutes **[13]**.

Figure II.12. Position of viscometer in bath water   **Figure II.13. Position of viscometer in bath oil**

The oil begins to flow by gravity from the upper reservoir through the capillary into the lower reservoir. When the liquid level reaches the upper mark, the stopwatch is started. The stopwatch is disconnected when the liquid level reaches the lower mark.

After filling, the diagrams below show the situation of the Ubbelohde device when the stopwatch is started and stopped.
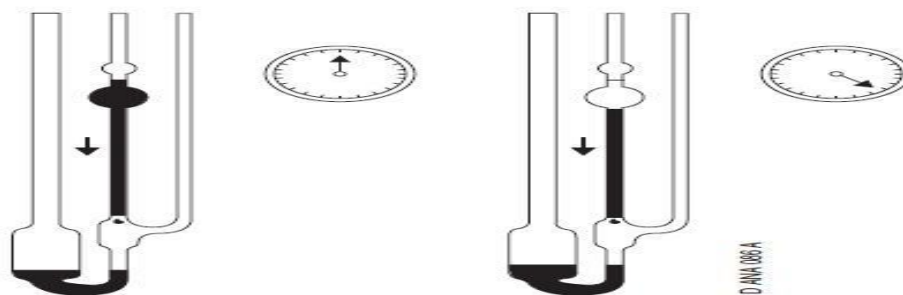
Figure II.14 Measurement of the flow time of a given volume of product at fixed temperature through a capillary tube

II.3.1.4. Determination of the average molecular weight according to ASTM-D 2502

The method ASTM-D 2502 **[13]** is a method for estimating the mass-average molecular mass of oils from the measurements of their viscosities 37 .78 and 98.89 ° C. It is applicable to oils having a molecular mass of between 250 and 700 g / mol.

The average molecular weight is obtained by a chart, see appendix 2, linking the viscosity to 98.89 ° C. and a parameter H, determined from the viscosity at 37.78 ° C., which is shown in appendix 3.

II.3.1.5. Point of flow and cloud point measurement
II.3.1.5.a. Measurement of pour point

The ASTM standard in D97 **[13]** describes a pour point method for petroleum products. The pour point was in a refrigerator.

II.3.1.5.a.1. Apparatus
A test tube, placed in a jacket forming an air bath;

- A jacket.
- A cooling bath.
- A thermometer for the test tube and an ordinary thermometer for the refrigeration bath.

II.3.1.5.a.2. Description

The vial, cylindrical of the clear glass, the flat bottom of outside diameter 33.2 to 34.8 mm, and 115 to 125 of height. The inside diameter of the flask may range from 30.0 to 32.4 mm, the vial will have a line to indicate a sample size 54 ± 3 mm above the inner bottom.

- The cap to fit the test flask, and centralized the test thermometer.
- Gasket to fit comfortably around the outside of the test flask. The pad can be made of rubber, cook, or any other material that is elastic enough to hang in the test flask and tough enough to take its shape. Its purpose is to prevent the test flask from touching the jacket.
- Required bath temperatures can be obtained by refrigeration if available, otherwise by appropriate refrigerant mixtures.
- Disc, cork or felt, 6mm thick to fit deep inside the jacket.
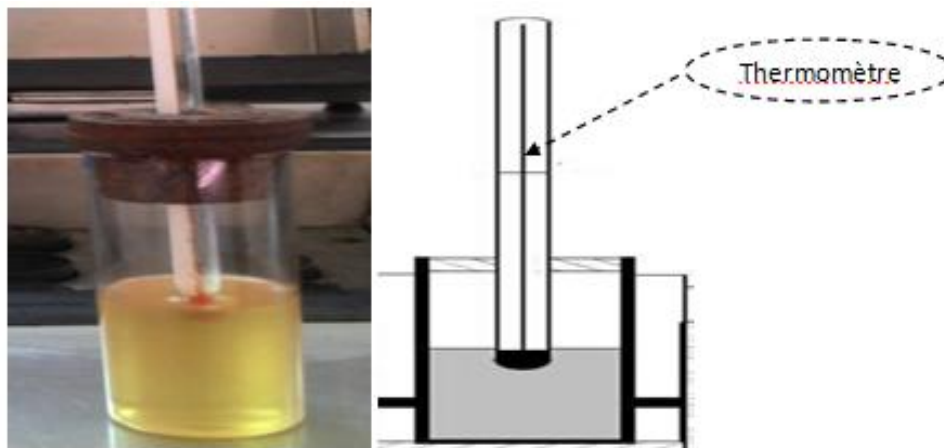
Figure II.15. Location of the pour point thermometer

The sample is then heated to 50 ° C. before being gradually cooled with increasingly cooler cooling baths.
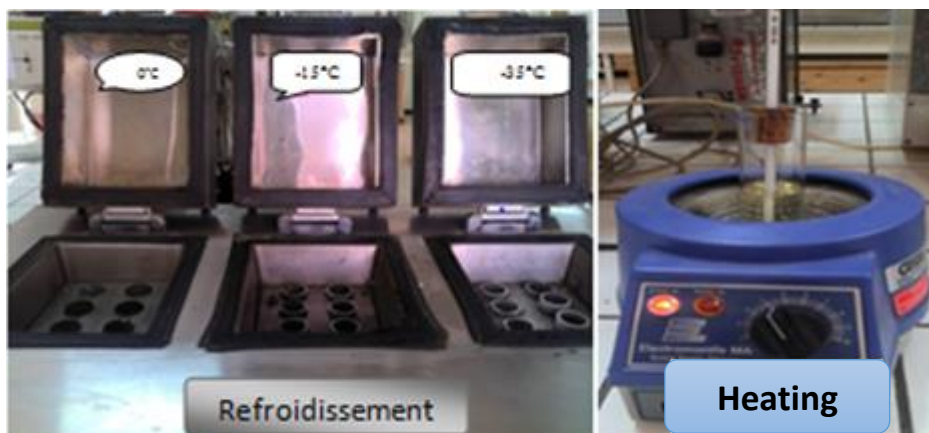
Figure II.16. Measurement of the pour point

Measurements are preferably started at a temperature preferably above 9 ° C. and less than 15 ° C, and the test tube is examined whenever the temperature drops by 3 ° C.

As soon as the oil no longer flows when the tube is tilted, the test tube is held in the horizontal position for 5 seconds counted per stopwatch.

Figure II.17. Frozen tube with stopwatch

The pour point is reached if, under these conditions, the surface does not deform. It is the temperature indicated at this time by the thermometer increased by 3 ° C.

