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Review

Recovery of Base Oil from Used Oil through Solvent Extraction Followed by Adsorption: A Review

Aftab Hasnat Khan[1], **Nadia Batool**[2], **Abdul Aziz**[2*]

[1]Department of Chemistry, Pakistan Institute of Engineering and Applied Sciences (PIEAS), Nilore, Islamabad, Pakistan
[2]Chemistry Division, Chashma Nuclear Power Complex Unit-2, Mianwali, Pakistan
E-mail: aziz1ctn@gmail.com

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Abstract: Base oil is the major component of lubricating oil, which is present in its original form and used lubricating oil in a sufficient amount. This review article focuses on the recovery of base oil from used oil through solvent extraction followed by adsorption. Different solvents and various solvent-to-oil ratios are studied. Solvent selection is based on its ability to selectively dissolve the base oil from used oil and to reject the impurities in the solution. The percentage yield and quality of extracted oil usually depend upon the nature of the solvent, solvent-to-oil ratio, and nature of impurities. Various solvents and solvent-to-oil ratios are studied. However, results show that methyl ethyl ketone (MEK) is the best solvent because it gives the maximum sludge removal, is more easily recovered, and also has a low cost. A two-phase system occurs after adequate mixing of solvent and oil. Before solvent addition, oil is dehydrated to remove water present as impurities, and then light hydrocarbons are removed using vacuum distillation. After solvent addition, the extracted phase is separated, leaving behind the sludge phase, which consists of aggregates of impurities. The solvent is evaporated from the extracted phase. The extracted oil is put in contact with a suitable adsorbent to absorb the aromatic contents and heavy metals in it. In the end, additives are added to the recovered oil for the formulation of a new lubricant.

***Keywords*:** lubricant, solvent extraction, adsorption, activated carbon

1. Introduction

Technology has proven indispensable for human needs in the present world of modernization. However, the world is also suffering from negative impacts resulting from the advancement of technology. Today, protecting the environment and natural resources, as well as preserving energy resources, has become the biggest human need. Our natural resources and environment are suffering from environmental pollution in the present world. So, there is a big need in the present world to minimize environmental pollution [1]. Lubricating oils are essential for moving components in heavy machinery on an industrial scale. They are replaced with new ones after a certain time when they get contaminated [2]. Improper ways of disposing of used oil go in that direction, where environmental pollution poses a serious threat to living and non-living things such as air, soil, water, etc. Different types of vehicles and industrial operations consume a large quantity of lube oil every day [3]. According to a recent survey, it is estimated that 274,000 tons of waste lubricating oil are produced every year only from vehicles, which is not being discarded properly in Pakistan [4]. Landfilling and incineration are common options to tackle used oil in an easygoing way in Pakistan or

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other developing countries. These methods are faster ways to deal with large amounts of waste oil. However, these disposal methods not only pose an imminent threat to local environments, but they have also been suffering from the loss of valuable resources found in used oil [5]. Therefore, there is a requirement to introduce or devise a technology that would extract valuable material from used oil, leaving behind the minimum amount of hazardous waste. Re-refining is the best strategy to utilize the used oil, with many benefits [6]. Producing base oil from crude oil is nine times more costly than the production of base oil from used oil because crude oil contains more impurities than used oil [7]. Hence, re-refining is also an economical process for the production of base oil, in addition to getting rid of the dumping of used oil. There are many re-refining processes for used oil, and there are chemical and physical treatments for used oil as well. The acid clay method [8], the ionizing radiation method [9], the inorganic membrane method [10], vacuum distillation [11], solvent extraction [12], and thin film evaporation [13] are the most commonly applied re-refining technologies. Most of these methods accompany finishing steps like adsorption [14] and hydro-treating [15]. However, solvent extraction is the most efficient process for the recovery of base oil from used oil because it is low-cost, and sufficient recovery of base oil is made using this process compared to the other processes. It is also a reliable method for recovering base oil from used oil [16].

2. Introduction to lubricants

A lubricant material is used to reduce the friction between the surfaces of moving parts when they come into contact with each other [17]. Lubricating oils lubricate the surfaces of moving components in such a way that they interpose a film between the surfaces of moving units. They alter mechanical friction into fluid friction. In this way, they help to save the physical health of moving bodies [18]. They are heat transfer agents because when two different bodies are moving along with each other, an enormous amount of heat is produced, which needs to be carried away from the system [19]. Lube oils also do the job of power transmission between the components in a system. They are cleaning agents too; they flush away the tear and wear of engines, dirt, salt, ash, carbonaceous particles, degradative components resulting from base oil or chemical additives, and other metallic and non-metallic impurities. Internal parts of the engine get cooled by lubricating oil that can't be directly cooled by the engine water-cooling system [20]. There are three major classes of lubricating oils: lubricating greases, automotive oils, and industrial lubricating oils [21].

2.1 *Types of lubricating materials*

Lubricants exist in liquid, gas, and solid forms [22]. However, liquid lubricants are the most common among the others. Solid and gas lubricants are used under special conditions. For example, solid lubricants work well under extreme conditions, and they are introduced to overcome the drawbacks that we experience when using liquid lubricants [23]. They have high shear strength. Solid lubricant is used in powder and suspension form or as lubricating grease, which is semi-solid. Solid lubricants can be classified into metal solids, polymers, carbon, graphite, ceramics, and cement [24]. Modern solid lubricants are polymers in nature [25]. Polytetrafluoroethylene (PTFE), nylon, and synthetic polymers are examples of polymeric solid lubricants. Polymeric solid lubricants have lighter weights and low thermal conductivity [26]. Gas lubricants, usually helium and nitrogen, are used where a very small film thickness is required between the surfaces of the moving parts. Advantages related to gas lubricants are a large temperature range, very low friction due to low viscosity, and no vaporization or degradation. However, a special design is required to use gas lubricant [27]. Liquid lubricants are composed of mineral or synthetic base oils and chemical additives. Chemical additives are added to mineral-based oils to enhance the performance features of liquid lubricants [28]. A major source of liquid lubricants is crude oil. Evaluation of crude oil parameters is important before the process of mineral base oil extraction from them. Liquid lubricants are classified according to crude oil chemistry. Liquid lubricants are trending in the market due to their compatibility with lubricating system design. They are easy to use, and the lubricating system does not require special design, as in the case of a gas lubrication system [29]. However, liquid lubricants' performance does not remain stable for a long time because they are more prone to contamination as compared to gas and solid lubricants. Therefore, chemical additives are also added to liquid lubricants to keep them in a stable state as well as for their better performance. The general properties of lubricants comprise a high boiling point, thermal stability, low freezing point, high viscosity index, oxidation resistance, hydraulic stability, corrosion prevention, and demulsibility [30].

