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Research project Title: Recycling of used lubricating oil to base oil using solvent extraction and clay adsorption

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Acronyms

TAN: Total acid number

SAE: Society of automotive engineers

ASTM: American society for testing and materials

ppm: part per million

ULO: Used lubricating oil

AOAC: Association of official analytical chemist

KOH: Potassium hydroxide

TBN: Total base number

SD: standard deviation

ANOVA: Analysis of variance

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Abstract

The management of waste lubricating oils is particularly important because of the large quantities are generated through transport and Industrial activities. These waste oils have detrimental effect on the environment if not properly handled, treated or disposed to the environment. Thus, it is imperative to find more environmentally sustainable ways to dispose used lubricating oils through recycling. The main aim of the study was to determine the optimum extraction condition for the extraction of base oil from used lubricating oil. For this, first four different solvents namely ethanol, isopropanol, n-butanol and hexane were examined for maximum oil yield. With the solvent that gives maximum oil yield solvent to oil ratio 1:1, 2:1, 3:1 and 4:1 and extraction time 24hr, 48hr, 72hr and 96hr were considered for optimization. From the experiment it was found that a maximum yield of 74.55% was obtained using n-butanol solvent. On the basis of n-butanol solvent further experimental investigation was performed by implementing full factorial experimental design. According to the study a maximum oil yield of 86% was obtained at 4:1 solvent to oil ratio and 96hr extraction time and a minimum oil yield of 21% was obtained at 1:1 solvent to oil ratio and 24hr extraction time. ANOVA analysis was showed significant effect of solvent to oil ratio and extraction time on oil yield (P value < 0.005). When a solvent to oil ratio increased from 1:1 to 4:1, the oil yield increased from 25.1% to 86% for 96hr extraction time. A sharp increase of percentage oil yield was observed as the solvent to oil ratio increased. On the other hand no significant percentage oil increment was observed with increment of extraction time above 48hr. Therefore, the optimum extraction time is 48hr. Quadratic regression model equation was obtained to describe the effect of operatic conditions on base oil yield. The properties (specific gravity, water content, ash content, kinematic viscosity, viscosity index, flash point, pour point and metal content) of the base oil extracted under a condition that gave maximum oil yield were determined and compared to those of virgin lubricating oil and base lubricating oil. The result obtained indicated that the properties of recycled oil were within the standard of base lubricating oil. Therefore, the recycled oil can be used as base oil which is the fundamental component in manufacturing of different types of lubricating oil. Based on the preliminary feasibility study, the production process was found to be feasible at 3:1 solvent to oil ratio with rate of return on investment equal to 22.4% and payback period of 2.8 years. According to the study also the process is not economically feasible for solvent to oil ratio more that 3:1 and therefore, the optimum solvent to oil ratio is 3:1.

Key words: Used lubricating oil, Solvent extraction, Base oil, Yield, Viscosity index

Chapter one

Introduction

1.1 Background

Lubricating oils are viscous liquids that are used for lubricating moving part of engines and machines. These oils are derived from petroleum base feedstock which consists of mainly of complex mixtures of hydrocarbon molecules. These hydrocarbon molecules generally range from low viscosity oils having molecular weights as low as 250, up to very viscous lubricants with molecular weight as high as 1000 [1].

Lubricating oils help to protect rubbing surfaces, reduce friction between moving and connected parts, eliminate buildup of temperature on the moving surfaces and keep the engine clean [2]. In the process, they serve as a medium to remove high buildup of temperature on the moving surfaces. Further buildup of temperature degrade the lubricating oils, thus leading to reduction in properties such as: viscosity index, specific gravity, etc. Dirts and metal parts worn out from the surfaces are deposited into the lubricating oils. With increased time of usage, the lubricating oil losses its lubricating properties as a result of over-reduction of desired properties, and thus must be evacuate and a fresh one replaced [1].

The main constituents of waste lubricating oils are the base oil, degraded additives (anti-wear elements, metal deactivators, corrosion inhibitors, friction modifiers, viscosity index improvers, demulsifying or emulsifying etc), metallic debris, oxidation products and carbon soot [3].

The disposal of used lubricating oils has now become a major problem. The contaminants in waste oil have adverse environmental and health impacts. If put into storm water drains or sewers, they can affect waterways and coastal waters, when dumped in soil or sent to landfill, they can migrate into ground and surface waters [4]. Roughly one gallon of used engine oil would contaminate one million gallon of water including fauna and flora [10]. Another poor use of used lubricating oil is for energy generation through incineration. This leads to emission of probably carcinogenous products, contribute to environmental pollution [4].

Recycling of used lubricants is now attracting more attention than before. This is partly because of the fear of dwindling of world oil reserves and more as a result of the environment concern which it posses and also from waste - to - energy point [1]. Several methods of recycling waste lubricating oil have evolved over time from the period which recycling of waste oil began during late 1930s. These methods include; acid method, distillation/clay method, acid/clay method, solvent/clay method and others [5].

Solvent extraction followed by adsorption has been found to be one of the competitive processes for recycling of used lubricating oil [6]. Therefore this study aims to recycle used vehicle lubricating engine oil which is considered as waste in different car service stations using solvent/clay treatment method and determine optimum operating condition for the refining process.

1.2 Statement of the Problem

Motor oil is used for lubricating various internal combustion engines. During normal use, many impurities such as dirt, metal scrapings, water and chemicals can get mixed in the oil, so the oil will be ineffective for further application as a result lubricating oils must be replaced on a regular basis in all operating equipment [7].

The management of waste lubricating oils is particularly important because of the large quantities are generated through transport and Industrial activities. These waste oils have detrimental effect on the environment if not properly handled, treated or disposed to the environment [8]. It is a common practice in Ethiopia that used oil is disposed in to ground, gutters, water drains, open plots and farms. This leads to pollution of streams, ground water, and lakes and also used oil dump on the ground reduces soil productivity and makes the plants grown on the soil to be unsuitable for food and forage.

Since Ethiopia is aiming to become a middle income country in 2030 and developing a green economy meeting the sustainable development goals, is facing challenges on its environment from the transportation sector; according the data of transport authority of the country estimates that the total number of registered vehicles including motor bicycle in the country as of July 07 2019 is 1,071,345 [9]. These cars are responsible for using a round 32 million liters of oil per year. About 80% – 90% of lubricating oil will be considered as waste after it had been used.

Thus, it is imperative to find more environmentally sustainable ways to dispose used lubricating oils through recycling. Presently recycling of the waste lubricating oil is considered as the best method to address the environmental challenge posed by the indiscriminate waste engine oil disposal. In addition, recycling is also considered as an effective means of conserving the depleting world crude reserve and job creation through the construction and operation of recycling plants.

Recently a number of treatment technologies have been developed that promise to solve technical, economic and environmental problems associated with used lubricating oil recycling. Therefore this research aims to recycle used lubricating oil using solvent clay treatment method, thereby reducing environmental pollution and also minimize importation of lubrication oil.

1.3 Objectives

1.3.1 General objective

The general objective of the study was to determine optimum extraction condition for recycling of used lubricating oil to base oil using solvent extraction followed by clay adsorption.

1.3.2 Specific objectives

The specific objectives of this study were

- ✓ To characterize used lubricating oil.
- ✓ To investigate the effect of process parameters (solvent type, solvent to oil ratio and time) on yield.
- ✓ To evaluate the physiochemical properties of treated lubricating oil.
- ✓ To compare the physiochemical properties of treated lubricating oil with both virgin oil and base lubricating oil.
- ✓ To perform techno-economic feasibility study

1.4 Significance & Beneficiaries

Since the current disposal practice of used lubricating oil in our country is just dumping in drains, ground, rivers and lagoons this poses serious environmental and health hazards. Thus, once this research is implemented to practice it will have proper utilization of the waste lubricating oil. Furthermore, the study has significant role to overcome the challenge of environmental issues as well as economic related to lubrication oil. It will have also role in saving foreign currency by reducing the import of lubricating oil to the country. up on the establishment of recycling of used lubricating oil plant the community will be benefited.

Chapter two

Literature review

2.1 Historical background of lubricating oil

Lubricating oils (LOs) are conventionally obtained from crude oil, so that, chemical composition of LOs consists on average of about 80–90% base oil and about 10–20% chemical additives and other compounds [11]. In the process of manufacturing lubricating base oils, it is necessary to remove the aromatic hydrocarbons from lube oil cuts to improve several lubricating properties. Lube oil cut normally contains saturated and unsaturated compounds. The separation is based on liquid-liquid extraction by a cyclic and polar solvent [12]. The composition of crude petroleum from different sources varies tremendously, but a typical make-up is as follows [13]:

Gases = 5%,

Gasoline = 35%,

Kerosene = 10%,

Diesel = 20%,

Lube oil = 2%

Tar, Bitumen 28%

The conventional steps in lubricating oil manufacture are pretreatment of the crude oil charge, followed by distillation of the crude in two steps (an atmospheric tower and vacuum tower), deasphalting (as required by the nature of the crude oil charge), dewaxing, solvent extraction, filtering and blending including mixing various additives with the final lubricating oil. The prime objective in the production of lubricating oil is the separation of wax distillate and cylinder stock without any decomposition or cracking of the lubrication oil fractions, thus a vacuum distillation unit is used to separate the wax distillate and the bottom stock at a lower temperature. The properties which make the high boiling paraffin hydrocarbon suitable for lubricating manufacture include stability at high temperatures, fluidity at low temperature, only a moderate change in viscosity over a broad temperature range and sufficient adhesiveness to keep it in place under high shear forces. The desired fractions for the manufacture of the lubricating oil have high boiling points and its separation into various boiling points range cuts must be accomplished under reduced pressure. The vacuum tower produces some fuel oil overhead which is sold as a separate product or sent to another area of the refinery for further processing and blending. The two main products from the vacuum tower are wax distillate and cylinder stock which is the bottom product. Both streams contain desirable lubricating oil constituents as well as by-products. The wax distillate is charged directly to the dewaxing unit. The vacuum tower bottoms, or cylinder stock are charged to deasphalting unit. These two fractions from the basic stock for lubricating oil manufacture [14].

2.2 Characteristics of lubricating oil

All lubricants are characterized by some properties, which must possess certain properties which must meet the lubrication requirements in an engine. These properties include viscosity index, cloud point, flash point, pour point, total acid number (TAN), ash content, water content, corrosive properties, relative density, insolubles, carbon residue, metal content, total base number (TAN) etc.

2.2.1 Viscosity

Viscosity is defined as the force acting on a unit area where the velocity gradient is equal at a given density of the fluid [14]. Viscosity is strongly depending on the temperature. Viscosity is a very important property for grading lubricants. It is a measure of fluid resistance to flow and is strongly dependent on temperature. A decrease in the viscosity of engine oil indicates that the oil is contaminated [15]. Lubrication 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. The greater or higher the SAE viscosity numbers, the more viscous or heavier is the lubricating oil. Viscosity numbers are given in terms of saybolt second universal, SSU. The addition of certain additives is for the improvement of viscosity-temperature characteristics [14]. There are two related measures of fluid viscosity; Dynamic (or absolute) viscosity and Kinematic viscosity.

2.2.1.1 Dynamic (absolute) viscosity

Absolute viscosity is defined as a measure of internal resistance of a fluid. Absolute/dynamic viscosity is the tangential force per unit area required to move one horizontal plane with respect to another plane per unit velocity when maintaining a unit distance apart in the fluid [22].

2.2.1.2 Kinematic viscosity

Kinematic viscosity will be obtained by dividing the absolute viscosity of a particular fluid with the fluid mass density. Viscosity is a state function of temperature, pressure and density in that, viscosity varies inversely proportional to temperature. Viscosity testing always gives the contamination information as is used to indicate the presence of contamination in used engine oil. The oxidized products as well as polymerized products which are dissolved and suspended will result into increase of the oil viscosity, while the decreases in the viscosity of engine oils will indicate the presence of fuel contamination [25].

2.2.2 Pour point

The pour point indicates the temperature below which the oil loses its fluidity and will not flow or circulate in the lubricating system. Lubricating oils with low pour points show good quality [15]. It is very important to users of lubricants in low temperature environment [14]. Engine base oils may contain waxes and paraffins which solidify in cold temperatures; it have been noted that oils with high wax and paraffin content will always have a higher pour point [22]. The pour point of lube oil is highly affected by oil's viscosity; engine oils with high value of viscosity are normally characterized with high pour points. When starting the engine in cold weather the pour point of oil become an important variable. The oil must have the ability to flow well into the oil pump for a particular range of conditions and therefore be pumped to the various part of the engine even at low temperatures [23].

2.2.3 Specific gravity

From ordinary theory, we know that density of a substance is equal to the mass of a substance divided by the volume of the substance. Specific gravity is the ratio of the density of the material to density of the equal volume of water. The temperature at which the density is been measured must be known for density changes as temperature changes [14]. Specific gravity is highly influenced by the chemical composition of the lubricant oil. An increase in the amount of aromatic compounds in the lubricant oil will results in an increase in its specific gravity, while an increase in the saturated compounds will results in a decrease in the specific gravity [26].

2.2.4 Flash point

Flash point is the minimum temperature at which an oil gives off sufficient vapours to form an explosive mixture with air. The flash point test gives an indication of the presence of volatile compounds in oil and is the temperature to which the oil must be heated under specific conditions to give off sufficient vapor to form a flammable mixture with air [14]. A decrease in flashpoint reveals that the oil is contaminated through dilution of lubricating oils with unburned fuel. An increase in flashpoint indicates the evaporation of light components from the lubricating oil [16]. The flashpoints also portray the relative measure of safe properties of lubricating oils [15]. There are various methods of determining flash point of oils as contained in ASTM. Flash point, open cup is the temperature at which a flash appears on the surface of the sample when a small flame of specified size is passed across the cup at regular temperature intervals while the oil in the cup is being heated at a specified rate [16].

2.2.5 Water content

Water found in lubricating oil in service depends on where the automobile is being used. In almost all system, traces of water in the lubricant are unavoidable, arising from such sources as leaking oil coolers, engine cooling system leaks and in all types of machinery, from atmospheric condensation. Accordingly, the water content must not exceed the “action” levels (more than 0.5) recommended for the different grades of oil and application. In places where there are bad roads and drainage system, one is bound to see water as part of contaminants of the oil. The water in the radiator may also contribute to the presence of water in lubricating oils in use. The presence of excessive water contamination will affect the viscosity of the oil and this may give rise to emulsion formation and can also lead to gear tooth and bearing problems [1].

2.2.6 Total acid number

The total acid number is the measurement of acidity in oils used as lubricants. It is one of the crucial chemical properties that gives stability to lubricating oils. Lubricating oil is said to be stable if it can resist oxidation that yields acids, lacquers and sludge [17]. TAN indicates the amount of alkali in milligrams that is required to neutralize the acids in one gram of oil. Normally, acidity increases with the oxidation of lubricating oils [16].

2.2.7 Total base number

Total base number (TBN) is a measurement of basicity and is expressed in terms of the equivalent number of milligrams of alkali (most especially potassium hydroxide) per gram of oil sample. TBN generally ranges from 6 to 80 mg KOH/g in modern lubricants, 7–10 mg KOH/g for general internal combustion engines and 10–15 mg KOH/g for diesel engine operations. Oils and lubricants have a base reserve designed to neutralize the acids produced after the combustion process in order to avoid corrosion of engine components [18]. A low Total base number (TBN) indicates that the oil has to be changed. TBN is determined by acid-base titration using HCIO4 as the titrant [15].

2.2.8 Ash content

When the lubricating oil is completely burned, the remaining solid is called ash and it shows the oil purity [19]. This method consists of measuring the amount of ashforming material in the oil which provides an idea about the quality of the product and the adequacy for any application since the ash represents the impurities and unwanted components [38].

2.2.9 Carbon residue

This evaluates the solid residue obtained when the oil is heated to complete vaporization and it refers to the amount of deposit formed [19].

At temperatures of 300°C or more in the absence of air, oils may decompose to produce low molecular weight fragments from the large molecular weight species typically found in mineral oils. The fragmented or ‘cracked’ hydrocarbon molecules either recombine to form tarry deposits (asphaltenes) or are released to the atmosphere as volatile components. The deposits are undesirable in almost all cases and most lubricating oils are tested for deposit forming tendencies. The carbon residue is determined by weighing the residue after the oil has been heated to a high temperature in the absence of air. The carbon residue test can be used to evaluate the characteristic of engine oils to depositing carbonaceous material in internal combustion engines. It is important to note that the carbon residue value of engine/motor lubricating oil is regarded as indicative of the amount of carbonaceous deposits that the engine oil would form in the combustion chamber of an engine [27]. The carbon residue parameter is of little importance in the case of synthetic oils because of their good thermal stability. It is also infrequently used in characterizing well refined lubricants.

2.2.10 Metal content

The metal content of base oil is a very important parameter as the metal content in an oil sample as it can increase the rate of corrosion of the metal parts it is in contact with. The metal content of oil mainly come from the additives added to the oil. Calcium comes mainly from detergents and dispersants, sulphur from extreme pressure additives and Zinc is introduced to base oil in the form of additives packaged as anti-oxidant, corrosion inhibitor, anti-wear, detergent [33].

Calcium: Used as a detergent and dispersant additive to maintain suspension of particulate matter, along with maintaining a reserve alkalinity. Concentration levels vary greatly depending on oil brand.

Zinc: Another anti-wear, anti-oxidant, and corrosion inhibitor additive also commonly found in bearing alloys. Concentration levels vary greatly depending on oil brand.

Chromium: The source of chromium wear metals is almost always from piston rings, which are used to form a tight seal between the moving piston and stationary cylinder wall. These rings have to reliably create a tight seal between the piston and the cylinder wall while travelling at up to 4,000+ feet per second and dealing with peak pressures of over 2,000 psi (136 Bar) depending on the engine design and usage.

Lead: Lead is a soft, sacrificial wear metal used on surfaces such as bearings. Lead based Babbitt alloys. Commonly found in main crankshaft journal bearings and contaminated fuels. Other sources include leaded fuels and gasoline octane improvers.