II.3.1.6. Color Measurement

Colors ASTM D1500 **[13]** has an international standard specifying a method for the visual determination of the color of certain petroleum products, such as lubricating oils, heating fuels, diesel engine fuels and oil waxes. It is limited to products which do not contain artificial colors.

The determination of the color of the petroleum products is carried out using a comparative colorimeter. A sample of the liquid was compared transparently with colored glass standards. The scale varies from 0.5 to 8 in steps of 0.5 ranging from light to dark.

Figure II.18. Colorimeter comparative with scale

II.3.1.6.a. Description

Colorimeter comprising a metal housing with a standard color disk, prism with 2 fields of view, test tube housing, light source, delivered with 2 test tubes.

II.3.1.6.b. Procedure

Place a control vessel, filled to a depth of at least 50mm with distilled water, of the colorimeter through which standard glass will be observed. The sample is then placed in its container in the other compartment. The containers are covered to exclude all outside light.

The light source is fed by comparing the color of the sample to that of the standard color disc.

II.3.2. The Elementary analyzes

II.3.2.1. Determination of sulfur content

ASTM D 1662 **[13]** is an international standard that prescribes a method for determining the sulfur content.

Figure II.19. Appareil de teneur en soufre

II.3.2.1.a. Equipment

- An X-ray generator.
- A sample chamber.
- A lever for opening the sample chamber lid.
- Built-in printer with recording paper.
- Control panel for operation (data input).
- Control screen to display the various messages and view the results.

- A set of templates (A and B) used to prepare and mount the box (ref. 38 0006).
- Set of box frames (internal and external elements) (ref.903 800 0100 Horiba).
- Disposable sample boxes (ref. 903 800 0300 Horiba).
- Canopy windows (transparent film ref.903 800 0200)

II.3.2.1.b. Method of analysis

The instrument is turned on and thirty (30) minutes are allowed for stabilization, after selecting a measurement program that includes the number of the calibration curve; The analysis time is one hundred (100) seconds and the number of measurements to be performed for each analysis is three times. The prepared sample is placed in the sample chamber by directing the glass portion downward and the sample chamber cover is closed.

II.3.2.1.b.1. Carbon distribution analysis (n-d-M analysis)

Based on the measured values for RI, SG and sulfur, aromatic, naphthenic and paraffinic contents were identified on the basis of ASTM D-3238 which is expressed as follows:

First, the factors **v** and **w** should be calculated from the observed SG and the refractive index (RI) using Eqs. (2) and (3).

$$v = 2.5(RI^{20} - 1.4750) - (SG^{20} - 0.8510) \qquad (II.2)$$

$$w = (SG^{20} - 0.8510) - 1.1(RI^{20} - 1.4750) \qquad (II.3)$$

Calculus of the percentage of aromatic hydrocarbons (C_A %) From v and the molecular weight (Mw) were carried out using Eqs. (4) or (5):

$$C_A\% = 430v + \frac{3660}{Mw} \quad \text{for positive v} \qquad (II.4)$$

$$\frac{1}{C_A}\% = 670v + \frac{3600}{Mw} \quad \text{for negative v} \qquad (II.5)$$

The percentage of aromatic and naphthenic hydrocarbons (C_R %) is calculated from w, Sulfur content and the molecular weight using Eqs(6) or (7).

$$C_R\% = 820w - 3s + \frac{10000}{Mw} \quad \text{for positive w} \qquad (II.6)$$

$$C_R\% = 1400w - 3s + \frac{10600}{Mw} \quad \text{for negative w} \qquad (II.7)$$

In which, "s" is sulfur content in mass percent.

Eqs. (8) and (9) are used for the calculation of naphthenic Hydrocarbons ($C_N\%$) and the percentage of paraffinic ones $C_P\%$).

$$C_N\% = C_R\% - C_A\% \tag{II.8}$$

$$C_P\% = 100 - C_R\% \tag{II.9}$$

II.3.2.1.b.2. viscosity index analysis

Petroleum products according to ASTM – D341. The viscosity index of the feed stock fraction was calculated according to ASTM – D2270 by applying the procedure for oils of zero to 100 VI. In this procedure Equation (II.1) was used.

$$\text{VI} = \frac{L-U}{L-H} \times 100 \tag{II.10}$$

Where

U : kinematic viscosity at 40 °C of the oil whose viscosity index to be calculated (cst).

L : kinematic viscosity at 40°C of an oil of zero viscosity index having the same kinematic viscosity at 100°C as the oil whose viscosity index is to be calculated (cst).

H :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 (cst).

Chapter 3: Results and Discussions

III.1.The Factors effecting the noted observations of the tests

The experimental factors which were investigated in this study were selected based on their influential rank in affecting used oil recovery ability of the solvent extraction process and availability of required facility that allow monitoring and controlling.

Several categorical factors were nominated for the laboratory experiment. These are solvent-to-used oil sample ratio and solvent type. Solvent-to-used oil sample ratio and solvent type have significant influence on the effectiveness of the re-refining method.

III.1.1. Effect of washing and settling time

The amount of impurities removal increases by increasing settling time of the washed oil. Rate of settling was maximum during initial 48 hours. However it continued up to 48 hours. Impurities aggregate and form sludge which sediments out. Further increase in time has negligible effect on sludge settling.

III.1.2. Selection of Solvent

The solvents were selected according to Burrel's classification i.e. solvents with high capacity (alcohols), moderate capacity (ketones) and low capacity (hydrocarbons). Use of these solvents is based on their capacity to precipitate sludge.

- Observations indicate that no sludge formation takes place when sulfuric acid was used.

Figure III.1 No results using sulfuric acid

The Acetic acid precipitate a good quantity of heavy impurities same as effect of the water.

Figure III.2. Sludge formation using acetic acid

Petroleum ether and gasoline acted as a diluent, they keep the soot particles suspended on the mixture.

Figure III.3. Gasoline and petroleum ether suspended the particles in the oil

Propanol and butanol separate the asphalt from the oil with excellent performance and the color is changed to light one. The only noted difference betwin the tow solvents is that the resulted asphalt for butanol is so hard but with the propanol contains a quantity of oil.