2.2 *Lubricating oil composition*

A major component of lubricating oil is base oil, which is derived from petroleum or synthetic oil [31]. Lubricating oils are organic in nature. Base oil itself performs most of the functions of lubricant material, but it can only do a part of the job and needs a helping material to do its job completely [32]. Chemical additives are added to enhance the life expectancy of equipment and to promote the stability and performance of lubricating oils. They also suppress the bad qualities of lubricants. The nature and quantity of chemical additives depend upon the nature of the lubricant type (engine oil, hydraulic oil, compressor oil, etc.) and operating conditions (temperature, pressure, and other environmental factors) [33]. The composition of typical lubricating oil is given in Table 1.

Table 1. Composition of typical lubricating oil [34]

	Ingredients	wt.%
	SAE 30 or 40 base oil stock	71.5 to 96.2
Additives	Metallic detergents	2.0 to 10
	Ashless dispersant	1.0 to 9.0
	Oxidation inhibitor	0.5 to 3.0
	Antioxidant or antiwear	0.1 to 2.0
	Friction modifier	0.1 to 3.0
	Pour point depressant	0.1 to 1.5
	Antifoam	2 to 15 ppm

Note: SAE = Society of Automotive Engineers, United States. An oil rated as SAE oil (for example, SAE 5W30) denotes its viscosity; and wt.% = weight percentage

2.3 *Base oil*

Base oil is composed of paraffinic compounds with a minor contribution of naphthenic and aromatic compounds [35]. They are produced from a petroleum fraction by distillation at boiling temperatures of 300 °C and 400 °C [36]. The base oil can be produced synthetically. Usually, they are classified into three main types: mineral oils, modified mineral oils, and synthetic oils, based on their manufacturing strategy. Mineral oil molecules are similar to those of crude oil, while in modified mineral oils, base oil extracted from crude oil undergoes some modification processes like de-waxing, hydro-treating, etc. to improve the quality of the base oil [37]. Synthetic base oils are made by the polymerization process. Two other main types of base oils are also made based on their composition, i.e., paraffinic base oil and naphthenic base oil. Paraffinic base oil is produced from paraffinic crude oil. They have alkanes as a major component, of which long straight-chain alkanes are most dominant. They have a high viscosity index, high resistance to oxidation, a high flash point, and contain low-temperature properties [38]. Aromatic contents in paraffinic base oils affect the viscosity index of mineral base oils badly. Therefore, they need to be removed from paraffinic base oil before their modification into the final shape. Naphthenic base oil contains high portions of alicyclic compounds that have closed-ring methylene groups. They are wax-free or have little wax content. They are used where low-temperature properties are needed and the viscosity index is little considered. They have better solvent power but less resistance to oxidation as compared to paraffinic ones [39]. The American Petroleum Institution (API) introduced a simple classification system for mineral-based oil given in Table 2.

Table 2. API classification of base oil and related production methods [40]

Group	Sulfur (wt.%)	Saturates (wt.%)	Viscosity index	Main production methods
I	> 0.03	< 90	80 to 120	Solvent extraction
II	≤ 0.03	≥ 90	80 to 120	Hydrocracking, isomerization, and solvent extraction
III	≤ 0.03	≥ 90	≥ 120	Hydrocracking and isomerization
IV		Polyalphaolefins		Synthetics
V		All the others		Solvent extraction, hydrogenation, and synthetics

2.4 *Chemistry of crude oil*

A major source of base oil is crude oil. Therefore, it is important to discuss the chemistry of crude oil for a better understanding of base oil classification and the physical and chemical properties of base oil. Crude oil is organic in nature with a small amount of metallic elements such as nickel, vanadium, and iron. It comprises hydrocarbon compounds. However, small amounts of elements like nitrogen and sulfur are also mixed with hydrocarbons in crude oils [41]. Crude oils have complex combinations of hydrocarbons, from simple methane molecules to asphaltenes, whose structures have not been mapped thoroughly yet. However, the respective crude oil doesn't need to contain all those properties that are compatible with a lubricant [42]. Isoparaffins, naphthenes, and aromatics are desirable lube oil molecules. Meanwhile, multi-ring aromatics, heterocyclic compounds containing nitrogen and sulfur, and normal paraffins are non-desirable lube oil molecules found in crude oil [43].

2.5 *Lubricating oil additives*

Additives are added to the base oil to enhance certain properties of the lube oil. Additives are specific to lubricant oil, which implies that it is not necessary for lubricant that perform its action efficiently for more than one lubricating oil system [44]. Their concentration may reach 30% by weight in lubricating oil [45]. Table 3 depicts different types of additives, their functions, and examples.