Copper: Copper is widely used due to its high ductility and thermal conductivity. It is mainly utilized in bushings and bearings such as: crankshaft journal bearings, connecting rod bearings, camshaft bushings, piston wrist pin bushings, thrust washers, and even heat exchangers (oil coolers).

Iron: This is the only wear metal that accurately and linearly increases with the length of time the oil has been in service. It has many sources inside of an engine, most commonly coming from cylinder liners, camshaft lobes, crankshaft journals, and oil pumps.

Nickel: Though not very widely used anymore, Nickel can be found in certain alloys of steel for internal engine parts, and also is used as a coating on bearings.

Magnesium: Also used as a detergent and dispersant additive to maintain suspension of particulate matter, and occasionally used in certain alloys of steel. Concentration levels vary greatly depending on oil brand

Molybdenum: This is most commonly used as an anti-wear/anti-scuff additive and has an effect commonly called “Moly plating” where over time, a thin and microscopic layer of Molybdenum tends to form between contact surfaces, thereby creating a lower coefficient of friction between the two parts. Concentration levels of Molybdenum vary greatly depending on the formulation of each specific oil brand, and viscosity. (http://www.bobistheoilguy.com/engine-oil-analysis/, June 7, 2016).

2.3 Used lubricating oil and its composition

Used lubricating oil refers to the engine oil, transmission oil, hydraulic and cutting oils after use. It is also refers to the degradation of the fresh lubricating components that become contaminated by metals, ash, carbon residue, water, varnish, gums, and other contaminating materials, in addition to asphaltic compounds which result from the bearing surface of the engines [20].

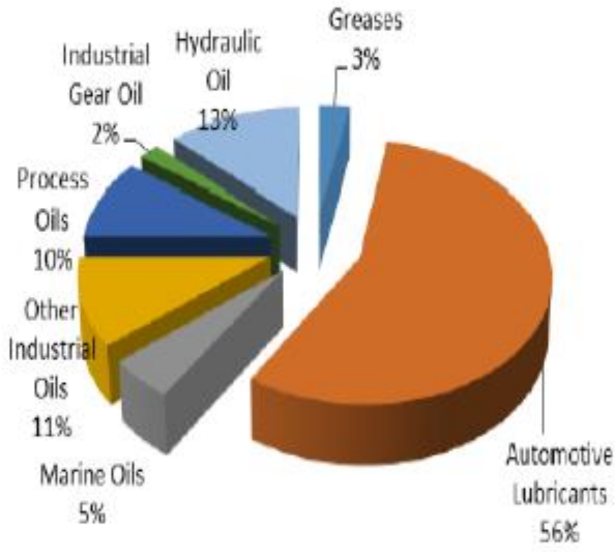


Fig 2.1 Percentage of contribution of used lubricating oil [20]

After a certain period of useful life, the lubricating oil loses its properties and cannot be used as such in machinery. Buildup of temperature degrades the lubricating oil, thus leading to reduction in properties such as: Viscosity, Specific gravity, etc. Dirt and metal parts worn out from the surfaces are also deposited into the lubricating oils. With increased time of uses, the lubricating loses its lubricating properties as a result of over reduction of desired properties and thus must be replaced with fresh one. It is surprising to know that base oil never gets spoiled, it only gets dirty. Lubricating oil becomes unfit for further use for two main reasons: accumulation of contaminants in the oil and chemical changes in the oil [20]. The main contaminants are listed below.

2.3.1 Combustion products

Water: Fuel burns to CO_2 and H_2O. For every liter of fuel burnt, a liter of water is created. This normally passes out through the exhaust when the engine is hot, but when cold it can run down and collect in the oil. This leads to sludge formation and rust. Water from leakage of the cooling system

Soot and carbon: These make the oil go black. They form as the result of incomplete combustion, especially during warm-up with a rich mixture.

Lead: Tetraethyl lead, which used to be used as an anti-knock agent in petrol, passes into the oil. Typical used engine oil may have contained up to 2% lead, but today any lead comes from bearing wear and is likely to be in the 2 - 12 ppm range.

Fuel: Un burnt gasoline or diesel can pass into the lubricant, again especially during start-up [20].

2.3.2 Abrasives

Road dust: This passes into the engine through the air-cleaner. Composites of small particles of silicates, wear metals, iron, copper and aluminum are released due to normal engine wear [20].

2.3.3 Chemical products

Oxidation products: Some of the oil molecules, at elevated temperatures, will oxidize to form complex and corrosive organic acids. Composition of used oil consists of four major groups, which have average values of 76.7% saturates, 13.2% monoaromatics, 3.7% diaromatics and 6.5% polyaromatic-polar material [20].

2.4 Environmental impacts of used lubricating oil

Used lubricating oils are classified as hazardous wastes, and constitute a serious pollutant problem not only for the environment, but also for human health due to the presence of harmful contaminants, such as heavy metals, polychlorinated biphenyls (PCBs) and polycyclic aromatic hydrocarbons (PAHs) [11]. The presence of degraded additives, contaminants, and by-products of degradation render waste oils more toxic and harmful to health and environment than virgin base oils. If put into storm water drains or sewers, they can affect waterways and coastal waters [4]. When dumped in soil or sent to landfill, they can migrate into ground and surface waters though numerous land treatment processes. In addition, uncontrolled used oils are a threat to plant and animal life, which can further result in economic losses, for example, recreation and fishing industries. For example, used oil from internal combustion engines generally accumulates a variety of contaminants, which increase the oil's toxicity [3].

Improper application of used oil for multiple customary purposes also leads to various environmental degradations and health effects. Some local uses of changed oil and its direct and indirect adverse effects are shown in Table below [21]

Table: 2.1 Environmental impact assessment of used lubricating engine oil [21]

Local uses of used oil	**Road construction**	**Environmental effect**
Road construction	On the ground	Soil pollution
Rust prevention	On a metal device	Stains on contact
Old engines emergency lubricant	Automobiles, generators	Air pollution, waste
Wood preservation	Timber; roofing, fencing	Land pollution
Mixed with grease for gear oil	Gear box lubricant	Spills; Soil pollution
Production of grease	Automobile lubricant	Stain on contact
Burning, Boilers, furnaces	Burners, bakery, incinerators	Off-gas, air pollution
For pest, weed, and dust control	Garden, workshops	Soil pollution
Hydraulic oil	Props, Lifts, Jacks	Spills
Ball joint oil and nuts loosing oil	Ball and socket joints, nuts	Stains on contact
Block and Balustrade mold lubricant	Block, bricks, balustrade molds	Spills
Medication	Wound and cuts	Additional Health effect
Dust and tick control	Land, floor	Land pollution, Stains
Road construction	On the ground	Soil pollution

There are many harmful constituents in used oil that may cause cancer or other health problems if they are inhaled or ingested. For example, it was reported that burning used oil tagged as the top source of airborne lead emissions. Typical levels of contaminants found in used oils are also summarized in Table below [22]

Table 2.2: Typical levels of contaminants in used lubricating oil [22]

Category	Compounds	Automotive Used Oil Concentrations [ppm]
Metal	Cadmium	5 – 25
	Chromium	50 – 500
	Arsenic	2
	Barium	3 – 30
	Zinc	100 – 1200
	Lead	100 - 1200
Chlorinated hydrocarbons	Dichlorodifluoromethane	1000 - 4000
	Trichlorotrifluoroethane	
	Tetrachloroethylene	
	1,1,1- Trichloroethane	
	Trichloroethene	
	Total Chlorine	
Other organic compounds	Benzene	100 – 300
	Toluene	500 – 5000
	Xylene	500 – 5000
	Benzo(a)anthracene	10 – 50
	Benzo(a)pyrene	5 – 20
	Naphthalene	100 – 1400
	PCB’s	NG – 20

2.5 Sources of used lubricating oil

By far 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 [20]

2.6 Used lubricating oil management options

Management of waste oils is a growing concern particularly in industrial and urban areas. Generation of waste oils is closely linked with increase in population of automobiles and industries. When additives and foreign substances, such as metal powder, chips and other particles, are mixed with lubricating oil, aging, degrading and failure will likely occur, leading to mechanical fault and degraded performance. In such cases, the oil is replaced to improve the performance. The used, spent or waste oils should be collected and recycled not only to prevent the environment pollution but also to preserve natural resources [3].

Improper used oil disposal is simply a waste of a valuable resource. Every gallon of used motor oil not recovered results in the need to drill for more oil and in some cases it results in increases in oil import. Today, however, most of the crude petroleum produced throughout the world contains very little of the special hydrocarbon chains necessary for motor oil. As a result, refining crude petroleum to produce virgin lube oil is an elaborate, complex, and expensive process that requires nearly three times energy as much as refining used oil [20]. The amount of crude oil required to produce a certain volume of lubricating oil is nearly 9 times higher to produce the same volume of lubricating oil from used oil [2]. Lube base oil can be recovered and 'regenerated' to the quality equal to or better than its original virgin form [20].

A large range of waste oils can be recycled and recovered in a variety of ways, either directly or after some form of separation and refinement. As per the waste management hierarchy, the first option is to conserve the original properties of the oil allowing for direct reuse. Other options could include recovering its heating value and/or using in other lower level applications. Certain types of waste oils, lubricants in particular, can be reprocessed allowing for their direct reuse. The use of waste oils, after treatment, can be either as lube base stock comparable to refined virgin base oil or as clean burning fuel [3].

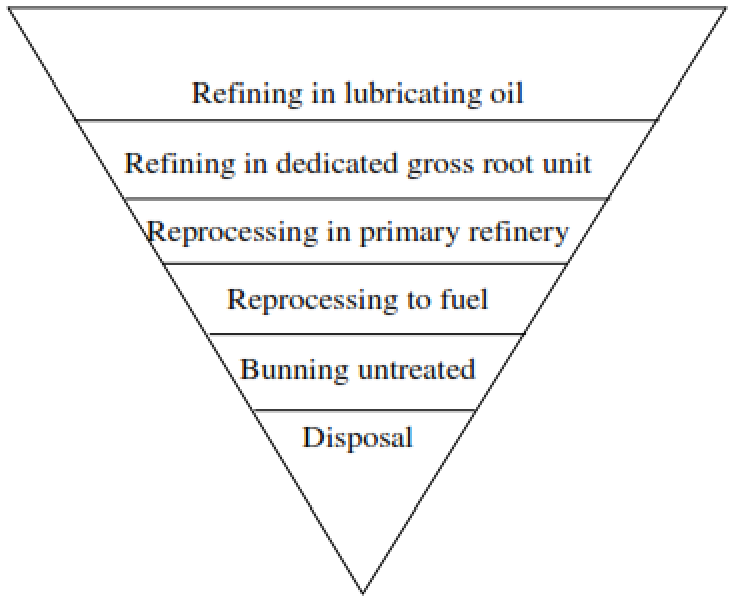


Fig 2.2: Waste oil hierarchy [28]

2.7 Used lubricating oil treatment technologies

Used oils have been recycled for the past four decade. The idea of recycling used lubricating oil was presented in the year of 1930. Initially the used lubricating oils were burnt to produce energy, and later these oils were reblended to engine oils after treatment. Due to the increasing necessity for environmental protection and more stringent environmental legislation, the disposal and recycling of waste oils has become very important. The recycling of waste lubricating oils can be accomplished through three basic methods, which are reprocessing, re-refining and incineration [3].

2.7.1 Reprocessing

The objective of re-processing is to produce a finished fuel oil that is low in basic sediment and water content, and that will not clog burners, foul boiler tubes, or cause sediment build-up in customer tanks. As such, the process requires filtration and removal of coarse solids that can pose environmental hazard or operational problems. Treatment options include mainly physical processes like settling, filtration, or a combination of these operations. Unfortunately, these processes alone are not sufficient to remove all chemical contaminants in the oil, and inclusion of further treatment processes such as clay contacting and distillation would reduce the competitive advantage of waste oil processors [28].

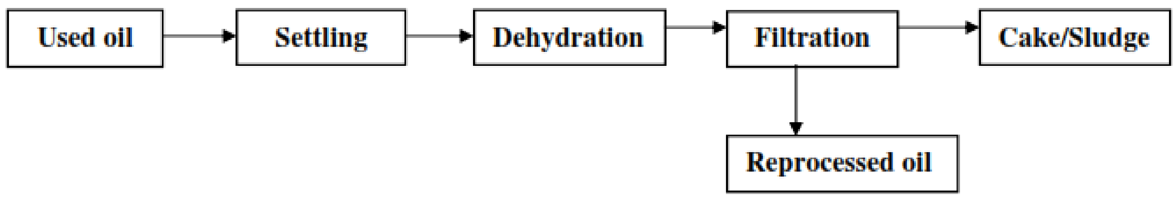


Fig 2.3 Block flow diagram for used oil reprocessing [28]

2.7.2 Refining

The main way for ULOs recycling is the so-called re-refining, which consists of recovering the original base oil to be reused in the formulation of new products [11]. Re-fining is the use of distilling or refining processes on used lubrication oil to produce high quality base stock for lubricants or other petroleum products. The use of this method has increased tremendously in developed countries, some countries reaching up to 50% of the country's need for lubricating oil. There are various methods developed by different western countries in the treating of used lubricating oil for reuse. It requires the conversion of waste oil to a product with similar characteristics to those of virgin oil. The process typically involves, but is not limited to, pre-treatment by heat or filtration, followed by either vacuum distillation with hydrogen finishing or clay, or solvent extraction with clay and chemical treatment with hydro-heating. Vacuum distillation followed by clay contacting offers a less polluting and more economic solution to the re-refining process, particularly for small-scale plants with a capacity range between 10000 and 30000 tons. The resulting residual by-product is well compacted and baled in thick plastic sheets prior to disposal in landfills [3]. There are various methods developed in the treating of used lubricating oil through refining.

2.7.2.1 Acid Clay method

Historically, the most successful technology for treating used oils was the acid-clay process developed in Washington State [29]. This process was capable of producing good quality lubrication stocks, but also a large volume of acid-sludge that is contaminated with petroleum [1]. In this process which has been widely used by re-refining facilities, used oil is initially subjected to filtration and dewatering mechanisms. Light products (Ethane, Methane etc.) are removed at the initial distillation step. It is then contacted with sulphuric acid which extracts oxygen compounds, asphalt, resin derivatives, other nitrogen and sulphur based compounds and metal contaminants from the oil. At the end of this process, desirable concentrations of paraffin and naphtalene molecules remain in the oil [30].

Acid clay process utilizes many of the following basic steps

i. Removal of water and solid particles by settling
ii. Vacuum distillation to remove light hydrocarbon
iii. Sulphuric acid treatment to remove gums, greases etc
iv. Alkaline treatment to neutralize acid
v. Water washing to remove soap
vi. Clay contacting to bleach the oil and adsorb impurities
vii. Filtering to remove clay and other solids [31].

In the conventional acid clay process the used lubricating oil is settled or filtered after collection. Then, the next step of re-refining involved removal of water from the used oil by atmospheric distillation. Thus, the used oil was distilled up to 200°C and furthered fractionated under vacuum (5mmHg) to eliminate the light hydrocarbons. The residual fraction over 350°C was obtained. The dehydrated oil (feed oil) was collected and sent to the next steps for further treatment. In the acid treatment, feed oil is treated with 98% concentrated sulphuric acid with a ratio 4:1, respectively. The mixture is then stirred for one hour at 50°C. The mixture was then allowed to cool and kept undisturbed for 24 hours for deasphalting and settling of acid sludge from acid treated oil. The next step of acid treatment was the clay percolation; an adsorption process would take place. Percolation technique is carried out via a continuous process. The treated oil by acid passed through adsorbent of activated clay in a double jacket long glass column. For each process, re-refined base-oil will be collected and analyzed [32].

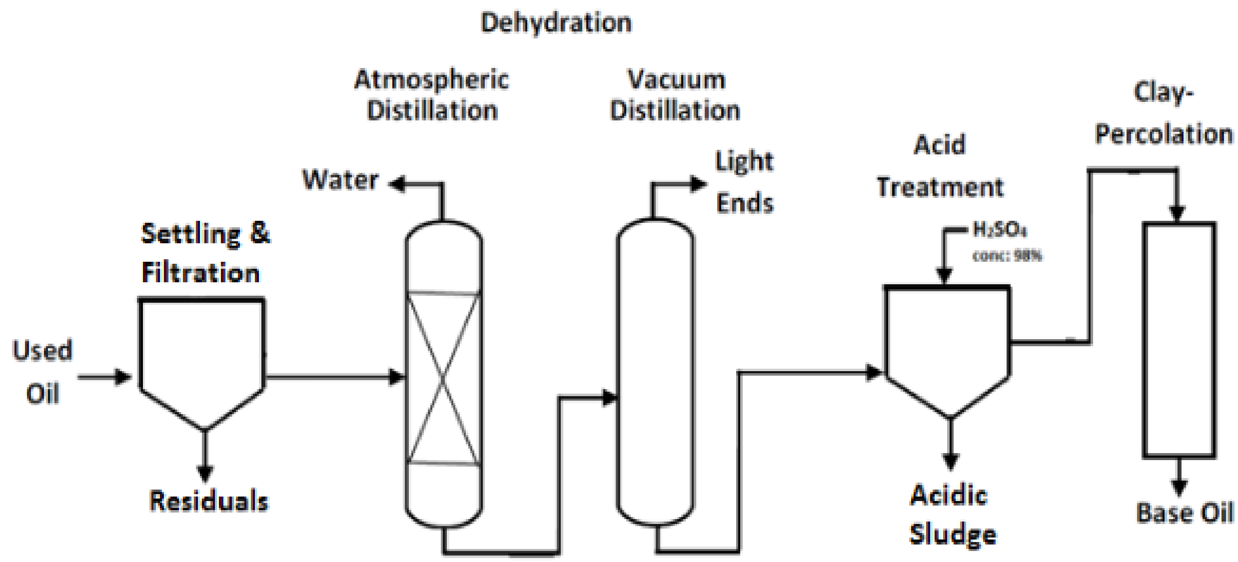


Fig 2.4 Process flow sheet for used oil recycling using acid clay method [32]

Acid-Clay Process for used oil recycling is the first used oil re-refining practice. Regardless of some controversies, it has several features and disadvantages that distinguish it from other technologies.