Propanol and butanol separates the asphalt from the oil
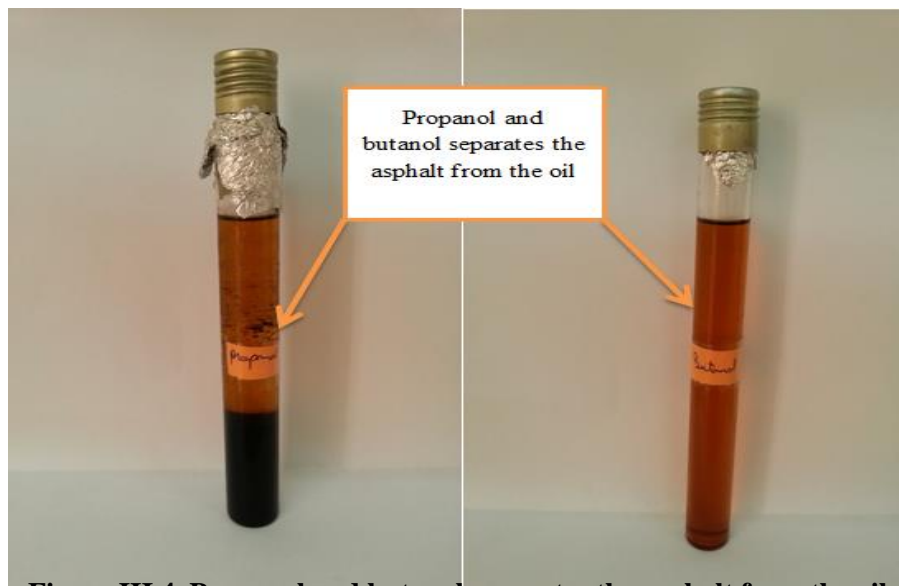
Figure III.4. Propanol and butanol separates the asphalt from the oil

The same observations are noted for using a mixture betwin propanol +gasoline,butanol+gasoline and MEK+ gasoline.

Bisolvents propanol+ gasoline, butanol+ gasoline separates all the asphalt from the oil
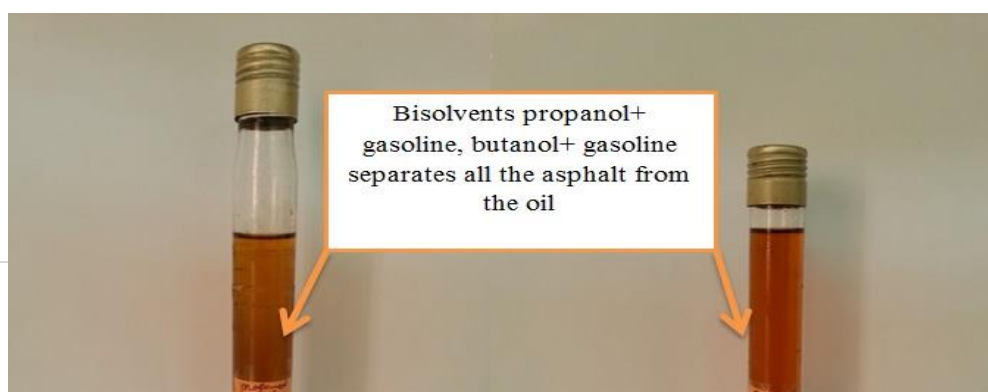
Figure III.5: Bisolvents propanol+gasoline, butanol+ gasoline separate the asphalt from the oil

Figure III.6. MEK separates the sludge from the oil

◆ Binary Solvent Process

In order to perform purification and energy recovery simultaneously, it is the best practice to mix gasoline with MEK , gasoline with propanol and gasoline with butanol as one chemical system . Those products are perfectly compatible to mix with each other in any ratios without having any negative effects.

III.2. Effects of experimental factors on the results

The experimental data on M, SG, RI at 20°C and the sulfur content for regenerated oil are given in table III.1, respectively at different type of the solvent.

The washing and settling time, type of solvents used and the mixing ratio applied for different tests has shown significant effects on the yield of recovered oil. Density, viscosity, flow point, refraction index, color and sulfur…, are also influenced with different intensity as the type and amount of the experimental factors are varied.

Table III.1: Properties of treated used oil

	(RI) (20°c)	(SG)	Kinematic Viscosity (40°c)	Kinematic Viscosity (100°c)	Viscosity Index (IV)	pour point (°C)	yields (%)	Sulfur (S) (%)	color
Virgin oil	-	-	108	14.3	135	-30	-	-	4.5
Propanol	1.4975	0.8950	120.49	11.35	75	-8	81.62	0.041	<5
Pro+gasoline	1.5028	0.8956	124.1	11.5	66	-11	85.8	0.041	<8
Butanol	1.5120	0.8971	125.2	10.9	60	-15	87.5	0.047	<8
But+gasoline	1.5135	0.8965	126.8	11.1	62	-13	88.6	0.041	<8
MEK	1.4989	0.8960	123.5	11.2	68	-10	92.1	0.044	<6

III.2.1. Effect of solvent type

From the experimental result, as it shown in the table III.1 the types of solvent have significant effect on the properties of the treated oil such as yield, kinematic viscosity, the density, flow point, refraction index, color and sulfur.

III.2.1.1. Effect of solvent type on the yield of regenerated oil

The amount (percentage) of yield which was acquired from the experiment by varying the type of solvent was different. The yield with bisolvent type MEK+gasoline was higher than the other solvents. The yield of treated oil with butanol mixed with gasoline is 87.5% and it is so close to the yield when we used the butanol a lawn which is 88.6%, the same results for propanol, propanol+gasolin.As result it is better to use bisolvent rather than solvent in order to minimize the cost of the procedur.gasoline is a cheap and available product in our country plus it is easy to handle and to be recovered. The figure III.7 shows clearly the effect of solvent's type on the yields of the treated oil.
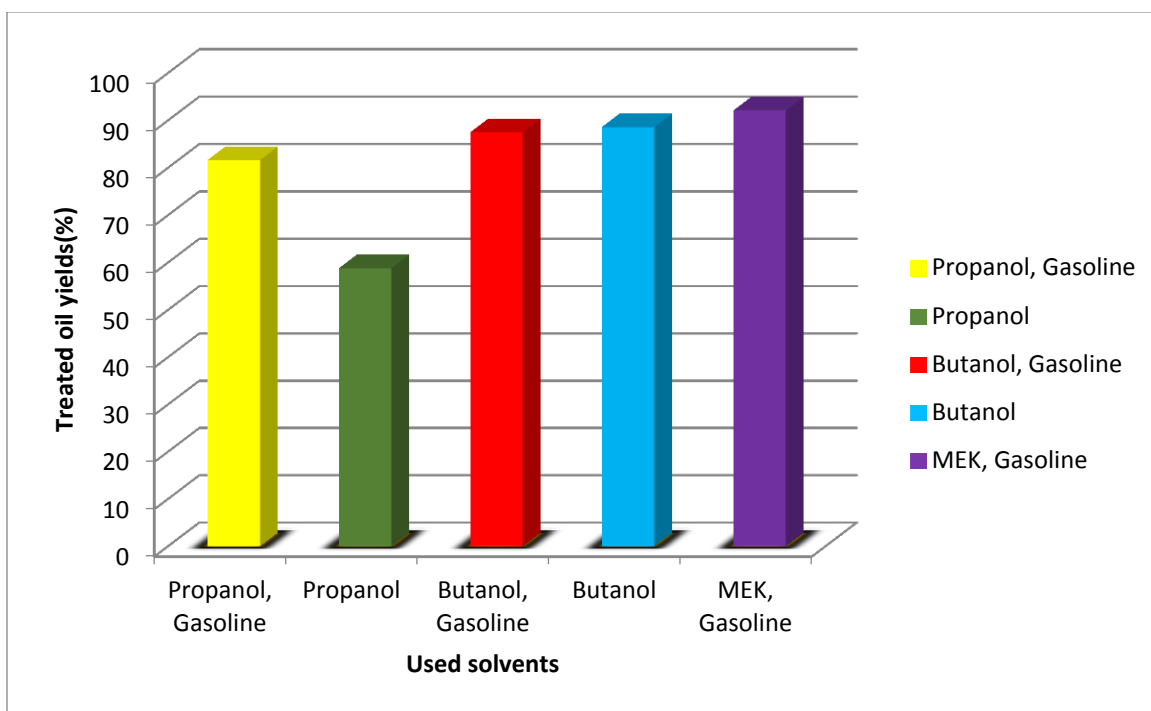
Figure III.7. Effect of solvent on the used oil's yield