Table 3. Types, functions and examples of lubricating oil additives [46]

Types of additives	Function	Examples
Viscosity index improvers	Maintain the viscosity of the oil at the required levels at higher temperatures by providing a stable film	Methacrylate polymers and acrylate polymers
Detergent	Act as a neutralizing agent for acidic combustion products produced in internal combustion engines	Metallo-organic compounds: calcium, magnesium, sodium, barium sulphonates, phenolates, and phosphates
Dispersant	Keep the sludge particles (dirt, varnish, water, etc.) in a stable, dispersed form	Long-chain hydrocarbon succinimides (polyisobutylene succinimides)
Oxidation inhibitor	Prevent the mineral oil from being oxidized, and form protective films on metal to prevent acidic corrosion	Zinc dithiophosphate (ZDP), alkyl sulfides, and aromatic sulfides
Rust and corrosion inhibitors	Prevent the metal surface from corrosion and rust by forming a barrier film over the metal surface with a high polar attraction towards the metal surface	Alkaline compounds, organic acids, and esters
Anti-foam	They can destroy foam by keeping the interaction with air bubbles in foam and coalescing and collapsing them	Dimethylsilicones and dimethylsiloxanes
Pour point depressant	Prevent the formation of wax by keeping the wax molecules apart to form agglomerates	Alkylaromatic polymers and methacrylate polymers

3. Used oil

Lubricating oil performance decreases sharply with time due to contamination resulting from the tear of the engine, the local environment, and other operations occurring in the vicinity of the lubricating system [47]. Used oil contains certain impurities like wear and tear of the engine, dirt, salt, varnishes, gum, moisture, un-saturates, asphaltic compounds, phenolic compounds, acidic compounds, combustion products, and alcohols. These ingredients in used oil make lube oil unfit for further use and need replacement with a new one [48]. The effect of impurities on lube oil's physical and chemical properties is shown in Table 4.

Table 4. Properties of fresh and used lube oils [49]

Properties	Virgin lube oil	Used lube oil
Specific gravity at 20 °C	0.882	0.910
Flash point (°C)	220	190
Total acid number (mg KOH/g oil)	2.20	4.40
Total base number (mg HCl/g oil)	4.70	1.70
Viscosity at 40 °C (cSt)	139	115
Viscosity at 100 °C (cSt)	16	14.8
Carbon residue (wt.%)	0.82	0.300
Water volume (%)	-	12.3
Sulfur (wt.%)	0.32	0.42
Lead (ppm)	0	7,535
Zinc (ppm)	1,664	1,097
Calcium (ppm)	1,210	4,468
Phosphorus (ppm)	1,397	931
Magnesium (ppm)	675	309
Barium (ppm)	37	297
Iron (ppm)	3	205
Sodium (ppm)	4	118

3.1 *Used oil treatment technologies*

Three main treatment technologies are used to tackle used oil. These are all methods of reusing waste oil to extract valuable sources from the used oil.

3.1.1 *Reprocessing*

Reprocessing introduces filtration and dehydration to remove sediments and water from used oil to make it reusable or more likely to be used as secondary fuel [50]. This process of treating used oil is suitable when it contains low basic sediments and low water contents [51]. Solid contaminants removed through the filtration process pose environmental hazards and operational problems. Commonly used steps for reprocessing are shown in Figure 1. However, such methods are not enough to eliminate all types of chemical contaminants in the oil. They require a solid finishing step like clay contact or distillation to completely remove contaminants [52].

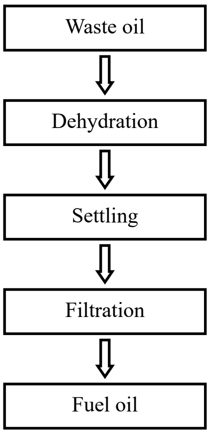

Figure 1. Used oil reprocessing steps

3.1.2 *Incineration*

With highly contaminated used oil with impurities like polychlorinated biphenyls (PCB) and polychlorinated terphenyls (PCT), the process of re-refining or re-processing could not remain suitable or efficient. Hazardous waste incinerators are used for this purpose to destroy the organics present in used oil as well as neutralize the acid compounds. Cement factories' kilns can also be used for controlling high-temperature incineration, having temperature ranges between 2,000 °C and 2,500 °C [53]. However, incineration results in a huge amount of ash, which consists of heavy metals, and it releases carcinogenic substances into the environment; hence, it pollutes the environment and is used as a last option [54].

3.1.3 *Re-refining*

Used oil comprises serious contaminating components but with a sufficient amount of valuable material, such as base oil in safe form. The idea of recycling used oil is not a new one among used oil treatment technologies [55]. It has been practiced since 1930. Re-refining and re-processing are the main recycling and regeneration methods. However, the advantage of re-refining over re-processing is that it renders the properties of re-refined oil closer to those of virgin oil. The re-refining process removes physical, mechanical, and, most importantly, chemical impurities from used oil with different technologies [56]. The flow diagram for re-refining is presented in Figure 2. This method is becoming more prominent in developing countries, and they are meeting the demands of lubricating oil by 50% using it [57]. This method is not only a cheap method of producing lubricating oil but also an easy and more suitable way to get rid of used oil containing a lot of pollutants [58].

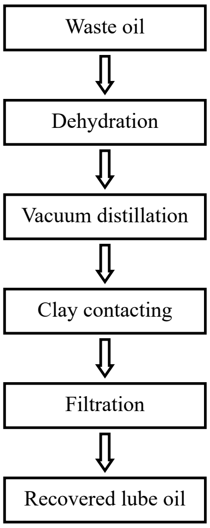

Figure 2. Flow diagram of the re-refining process

4. Re-refining technologies

Physical and chemical processes, or combinations of both, are used to treat used oil. Criteria for process selection depend upon the capital and operational cost of the treatment process and the nature of the used oil (industrial, automotive, etc.) [59]. Solvent extraction, acid clay method, vacuum distillation, radiation technology, membrane method, thin film evaporation, etc. are applied by different countries. Hydro-treating in combination with suitable adsorbents (clay, activated carbon, etc.) and the de-waxing process are used as finishing steps with oil-regenerating methods. However, solvent extraction and acid clay methods are commonly applied in different countries because these methods do not require high costs to run [60].