Features

- It requires low capital investment which makes it most cost effective for small and tiny scale plants
- The overall process is non-sophisticated and very simple to operate.
- Its operating cost is lower

Disadvantages

- It causes environmental pollution due to generation of acid sludge and acid gas emission.
- Generates acid sludge which is quite toxic and requires of expensive disposal techniques
- High operation costs, continuous clay consumption.
- Gives Lower yield due to loss of oil in sludge as well as clay since higher dosage of clay is required.
- Inconsistent quality.

- The sulphur and PAHs in the oil cannot be separated, the contaminants in the oil remain in the base oil.
- Life span of the equipment used in acidic environment is reduced.
- Obsolete process [42].

2.7.2.2 Solvent Extraction Method

Used lubricating oils may be re-refined by treatment with an organic solvent that dissolves base oil and flocculates the major part of additives and particulate matter. Solvent extraction is one of the cheapest and more efficient processes for used oil recycling. Used lubricating oils may be re-refined with organic solvents that dissolve base oil and segregate the additives and solid particles [34]

Solvent extraction became very popular because it overcame the major problem that came along with acid treatment which is acid sludge generation and the solvent could be recovered using distillation for reuse [33].

Solvent extraction re-refining processes include a precleaning stage to partially overcome problems associated to physical treatments. Afterwards, a solvent extraction operation is used to selectively extract base oil from the rest of ULOs components, which is finally obtained by distillation either under atmospheric or vacuum conditions. This process does not require additional finishing and it is competitive even for medium scale production. Additionally, this technology is interesting in terms of energy and natural sources conservation, as it allows the recovery of most base oil for reuse [11]. The main processes that are take place in solvent extraction method are pretreatment dehydration, diesel striping, extraction and separation.

Under pretreatment stage, used lubricating oil is stored for several days to allow large suspended particles to settle under gravity or filtered using vacuum filter since it contains different types of solid materials.

Then after the pretreatment of the used oil, it must be dehydrated in order to remove the water in the used oil. The oil is heated in a heating mantle at a temperature of 120^0C-140^0C to remove the water that has been mixed into it.

The dehydrated used oil is then fed continuously into a vacuum distillation for fractionation at a temperature of above 240^0C. Lighter oils boil off first and are removed. Other heavier components will do not boil in the conditions used.

The lubricating oil fraction obtained by vacuum distillation will be mixed by agitation with the solvent in an appropriate ratio. The lubricating oil and solvent mixture will be allowed to settle in the separation flask for four hours. The aromatic content and degraded additives present in the lubricating oil fraction are settle at the bottom and the lubricating oil fraction and solvent mixture layer forms at the top. Finally separation of oil from solvent will be take place by distillation either under atmospheric or vacuum conditions. The lubricating oil produced at this stage is similar to that of the base lubricating oil [36].

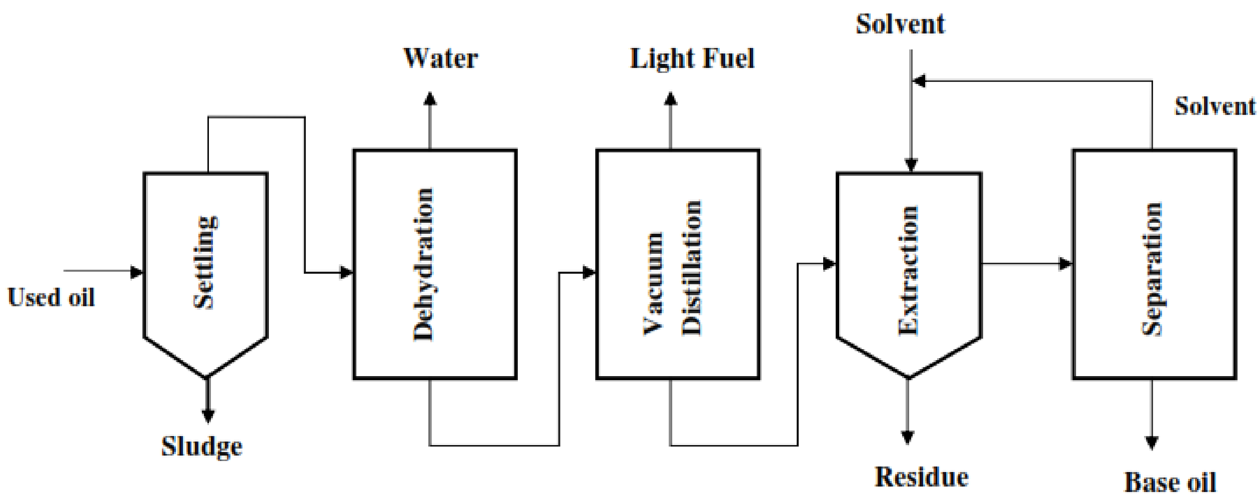


Figure 2.5: Process flow sheet for recycling of used lubricating oil using solvent extraction method [36]

Solvent extraction method has several features and disadvantages that distinguish it from other technologies.

Feature

- Produces good quality base oil
- The process is carried out under lower pressure and temperature compared to other technologies
- The process has high product operational efficiency
- Solvent is recyclable.

- Small quantities of waste and contaminants are generated and waste disposal cost is low.

Disadvantages

- Based on the waste oil used, the solvent costs can be high.
- If it is operated at high pressure, it requires highly skilled operator [42].

2.7.2.3 Vacuum distillation

The basic steps in vacuum distillation are pretreatment of oil to remove impurities which can result in fouling and corrosion of equipment followed by distillation where water and light hydrocarbons are separated then vacuum distillation using thin film or a conventional vacuum column. Vacuum distillation is followed by hydro treating of distillate with high pressure and temperature in the presence of catalyst for the purpose of removing chlorine, sulfur, nitrogen and organic component. Hydro treated oil is further fractionated under high vacuum in to components of industrial hydraulic and mother oil. The resulting residue from the vacuum distillation treatment can be used for road and bitumen production [33].

2.7.2.4 Hydrogenation

As the name implies the process involves hydrogenation. Used oil and hot hydrogen was heated and mixed in a pressurized mixing chamber. The mixture was transferred to a separator routed to a residue stripper and processed in a catalytic reactor to remove soluble metals and passed through hydro-finishing reactor for dechlorination, desulphurization and other processes. The treated hydrocarbons resulted in products of improved odor, chemical properties and color. The residue resulting from the process is a high boiling range of hydrocarbon product fractionated in to neutral oil products with varying viscosities which can also be used to blend lubricating oil [33].

2.7.2.5 Membrane Technology

Another method used for regeneration lubricating oils was membrane technology. Three types of polymer fiber membranes [polyethersulphone (PES), polyvinylidene fluoride PVDF), and polyacrylonitrile (PAN)] were used for recycling the used/waste engine oils. This process is carried out at 0.1 MPa pressure and 40°C and is a continuous operation which removes metal particles or dusts from used/waste engine oil and also improves the recovered oils liquidity as well as flash point. Despite the above mentioned advantages, the expensive membranes may get damaged and fouled by large particles [35].

2.8 Factors affecting solvent extraction

Solvent extraction is one of the cheapest and more efficient processes for used oil recycling [34]. However, the efficiency of the extraction process can be affected by different factors such solvent type, solvent to oil ratio, time, extraction temperature and other.

2.8.1 Solvent Type

Waste lubricating oils may be redefined by treatment with an organic solvent that dissolves base oil and flocculates the major part of additives and particulate matter. This operation is intended to substitute the classical reaction with sulfuric acid, which generates an acid sludge, creating difficult disposal problems [37].

Several factors such as high molecular weight, solubility, sludge formation as well as ease of recovery were put into consideration before solvent choices were made. The ability of a polar solvent to form flocs from pre-treated lubricating oil depends on solubility parameters [33]. As is usually the case, it is desirable for solvents to be cheap, noncorrosive, nonflammable, non explosive, non toxic, easily removable, and easily recoverable. It obviously may be impossible to meet all these objectives. The characteristics of the matrix to be extracted, mass transfer mechanisms also have to be considered in developing an optimal extraction system.

Waste motor oils are composed of (a) unchanged base oil molecules that one intends to recover; (b) oxidized base oil molecules; and (c) polymers, such as polyolefins and polymethacrylates, generally introduced in motor oils as viscosity index improvers, pour point depressants, and dispersants; (d) other additives such as dispersants, detergents, antifoaming and extreme pressure agents, and others, including a great variety of chemical products such as, for example, succinimides, sulfonates, phosphonates, dialkyldithiophosphates of calcium, zinc, and barium; phosphites; amines; phenols; silicones; carboxylic acid salts; and many others; (e) water, originating from fuel combustion in the engine and accidental contamination by rain; (f) light hydrocarbons from incompletely burned gasoline and diesel oil; (g) carbonaceous particles formed by partial coking of fuels, graphitic particles from graphitic oils, and metallic particles produced by motor wear. The bulk of these particles are kept in stable dispersion by the dispersant additives. If a solvent is to be designed to recover the base oil, separating it from the additives and particulate matter, the solvent must have the following properties: (1) it must be miscible with the base oil contained in the waste oil being processed; and (2) when mixed with the waste oil, it must reject from the solution the additives and the dispersed particles (or part of them), allowing their aggregation to particle sizes big enough to separate from the liquid by sedimentation. A liquid meeting requirement 1 and 2 will be called in this paper an extraction-flocculation solvent [37].

A large number of other pure organic liquids and their multicomponent solutions exhibit those properties and have potential utility for separating the base oil from the additives and other undesirable impurities in waste oils. Ketones and alcohols are the most efficient solvents for extraction since they are miscible with the base oil on the one hand and focculate some of the additives and carbonaceous compounds by applying an antisolvent effect on the other hand. The capability to segregate sludge is closely related to the difference between the solubility of the solvent and the polyisobutylene, a viscosity improver additive [37].

2.8.2 Solvent to oil ratio

Solvent to oil ratio has a very prominent effect on the quality of oil produced (its characteristics) as well the percentage of oil recovered from a pre-treated oil sample. Usually the higher the solvent to oil ratio the greater its ability to remove contaminants in the form of sludge from the oil sample [33].

Increasing the solvent to oil ratios increases the sludge forming capabilities. However, the viscosity of the oil decreases as solvent to oil ratio increases this is because the sludge removed from the treated oil contains aromatic compounds, aromatic compounds with high molecular weights have been removed, leaving behind paraffinic compounds of relatively lower viscosity when compared to its aromatic counterparts leading to oil which has lower viscosities [31].

It is clear that as the solvent oil ratio increase, the maximum percent sludge removal increase and the raffinate oil decreased. This is because as the oil to solvent proportions increases the medium mutual solubility of the oil in the solvent increases. The removal of sludge increases as the solvent: oil ratio increases till 3:1 [7].

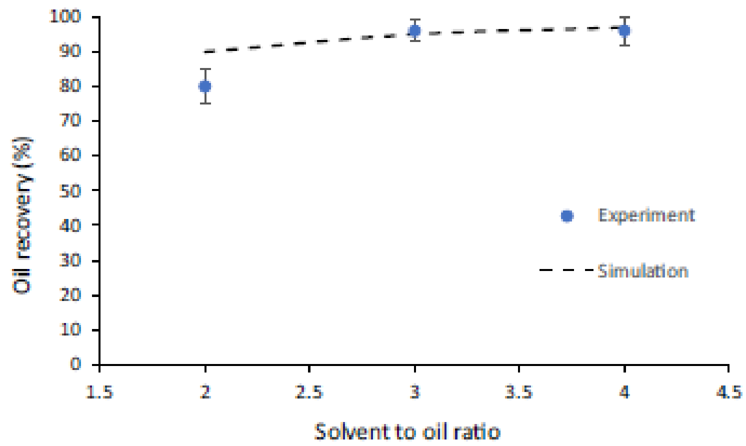


Figure 4.1 Percentage of oil recovery versus solvent-to-oil ratio [38]

2.8.3 Extraction time

In general, a prolonged extraction time results in an increased yield of the oil until equilibrium is reached. Thereafter, the concentration of compound will not increase further but there will have greater liability for degradation. Prolonged extraction time is also not desirable from an economic standpoint of labor and energy requirements. Therefore it is essential to find an optimum extraction time [9].

In waste lubricating refining techniques specifically acid/clay and solvent extraction methods 24 to 72 hours of reaction time/extraction time is recommended [37].

2.8.4 Temperature

Temperature generally affects both the equilibrium and mass transfer rate of the extraction process. The higher temperature results in greater solubility of compounds in the solvent [9]. However, in solvent extraction method the process of refining used lubricating oil is performed by through a process of extraction and flocculation technique at higher temperature above room temperature enhances the rate of sol vent vaporization this leads to decrease the extraction yield [37].

Chapter 3

Materials and Methods

The experimental work was done in laboratory of chemical engineering department of Adama science and Technology University, Adama Ethiopia, school of chemical and bio engineering of Addis Ababa institute of technology and chemistry department of science faculty of Addis Ababa University, Addis Ababa Ethiopia.

3.1 Materials and Equipment

Materials used during the experiment were used lubricating oil, n-butanol (99.9%), hexane (99.9%), ethanol (99%), isopropanol (99%) potassium hydroxide, distilled water, filter paper and bleaching earth clay. All the chemicals and reagents are purchased from chemicals and laboratory equipment suppliers found in Addis Ababa.

The equipment used were vacuum pump, heating mantle, flask, beaker, condenser, measuring cylinder, thermometer and funnel are purchased from chemical and laboratory equipment suppliers found in Addis Ababa while chiller, centrifuge, Furness, vibro viscometer, balance, density bottle, oven and deep freezer are obtained from Adama Science and Technology University (Chemical engineering department) and Addis Ababa University (school of chemical and bio engineering and Chemistry department).

3.2 Experimental Methods

3.2.1 Raw material characterization

Used lubricating oil (15W 40) for diesel engine vehicle for this experiment was obtained from SCANIA truck after the car made one complete trip to Djibouti (which is around 2500km). The physiochemical properties such as kinematic viscosity, specific gravity, color measurement, pour point, flash point, ash content, total acid number, water content and metal content of used lubricating oil were determined.

3.2.1.1 Determination of Specific gravity

The density of used oil was determined using density bottle method. A clean and dry density bottle of 25ml capacity at 25^0C was weighed in gm. Then the bottle was filled with water and reweighed at 25^0C. Used oil was brought to 25^0C and the water was substituted with this used oil after drying the density bottle and weighed again and the specific gravity is determined by the following formula. (A.O.A.C official method 920.212, 2000).

$$Specific\ gravity\ at\ 25℃ = \frac{A-B}{C-B} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots. (3.1)$$

Where: A= weight in gm of density bottle with used lubricating oil at 25^0C

B= weight in gm of density bottle at 25^0C

C= weight in gm of density bottle with water at 25^0C

3.2.1.2 Determination of Water content

Water content is the amount of water present in the lubricant. It was estimated by putting a measured amount of used oil in an oven at 105°C for 1 hour. The sample was removed from the oven and cooled in a dissector and weighed. The process was repeated until a constant weight is observed and the water content of the oil is determined by

$$water\ content\ of\ the\ used\ lubricating\ oil = \frac{w_1 - w_2}{w_1} * 100\% \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (3.2)$$

Where: w_1 = Original weight of the used lubricating oil before drying

w_2 = Weight of the used oil after drying

3.2.1.3 Determination of kinematic viscosity

Viscosity is a very important property for grading lubricants. It is a measure of fluid resistance to flow and is strongly dependent on temperature. Kinematic viscosities of the oil at 40°C and 100°C are usually used to characterize oil quality. A kinematic viscosity of used lubricating oil was measured indirectly using vibro viscometer by determining dynamic viscosity. Initially, a sample was heated at a temperature of 40^0C. Known quantity of used lubricating oil was measured and fed to a sample holder of the vibro viscometer. A sensor of the viscometer is immersed to the oil and then a dynamic viscosity of used lubricating oil was displayed on the vibro viscometer screen at a temperature of 40^0C. And then the kinematic viscosity was calculated by

$$\nu = \frac{\mu}{\rho} \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots. (3.3)$$

Where: μ = dynamic viscosity of used lubricating oil

ρ= density of used lubricating oil

3.2.1.4 Total acid number

The total acid number is the measurement of acidity in oils used as lubricants. It is one of the crucial chemical properties that gives stability to lubricating oils. Lubricating oil is said to be stable if it can resist oxidation that yields acids, lacquers and sludge. Normally, acidity increases with the oxidation of lubricating oils [15]. Total Acid Number (TAN) of the used oil was determined using ASTM D974. It is the quantity in milligrams of potassium hydroxide (KOH) per gram of oil necessary to neutralize acidity. Two grams of used lubricating oil was weighed and mixed with 100 mL of the titration solvent (toluene and isopropyl alcohol containing a small amount of water) and 0.5 mL of the indicator solution (phenolphthalein) and swirled until the sample was entirely dissolved by the solvent. The mixture assumes black color and titrated with 0.1 M KOH solution in increments and mixed vigorously near the end point i.e. pink color. To observe the end point of dark-colored oil, the flask was shaken vigorously to produce momentarily slight foam and the color change occurs under a white fluorescent. The neutralization number or Total Acid Number (TAN) is calculated as;

$$TAN\left(\frac{mg\ KOH}{g\ sample}\right) = \frac{[(A-B)*M*56]}{W} \dots\dots\dots\dots\dots\dots\dots..3.4$$

Where; A = KOH solution required for titration of the sample, mL,

B = KOH solution required for titration of the blank, mL,

M = molarity of the KOH solution

3.2.4.5 Flash point

Flashpoint is the temperature to which the oil must be heated under specific conditions to give off sufficient vapor to form a flammable mixture with air [15]. Flash point was determined by open cup method. The oil is taken in the open cup and heated. The temperature at which the vapor given out by the heating oil catches fire momentarily when exposed to the ignition source is noted as flash point temperature. Used lubricating oil sample was introduced into a beaker and then heated on heating mantle to determine the temperature at which a flash will appear on the surface of the sample when exposed to the ignition source. This temperature was measured using a thermometer and recorded as the flashpoint (ASTM D92).