III.2.1.2. The effect of the solvent on the RI of the regenerated oil

The quality of regenerated oil can be deduced from the refractive index. The lower the refractive index the better the quality. Refractive indexes for hydrocarbons vary from 1.35 to 1.60; however, aromatics have refractive index values greater than naphthenes, which is in turn greater than paraffins. Paraffinic oil have lower refractive

index values The lubricants have a refractive index values of the order of 1.470 to 1.550. The figure III.8 shows clearly the effect of solvent's type on the (RI) of the treated oil.

Figure III.8. The effect of the solvent on the RI of the regenerated oil

III.2.1.3. The effect of the solvent on the SG of the regenerated oil

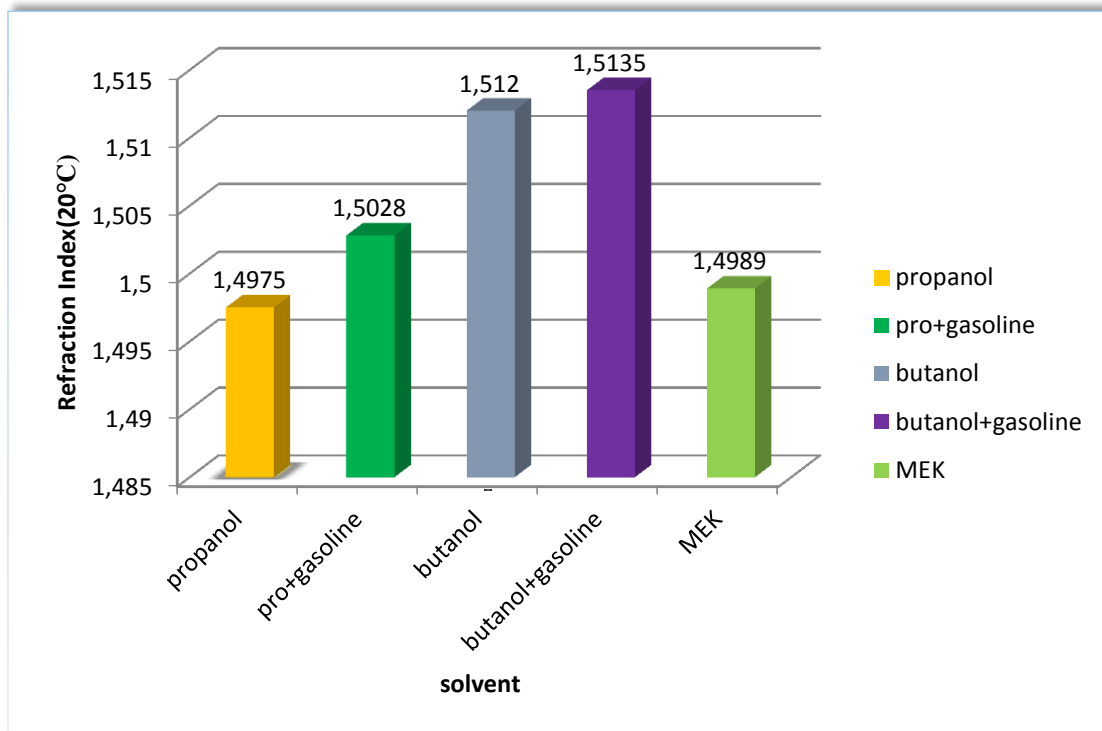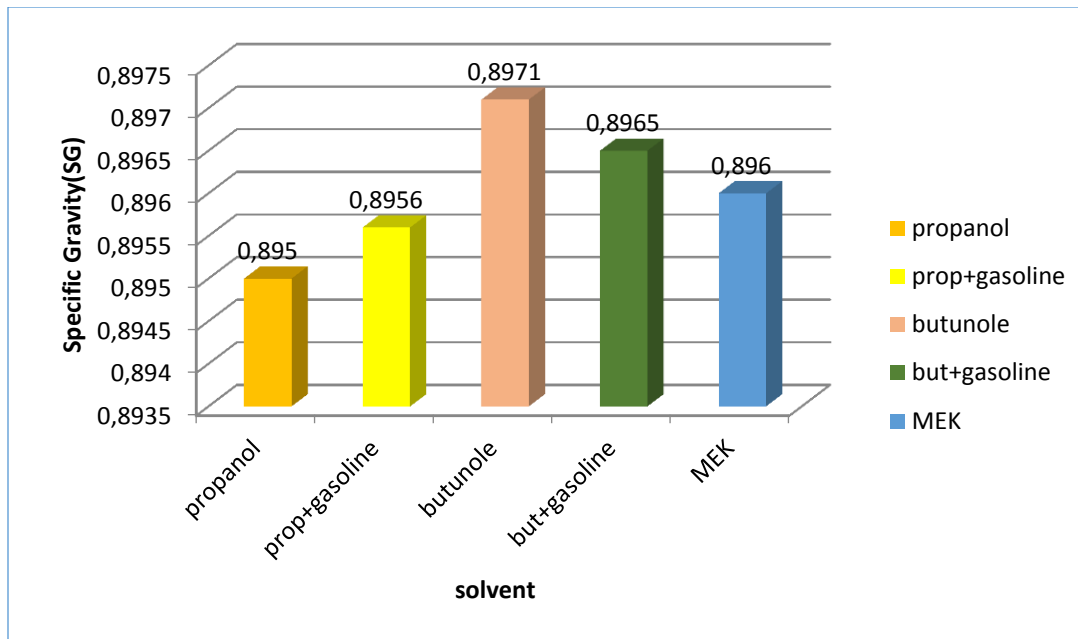Figure III.9 shows that predominantly paraffinic oil have a density of between 0.895 and 0.8971.