4.1 *Acid clay method*

In Figure 3, the acid clay method is shown. In this method, an acid like H_2SO_4 is used as a solvent, which causes the solvent-flocculation of undesirable components in used oil like asphaltenes, metals, oxidation products, etc. Poly-aromatic hydrocarbons (PAHs) and heavy metals present in used oil that can't be removed by acid treatment methods are treated with clay [61]. The acid amount is important because if it is increased, it will have significant effects on product yield, kinematic viscosity, and some other physiochemical properties. However, it is ineffective beyond a certain limit. Clay is used as a helping hand with acid because it acts as an adsorbent material [62]. Sometimes activated carbon is also used instead of clay as an adsorbent with acid. Activated carbon is mostly introduced for the adsorption of organic compounds. However, clay is also a sophisticated adsorbent that occurs in nature because it does the same job for waste oil re-refining as it does for crude oil refining. It is a popular adsorbent because of its great number of pores and large surface area. It is used to remove heavy metals, color, and some other undesirable impurities found in used oil [63]. Sulphuric acid [64], acetic acid [65], formic acid, and phosphoric acid [66] were used by different researchers in the acid clay method. The acid clay method is one of the lowest-cost techniques applied for used oil recycling. However, certain disadvantages associated with this method, which decrease its use in most countries, are incomplete removal of metals, low product yield, a higher percentage of clay required to reclaim the color of extracted oil, and most importantly, acid sludge produced from this method, which causes environmental pollution [67].

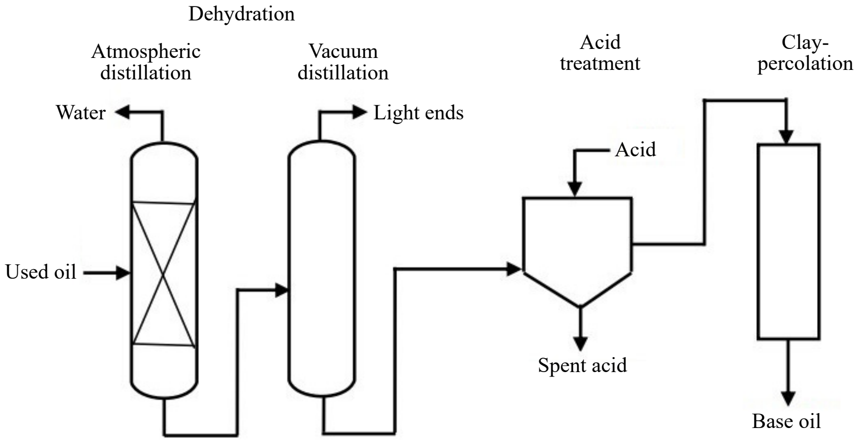

Figure 3. Flow chart of the acid clay method

4.2 *Solvent extraction*

Solvent extraction is defined as a process that distributes the solute particles between two immiscible liquids. It is also defined as a separation technique used to extract valuable material from two immiscible liquids created by the addition of a foreign substance called an extracting agent or solvent. Solvent extraction is an environmentally friendly method for the extraction of base oil from used oil. It is also a reliable, economical, and most suitable method with a good product yield [68]. The solvent should have the ability to base oil extraction selectively from used oil and to reject the impurities in the solution so that they can aggregate [69]. Solvent selection is based on its ability to act as an extractant as well as a flocculent. Furthermore, the solvent should have a low cost, a low boiling point, and be easily recovered [70]. Hydro-treating, de-waxing, or adsorption work with the solvent extraction process as a finishing step to remove some impurities that are not yet part of the extracted oil. The finishing step also helps to reclaim the original color of the extracted lube oil. Some flocculation agents are added separately to the solvent to help, and hence, it increases product yield [71]. Overall, the quality and percentage yield of recovered oil depend upon the nature of the solvent, solvent-to-oil ratio, and nature of impurities [72]. The solvent extraction process comprises sedimentation, filtration, distillation, solvent extraction, settling, and solvent evaporation steps. Solvent, either single or composite, is used as an extractant. Solvents from the alkane, alcohol, and ketones families are the most popular solvents in base oil recovery, utilized as single solvents or composite solvents [73]. However, this solvent should have very high purity because impurities in it affect the extraction process. Supercritical ethane [74] and supercritical propane [75] have also been used as extracting agents where the above-mentioned solvents do not work. The use of liquefied petroleum gas (LPG) condensate and stabilized petroleum (SP) condensate as extracting materials is a new approach to extracting base oil from used oil, but it comes at a high cost for such solvents [76].

4.2.1 *Evaluation of solvent extraction efficiency*

Solvent extraction performance depends on percentage removal, percent oil loss (POL), and product yield. The process is depicted in Figure 4.

4.2.1.1 *Percentage sludge removal*

After solvent evaporation, wet sludge (w_{wet}) is obtained that needs to be washed out with the same solvent to obtain any remaining base oil portion. Percentage sludge is collected using the following Equation 1, where w_{dry} is the weight of dry sludge and w_{oil} is the weight of oil [77].

$$\text{Sludge removal (\%)} = \frac{w_{dry}}{w_{oil}} \times 100 \tag{1}$$

4.2.1.2 *Percent oil loss (POL)*

This factor describes how much oil is trapped by the sludge. Solvents with a greater ability to form sludge also suffer from higher POL [78].