3.2.4.6 Pour point

The pour point indicates the temperature below which the oil loses its fluidity and will not flow or circulate in the lubricating system. Lubricating oils with low pour points show good quality. 20 mL of used lubricating oil sample was introduced into a container and then chilled at a specific rate and checked at intervals of 5 min until the oil stopped flowing. The temperature at which this occurred was recorded as the pour point (ASTM D97).

3.2.4.7 Color

It is normal to see oil color change without experiencing any lubricant issues. There are several factors that can cause an oil to change color after use including oxidation, deposits and contamination. Visual inspection was used to detect the color of used lubricating oil.

3.2.4.8 Ash content

Ash content of used lubricating oil was determined using a furnace. A 20 g of oil was added in a burning cup. Then, the sample was placed in a furnace. A furnace was located at a temperature of 500^0C for 1 hour and after burning the residue sample was weighted and ash content was calculated using a formula

$$Ash\ Content\ (\%) = \frac{Final\ mass\ of\ sample\ after\ burning}{Initial\ mass\ of sample} * 100\% \quad \ldots\ldots\ldots\ldots\ldots..(3.5)$$

3.2.4.9 Metal content

The metal content of different metals found in the used lubricating oil samples was obtained by atomic absorption spectrometry (Analytikjena ASS Zeenit 700P). Metal concentrations were determined from the calibration curve that is obtained from standard solutions. These tests were conducted at Addis Ababa University, science faculty chemistry department.

3.2.2 Raw material preparation

Used motor lubricating oil that was freshly collected was let to settle for 24hrs before filtering. Filtration of the used oil was carried out to remove impurities such as sand, metal chips, micro impurities that contaminated the lube oil. This was done using a vacuum pump, funnel, and a filter paper. After that, the used lubricant was taken at 1 liter per batch within an open flask on a heating mantel for dehydration for about 90 min. This process significantly reduced impurities in waste oil such as antifreeze, water and other solvents. After the used lubricant was dehydrated the flask was further distilled at a temperature of 250°C to remove the light fuel and the hydrocarbons, Vacuum pump was used to elevate the temperature. The distilled oil will leave to cool down and settle for at least 24 hr.

3.2.3 Base oil extraction

2 gram of KOH was added to 300ml of four different solvents (hexane, ethanol, isopropanol and butanol) in a beaker. The mixture was strongly mixed until all the KOH dissolved into the solvent solution and ready for solvent extraction. Then, 100 ml of used oil sample was mixed with the solvent/KOH mixture in a beaker for 25 min under continuous stirring, this is to ensure perfect mixing and at the same time preventing any losses of oil to the sludge. After strong agitation, it was then left under room temperature for 72 hr to allow extraction- flocculation. The solvent was then separated by distillation.

The extracted oil was then mixed with a fixed amount of 15 wt% clay into a beaker. The mixture was then subjected to intense agitation for 10 min. It was then left at room temperature for four hour to allow gravity settling. After that, the recovered oil was separated from the adsorbent mixture and the oil sample was then collected. The yield of oil is calculated on the basis of initial volume of used oil taken.

$$Yield = \frac{Recoverd\ oil}{Used\ oil\ taken} * 100\% \ldots \ldots \ldots \ldots \ldots \ldots \ldots . (3.6)$$

With the solvent that yields a maximum oil, the effect of solvent to oil ratio (namely 1:1, 2:1, 3:1 and 4:1) and extraction time (24 hrs, 48 hrs, 72 hrs and 96 hrs) was studied by implementing full factorial experimental design.

3.2.4 Product Characterization

The physicochemical properties such as kinematic viscosity, specific gravity, color measurement, pour point, flash point, ash content, total acid number, water content, and metal content for the recycled oil which was obtained at optimum operating condition was determined.

3.2.4.1 Determination of Specific gravity

The density of recycled oil was determined using density bottle method. A clean and dry density bottle of 25ml capacity at 25^0C was weighed in gm. Then the bottle was filled with water and reweighed at 25^0C. The recycled oil was brought to 25^0C and the water was substituted with this recycled oil after drying the density bottle and weighed again and the specific gravity was determined by the following formula. (A.O.A.C official method 920.212,2000).

$$Specific\ gravity\ at\ 30°C = \frac{A - B}{C - B} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots. (3.7)$$

Where: A= weight in gm of density bottle with recycled oil at 25^0C

B= weight in gm of density bottle at 25^0C

C= weight in gm of density bottle with water at 25^0C

3.2.4.2 Determination of kinematic viscosity

A kinematic viscosity of the recycled oil was measured indirectly using vibro viscometer by determining dynamic viscosity. Initially, a sample was heated at a temperature of 40^0C. Known quantity of recycled oil was measured and fed to a sample holder of the vibro viscometer. A sensor of the viscometer was immersed to the recycled oil and then a dynamic viscosity of recycled oil was displayed on the vibro viscometer screen at a temperature of 40^0C. And then the kinematic viscosity was calculated by

$$\nu = \frac{\mu}{\rho} \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots. (3.8)$$

Where: μ = dynamic viscosity of recycled oil

ρ= density of oil recycled oil

3.2.4.3 Determination of Water content

Water content is the amount of water present in the lubricant. It was estimated by putting a measured amount of recycled oil in an oven at 105°C for 1 hour. The oil was removed from the oven and cooled in a dissector and weighed. The process was repeated until a constant weight was observed and the water content of the oil is determined by

$$water\ content\ of\ the\ recycled\ oil = \frac{w_1 - w_2}{w_1} * 100\% \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots (3.9)$$

Where: w_1 = Original weight of the recycled oil before drying

w_2 = Weight of recycled oil after drying

3.2.4.4 Total acid number

Total Acid Number (TAN) of recycled lubricating oil was conducted using ASTM D974. It is the quantity in milligrams of potassium hydroxide (KOH) per gram of oil necessary to neutralize acidity. Two grams of recycled oil sample was weighed and mixed with 100 mL of the titration solvent (toluene and isopropyl alcohol containing a small amount of water) and 0.5 mL of the indicator solution (phenolphataline) and swirled until the sample was entirely dissolved by the solvent. The mixture assumes yellow color and titrated with 0.1 M KOH solution in increments and mixed vigorously near the end point i.e. pink color. To observe the end point the flask was shaken vigorously to produce momentarily slight foam and the color change occurs. The neutralization number or Total Acid Number (TAN) is calculated as;

$$TAN\left(\frac{mg\ KOH}{g\ sample}\right) = \frac{[(A - B) * M * 56]}{W} \dots\dots\dots\dots\dots\dots\dots..3.10$$

Where; A = KOH solution required for titration of the sample, mL, B = KOH solution required for titration of the blank, mL,

M = molarity of the KOH solution

3.2.4.5 Flash point

Flash point was determined by open cup method. The oil was taken in the open cup and heated; the temperature at which the vapor given out by the heating oil catches fire momentarily when exposed to the ignition source is noted as flash point temperature. Recycled oil sample was introduced into a beaker and then heated on heating mantle to determine the temperature at which a flash will appear on the surface of the sample when exposed to the ignition source. This temperature was measured using a thermometer and recorded as the flashpoint (ASTM D92).

3.2.4.6 Pour point

20 mL of recycled lubricating oil was introduced into a container and then chilled at a specific rate and checked at intervals of 5 min until the oil stopped flowing. The temperature at which this occurred was recorded as the pour point (ASTM D97).

3.2.4.7 Color measurement

Visual inspection was used to detect the color of recycled lubricating oil.

3.2.4.8 Ash content

Ash content of recycled lubricating oil was determined using a furnace. A 20 g of recycled oil was added in a burning cup. Then, the sample was placed in a furnace. A furnace was located at a temperature of 500^{0}C for 1 hour and after burning the residue sample was weighted and ash content was calculated using a formula

$$Ash\ Content\ (\%) = \frac{Final\ mass\ of\ sample\ after\ burning}{Initial\ mass\ of recycled\ oil\ sample} * 100\% \quad \ldots\ldots\ldots\ldots\ldots..(3.11)$$

3.2.4.9 Metal content

The metal content of different metals found in the recycled lubricating oil samples was obtained by atomic absorption spectrometry (Analytikjena ASS Zeenit 700P).

3.3 Experimental Design

Factorial design is used to investigate the effect of each factor. In a factorial experiment all the possible combinations factor levels would be tested and it would be possible to determine the effect of individual factors and to assess the effect of change of two or more variable at a time (Zivorad, 2004).

The analysis was performed by utilizing Design Expert software using general factorial design method. This method of experiment design helps to differentiate the significance of the main and the interaction factors. The soft-ware also used to develop the mathematical model that will describe the effect of main and interaction factors on the Response.

Factors: 2 factors were investigated as mentioned earlier; these are: solvent to oil ratio and time of extraction.

Factor Levels: For each factor four levels are considered.

Replicates: Each independent experiment is repeated two times.

Number of runs: For "m" levels, "n" factors and "k" replicate, the number of experimental runs that need to be performed is equal to $\mathbf{k*m^n}$ (Zivorad, 2004). In this study m=4, n=2 and k=2 thus, $\mathbf{2*4^2 = 32}$ experimental runs were performed.

The factors and their levels

The levels that were selected for each factor were:

1. Solvent to oil ratio: 1:1, 2:1, 3:1 and 4:1
2. Extraction time: 24hr, 48hr, 72hr and 96hr

Model equation

Finally, Regression models were established for the dependent variables to fit the experimental data for the response using Design expert 7.0.0 software.

Chapter Four

Result and Discussion

This part discusses the results obtained from the experiment performed on the used lubricating oil. The experiments were performed in three steps.

- Step 1 (Used oil characterization): An experiment was conducted to assess the quality of used lubricating oil. Under this experiment the specific gravity, kinematic viscosity, water content, total acid number, flash point, pour point, color measurement, ash content, and metal content of the used lubricating oil were determined.
- Step 2 (Base oil extraction): In this step the base oil was extracted using solvent extraction method following clay treatment.
- Step 3 (product characterization): In this step base oil which was extracted at a condition that gives maximum oil yield was characterized. Under this step specific gravity, kinematic viscosity, water content, total acid number, flash point, pour point, color measurement, ash content and metal content of the base oil was determined.

4.1 Raw material Characterization

The properties of used lubricating oil are the function of length of use. Depending on the work load the oil is discarded after 2000km - 5000km is used. Used lubricating oil was characterized prior to recycle the base oil. Specific gravity, kinematic viscosity, water content, total acid number, flash point, pour point, color measurement, ash content and metal content of the used lubricating oil were determined and the results are given below.

4.1.1 Specific gravity

The specific gravity of the oil was calculated using equation (3.1) and the result is summarized in the table below

Table 4.1 Specific gravity

Item	Value		
	Ran 1	Ran 2	Ran 3
A= weight in gm of density bottle with oil at 30^0C	41.57	41.24	41.36

B= weight in gm of density bottle at 30^0C	13.60	13.60	13.60
C= weight in gm of density bottle with water at 30^0C	43.61	43.8	43.75
Specific gravity	0.932	0.921	0.925
Mean±SD	0.926±0.06		

From the above used oil analysis, the specific gravity was in agreement to those of a literature since (Adewole et.el, 2019) and (Doaa et.el, 2018) who reported a specific gravity of 0.928 and 0.92 respectively. As also expected, the value obtained is higher to the value of the virgin oil (SAE 40) due to contamination and oxidation.

4.1.2 Water content

The water and volatile matter of the oil was determined by oven method. 5 gm of oil was taken and put in oven and the weight was recorded every one hour and the result obtained is summarized in the table below.

Table 4.2 Experimental result for water content

Time (hr)	0	1	2	3	4
Weight of sample (gm)	5.0	4.93	4.91	4.86	4.86

Using equation (3.2) the water content of the oil

$$Water\ content \quad = \frac{5.0gm - 4.86gm}{5.0gm} * 100\%$$

$$= 2.8\%$$

The water content of used oil is higher than the result reported by (Doaa et.el, 2018) which was 0.25% the rise in water content was caused by may be the difference in time that the oil is used before it is changed [1].

4.1.3 Ash content

Ash content represents the incombustible component (inorganic matter) remaining after the sample is completely burned. The ash content was determined using furnace at 500^{0}C and the result is summarized in the table below.

Table 4.3 Experimental result for ash content

Items	Run 1	Ran 2	Ran 3
Mass of oil (sample)	20.13	20.05	20.04
Mass of empty crucible	42.94	42.94	42.94
Mass of crucible + sample after burning	43.28	43.26	43.27
Ash content (%)	1.69	1.60	1.65
Mean ± SD	1.65±0.05		

An average value of 1.65% ash content was obtained for used oil sample. Higher value of ash content in used lubricating oil is due to the concentration of the contaminants in the oil such as dirt, iron oxide, wear metals and corrosion products as well as the concentration of ash producing additives which are built during the course of lubrication inside the engine [38].

4.1.4 Kinematic Viscosity

The kinematic viscosity of the oil at 40^{0}C and 100^{0}C was determined indirectly by determining dynamic viscosity at the specified temperatures using vibro viscometer and then dividing by density of used lubricating oil. The dynamic viscosity obtained is summarized in the table below.

Table 4.4 Dynamic viscosity

Temperature (^{0}C)	Dynamic viscosity (mPa.s) (cP)
40	105
100	9.65

Using equation (3.6) and density of oil 0.926gm/cm^3, the kinematic viscosity of oil is

Table 4.5 Kinematic viscosity

Temperature (^{0}C)	Kinematic viscosity (mm^2/s) (cSt)
40	113.4
100	10.4

From the above analysis, it can be seen that there is an increase in kinematic viscosity of used lubricating oil this is due to metallic scrapping act as catalysis at high combustion temperature and oxygen vicinity, produce an asphalt like sludge which increases the viscosity during its application period [43].

The result obtained is in agreement to those of literature since Adewole B. et al 2019 and Ousman et al 2017 who reported a kinematic viscosity of used lubricating oil at 40^{0}C and 100^{0}C of 105.3cSt and 11.4 cSt and 107.5 and 12.9cSt respectively.

4.1.5 Flash Point

Flashpoint is the temperature to which the oil must be heated under specific conditions to give off sufficient vapor to form a flammable mixture with air. It gives an indication of the presence of volatile compounds in the oil. A decrease in flashpoint reveals that the oil is contaminated through dilution of lubricating oils with unburned fuel. An increase in flashpoint indicates the evaporation of light components from the lubricating oil [15]. Open cup method was used to determine the flash point of the oil; the result is summarized in the table below.

Table 4.6 Experimental result for Flash point

Run	Flash point (^{0}C)	Mean ± SD
1	210	208±3^{0}C
2	210	
3	205	

The flash point determined 208^0C which is slightly lower than the literature value reported by (Adewole et al, 2019) which is 212^0C. This tells as lower amount of light fuel (diesel) is found in the used lubricating oil used in this study.

4.1.6 Pour point

The pour point of the oil was determined using deep freezer and digital thermometer. The temperature controller was tuned to maximum. The sample was placed and checked every two minutes. It was obtained that the oil stops flowing at -24.3^0C. Therefore, the pour point of the oil can be taken as -24.3^0C. Nancy Z. and Hosni T., 2020 reported a value of -24^0C which is nearly in agreement to the value obtained in this study.

4.1.7 Total acid number

The total acid number was determined using titration. After the required solutions were prepared with the specified concentration, titration to end point was done and the following result is obtained.

Table 4.7 Total acid value

Run	Volume of KOH for blank	Volume of KOH for sample	Mass of sample (g)	Total acid number
1	24.5	22.8	2	4.76
2	24.5	23.1	2	3.92
			Mean ±SD	4.34±0.59

An average value of 4.34% total acid value was obtained for used oil sample. Higher acid value was obtained when compared to Miruna D. and Sorin A., 2020 which is 3.1. Total acid number increases due to oxidation of lubricating oil [19]. Higher acid concentration in used lubricating oil can cause corrosion in automotive engine [15]

4.1.8 Color

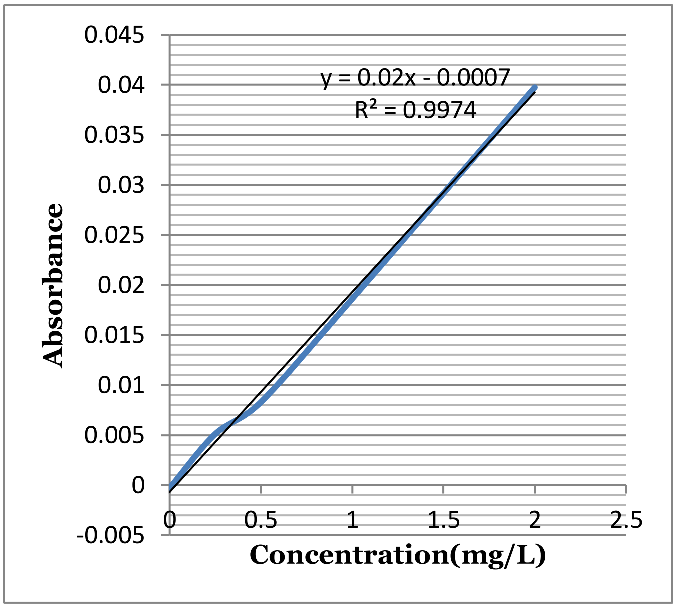
Visual inspection was implemented to determine the color of used lubrication oil. According to the result obtained the color of used lubricating oil was considered as deep black color.

4.1.8 Metal content

A standard solution for the metals Ca, Fe, Pb and Zn was first prepared and a calibration curve was established.