FigureIII.9. The effect of the solvent on the SG of regenerated oil

III.2.1.4. The effect of the solvent on the VI of the regenerated oil

The figure III.10 shows the effect of the solvent type on the IV of regenerated oil.

Viscosity index is an empirical number indicating variation of viscosity of oil with temperature. A low VI value indicates large variation of viscosity with temperature that is characteristic of aromatic oils. Similarly, paraffinic hydrocarbons have high VI values.

The decrease in oil viscosity index related to the rise in naphthene – aromatic and polar aromatic content and the increase in saturates content in the regenerated oil.

The maximum value of VI of the treated oil was obtained using propanol as solvent, with the value of 75, is lower than the virgin oil. Deterioration in viscosity is due to the degradation of some compounds paraffinic.
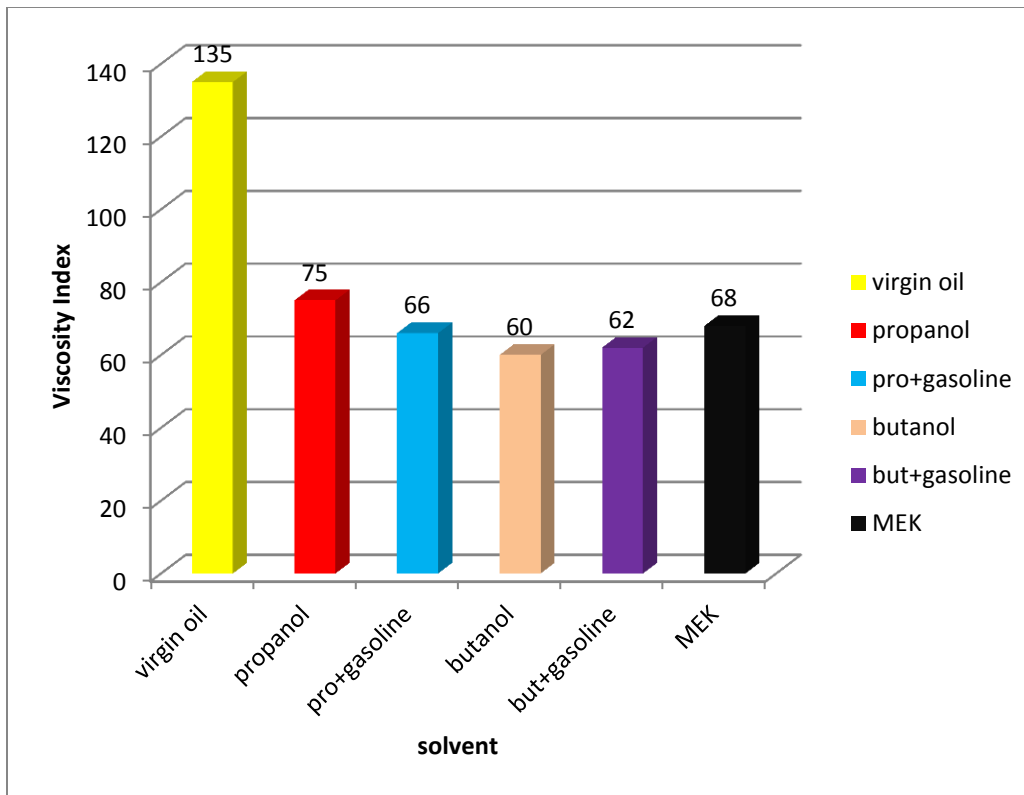
Figure III.10. The effect of the solvent on the (VI) of the regenerated oil

III.2.1.5 The effect of the solvent on the pour point of the regenerated oil

Pour point represents the lowest temperature at which an oil can be stored and still capable of flowing under gravity. Pour point is one of lower temperature characteristics of heavy fraction. Presence of wax and heavy compounds increase the pour point of lubricating oil.

The lowest pour point was obtained for the oil treated with butanol with -15 °C, the concentration of aromatics in the oil reduced the pour point. For commercial formulation of engine oils the pour point can be lowered to the limit of -25 and -40 °C. This is achieved by using pour point depressant additives that inhibit the growth of wax crystals in the oil **[14]**.

The figure III.11 shows clearly the rising of pour point of all regenerated oil products, this is due to the degradation of the additives at high operating temperature of the engine.
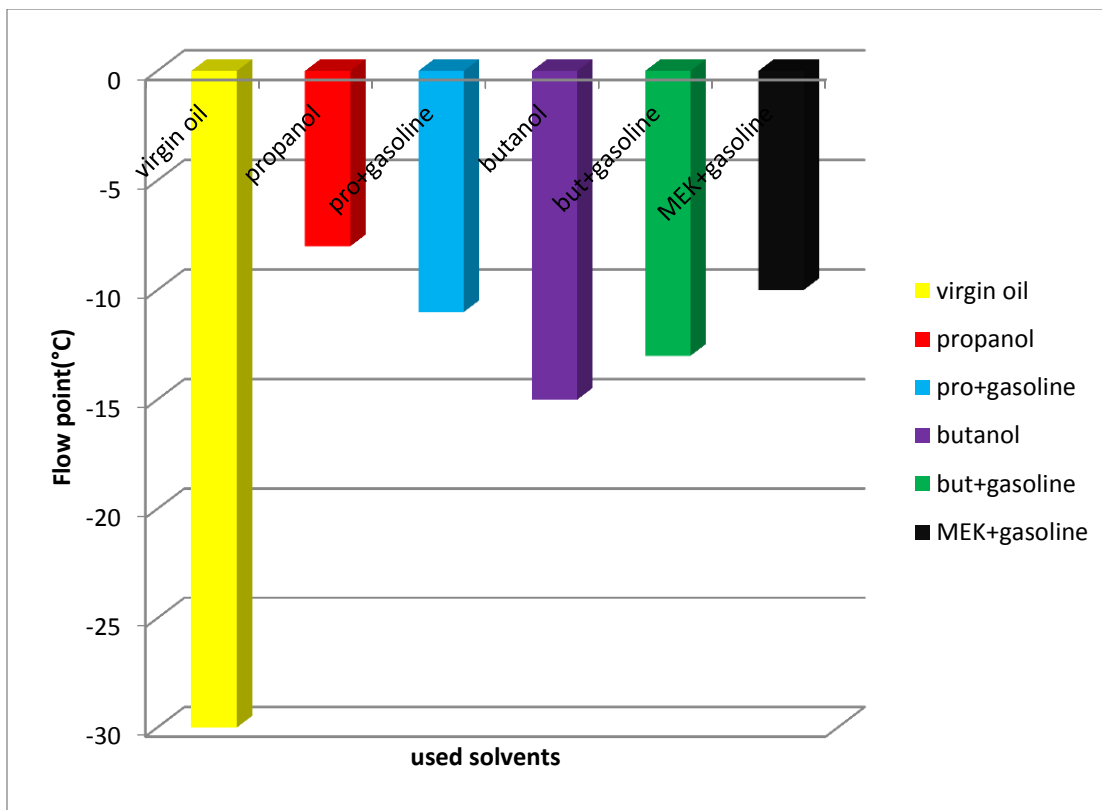
Figure III.11. The effect of solvents on the pour point of treated oil