$$\text{POL}\ (\%) = \frac{W_{wet} - W_{dry}}{W_{oil}} \times 100 \tag{2}$$

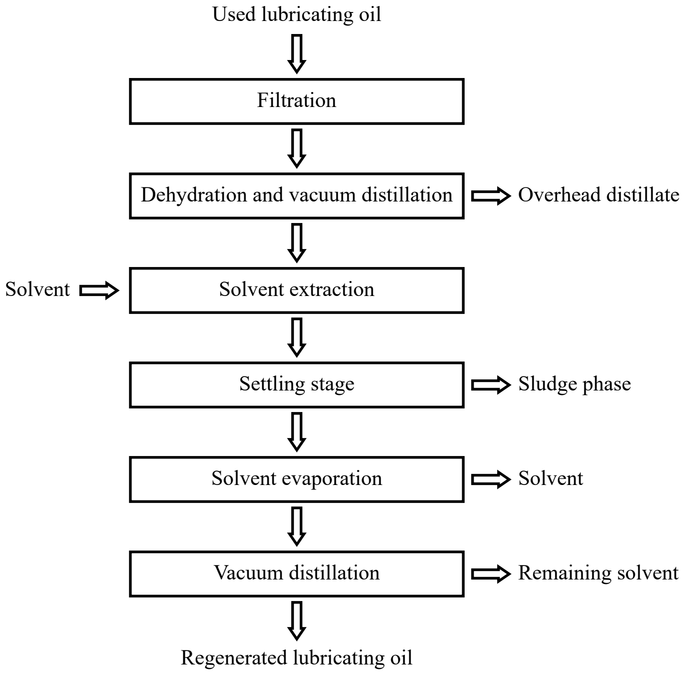

Figure 4. Regeneration of lubricating oil through solvent extraction

5. Methodology

Having high product yield, maximum sludge removal, and low running costs, solvent extraction is one of the best technologies to recover base oil from used oil [79] and is discussed in this review article with adsorption as a finishing step. In the solvent extraction process, there are the following physical and chemical treatment steps; the first three steps are pre-treatment steps before solvent addition.

5.1 *Sedimentation*

Sedimentation sets out the suspended particles under the influence of gravity or with the help of a centrifuge. In gravity, a sedimentation sample of used oil is allowed to settle for 24 to 48 hours, while a centrifuge makes faster sedimentation at 4,000 rpm. Used oil is separated from sediments [80].

5.2 *Filtration*

In this process, lubricating oil is first filtered to remove debris and other solid particles. However, when these impurities become fine particles, simple filtration is ineffective, and only sedimentation is required [81].

5.3 *Dehydration*

Moisture becomes part of lubricating oil with time. It comes mainly from the local environment where used oil is collected or as a result of the combustion of fuel in a combustion engine. Dehydration is carried out using a simple distillation process in which a sample of used oil is heated to 120 °C, ensuring complete evaporation of water. Water removal is necessary from used oil because it is not required in the formulation of new lubricants as it lowers its viscosity [82].

5.4 *Vacuum distillation*

The vacuum distillation step has two objectives. First, we need to remove the light hydrocarbons that can be in the form of gasoline or can be degradative components of chemical additives in lube oil. Second, it is necessary to collect the base oil residues that used oil contains [83]. A vacuum distillation setup is devised with a maximum temperature of 240 °C and a pressure of 5 mmHg. All the light hydrocarbons were removed at a temperature of 140 °C, while the base oil residue fraction was collected at 240 °C [84].

5.5 *Solvent extraction*

Solvent is added to a sample of used oil in a canonical flask. Different solvent-to-oil ratios are tested. The extractant, either as a single solvent or as a composite solvent, is used to extract the base oil from used oil [85]. After solvent addition, a mixture of solvent and used oil is stirred over the hot plate for 30 minutes at 500 rpm. This mixture, after stirring, is settled for 24 to 48 hours in a separatory funnel. A two-phase system occurred after the corresponding time set for setting. One is the extract phase, which contains solvent and base oil, while the other is the sludge phase, which consists of aggregates of impurities. The two phases are well distinguished from each other because of their different colors and textures. The extract phase is a brownish-black liquid, while the sludge phase is semi-solid with physical, mechanical, and chemical impurities. The sludge phase is decanted and washed with the same solvent to get the remaining base oil [86].

5.6 *Solvent stripping*

The solvent is evaporated from the solvent phase at a temperature slightly higher than the boiling point of the solvent using a simple distillation setup. Recovered oil still may have some contaminants, like heavy metals and PAHs, that need to be removed to retain the original color of the base oil [87].

5.7 *Adsorption*

PAHs and heavy metals that are part of recovered oil decolorize the recovered oil. A solution to get rid of such types of impurities is their adsorption on a suitable adsorbent. Adsorption can be carried out in two ways. First, just mix and stir the adsorbent, adding 5% w/w to the 40 grams of recovered oil and allowing it to settle for 3 to 4 hours. Adsorbent-treated oil is filtered to remove any adsorbent particles. The second strategy is to fill the column (used in column chromatography) with a suitable adsorbent. Here, important things to be considered are the particle size of the adsorbent, the amount of adsorbent, the nature of the eluent, and the amount of oil [88]. The most commonly used adsorbents for base oil recovery are activated carbon [89], silica gel, alumina, chitosan powder [90], magnesite rock, rice husk, etc. [91, 92]. Activated carbon is the best adsorbent for organic compounds like PAHs [92].

6. Parameters influencing the solvent extraction process

The following factors affect the percentage of sludge removal, POL, product yield, and the quality of oil employed in the solvent extraction process for base oil recovery.

6.1 *Nature of the solvent*

Before selecting a solvent in the solvent extraction process for base oil recovery, one should be careful about the number of carbon atoms in the solvent. There should be three to five carbon atoms in a solvent. Solvents having carbon atoms less than three are not able to extract the base oil because their solubility parameters are not comparable to those of the base oil, and solvents having carbon atoms greater than five prevent the impurities from aggregating. The second thing is the ability of the solvent to make hydrogen bonds [93]. According to Burrell's classification [94], alcohols have the highest capacity to make hydrogen bonds. Ketones have a moderate capacity to make hydrogen bonds, while hydrocarbons have the lowest capacity to make hydrogen bonds. As the solvent has to perform its job as a flocculent, it has a greater ability to make hydrogen bonding show better results. But the boiling point and cost of the solvent are also important factors in the selection of a solvent in the solvent extraction process to make it efficient and economical [95]. A solvent having a lower boiling point is preferred because it is easily recovered in the solvent evaporation step. Therefore, methyl ethyl ketone (MEK) is preferred to use as a single solvent rather than alcoholic solvents by many researchers because it has a low boiling point and is easily recovered [96].