Table 4.8 standard calibration table

		Absorbance			
Sample	Standard concentration (gm/L)	Ca	Fe	Pb	Zn
Ca	0	-0.00029			
	0.25	0.00512			
	0.5	0.00828			
	1	0.01861			
	2	0.03975			
Fe	0		-0.01298		
	0.25		0.00072		
	0.5		0.01549		
	1		0.04510		
	2		0.09587		
Pb	0			0.00010	
	0.25			0.00396	
	0.5			0.00885	
	1			0.01417	
	2			0.01822	
Zn	0				-0.00243
	0.25				0.11735
	0.5				0.23053
	0.75				0.34938
	1				0.47639



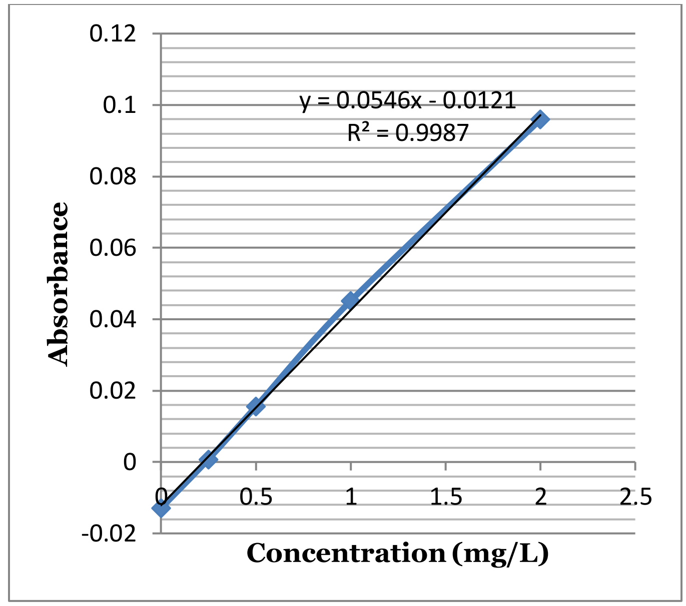
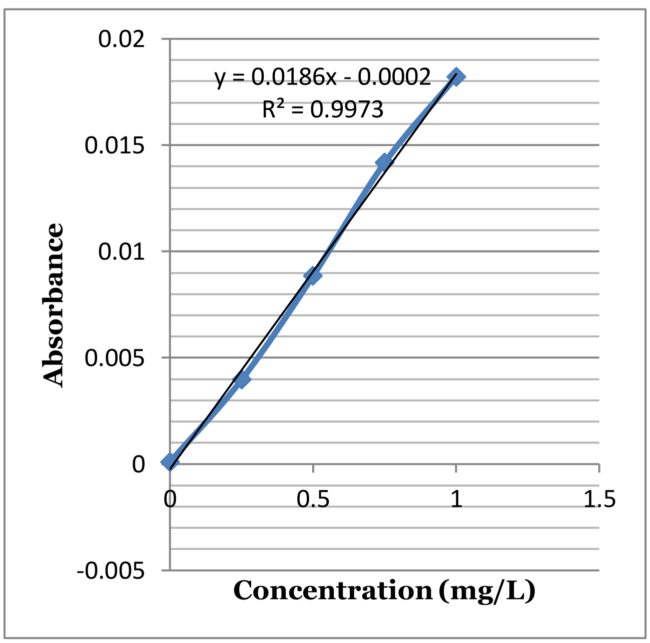


Figure 4.1 Calibration curve for calcium (Ca)

Figure 4.2 Calibration curve for iron (Fe)



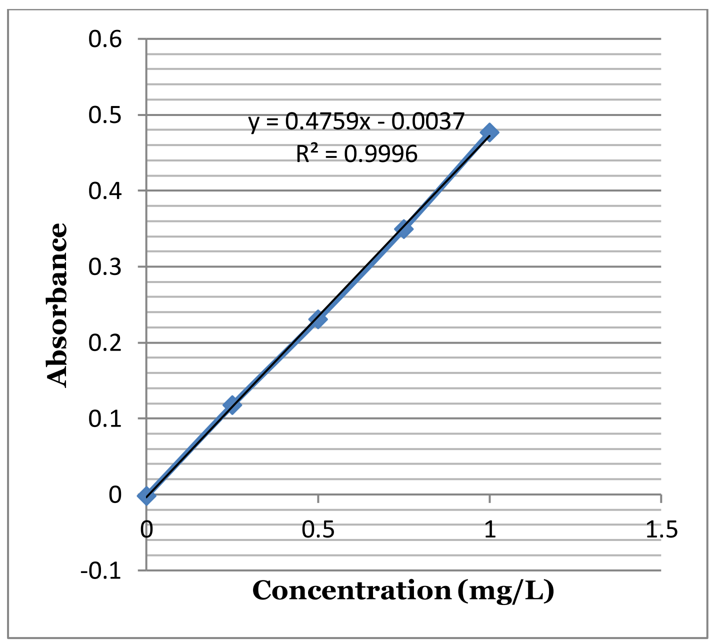


Figure 4.3 Calibration curve for lead (Pb)

Figure 4.4 Calibration curve for zinc (Zn)

As it can be seen in the graph the correlation coefficients for all metals are greater than 1, therefore, the corresponding metal concentration for used oil sample were red and summarized in the table 4.10 below.

Table 4.9 Metal content of used lubricating oil

Metals	Concentration in mg/L (ppm)
Calcium	224.1
Iron	703.3
Lead	373.5
Zinc	53.5

According to the analysis 224.1ppm calcium, 703.3ppm iron, 373.5ppm lead and 53.5ppm zinc was obtained for used lubricating oil sample. The metal content of lubricating oil is a very important parameter as the metal content in an oil sample as it can increase the rate of corrosion of the metal parts it is in contact with [33]. Trace amount of metal content is available in virgin lubricating oil [37]. Calcium comes mainly from additives added to the oil which are depressants (pour point depressant) and detergents. Zinc is introduced to base oil in the form of additives packaged as antioxidant, corrosion inhibitors and anti-wear [33]. Iron and lead are introduced to oil due to any engine is mostly made of iron and lead and during combustion in the engine chamber of any fuel, thinny parts of these metals are found in part per million [19].

4.2 Base oil Extraction

4.2.1 Vacuum Filtration

Filtration of the used oil was carried out to remove impurities such as sand, metal chips, micro impurities that contaminated the lube oil. This was done using a vacuum pump, funnel, and a filter paper. 10 liters of used lubricating oil was taken and filtered. A batch filtration (1liter per batch) was implemented to accomplish the task required in this step.

(a)

(b)

(c)

Figure 4.5: (a) Vacuum filtration setup (b) used oil sample feed to vacuum filtration (c) filtered used oil sample

4.2.2 Dehydration

The filtered oil sample was allowed to dehydrate to remove water added to the oil during combustion. The dehydration process was carried out by placing measured quantity of filtered used lubricating oil in a beaker and applying simple atmospheric distillation set up at a temperature of 140^0C. The dehydration process was done for 90 minutes.

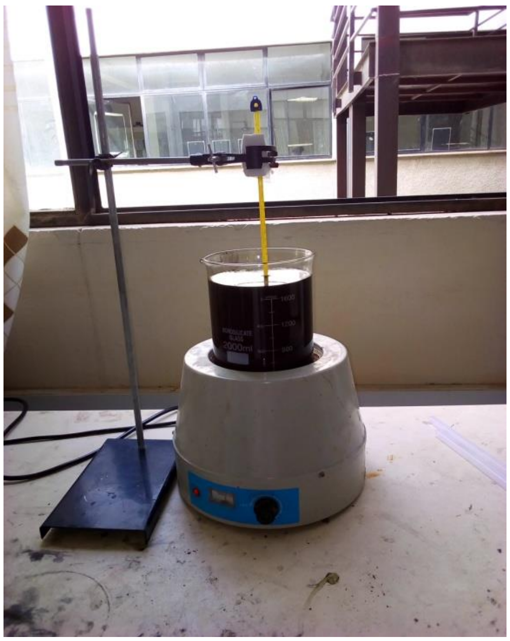


Figure 4.6 Dehydration process

4.2.3 Vacuum Distillation

The dehydrated used lubricating oil was further distilled to eliminate light fuels at a temperature of 250^{0}C. A vacuum pump was connected to elevate the temperature to the boiling point of light fuels. The distillation process was continued until last drop of condensed light fuel was seen. The distillation process was took place with two different setup as in the figure below.

Setup 1

Setup 2

Figure 4.7 Vacuum distillation

Light fuel (diesel) recovered under this stage for four consecutive batches are summarized in the table below.

Table 4.10 Recovered diesel fuel

Batch	1	2	3	4
Used oil sample (gm)	300	350	350	350
diesel recovered (gm)	12.8	14.2	15.1	13.2
Percentage recovery	4.3%	4.1%	4.3%	3.7%

$$\text{Average percentage recovery of diesel fuel} = \frac{4.3\% + 4.1\% + 4.3\% + 3.7\%}{4} = 4.1\%$$

Used lubricating oil can be used as an alternative fuel in variety of engine configuration and furnace [38]. Presence of such amount (4.1%) diesel fuel can facilitate the application of used oil for such purpose. However, combustion of used lubricating oil for energy utilization will result pollution and gas emission for greenhouses [28].

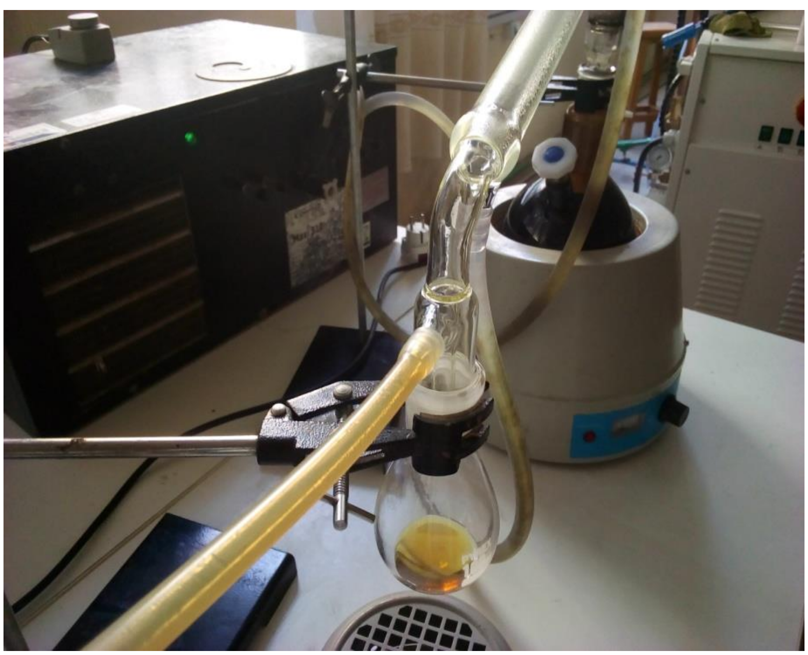


Figure 4.8 Light fuel (diesel) recovery

4.2.4 Oil Extraction

The extraction of oil treated by proposed solvents have been investigated and the percentage oil yield was calculated using equation (3.11) and the result is summarized in the table below.

Table 4.11 Percentage oil yield

No. run	Factor	Oil Yield (%)			Mean$\pm$ SD (%)
	Solvent type	Replicate 1	Replicate 2	Replicate 3	
1	Ethanol	6.12	5.20	5.85	5.72±0.47
2	Hexane	No phase separation	No phase separation	No phase separation	No phase separation
3	n-butanol	73.82	78.09	71.74	74.55±3.24
4	Isopropanol	28.25	23.76	25.56	25.86±2.26

According to the result obtained a maximum yield of 74.55% was achieved when n-butanol solvent was applied followed by a yield of 25.86% was obtained when isopropanol solvent was used. On the other hand a minimum oil yield of 5.72% was obtained with ethanol solvent was implemented. In contrary when hexane solvent was used no phase separation is seen, this is because the extraction process was conducted at room temperature due to the fact that solvent is miscible to base oil and slightly miscible with impurities. Thus applying hexane as a solvent at this temperature is not appropriate may be at high temperature. However, an increase temperature decreases capability to segregate sludge from waste oils and also increase the mutual solubility of solvent and non-polar and polar macromolecules from additive package [37] and also increases the rate of solvent vaporization. When hexane is mixed with used oils, the liquid hydrocarbons at room temperature keep stable the solution of macromolecular and other additives as well as the dispersion of carbonaceous and other particles. No destabilization is observed after centrifugation time or many days of gravity settling, no sludge settles at the bottom of test tubes [13].

From results obtained it can be said that as the number carbon atom increases in alcohol the rate of extraction increases and hexane which is non-polar organic solvent cannot be used as flocculation and extraction solvent in the case of refining used lubricating oil.

Oil yield of 83.5% was reported V. katiyar and S. Husain (2010) using 1-butanol solvent at a temperature of 20^0C, 3:1 solvent to oil ratio and 24 hour extraction time. A. Amer and et al. (2014) reported 79.2% oil yield using butanol solvent at 3:1 solvent to oil ratio, 25^0C and 24 hour extraction. Additionally oil yield of 29.5% was reported by A. Amer and et el. using solvent type propanol, solvent to oil ratio 3:1, 25^0C and 24 hour extraction temperate and time respectively.

4.2.5 Optimization of extraction conditions for maximum oil yield using n-butanol solvent

The experimental design selected for this study was full factorial design. The considered parameters during the study were solvent to oil ration and extraction time. The complete design matrixes with values obtained are shown in table 4.12 below.

Table 4.12 Experimental values of oil yield obtained

Run	n-butanol to oil ratio	Extraction time (hr)	Experimental oil yield (%)		
			Replicate 1	Replicate 2	Mean
1	1:1	96	25.2	24.9	25.1
2	4:1	96	85.6	86.4	86.0
3	2:1	24	39.5	41.1	40.3
4	4:1	24	81.3	80.1	80.7
5	1:1	24	21.3	20.7	21.0
6	3:1	24	68.1	67.8	68.0
7	4:1	48	83.5	84.7	84.1
8	1:1	72	24.5	24.9	24.7
9	2:1	96	50.1	49.7	49.9
10	3:1	72	73.9	73.1	73.5
11	3:1	48	73.2	72.9	73.1
12	4:1	72	84.4	85.1	84.8
13	2:1	72	47.7	46.5	47.1

14	3:1	96	74.7	73.9	74.3
15	1:1	48	24.0	25.0	24.5
16	2:1	48	46.8	45.3	46.1

From table 4.12, the maximum yield is 86.0% at experiment no 2 while the minimum yield is 21.0 at experiment no 5. Therefore, it can conclude that maximum amount of oil yield was obtained at 4:1 solvent to oil ratio and 96 hour extraction time.

Previously conducted study by V. Katiyar and S. Husain, 2010 showed that oil yield of 84% was obtained using n-butanol solvent at 4:1 solvent to oil ratio and 24 hour and 25^0C extraction time and temperature respectively.

4.2.6 Development of regression model equation

The model equation that correlates the response (oil yield) to extraction process variables in terms of actual vales after excluding the insignificant terms is given below. The analysis of variance (ANOVA) obtained from design expert software 7.0.0 which tells the significance of parameters is shown in table 4.8 below

Table 4.13 Analysis of variance (ANOVA)

Source	Sum of squares	Degree of freedom	Mean square	F value	P value Prob > F	Remark
Model	3734.15	5	746.83	131.30	<0.0001	Significant
A-Solvent to oil ratio	3191.54	1	3191.54	561.11	<0.0001	*
B-Extraction time	33.67	1	33.67	5.65	0.0452	*
A^2	491.78	1	491.78	93.83	<0.0001	*
AB	0.90	1	0.9	0.13	0.7023	**
B^2	5.91	1	5.91	0.87	0.3421	**
Residual	39.82	7	5.69			
Lack of fit	39.82	3	13.27			

Pure error	0	4	0			
C or Total	3773.97	12				

The model F- value of 131.30 implies the model is significant. Value of “prob>F” less than 0.05 indicates the terms are significant. In this case A-Solvent to oil ratio, B-extraction time and A^2-square of solvent to oil ratio are significant model terms. Values greater than 0.1000 indicates the model terms are not significant. Hence AB-interaction between solvent to oil ratio and time and B^2-the square of time are not significant model terms.

From Design expert software model statistics summary, quadratic model was suggested as shown in the table 4.9 below. The quality of the model developed could be evaluated from their coefficients of correlation. The value of R-squared for the developed correlation is 0.9855. It implies that 98.95% of the total variation in the oil yield is attributed to the experimental variables studied.

Table 4.14 Model statistics summary

Source	Standard deviation	R-squared	Adjusted R-squared	Predicated R-squared	Press	Remark
Linear	7.41	0.8546	0.8255	0.6542	1305.01	
2F1	7.80	0.8548	0.8064	0.6366	1371.30	
Quadratic	2.38	0.9895	0.9819	**0.9015**	**371.86**	**Suggested**
Cubic	1.41	0.9974	0.9937	0.6934	1157.21	

The final model equation in terms of actual factor is presented by equations 4.1 for representing the variation of percentage oil yield with independent factors:

$$Oil\ yield = -21.97315 + 43.23981 * Solvent\ to\ oil\ ratio + 0.069792 * Extraction\ time - 4.48858 * Solvent\ to\ oil\ ratio^2 \quad \ldots\ldots\ldots\ldots (4.1)$$

4.2.7 Experimental oil yield versus predicated oil yield by model equation

The table 4.15 below shows the relation between the actual value of the experiment and value predicated by the model equation developed by the design expert software 7.0.0

Table 4.15 Difference between the experimental (actual) value and predicated value

Ran	Actual oil yield (%)	Predicated oil yield (%)	Residual
1	25.1	23.5	1.6
2	86.0	85.9	0.1
3	40.3	48.2	-7.9
4	80.7	80.8	-0.1
5	21.0	18.5	2.5
6	68.0	69.0	-1.0
7	84.1	82.5	1.6
8	24.7	21.8	2.9
9	49.9	53.3	-3.4
10	73.5	72.4	1.1
11	73.1	70.7	2.4
12	84.8	84.2	0.6
13	47.1	51.6	-4.5
14	74.3	74.0	0.3
15	24.5	20.1	4.4
16	46.1	49.9	-3.8

From model statistics summery the value of correlation coefficient was found 98.95%. This high value of correlation coefficient indicated the significance of the model.

4.3 Effect of process parameters on percentage oil yield

4.3.1 Effect of solvent to oil ratio on percentage oil yield

The effect of solvent to oil ratio on oil yield for 24 hour, 48 hour, 72 hour and 96 hour extraction time is shown in the figure (4.1) below.