III.2.1.6. The effect of the solvent on the sulfur content of the regenerated oil

Sulfur is the most important heteroatom that may be present in petroleum products as dissolved free sulfur and hydrogen sulfide (H2S). It may be also be present as organic compounds such as thiophenes, mercaptanes, alky sulfates, sulfides (R-S-R'), disulfides (R-S-S-R). Its presence is undesirable for the reasons of corrosion, bad odor, poor burning and air pollution. In addition presence of sulfur in lubricating oils lowers resistance to oxidation and increases solid deposition on engine parts **[14].** The figure III.12 clearly shows that the sulfur content is very low for all the oils obtained, however the oils have been desulfurized by treatment with the clay.
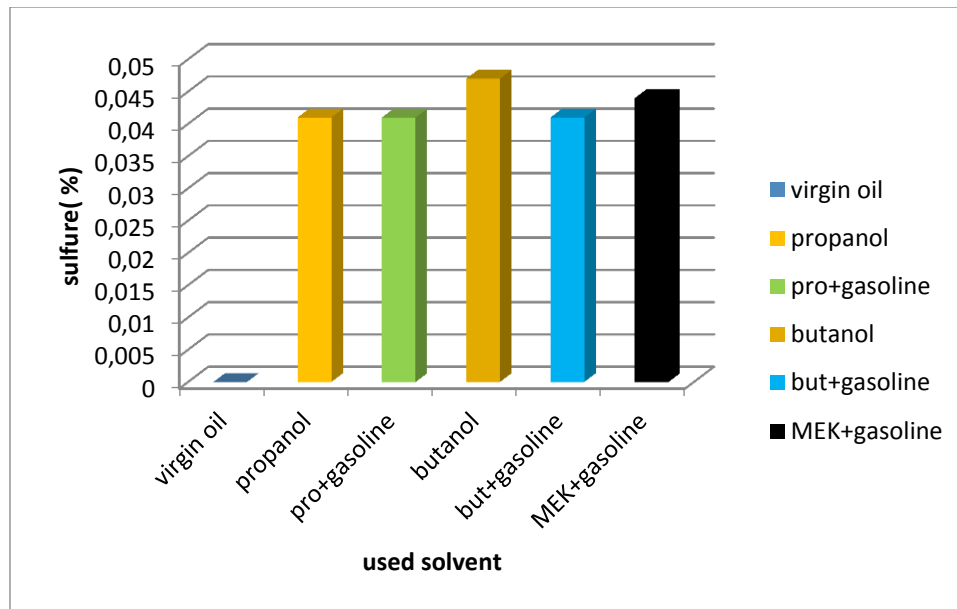
FigureIII.12. The effect of solvents on sulfur content of the treated oil

III.2.1.7. Effect of the solvent on molecular weight of regenerated oil

The relative molecular weight was estimated from kinematic viscosity at 40 and 100 °C. The function H of the viscosity at 40 °C was then obtained from the table given in ASTM D 2502.The H value and the viscosity at 100 °C were then used to estimate the molecular weight from the correlation graph of ASTM D 2502.   The figureIII.13 shows the values of molecular weight, the regenerated oils obtained by various solvents treatment have molecular weights of from 500 to 550.
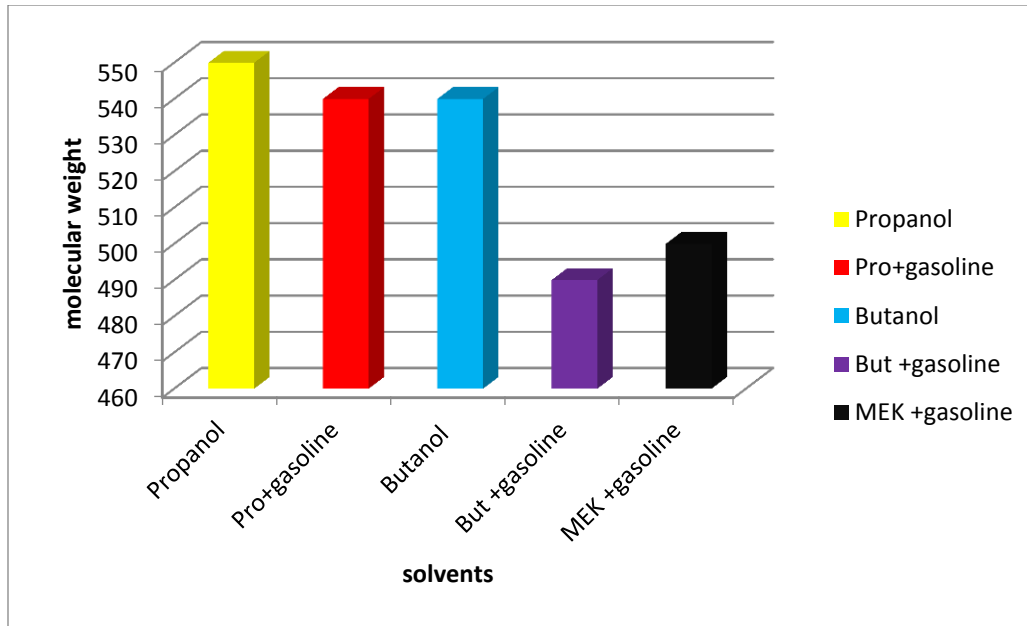
FigureIII.13. The effect of solvents on the molecular weight of the treated oil

III 2.1.8 The effect of the solvents on Distribution of carbons in paraffins (% C_P), naphtens (%C_N) and aromatics (%C_A)

Based on the experimental values regenerated oil, the aromatic, naphthenic and paraffinic contents of each mixture were calculated by ASTM D 2335 that is described in the chapter II.