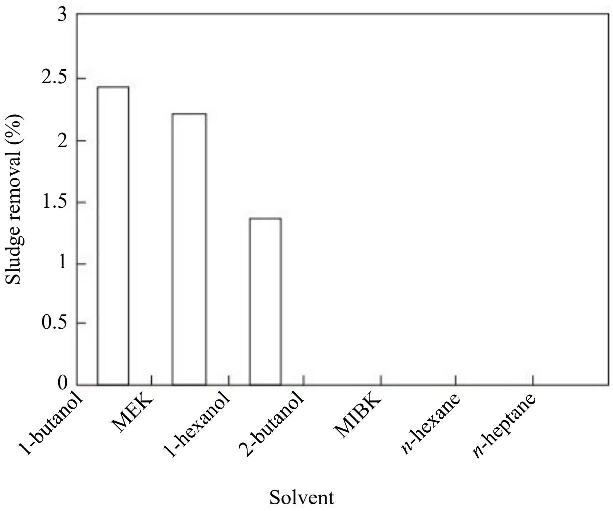

Figure 5. Sludge formation ability of different solvents. Note: MIBK = methyl isobutyl ketone

Figure 5 indicates that 1-butanol shows maximum sludge removal because of its high polarity as compared to all other solvents given here [97].

6.1.1 *Composite solvent as an extractant*

The composite solvent approach as an extractant shows better performance in percentage yield and properties of recovered oil than a single solvent used as an extractant. In the composite solvent approach, different solvents have different properties that dominate over the properties of a single solvent, which acts as an extractant-flocculent [98]. Results of the single solvent and composite of 38% 1-butanol, 37% 2-propanol, and 25% butanone are shown in Table 5.

Table 5. Comparison of properties of re-refined oil with composite solvent and single solvent [98]

Properties	Fresh oil	Used oil	Recovered oil with a single solvent	Recovered oil with a composite solvent
Flash point (°C)	200	120	130	150
Viscosity (cSt)	90	120	98	94
Ash (wt.%)	0.01	2.02	0.15	0.09
Specific gravity	0.90	0.93	0.858	0.88
Pour point (°C)	- 8	- 30	- 18	- 15
Fe contents (ppm)	-	50	30	13

It is clearly shown in Table 5 that a composite solvent shows better efficiency, but a problem arises in the solvent stripping step when using a composite solvent because each solvent has a different boiling point, and hence, recovery of individual solvents is not possible [99].

6.2 *Solvent-to-oil ratio*

The solvent-to-oil ratio is another important factor in the base oil recovery process through the solvent extraction process because we have to prevent our solvent from being saturated with a base oil and also need to prevent the impurities from entering the extracted oil. Solvent-to-oil ratios of 1:1 and 2:1 are not suitable because the solvent becomes saturated. Different solvent-to-oil ratios like 3:1, 4:1, 5:1, and 6:1 are commonly applied. The best results are obtained with a solvent-to-oil ratio of 3:1 or 4:1 [100]. The greater the solvent amount, the greater its solvency power. However, a solvent-to-oil ratio greater than 4:1 does not remain efficient because, under these conditions, some impurities also get dissolved in the solvent. The comparison of different solvent-to-oil ratios is given in Table 6. Maximum sludge removal and maximum oil recovery are two basic goals in solvent extraction, which are achieved by optimizing the choice of solvent and solvent-to-oil ratio [101].

Table 6. Comparison of different solvent-to-oil ratios against recovered oil properties [102]

Property	Fresh oil	Used oil	1:1	2:1	3:1	4:1
Density (kg/m^3)	895	912	885	905	895	903
Viscosity at 40 °C	131	38.3	49	61	72	69
Viscosity at 100 °C	14	6.1	7.5	8.3	10.3	9.57
Water content	0	1.5	1.2	0.76	0.34	0.42
Ash content	0.463	0.952	0.843	0.706	0.515	0.564
Flash point	242.5	178	195	211	220	218
Pour point	-14	-6	-7	-8	-11	-10
Carbon residue	1.3549	3.527	3.125	2.867	1.961	2.034
Color code	0.042	0.53	0.47	0.44	0.38	0.39
Total base number (mg HCl/g oil)	3.1	4.7	4.4	3.95	3.4	3.55
Total acid number (mg KOH/g oil)	2.4	5.8	5.1	4.3	3.7	3.9

6.3 *Mixing speed and time*

Waste oil is mixed with solvent at different solvent-to-oil ratios. The engine can tear and wear some suspended particles, which come from the local environment. These particles can be removed by free sedimentation, in which they are left to settle under gravity, or by centrifugation, in which the solvent-to-oil mixture is stirred quickly for a short duration [103]. The mixing speed of the solvent-to-oil mixture also matters because, at high speeds, suspended particles settle down very quickly and are still suspended after free sedimentation. Therefore, there is a direct relationship between mixing speed and product yield, as presented in Figure 6 [104]. In addition to mixing speed, mixing time also plays a role in the sedimentation of unwanted suspended particles, which is evident from Figure 7. A larger mixing time provides an opportunity for more suspended particles to settle down, and hence, product yield increases with increasing mixing time [59].