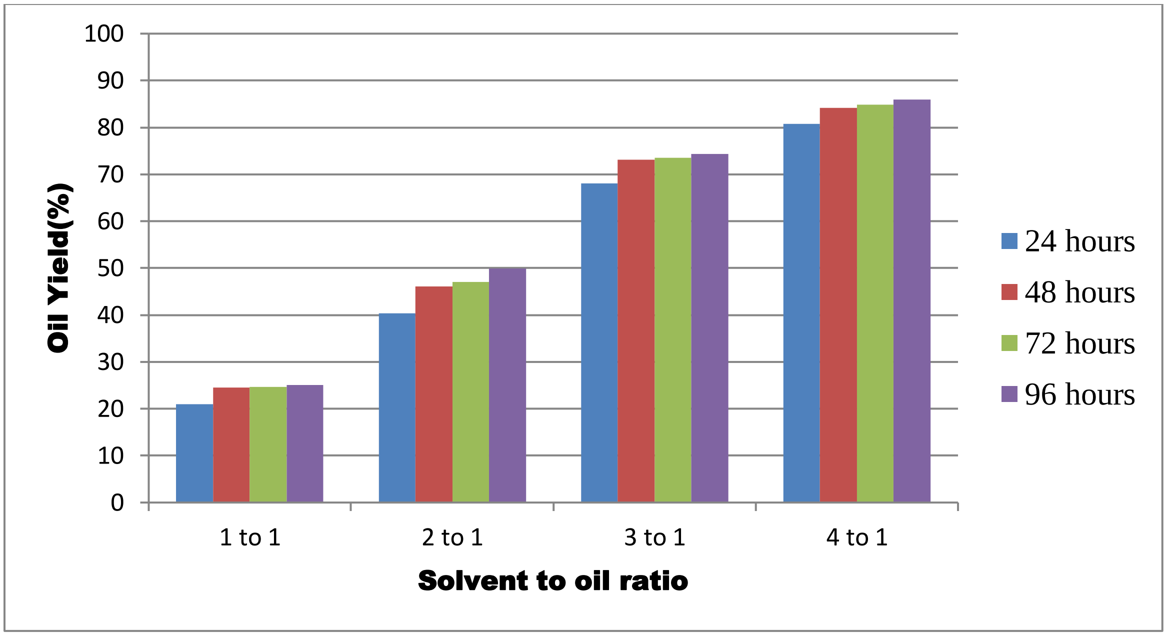


Figure 4.9 Effect of solvent to oil ratio on oil yield for four different extraction times

Solvent to oil ratio plays a great role on oil yield (figure4.1). A sharp increase of percentage oil yield was observed as solvent to oil ratio increases. For 24hrs, 48hrs, 72hrs and 96hrs extraction time as solvent to oil ratio increases from 1:1 to 4:1 the oil yield increase from 21% to 80.7%, 24.5% to 84.1, 24.7% to 84.8% and 25.1% to 86% respectively i.e the yield of larger solvent to oil ratio is 59.7% for 24 hrs extraction, 59.6% for 48 hrs extraction, 60.1% for 72 hrs extraction and 60.9% for 96 hrs extraction higher than smaller solvent to oil ratio. As it can be seen, the oil recovery was much higher with increasing solvent to oil ratio this is because at lower solvent to oil ratio the base oil might be saturated in the extraction phase leading to low oil recovery and greater oil losses [33]. Increasing solvent to oil ratio improves the mutual solubility of the oil in the solvent resulting in decreasing the oil lose in the sludge and increasing the oil recovery values [38]. However, increasing a solvent to oil ratio above a certain limit will not be economically feasible. Previously conducted studies show that solvent to oil ratio has a great effect on the extraction yield, specifically M. Hussein and et al. 2014 reported a result which is in agreement to the result obtained in this study. Additionally Doaa I. Osman and et al 2017 reported a similar result.

4.3.2 Effect of extraction time on percentage oil yield

The effect of extraction time on oil yield for solvent to oil ratio 1:1, 2:1, 3:1 and 4:1 is show in the figure (4.2) below.

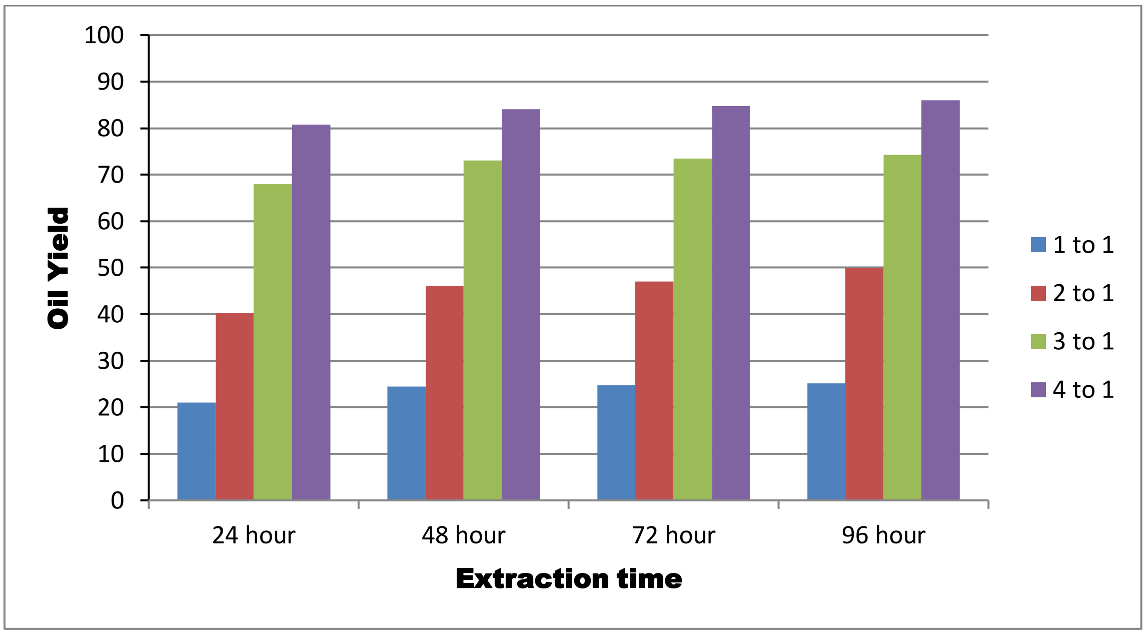


Figure 4.10 Effect of extraction time on oil yield for four different solvent to oil ratio

The percentage oil yield was directly related to extraction time i.e the yield increased as extraction time increased. However, increasing the time above some level may be wastage of time and cost. Therefore, the optimum contact time between the solvent and the oil was investigated in this study. According to the result obtained at 24 hour extraction time and 4:1 solvent to oil ratio the oil yield was 80.7%. The yield increased by 3.4% as the extraction time increased from 24 hour to 48 hour and it increased by only 0.7% and 1.2% as the time increased from 48 hour to 72 hour and from 72 hour to 96 hour respectively. No significant change in percent oil yield was shown with increasing the extraction time above 48 hour. Thus, the extraction process at a time higher than 48 hours does not lead to a higher yield of oil recovery i.e 98% of the maximum yield is attained at 48 hour extraction. Therefore, the optimum extraction time can be considered at 48 hour and extraction above this time may be wastage of time and cost.

4.4 Characterization of product (treated oil)

Using the process parameters that gave a maximum oil yield i.e. solvent type butanol, solvent to oil ratio 4:1 and extraction time 96 hours, oil was extracted and properties of oil were determined. The results obtained are summarized in the table (4.3) below.

Table 4.16 Properties of treated oil

No.	Oil properties	Treated oil	Untreated oil
1	Specific gravity	0.868	0.926
2	Water content (%)	0	2.8
3	Ash content (wt%)	0.08	1.65
4	Total acid number (mgKOH/g of oil)	0.54	4.34
5	Kinematic viscosity (cSt)	@40^{0}C = 74.9	@40^{0}C = 113.4
		@100^{0}C = 9.09	@100^{0}C = 10.4
6	Viscosity index	94.8	
7	Flash point (^{0}C)	230	208
8	Poor point (^{0}C)	-27.3	-24.3
9	Color	Yellow	Black
10	Metal content (ppm)		
	Ca	50.7	224.1
	Fe	45.0	703.3
	Pb	39.2	373.5
	Zn	18.8	53.5

Table 4.16 shows physical properties obtained for recycled oil. These properties are used to evaluate quality characteristics of recycled oil. The result obtained showed specific gravity of 0.868 which is lower than specific gravity of used oil due to the removal of contaminants during

recycling process. It was found that the percentage of ash in refined oil was 0.08% and it can be observed that the property of ash content for the extracted oil was improved. The ash reduction is a vital parameter in the extraction process since it accounts for the reduction of the concentration of the contaminants in the oil such as dirt, iron oxide, wear metals and corrosion products as well as the concentration of ash producing additives [38]. The total acid number is the measurement of acidity in oils used as lubricants. It is one of the crucial chemical properties that give stability to lubricating oils. Lubricating oil is said to be stable if it can resist oxidation that yields acids, lacquers and sludge. Normally, acidity increases with the oxidation of lubricating oils [15]. Total acid number of 0.54mgKOH/g of oil was obtained for recycled oil sample. This value indicates the acid concentration in recycled oil is decreased and thus, removes the cause of corrosion in engine. The kinematic viscosities of oil at 40^0C and 100^0C are 74.9cSt and 9.09cSt respectively which is lower than the result obtained for used lubricating oil which was 113.4cSt and 10.4 respectively. This shows that used oil has high viscosity due to oxidation and contamination beside that during the application period of lubricating oil the metallic scrapping act as catalysis at high combustion temperature and oxygen vicinity, produce an asphalt like sludge which increases the viscosity of used lubricating oil [43] and therefore the recycling process effectively removes the contaminants. The flash point value obtained for recycled oil is 230^0Cwhich is higher when we compared to the value obtained for used oil which is 208^0C. This shows that the recycling process tremendously removes light fuels during vacuum distillation. The result obtained showed also a pour point of -27.3^0C which is lower than the value obtained for used oil -24.3^0C. This increase in pour point of used lubricating oil is because of degradations of additives which were present in fresh oil as pour point depressants. Lubricating oils with low pour points show good quality. The refining process reduces the calcium content of used lubricating oil from 224.1ppm to 50.7ppm about 77% and iron content from 703.3ppm to 45ppm about 94% and lead content from 373.5ppm to 39.2ppm about 89% and zinc content from 53.5ppm to 18.8ppm about 65%. The metal content of base oil is a very important parameter as the metal content in an oil sample as it can increase the rate of corrosion of metal parts in contact with. Metal content of oil comes from additives added to oil. Calcium comes mainly from detergent and depressants zinc introduced to base oil in the form of additive packages as antioxidant and corrosion inhibiter [33]. Lead and iron are introduced since the block of any engine is mostly made from iron and lead the wearing of this parts leads to introduce these metals [19].

4.5 Comparisons of recycled lubricating oil with virgin and base lubricating oil

Comparisons of oil properties against both virgin lubricating oil and base lubricating oil specification are given in table 4.7 below.

Table 4.16 Comparisons of recycled lubricating oil with virgin and base lubricating oil

Oil properties	Measured value	Virgin lubricating oil (SAE 15W 40)	Base lubricating oil (SAE 30)
Specific gravity	0.868	0.870	0.87
Water content (wt%)	0	Nil	0
Ash content (wt%)	0.08	-	-
Total acid number	0.54		max 0.02
Kinematic viscosity at 40^0C	74.9	112.6	min 82.9
Kinematic viscosity at 100^0C	9.09	14.6	min. 9.5
Viscosity index	94.8	136.4	min. 90
Flash point (^{0}C)	230	230	min 215
Pour point (^{0}C)	-27.3	max. -18	max. -6
Metal content			

The properties of recycled oil were compared to both virgin lubricating oil SAE 15W 40 and base lubricating oil SAE 30 specified by society of automotive engineers. The specific gravity, water content, flash point, and pour point were within the specifications to virgin lubricating oil (SAE 15W 40). However, kinematic viscosity at 40^0C, kinematic viscosity at 100^0C, and viscosity index were lower than the specifications for virgin lubricating oil. On the other hand the specific gravity, water content, viscosity index, flash point and pour point were within the specifications to base lubricating oil specified by society of automotive engineers (SAE 30). And also slight deviations were seen to the kinematic viscosity at 40^0c and 100^0C to base lubricating oil. According to the result obtained the viscosity index which is a very crucial factor for lubricating oil is within the specifications for base lubricating oil (SAE 30). And the same is also true for the case of specific gravity, water content, flash point and pour point. Beside this, according to API base oil classification base oil having viscosity index greater than 80 and less than 120 is grade I base oil that can be used for producing motor oil, industrial oil hydraulic oil and lubricating grease. Therefore, from this it can be said that the recycled oil can be used as base oil which is the fundamental element in manufacturing of different types of lubricating oil that makes up 75% - 90% of the finished product.

Chapter five

Preliminary feasibility study

5.1 Market study and plant capacity determination

The annual production capacity of the plant is fixed based on the market study performed on number of vehicles available in the country obtained from transport authority of the country.

Table 5.1 Number of vehicles in Ethiopia at different years

Year	Number of vehicle
2015	587,454
2017	831,000
2019	1,071,160
2020	1,200,160

Source: Ethiopian transport authority

Based on the above data forecasting the number of vehicle for the next five years starting from 2023 using linear regression yields

Table 5.2 Forecasted number of vehicles in the country

Year	Number of vehicle
2023	1,655,013
2024	1,777,101
2025	1,900,000
2026	2,021,280
2027	2,144,000

To calculate the amount of oil that will be taken by the cars, let as assume the following

- ✓ Since larger vehicles takes an average of 20 liters of oil per one batch and smaller vehicles (motor bicycle) 2 liters, taking the average value 11 liters.
- ✓ It is recommended to change the motor oil for every 5,000km travel, based on this let as assume that every car will change motor oil every three month.

Therefore,

$$\text{Total amount of lubricating oil reqired at } 2023 = 1{,}655{,}013\text{veh} * 11\frac{\text{lit}}{\text{veh}} * 4$$

= 72,821,364 lit

Similarly

Table 5.3 Total Lubricating oil required for the next five years

Year	Quantity of lubricating oil (liter)
2024	78,192,444
2025	83,600,000
2026	88,936,320
2027	94,336,000

The plant production capacity can be fixed by taking some percentage from the market demand. Thus, taking 2% of the market demand as a market share the production capacity of the plant will be

Table 5.4 production capacity of the plant

Year	Quantity of lubricating oil to be produced (liter)
2023	1,456,427
2024	1,563,849
2025	1,672,000
2026	1,778,726
2027	1,886,720

$$Increasing\ rate = \frac{Final\ value - Initial\ value}{Initial\ value * Number\ of\ years}$$

= 5.9%

From the above data the maximum production capacity of the plant is 1,886,720 liter thus, the design capacity be taken as 1,900,000 liter and at the beginning the production capacity is 1,456,427liter with yearly increment of 5.9%.

5.2 Process technology and description

The main way for used lubricating oil recycling is the so-called re-refining, which consists of recovering the original base oil to be reused in the formulation of new products. Among different recycling (refining) technologies solvent extraction method followed by clay treatment is selected for this study. The process flow diagram for this technology is given in the figure below.

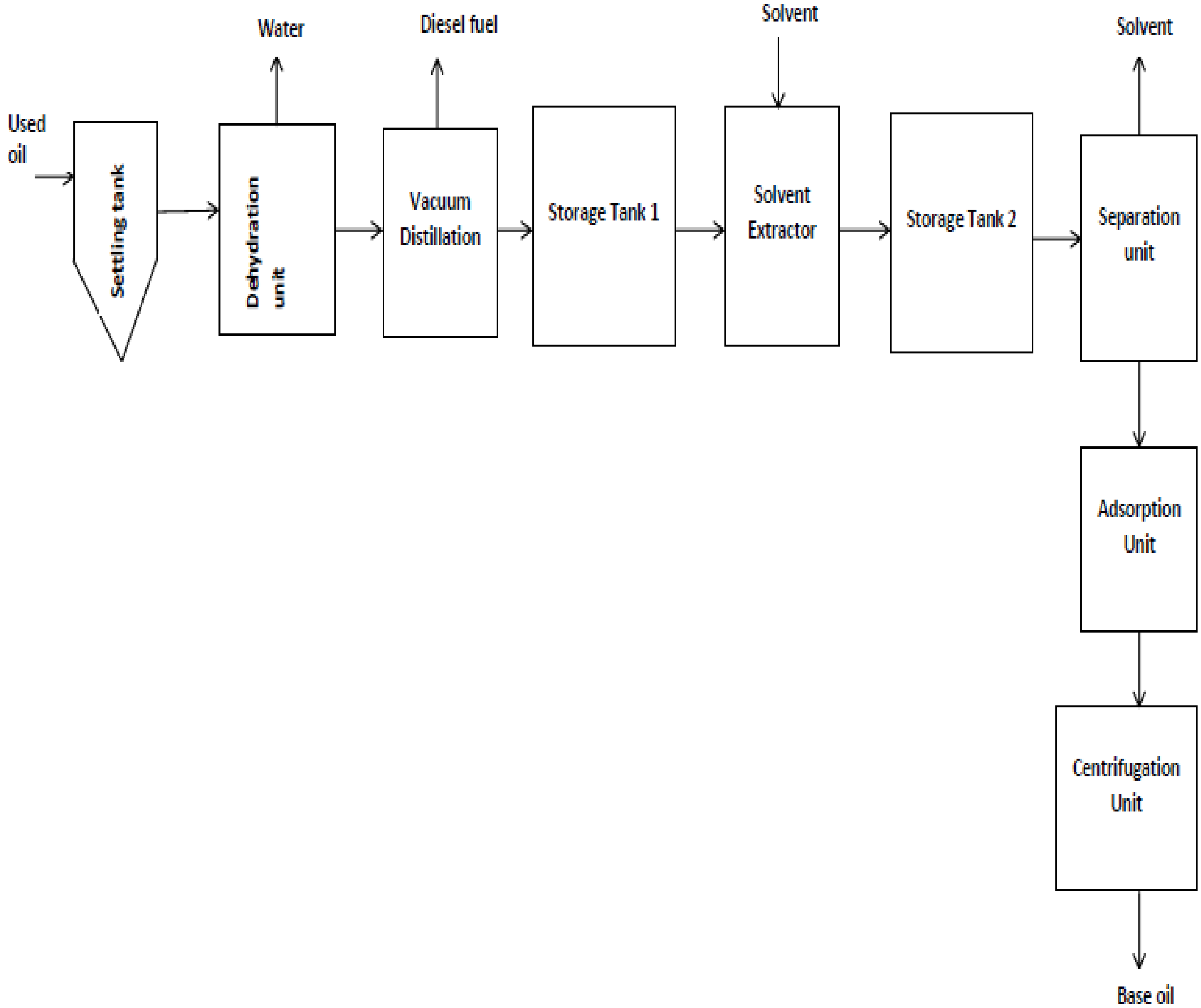


Figure 4.11 Process flow diagrams recycling of used lubricating oil

The main processes of this technology are settling, dehydration, diesel stripping, extraction and separation and clay treatment (adsorption). Under settling, used lubricating oil is stored to allow large suspended particles to settle under gravity followed by dehydration to remove the water in used oil. The oil is heated in a heater at temperature of 120^0C – 140^0C. The dehydrated used oil is then feed to vacuum distillation for light fuel removal at a temperature of 240^0C. The lubricating oil fraction obtained from vacuum distillation will be mixed by agitation with the solvent in appropriate ratio (4:1). The tank will allowed for flocculation and extraction for 48hr. the aromatic and degraded additives present in lubricating oil fraction are settle at the bottom and oil fraction and solvent mixture layer forms at the top. The separation of lubricating oil from solvent will take place under atmospheric pressure with simple batch distillation. Finally clay treatment will take place to improve the color of lubricating oil.