Table III.2: Shows the molecular weight (MW) and the Distribution of carbons in paraffines (% C_P), nahthenes (%C_N) and aromatics (%C_A)

	MW	C_A % wt	C_N % wt	C_P% wt
Propanol	550	12.68	22.79	66.34
Pro+gasoline	540	17.60	12.05	70.33
Butanol	540	26.88	15.38	64.52
But +gasoline	490	29.45	14.33	63.53
MEK +gasoline	500	13.67	21.28	64.94

Oil treated with propanol has a Paraffin content of 50, with a low aromatic content which demonstrates the best quality of oil product. The bed quality of regenerated oil has been obtained using butanol and mixture of butanol and gasoline as solvent; from where it was found a enough high amount of aromatic, 26.88 %wt and 29.45%wt respectively.

The figure III.14 shows clearly the distribution of carbons in paraffins (% C_P), naphtens (%C_N) and aromatics (%C_A) of different regenerated oil products.
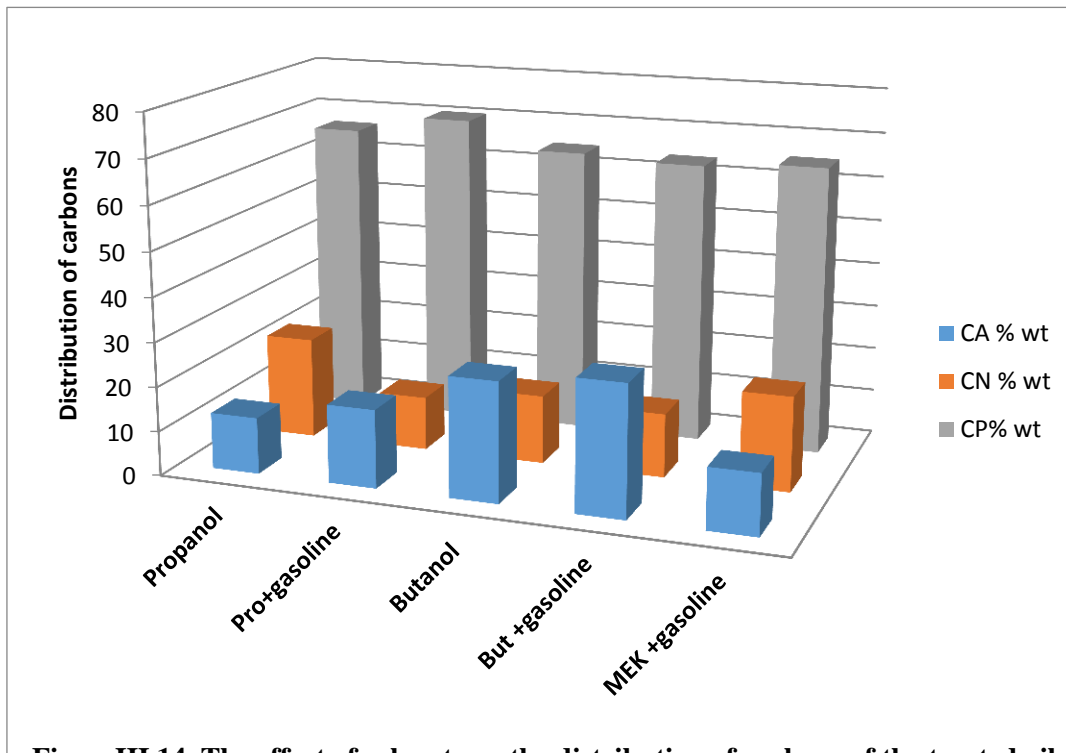
FigureIII.14. The effect of solvents on the distribution of carbons of the treated oil

Conclusion

Conclusion

Used motor oil is a high pollutant material that requires responsible management, because it causes damage to the environment and health diseases. Recycling of such contaminated materials is beneficial on the environment.

This research started from the aim of availing the lubricating, energy and heat hidden in the used motor oil. The properties of lubricating oil degrade over time, the viscosity and density increased because of contamination, the flash point decreased as a result of light ends. The metal content also increases, also the color get changed to black as a result of carbon and soot.

The washing and settling time applied for the sample has shown significant and excellent effects on the yield of recovered oils, the water precipitated most of the heavy impurities as sludge. After extraction with solvents, the results showed that the type of solvents effected on the yield. The maximum and minimum noted yields are 81.6% and 92.1% using propanol and MEK+gasoline respectively. The resulted properties using a pure solvents is the same when we used binary solvents. The yield using butanol 87.5% and butanol+gasoline gives 88.6%, so it is best economically to use binary solvents. The other properties (RI, VI, MW, KV…ect) of the oil were also characterized and gave good and acceptable result when it was compared with the virgin oil.

This experimental work proved that the use of water and binary solvents(extraction solvent+gasoline) is the best and cheapest process to recycle the used motor oil and preserve the natural resources.