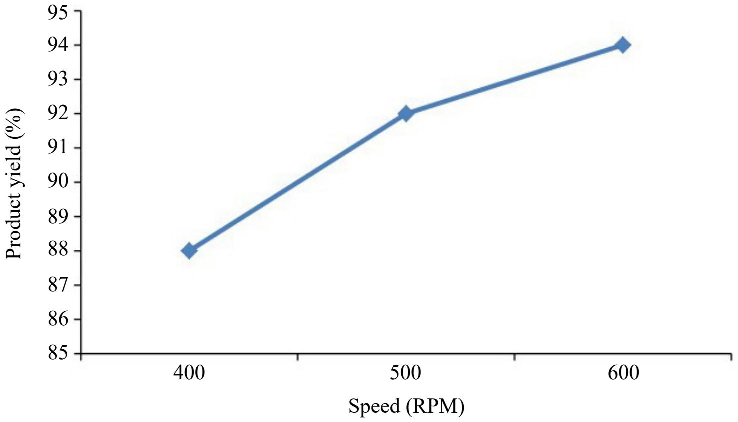

Figure 6. Effect of mixing speed on product yield

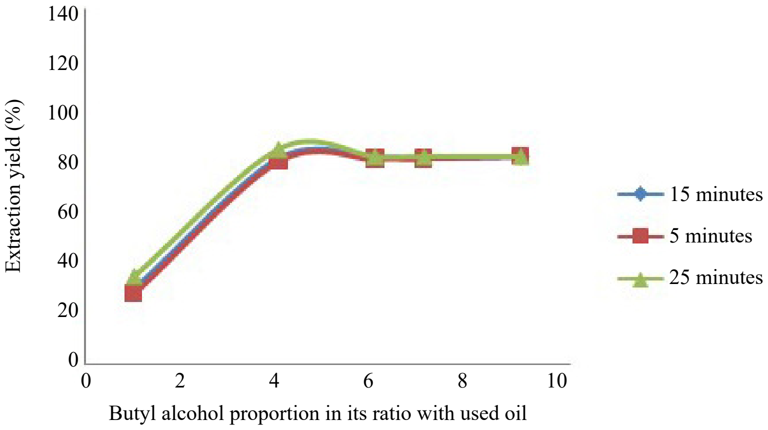

Figure 7. Effect of mixing time on product yield

6.4 *Extraction temperature*

The solubility of base oil increases slightly with an increase in temperature above 25 °C. This is primarily because heating reduces the Van der Waals forces between base oil molecules and separates base oil that has become trapped by impurities. However, above room temperature, co-extraction of sludge impurities with base oil in the solvent extraction process also started. Second, going beyond room temperature, the rate of evaporation of volatile solvents also increases [105]. In the case of MEK, which is a volatile solvent and has a boiling point of 80 °C, evaporation of MEK starts and goes faster between 40 °C and 60 °C. If the temperature is further increased from 60 °C, the rate of evaporation becomes faster. Hence, the best quality of oil with minimum solvent loss is achieved at ambient temperature (35 °C) [106].

6.5 *Flocculation agents*

Certain impurities in used oil are positively or negatively charged species. These charges remain stabilized in solution unless their charges are neutralized by adding any foreign substances that can neutralize them. Flocculants are inorganic and organic compounds that gather the destabilized particles together and agglomerate them, resulting in the thickening of sludge during treatment. Flocculants during used oil treatment by solvent extraction process help the solvent enhance separation efficiency, and hence, percentage sludge removal increases with a comparatively low amount of solvent [107]. It is found that sludge removal increases from 12.8% to 14.7% by using 2 grams of methyl ethyl amine (MEA) per kg of MEK. NaOH, KOH, and MEA have been used as flocculants in oil treatment by solvent extraction processes [108].

7. Parameters of lubricating oil

The following parameters of refined oil and used oil are usually analyzed, which are defined in this section. The values of these parameters (shown in Table 7) change due to contamination, so their evaluation from time to time is a way of checking the condition of lube oil.

Table 7. Parameter values of recovered oil with MEK solvent at a 3:1 solvent-to-oil ratio at 35 °C [109]

Parameters	ASTM	Used oil	Extracted oil
Total acid number (mg KOH/mg)	D 974	2.9	0.922
Pour point (°C)	D 97	-16	-15
Flash point (°C)	D 92	128	212
Ash content (wt.%)	D 874	0.93	0.61
Water content (wt.%)	D 95	2	0.08
Viscosity at 40 °C (cSt)	D 445	125.4	138.92
Viscosity at 100 °C (cSt)	D 445	14.8	15.9
Density at 20 °C (g/cm^3)	D 1289	0.66	0.899
Specific gravity at 20 °C	D 1289	0.9	0.8652
Aniline point	D 43	14 °C	10 °C

Note: ASTM = American Society for Testing and Materials; and D = miscellaneous materials

7.1 *Density*

Density is mass per unit volume. The density of lube oil is measured using either a hydrometer or a digital density meter. In the case of the hydrometer technique, a specific hydrometer is used for lubricating oil. The hydrometer is inserted into the sample at a slow pace. It then starts to float. The point at which the hydrometer rests represents the specific gravity of the sample. The hydrometer density is determined by using Equation 3.

$$\text{Density} = \text{specific gravity} \times 1\ \text{g/cm}^3 \tag{3}$$

Density measurement with a digital density meter is a more accurate and easier method. The density of used oil is lower than the density of fresh oil because of the degradation of base oil constituents or chemical additives. However, if aromatic contents are present as impurities or in the case of a mixture of different used lubricating oils, then the density of the used oil can be higher than that of fresh lube oil [110].

7.2 *Viscosity*

Viscosity is the resistance to flow of a liquid, or it can be described as interlayer friction between the molecules of a liquid, while kinematic viscosity is the resistance experienced by liquid molecules when they flow under the influence of gravity. Kinematic viscosity is measured by a two- or three-leg capillary glass viscometer. Fresh lubricating oil has a certain viscosity value. But used oil has a higher viscosity in some cases and a lower viscosity in some scenarios than that of fresh oil, depending on the environment. An increase in the viscosity of used oil shows the presence of compounds of oxidation and polymerization (these compounds can be both dissolved and suspended), or it may be the mixing of two or more lubricating oils. On the other hand, a decrease in the viscosity of used oil reveals the presence of light fuel oil. Temperature also affects viscosity. It decreases with an increase in temperature. It is determined at 40 °C and 100 °C [111].