5.3 Material balance

The material balance in plant design is necessary because this fixes the relative flow rates of different flow streams in the flow sheet.

Overall material balance

Basis: 1 hour operation

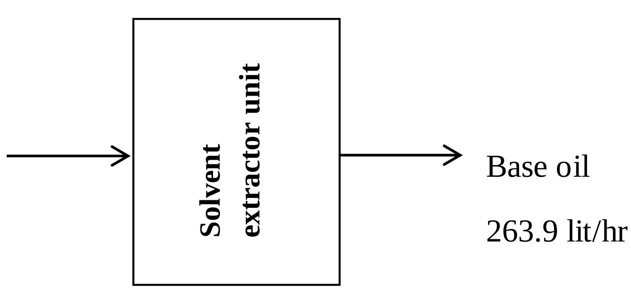
From market study plant annual production capacity is 1,900,000 lit/year

Assuming 300 working days per year

$$Base\ oil\ produced\ per\ year = 1{,}900{,}000 \frac{lit}{year} * \frac{1\ year}{300\ days} * \frac{1\ days}{24\ hr}$$

= 263.9 lit/hr

Balance on solvent extractor unit

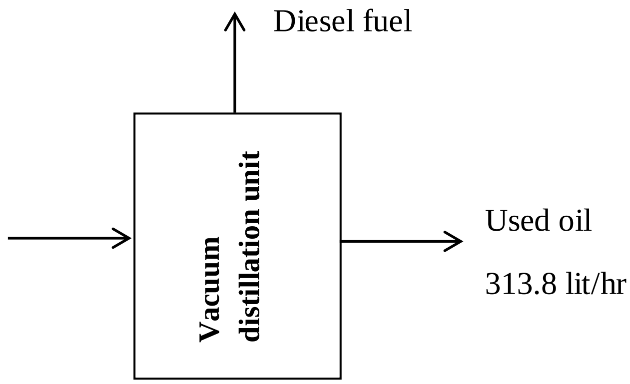


From the experimental work percentage recovery of base oil is 84.1%, therefore, amount of used oil feed to solvent extractor unit will be

$$Used\ oil\ feed\ to\ solvent\ extractor\ unit = \frac{263.9\frac{lit}{hr}}{0.841}$$

= 313.8 lit/hr

Balance on vacuum distillation unit

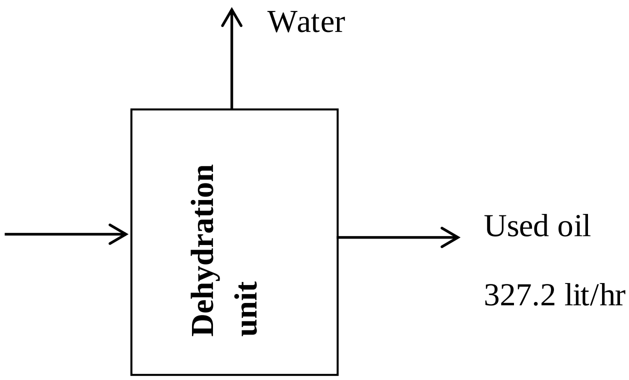


From experimental work average percentage recovery of diesel fuel is 4.1% therefore, used oil feed to vacuum distillation unit will be

$$Used\ oil\ feed\ to\ vacuum\ distilation\ unit = \frac{313.8\frac{lit}{hr}}{0.959}$$

= 327.2 lit/hr

Balance on dehydration unit



From experimental work water content of used oil is 2.8% therefore, amount of used lubrication oil feed to dehydration unit

$$Used\ oil\ requred\ for\ the\ process = \frac{327.2\frac{lit}{hr}}{0.972}$$

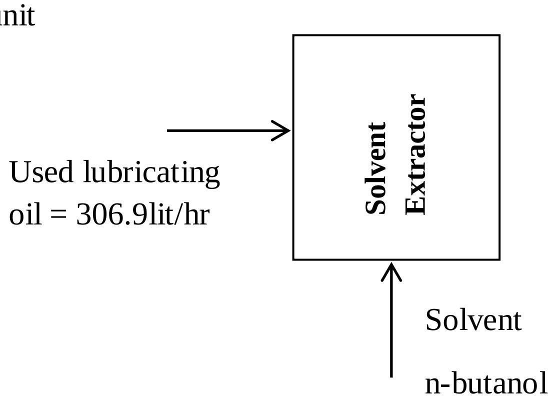
= 336.6 lit/hr

Yearly consumption of used will be then

$$= 336.6\frac{lit}{hr} * 24\frac{hr}{day} * 300\frac{day}{year}$$

= 2,423,520 lit/year

To determine amount of solvent (n-butanol) required let as perfume a balance on solvent extractor unit

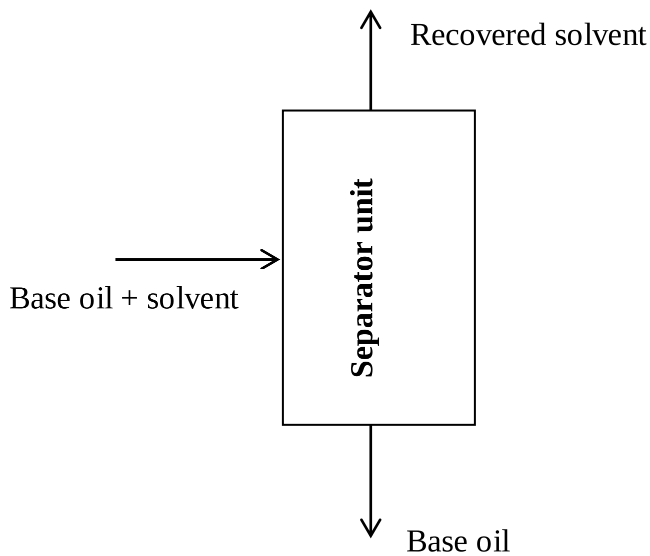


From experimental work taking 4:1 solvent to oil ratio as optimum value amount of n-butanol solvent required will be

$$Amount\ of\ n - butanol\ required = 4 * \frac{313.8 lit}{hr}$$

= 1,255.2 lit/hr

Balance on separator unit



From experimental work 99% solvent recovery was obtained therefore, amount of solvent recovered will be

$$Recovered\ solvent = 0.99 * amount\ of\ solvent\ feed\ to\ extractor\ unit$$

= 0.99*1,255.2 lit/hr

= 1,242.6 lit/hr

5.4 Equipment sizing and selection

From experimental work the optimum extraction time is 48 hours (2 days). The sizing of the equipment's will be based on this. Therefore, in storage tank 1 we should to store treated used lubricating oil (used lubricating oil ready for solvent extraction) required for 2 days that will make the process continuous after one batch operation.

Storage tank 1

$$Volume_{storage\ tank\ 1} = Volume\ of\ used\ lubricating\ oil\ required$$

$$= 313.8\frac{lit}{hr} * 48hr = 15{,}062.4lit = 15.1m^3$$

Taking 5% safety factor (assuming the tank is 95% full)

$$V_{tank\ 1} = \frac{15.9}{0.95} = 15.9m^3$$

- Select 16m^3 stainless steel storage tank

Settling tank

Assuming four (4) batches per day, this implies there will be eight (8) batches per 48 hour

$$Volume\ of\ settling\ tank = \frac{336.6\frac{lit}{hr} * 48hr}{8}$$

$$= 2{,}019.6 \text{ lit} = 2.02\text{m}^3$$

- Select 2m^3 stainless steel settling tank

Similar size (2m^3) of dehydration unit and vacuum distillation unit will be used

Solvent extractor unit

$$Volume\ of\ tank = Volume_{Used\ lub\ oil} + Volume_{solvent}$$

$$= 313.8 \text{ lit/hr*48hr} + 4(313.8 \text{ lit/hr*48hr})$$

$$= 75{,}312 \text{ lit} = 75.3\text{m}^3$$

- Select 76m^3 solvent extractor unit (a tank made up of stainless steel having agitation unit)

Storage tank 2

Since the percentage yield of base oi is 84.2%

Volume of storage tank 2 = 0.842*313.8lit/hr*48hr + 4(313.8lit/hr*48hr)

= 72,932.5lit = 72.9m^3

- Select 73m^3 stainless steel storage tank

Separation unit

Since we have eight (8) batches per 48 hour operation the size of separation unit will be one eighth of storage tank 2

$$Volume\ separation\ unit = \frac{73m^3}{8}$$

= 9.1m^3

- Select 10m^3 batch distillation

Adsorption unit

Assuming 100% solvent recovery in separation unit and 8 batches per 48hr, volume of adsorption unit will be

$$\text{Volume of adsorption tank} = \frac{0.842 * \frac{313.8lit}{hr} * 48hr}{8}$$

= 1,585.3lit = 1.6m^3

- Select 1.6m^3 adsorption unit (mixer)

Centrifuge

The adsorption process is done by taking 15wt% clay, volume of centrifuge will be

$$Volume\ of\ centrifuge = \frac{0.842 * \frac{313.8lit}{hr} * 48hr}{8} + 0.15 * \frac{0.842 * \frac{313.8lit}{hr} * 48hr}{8}$$

= 1,823.1lit = 1.82m^3

Since the centrifuge is cylindrical shape, the volume of cylinder will be

$$V = \pi r^2 h$$

Taking h = 2m

$$r = \sqrt{\frac{2m^3}{\pi * 2m}}$$

$$r = \sqrt{\frac{2 * 1.82m^3}{\pi * 2m}}$$

r = 0.56m

- Select a centrifuge having a diameter 1.12m (44in)

5.5 Cost estimation

5.5.1 Human resource cost estimation

Table 5.5 estimated human resource cost

Job title	Number of employees	Estimated salary(birr)	Total salary(birr)
General Manager	1	25,000	25,000
Production Manager	1	20,000	20,000
Technical Manager	1	20,000	20,000
Supervisor	1	12,000	12,000
Quality control expert	2	8000	16,000
Electrician	3	5,000	15,000
Mechanic	3	5,000	15,000
Operator in each unit	8	5,000	40,000
Labor workers	10	3,000	30,000
Accountant	4	5,000	20,000
Sells man	5	5,000	25,000
Administrator	1	7,000	7,000
Sore keeper	2	3,000	6,000
Secretory	1	3,000	3,000
Cleaner	3	2,500	7,500
Guard	2	2,500	5,000
Total	48		266,500

5.5.2 Equipment Costing

Table 5.6 estimated equipment cost

Equipment name	Amount needed	Unit price (in USD)	Total price (in USD)	Remark
Storage tank 1	1	31,500	31,500	Stainless steel construction
Settling tank	1	15,200	15,200	Stainless steel construction
Dehydration unit	1	15,200	15,200	Material of construction stainless steel
Industrial heater (Electric heater)	1	3,000	3,000	Working temperature 20^0C-500^0C Thermal efficiency 95%
Vacuum distillation unit	1	86,000	86,000	Complete vacuum distillation setup
Solvent extractor unit (Mixer)	1	162,400	162,400	Carbon steel
Storage Tank 2	1	49,000	49,000	Stainless steel
Separation unit (batch type distillation)	1	64,400	64,400	Complete distillation setup with reboiler, column, main condenser
Adsorption unit (mixer)	1	23,700	23,700	Carbon steel construction`
Centrifuge	1	26,200	26,200	Carbon steel construction
Centrifugal Pump	4	4,900	19,600	Discharge diameter 2in, cast iron

				construction
Gear pump	4	5,200	20,800	Flow rate 2 gallon per minute, cast iron construction
Total	18	486,700$	517,000$	

5.5.3 Components of fixed capital investment

Table 5.7 Components of fixed capital investment

Components	Cost (in USD)	Assumed % of total
Purchased equipment	517,000	25
Purchased equipment (installed)	186,120	9
Instrumentation (installed)	144,760	7
Piping (installed)	165,440	8
Electrical (installed)	103,400	5
Buildings	103,400	5
Yard improvement	41,360	2
Service facilities (installed)	310,200	15
Land	20,680	1
Engineering and supervision	206,800	10
Construction expense	248,160	12
Contractors fee	41,360	2
Contingency	165,440	8
Total	2,254,120	100

From the above table total cost **2,254,120$ (117,214,240 birr)** gives fixed capital investment

Total capital investment = Fixed capital investment + working capital

Usually working capital is 15% of total capital investment

Total capital investment = 117,214,240 birr + 0.15*Total capital investment

$$Total\ capital\ investment = \frac{117{,}214{,}240\ birr}{0.85}$$

Total capital investment = 137,899,106 birr

5.5.4 Operational cost

Raw material cost

n-butanol solvent – 56 Rupee/lit = 37 birr/lit

Bentonite clay – 1940 Rupee/ton = 1.30 birr/kg

Used lubricating oil – 3 birr/lit

Annual requirement n-butanol solvent

1,255.2lit/hr*48hr/batch*150batch/year = 9,037,440lit/year

Annual requirement of used lubricating oil

336.6lit/hr*48hr/batch*150batch/year = 2,423,520lit/year

Annual requirement of bentonite clay

0.15*0.842*313.8lit/hr*48hr/batch*150batch/year = 285,357kg/year

Thus annual cost of raw materials is

9,037,440lit/year*37birr/lit + 2,423,520lit/year*3birr/lit + 285,357kg/year*1.3birr/kg = **342,026,804birr**

Monthly labor cost is 266,500birr so annual labor cost will be **3,198,000birr**

Utility = 70USD/ton = 70 *(1,900,000lit*0.868/1000kg/ton) = 115,444USD = **6,003,088birr**

Maintenance = 6%*FCI = 0.06*117,214,240 birr = **7,032,854.4birr**

Property tax and insurance = 3%*maintenance = 0.03*7,032,854.4 = **210,985.6birr**

Since the current price of base lubricating oil is 3700USD/metric ton = 3.7USD/lit = 193birr/litter

Taking the selling price of the product 193 birr/litter, annual sales = 193birr/lit * 1,900,000litter = 366,700,000 birr

Selling cost = 1%*sales = 0.01*366,700,000 birr = **3,667,000 birr**

Administrative cost = 0.5%*sales = 0.005*366,700,000 = **1, 833,500 birr**

Total product cost is the sum of bold numbers = 363,972,232 birr

5.6 Profitability evaluation

Annual gross profit = Total sells – total product cost

Total sells = 366,700,000birr

Total product cost = 363,972,232 birr

Annual gross profit = 366,700,000 birr – 363,972,232birr

Annual gross profit = 2,727,768 birr

Net profit = Annual gross profit – annual income tax

Annual income tax = 35% of gross profit = 0.35*2,727,768 = 954,719birr

Net profit = 2,727,768 birr – 954,719 birr = 1,773,049birr

$$Rate\ of\ raturn\ on\ investment\ (ROR) = \frac{Net\ profit}{Tota\ capital\ investment} * 100\%$$

$$= \frac{1,773,049 birr}{137,899,106 birr} * 100\% \quad = 1.3\%$$

$$Payback\ period\ (PBP) = \frac{Depreciable\ fixed\ capital\ investment}{Net\ profit + Annual\ depreciation}$$

Depreciation = 10%FCI

= 0.1*117,214,240 birr = 11,721,424 birr

$$Payback\ period = \frac{117{,}214{,}240 birr}{1{,}773{,}049 birr + 11{,}721{,}424 birr}$$

= 8.7 years

The preliminary feasibility study shows that at 4:1 solvent oil ratio the plant is not feasible since the rate of return and payback period are 1.3% and 8.7 years respectively. This is due to higher solvent to oil ratio which is the key factor that made the production cost higher. According to the preliminary study the solvent cost accounts 92.4% of total production cost thus, the plant to be feasible we should have to reduce the solvent to oil ratio. Reducing the solvent to oil ratio from 4:1 to 3:1 can reduce the total cost of solvent by around 16%. Therefore, the plant feasibility study should be checked with3:1 solvent oil ratio.