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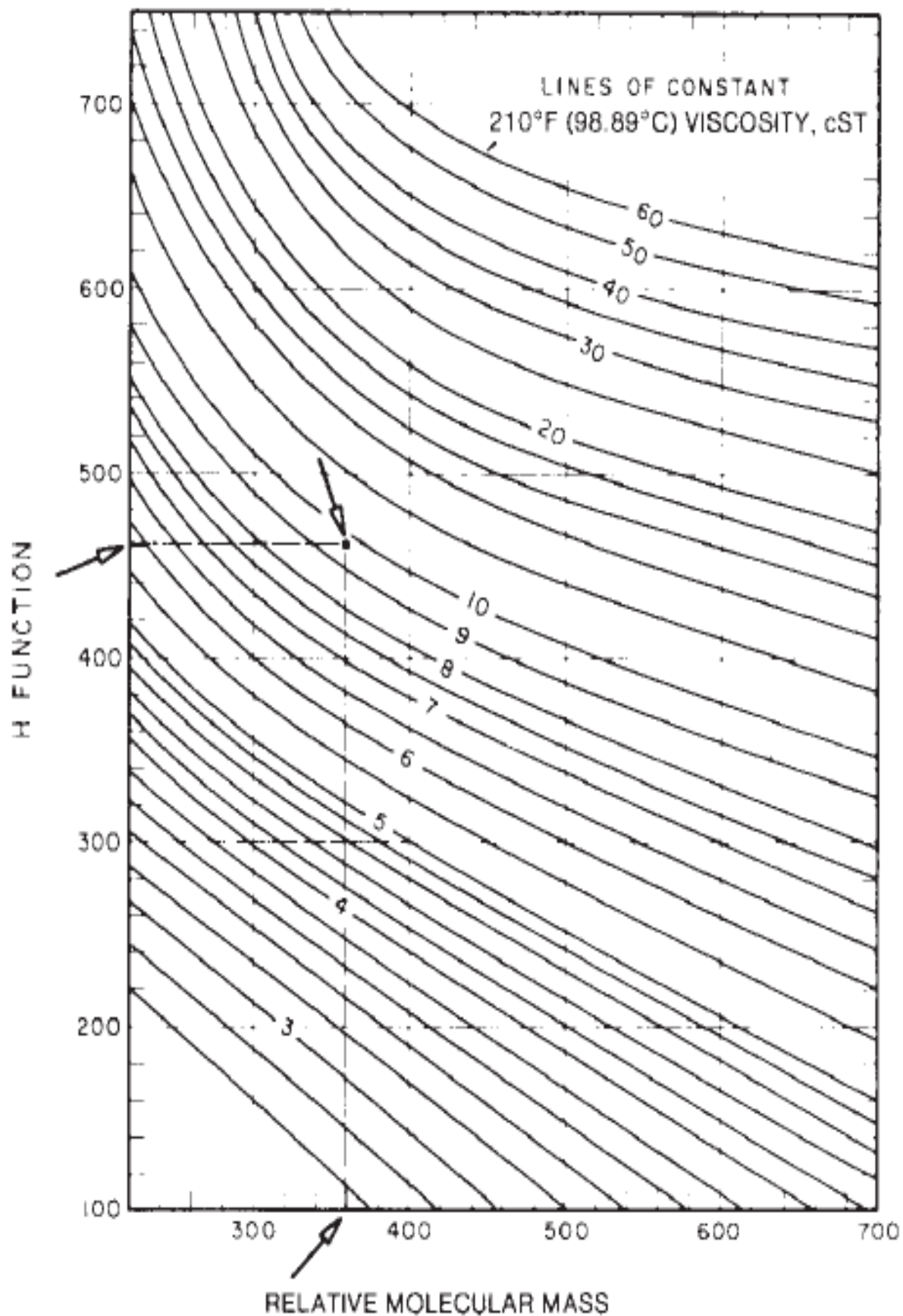
Annexes

Figure1: Viscosity-Molecular Weight Chart

TABLE 1: Tabulation of H Function

Kinematic Viscosity, cSt at 100°F (37.78°C)	H				
	0	0.2	0.4	0.6	0.8
2	-178	-151	-128	-104	-85
3	-67	-52	-38	-25	-13
4	-1	9	19	28	36
5	44	52	59	66	73
6	79	85	90	96	101
7	106	111	116	120	124
8	128	132	136	140	144
9	147	151	154	157	160
10	163	166	169	172	175
11	178	180	183	185	188
12	190	192	195	197	199
13	201	203	206	208	210
14	211	213	215	217	219
15	221	222	224	226	227
16	229	231	232	234	235
17	237	238	240	241	243
18	244	245	247	248	249
19	251	252	253	255	256
20	257	258	259	261	262
21	263	264	265	266	267
22	269	270	271	272	273
23	274	275	276	277	278
24	279	280	281	281	282
25	283	284	285	286	287
26	288	289	289	290	291

Kinematic Viscosity, cSt at 100°F (37.78°C)	H				
	0	0.2	0.4	0.6	0.8
33	314	314	315	316	316
34	317	317	318	319	319
35	320	320	321	322	322
36	323	323	324	325	325
37	326	326	327	327	328
38	328	329	329	330	331
39	331	332	332	333	333

H									
0	1	2	3	4	5	6	7	8	9

Abstract

The amount of used lubricating oil that is collected or dumped in our country is very large, because of the increase in population of automobiles and industrial facilities. The used lubricating motor oil is a highly pollutant material that requires responsible management. It may cause damage to the environment, if it's being disposed of appropriately.

In this study many tests carried out in the laboratory in order to find out an appropriate process to recycle used lubricating engine oil collected from a car wash station. By applying appropriate treatment: washing, settling, solvent extraction and distillation for solvent recovery, adsorption by bentonites, the base oil was obtained. The refined base oil was then characterized and compared with the virgin oil.

The type of solvent used and the mixing ratio applied for different samples has shown significant effects on the yield of recovered oil. MEK +gasoline solvent with mixing ratio of 6:1 gave the best result. A Maximum yield of 92,1% was obtained using MEK+gasoline binary solvent and mixing ratio of 6:1, minimum recovery yield is 81,62% was obtained using propanol and 6:1 mixing ratio.

A good quantity of The recycled engine oil obtained at the end of the process is ready to be used again or blended with other virgin base oils.

KEY WORDS:OIL, RECYCLING, SOLVENT, BENTONIT, SETTLING.

ملخص

إن كمية زيت التشحيم المستخدمة التي يتم جمعها أو إلقاءها في بلدنا كبيرة جدا، وهذا راجع لزيادة في عدد السيارات والمنشئات الصناعية. زيت التشحيم المستخدم هو مادة ملوثة التي تتطلب إدارة مسؤولة. وقد يتسبب في إلحاق الضرر بالبيئة عندما لا يتم التخلص منها بطريقة مناسبة.

في هذه الدراسة قمنا بالعديد من الاختبارات التي أجريت في المختبر من أجل معرفة عملية مناسبة لتجديد زيت تشحيم المحرك المستخدمة التي تم جمعها من محطة غسيل السيارات. من خلال تطبيق العلاج المناسب: الغسيل، والاستقرار، واستخراج المذيبات والتقطير لاستعادة المذيبات، الامتزاز من قبل البنتونيت، تم الحصول على النفط قاعدة. ثم تم وصف النفط قاعدة المكرر ومقارنتها مع النفط البكر.

وقد أظهر نوع المذيب المستخدم ونسبة الخلط المطبقة لعينات مختلفة تأثيرات كبيرة على محصول الزيت المسترد. ميثيل ايثيل + البنزين المذيبات مع نسبة خلط 6: 1 أعطى أفضل نتيجة. A تم الحصول على الحد الأقصى لمعدل 92،1٪ باستخدام ميثيل ايثيل سيتون + البنزين ثنائي المذيبات ونسبة الخلط من 6: 1، وحاصل الاسترداد الحد الأدنى هو 81،62٪ تم الحصول عليها باستخدام بروبانول و 6: 1 نسبة الخلط.

كمية جيدة من زيت المحرك المعاد تدويره التي تم الحصول عليها في نهاية العملية جاهزة للاستخدام مرة أخرى أو المخلوطة مع الزيوت الأساسية العذراء الأخرى.

الكلمات المفتاحية : زيت، تدوير، مذيب، بانتونيت، إستقرار.