7.3 *Viscosity index*

Lubricant oil can resist the change in its viscosity with a change in temperature at a higher temperature. A higher value of the viscosity index shows a better quality of lubricant oil. When lube oil becomes contaminated, its value decreases. Usually, viscosity index improvers are added as additives to a base oil to enhance the viscosity index of the lubricant oil. The viscosity index is measured by following the formula [112].

$$\text{Viscosity index} = \frac{(L-U)}{(L-H)} \times 100 \tag{4}$$

where U is the kinematic viscosity at 40 °C, L is the viscosity at 40 °C of oil with a viscosity index of 0, and H is the viscosity at 100 °C of oil with a viscosity index of 100. Here, L and H values are taken from the table regarding oil viscosity at different temperatures [43].

7.4 *Flash point*

The flash point is the lowest temperature at which a flammable mixture of fumes will ignite if a spark or other ignition source is present. This temperature is achieved when oil is continuously heated, and at a certain temperature, a flammable mixture of fumes is produced. But this is not the same for waste oil and fresh oil because impurities in used oil decrease the flash point of used oil, like gasoline impurities. The lower flash point of used oil is an indication of gasoline in it. An increase in the flash point of extracted oil shows that light components have been evaporated from it. It is determined by Cleveland’s open cup or closed cup apparatus. A sample of 10 ml in a beaker is placed on the cup and placed over a heated coil in the instrument. A flame source is brought in at intervals to determine the temperature at which garish appears on the surface, continuing the heat to lubricate oil in the beaker [113].

7.5 *Pour point*

It is defined as the lowest temperature below which oil stops flowing or moving. Their value should be very low for lubricating oil. In this test, the sample is first heated and then cooled at a specific rate. Oil flow was checked at intervals of 3 °C [114].

7.6 *Ash contents*

Ash contents are the incombustible materials in the used oil. They are inorganic in nature. Their presence is undesirable in used oil. So, there should be a maximum reduction in extracted oil from the solvent extraction step. The ash contents test is determined by placing the sample in a muffle furnace and heating it to 775 °C, then cooling and weighing the sample to determine the ash contents [115].

7.7 *Total acid number*

The total acid number is the amount of KOH needed to neutralize the acidic contents of the used oil. The combustion of engine fuel produces carboxylic acids and other acidic products. A higher total acid number value indicates the higher acidic contents of used oil, and hence, oil needs to change at this stage [116].

7.8 *Total base number*

The total base number is the amount of acid needed to neutralize the basic contents of used oil. Lubricating oils have a high alkalinity value due to the presence of highly alkaline chemical additives used as detergents or dispersants. A higher alkalinity of fresh lube oil is set to neutralize the acidic products coming from combustion so that prevention against corrosion can be made. However, its value gets lower when lubricating oil is used. This may be due to the degradation of chemical additives in used oil. Total base number measurement started by dissolving the sample of 2 grams of lube oil in 100 ml of solvent. A mixture of solvents like 2-propanol, toluene, and water in a ratio of 2:2:1 is used, respectively. The sample is titrated against HCl in glacial acetic acid. Before titrating the sample, two or three drops of p-naphtholbenzein indicator were added to the sample, and it gave the sample an orange color. The orange color of the sample changed to green or green brown when the end point was achieved. Then a blank titrant is prepared by adding only 10 ml of solvent without any sample or titration. The value of the total base number is calculated using Equation 4 [117].

$$\text{Total base number} = \frac{(Vs2 - Vs) \times 56.1 \times N}{Ws} \tag{5}$$

Here, $Vs2$ is the volume of titrant against the sample, Vs is the volume of titrant consumed against blank, N is the normality of titrant, which equals 0.0641, and Ws is the weight of the sample in grams.

7.9 *Metal contents*

There are many different sources of trace metals in used oil. For example, Ca, Zn, Ba, and P result from detergent dispersant agents, while Fe from engine wear and Pb come from gasoline [118]. So, there are a variety of metallic elements in used oil, and their analysis is performed by an atomic absorption spectrophotometer (AAS). Initially, extracted oil is heated at 200 °C and then calcined at 650 °C. Then ashes come into contact with HCl. After this, it is filtered and diluted with demineralized water, and then the sample is analyzed using AAS to determine the metal contents in it [119, 120]. Metal contents in used oil can also be determined by inductively coupled plasma (ICP) [121].

7.10 *Moisture contents*

Moisture contents in used oil may be the result of combustion products entering lube oil, or they may come from the local environment where used oil is collected and stored in drums. Moisture contents in used oil were found by Karl Fischer titration with the volumetric method. In this method, iodine produced from the chemical reaction of titer and methanol reacts with water. Lubricating oil is heated to remove water contents trapped or emulsified in it. One mole of iodine reacts with one mole of water, so the consumption of moles of iodine is directly related to the concentration of water present in used oil. Moisture contents in used oil are described in the form of percentage volume in this method [122].

7.11 *Aniline point*

The aniline point is defined as the temperature at which an equal volume of aniline and used oil is completely mixed and homogenized. A lower aniline point is an indication of a higher content of polyaromatics in used oil. In this test, 10 ml of both aniline and lube oil were mixed in a test tube. A mixture of two components is heated and then cooled. The temperature at which the mixture gets completely homogenized is the aniline point of the lube oil under testing [123].

8. Conclusion

It is estimated that one gallon of used oil contaminates nine million gallons of water. So, the major aim of the recovery of base oil from used oil is to make the environment clean. In addition to this, used oil contains a large portion of base oil in safe form, which can be recovered by the application of solvent extraction and adsorption methods. Lubricant oil used to reduce friction between surfaces of moving parts of an engine becomes contaminated with time. There are different methods applied for the recovery of base oil, and the most sophisticated method is solvent extraction. This review focuses mainly on solvent extraction used as a re-refining technology, with adsorption as a finishing step. Although solvent extraction gives a well-refined oil, this setup is only useful on a commercial scale. Extracted oil also needs the re-addition of chemical additives to be reused in compatible systems. Therefore, recycling is not a complete solution to meet the original quality of fresh lube oil, whatever the recycling method used. To meet the present industrial revolution, it is very much needed for technologists to prepare lubricating materials in the future that can sustain their features under enormous conditions for a very long time.

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Conflict of interest

All authors declare no conflicts of interest in this paper.

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