Updating the preliminary study for 3:1 solvent to oil ratio

From experimental study the oil yield was 73.1%

$$Used\ oil\ feed\ to\ solvent\ extractor\ unit = \frac{263.9\frac{lit}{hr}}{0.731}$$

= 361 lit/hr

$$Used\ oil\ feed\ to\ vacuum\ distilation\ unit = \frac{361\frac{lit}{hr}}{0.959}$$

= 376.4lit/hr

$$Used\ oil\ requred\ for\ the\ process = \frac{376.4\frac{lit}{hr}}{0.972}$$

= 387.2lit/hr

Yearly consumption of used will be then

$$= 387.2\frac{lit}{hr} * 24\frac{hr}{day} * 300\frac{day}{year}$$

= 2,787,840 lit/year

$$Amount\ of\ n - butanol\ required = 3 * 361 \frac{lit}{hr}$$

= 1083 lit/hr

Operational Cost

Raw material cost

n-butanol solvent – 56 Rupee/lit = 37 birr/lit

Bentonite clay – 1940 Rupee/ton = 1.30 birr/kg

Used lubricating oil – 3 birr/lit

Annual requirement n-butanol solvent

1,083lit/hr*48hr/batch*150batch/year = 7,797,600lit/year

Annual requirement of used lubricating oil

387.2lit/hr*48hr/batch*150batch/year = 2,787,840lit/year

Annual requirement of bentonite clay

0.15*0.731*361lit/hr*48hr/batch*150batch/year = 285,002kg/year

Thus annual cost of raw materials is

7,797,600lit/year*37birr/lit + 2,787,840lit/year*3birr/lit + 285,002kg/year*1.3birr/kg = **297,245,223birr**

Monthly labor cost is 266,500birr so annual labor cost will be **3,198,000birr**

Utility = 70USD/ton = 70 *(1,900,000lit*0.868/1000kg/ton) = 115,444USD = **6,003,088birr**

Maintenance = 6%*FCI = 0.06*117,214,240 birr = **7,032,854.4birr**

Property tax and insurance = 3%*maintenance = 0.03*7,032,854.4 = **210,985.6birr**

Since the current price of base lubricating oil is 3700USD/metric ton = 3.7USD/lit = 193birr/litter

Taking the selling price of the product 193 birr/litter, annual sales = 193birr/lit * 1,900,000litter = 366,700,000 birr

Selling cost = 1%*sales = 0.01*366,700,000 birr = **3,667,000 birr**

Administrative cost = 0.5%*sales = 0.005*366,700,000 = **1, 833,500 birr**

Total product cost is the sum of bold numbers = 319,190,651 birr

Assuming the size of unit operations are similar

Profitability evaluation

Annual gross profit = Total sells – total product cost

Total sells = 366,700,000birr

Total product cost = 319,190,651 birr

Annual gross profit = 366,700,000 birr – 319,190,651 birr

Annual gross profit = 47,509,349 birr

Net profit = Annual gross profit – annual income tax

Annual income tax = 35% of gross profit = 0.35*47,509,349 = 16,628,272.2birr

Net profit = 47,509,349 birr – 16,628,272.2 birr = 30,881,077birr

$$Rate\ of\ raturn\ on\ investment\ (ROR) = \frac{Net\ profit}{Tota\ capital\ investment} * 100\%$$

$$= \frac{30{,}881{,}077 birr}{137{,}899{,}106 birr} * 100\% \quad = 22.4\%$$

$$Payback\ period\ (PBP) = \frac{Depreciable\ fixed\ capital\ investment}{Net\ profit + Annual\ depreciation}$$

Depreciation = 10%FCI

= 0.1*117,214,240 birr = 11,721,424 birr

$$Payback\ period = \frac{117{,}214{,}240 birr}{30{,}881{,}077 birr + 11{,}721{,}424 birr}$$

= 2.8 years

The preliminary study shows that at 3:1solvent to oil ratio the plant is feasible since the rate of return and payback period are 22.4% and 2.8 years respectively. From the feasibility study it is possible to say that the plant is not economically feasible for solvent to oil ratio more than 3:1. Therefore, the optimum solvent to oil ratio is 3:1.

5.7 Plant site location

The site selection is decided based on the raw material availability, infrastructure, proximity to the market and other related facilities. Hence taking these things in to consideration the site for plant erection could be in Addis Ababa. The major source of raw material (used lubricating oil) for the plant is car service station. According to transport authority of the country 63% of the total cars available in country are found in nation's capital Addis Ababa. This leads to have large number of car service stations in the town that will guarantee continuous supply of raw material for the plant. Therefore, Addis Ababa is the ideal site for the plant.

5.8 Environmental impact

It is obvious that recycling of used lubricating oil is not only a way to convert waste to useable material but also a way to reduce the impact of used lubricating oil on the environment. Solvent extraction method is the most efficient and environmentally friendly when compared with other methods it will generate oil sludge that contains heavy metals, solid particles and other substances which will pollute soil, surface and ground water. Therefore, there must b economical and eco-friendly way to use/dispose this waste.

On way is that the sludge or waste byproduct of refining process of used lubricating oil can be used for asphalt production, pesticide carrier and weed killer [39]. Organic sludge obtained with polar solvents may be incorporated in asphalts or, better, used as a component of offset inks [37].

Thus, it is possible to provide this waste to asphalt production and pesticide production plant to be used as a raw material.

The other way is to use one of the disposal options among different alternatives. During the past years, a variety of oil sludge disposal methods have been developed such as land filling, land farming, incineration, solidification/stabilization and other [40]. Thus the problem can be solved by dumping the sludge at approved land fill areas. Waste disposal to a land fill remains the most common method of waste management because it is simple and relatively the most economical [41].

Chapter Six

Conclusion and Recommendations

6.1 Conclusion

In this study base lubricating oil was extracted from used lubricating oil using four different solvents namely ethanol, n-hexane, n-butanol and isopropanol. With the solvent that gives maximum oil yield, solvent to oil ratios (1:1, 2:1, 3:1 and 4:1) and extraction times (24hr, 48hr, 72hr and 96hr) were the considered parameters for optimization.

From experiment it was found that a maximum oil yield of 74.55% was obtained using n-butanol solvent followed by a yield 25.86% using isopropanol solvent. A minimum oil yield of 5.72% was obtained using ethanol solvent. When hexane solvent was used no phase separation was observed after mixing and 3 days of gravity settling meaning that no sludge settles at the bottom of the beaker. From the result obtained it can say that for alcohols the extraction yield increases with increasing molecular weight of the solvent and hexane which is non-polar organic solvent cannot be used as flocculation and extraction solvent in case of refining used lubricating oil.

On the basis of n-butanol solvent further experimental investigation was performed and it was found that a maximum oil yield of 86% was obtained at 4:1 solvent to oil ratio and 96hr extraction time. A minimum oil yield of 21% was obtained at 1:1 solvent to oil ratio and 24hr extraction time.

From design expert software analysis of variance (ANOVA) p value < 0.05 for solvent to oil ratio and extraction time indicated that the operating parameters have significant effect on oil yield.

Using optimum operating condition the oil was extracted and the properties of the oil were determined. Specific gravity, water content, ash content, kinematic viscosities at 40^0C and 100^0C, flash point, pour point and metal content were determined for quality analysis. The results were compared with virgin lubricating oil and base lubricating oil the results obtained indicated that the properties of recycled oil were comparable with that of base lubricating oil.

Therefore, the recycled oil can be used as base oil which is the fundamental element in manufacturing of different types of lubricating oil.

Finally, to check the feasibility of the process, rate of return on investment and payback period of the recycling process where evaluated. Based on the economic study the process was found to be feasible at 3:1 solvent to oil ratio with rate of return on investment equal to 22.4% and payback period of 2.8 years. According to the study also the process is not economically feasible for solvent to oil ratio more than 3:1.

6.2 Recommendations

Even if used lubricating oil has been recycled worldwide and used as a base oil in formulation of different types of lubricating oil, in our country the oil is not used in a way that would prevent environmental pollution moreover it becomes a major source of pollution. Beside that due to the lake of base oil there is no plant that produces lubricating oil. Therefore, we recommend the establishment of lubricating oil manufacturing plant that uses the recycled oil as base oil.

A certain properties of recycled lubricating oil deviate from virgin lubricating oil therefore, we recommend further study to be done blending ratio to blend recycle oil with virgin lubricating oil in order to enhance these properties and to be used in lubrication system.

Moreover, we recommend to test solvents outside of the solvents tested in this study like ketone solvents specifically methyl ethyl ketone.

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Annexes

Annex A: SAE 15W 40 virgin lubricating oil specifications

Technical Data Sheet (TDS)

PUMA Lubricants

Typical Physical Characteristics

Property	Temp	Units	Test Methods	15W-40
Viscosity Grade		SAE		15W-40
Kinematic Viscosity	@ 40°C	cSt	ASTM D445	112.6
Kinematic Viscosity	@ 100°C	cSt	ASTM D445	13.7
Viscosity CCS (15W)	@ -20°C	mPa.s (cP)	ASTM D5293	7000
Viscosity Index			ASTM D4292	130.1
Flash Point (COC)		°C	ASTM D92	218
Pour Point		°C	ASTM D97	-18
Density	@ 15 °C	kg/m^3	ASTM 4052	0.870

Annex B: Atomic Absorption spectrometry (AAS) metal content data

ADDIS ABABA UNIVERSITY

DEPARTMENT OF CHEMISTRY

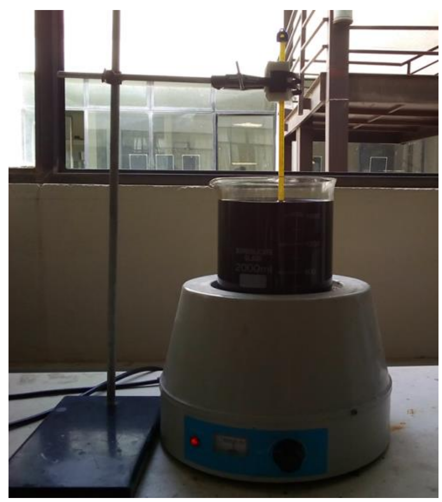
Sample data

	Concentration in mg/L							
Sample label	Ca	Remark (DF)	Fe	Remark (DF)	Pb	Remark (DF)	Zn	Remark (DF)
Raw 1	0.405	x10	1.251	x10	0.7940	x10	0.130	x10
Raw 2	0.563	x10	1.787	x10	0.8192	x10	0.101	x10
Rf1	1.089		1.069		0.8403		0.4063	
Rf2	1.103		0.8736		0.8540		0.4063	

Analysis done by: Dr. Weldegebriel Yohannes

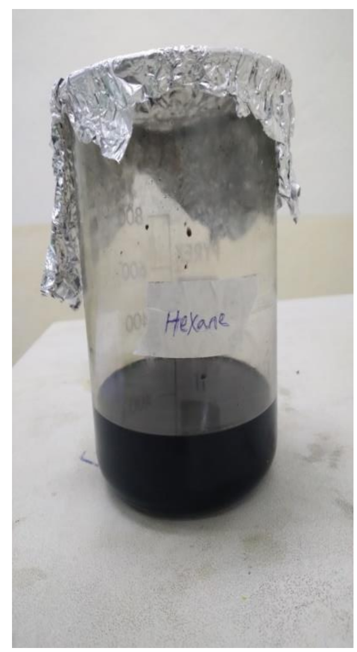
Annex C: Laboratory sample photos

Used lubricating oil (15w 40)

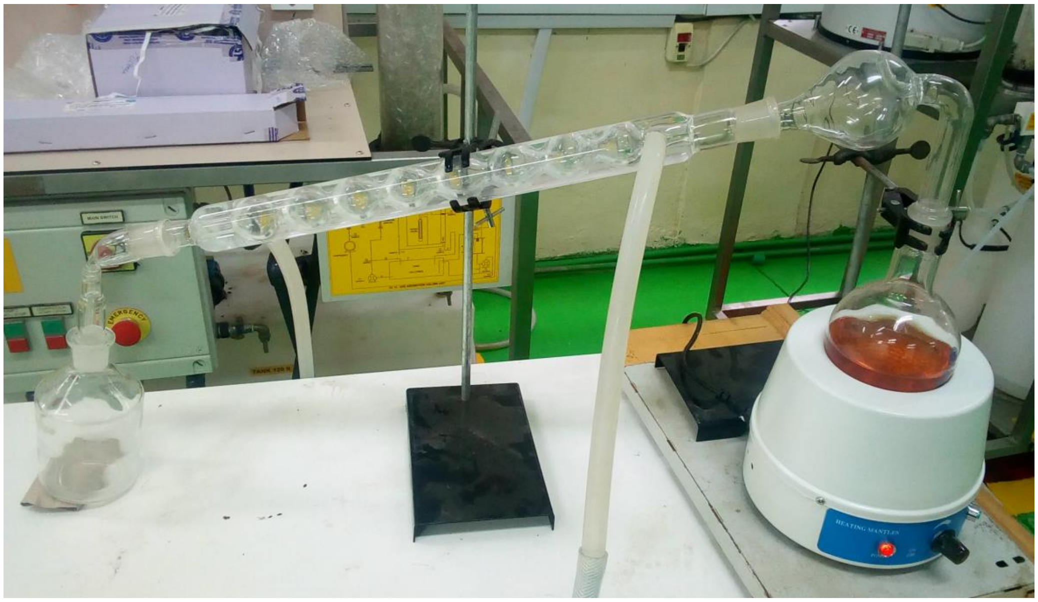


Dehydration process

Vacuum Distillation



Extraction and flocculation



Separation of solvent from oil

Recycled lubricating oil

Used lubricating oil sample

Dynamic viscosity determination


Pour point determination

Figure: Flash point determination

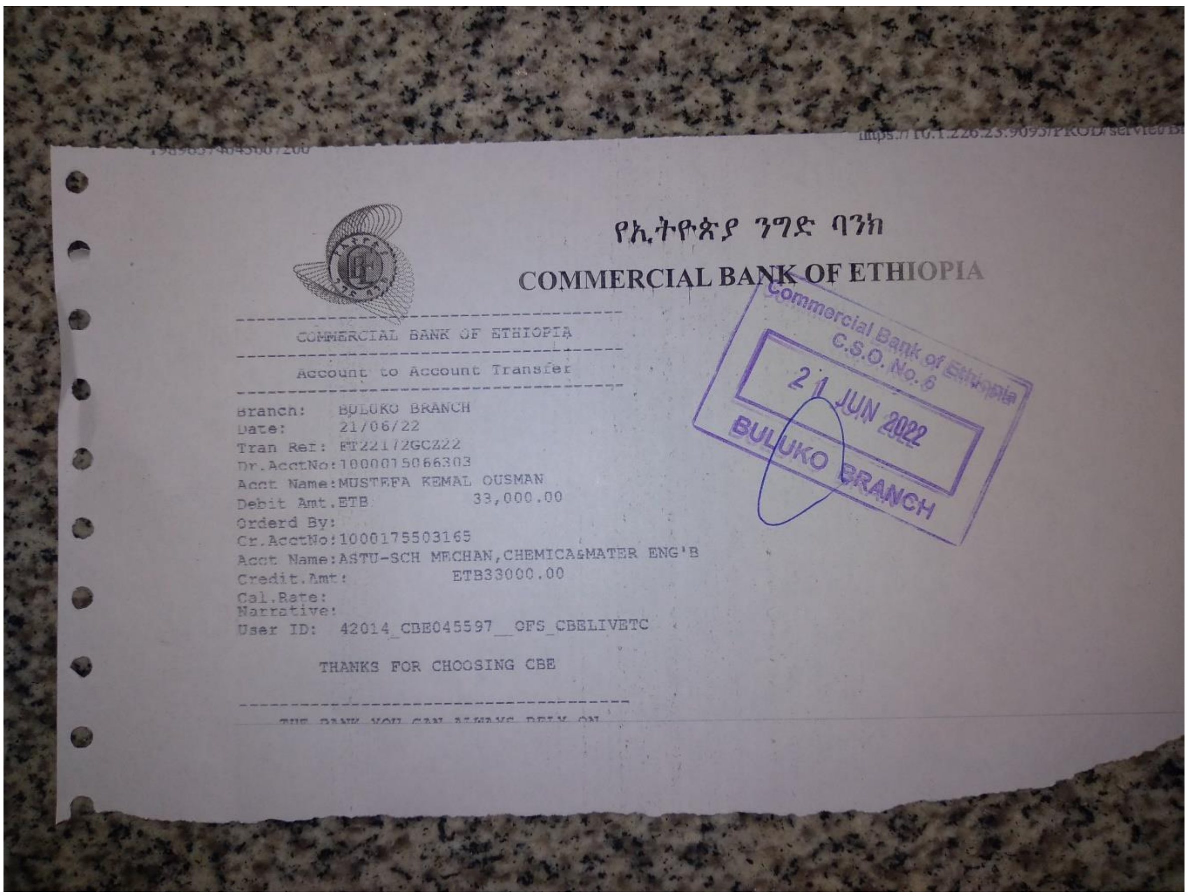
Ash content determination

Figure: Total acid value determination

Annex D: Budget utilization on sample and product characterization tests and limitation of the study

In this study ten (10) characterization parameters namely specific gravity, water content, kinematic viscosity, viscosity index, total acid number, flash point, pour point, color, ash content and metal content were tested for both raw material (used lubricating oil sample) and product (refined lubricating oil). As approved proposal a total of 34,200 birr was allocated for characterization test, that is 16,800 birr for sample characterization and 17,400 birr for product characterization. However we have only utilized 1,200 birr for metal content test and the remaining 33,000 birr was returned back to the university as the bank deposit slip shown below.

Carbon residue test was not conducted in this study due to unavailability of the testing equipment for the parameter.



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Account to Account Transfer

Branch: BULUKO BRANCH
Date: 21/06/22
Tran Ref: FT22172GCZ22
Dr.AcctNo:1000015066303
Acct Name:MUSTEFA KEMAL OUSMAN
Debit Amt.ETB 33,000.00
Orderd By:
Cr.AcctNo:1000175503165
Acct Name:ASTU-SCH MECHAN,CHEMICA&MATER ENG'B
Credit.Amt: ETB33000.00
Cal.Rate:
Narrative:
User ID: 42014_CBE045597__OFS_CBELIVETC

THANKS FOR CHOOSING CBE

Commercial Bank of Ethiopia
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21 JUN 2022
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Returned budget which was allocated for characterization test