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Base Oil Regeneration from Spent Lubricating Oil Using Solvent Extration Process: A Review

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Abstract: Lubricating oil is a viscous hydrocarbon component used to lubricate engine parts in automobiles. The presence of contaminants and residues such as zinc, iron, chromium, and polyaromatic hydrocarbons (PAHs) arising from wear and tear, additive degradation, thermal breakdown, and oxidation impair the quality of lubricating oil during service. Indiscriminate utilization of spent lubricating oil such as incineration for energy generation without prior treatment generates considerable amount of toxic gases and carcinogens which are environmentally catastrophic and detrimental to health. There is an increasing trend to regenerate and reuse discarded lubricating oil in response to environmental regulations and economic implications. Regeneration of base oil from spent lubricating oil entails using physical and chemical procedures to obtain base oil that can be reused in the formulation of new lubricants. Solvent extraction, hydro-treatment, and acid clay treatment are examples of traditional regeneration procedures. Many researchers have shown that solvent extraction followed by adsorption is a more efficient and effective procedure for recycling spent lubricating oil. This review paper provides an overview of the solvent extraction process as well as the impact of process variables on the process efficiency.
Key words: Spent Lubricating Oil, Solvent Extraction, Regeneration, Base Oil, Environmental Pollution

I. INTRODUCTION

Lubricating oil is a complex mixture of viscous hydrocarbon molecules. The high boiling point (>400°C) distinguishes lubricating oil from other fractions of crude oil (Speight and Exall, 2014), with molecular weight ranging from 250-100 (Bhushan et al., 2007). Lubricating oil's principal purposes are to minimize friction, inhibit corrosion, provide a medium for heat transfer, as well as serve as a suspending agent (Kannan et al., 2014; Mynin et al., 2004). Figure 1 depicts a typical lubricating oil composition.

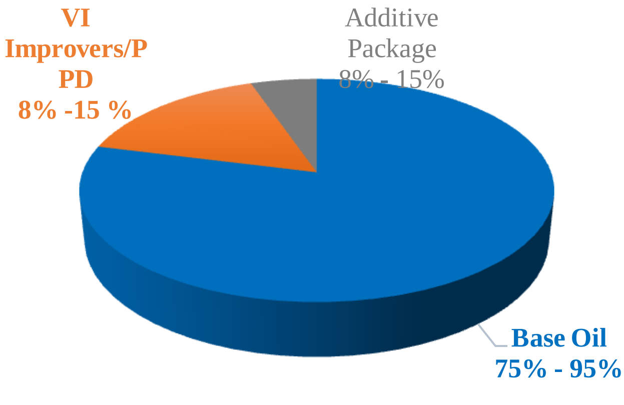

Figure 1. Typical Lubricating Oil Composition

1.2 Spent Lubricating Oil

Spent lubricating oil often referred to as used engine oil or waste oil is any lubricating oil refined from synthetic or crude components that has become contaminated by chemical or physical impurities during usage (Speight and Exall, 2014). Lubricating oil loses its functional properties during usage due to the presence of extraneous contaminants as well as products of oil deterioration. During usage, lubricating oil is exposed to a variety of conditions that can deteriorate its additive and base oil system (Speight and Exall, 2014). Factors such as oxidation, thermal breakdown, incompatible gases, entrained air, moisture and unintended mixing of a different fluid, additive depletion plays a key role in the degradation process (Speight and Exall, 2014)The products of these degradation processes renders the lubricating oil unfit for regular usage due to the presence of metallic fragments, ash content, lacquer, sludge, soot gel network, carbon residue, water, polycyclic aromatic hydrocarbons etc. Figure 2 presents the chemical composition of spent lubricating oil.

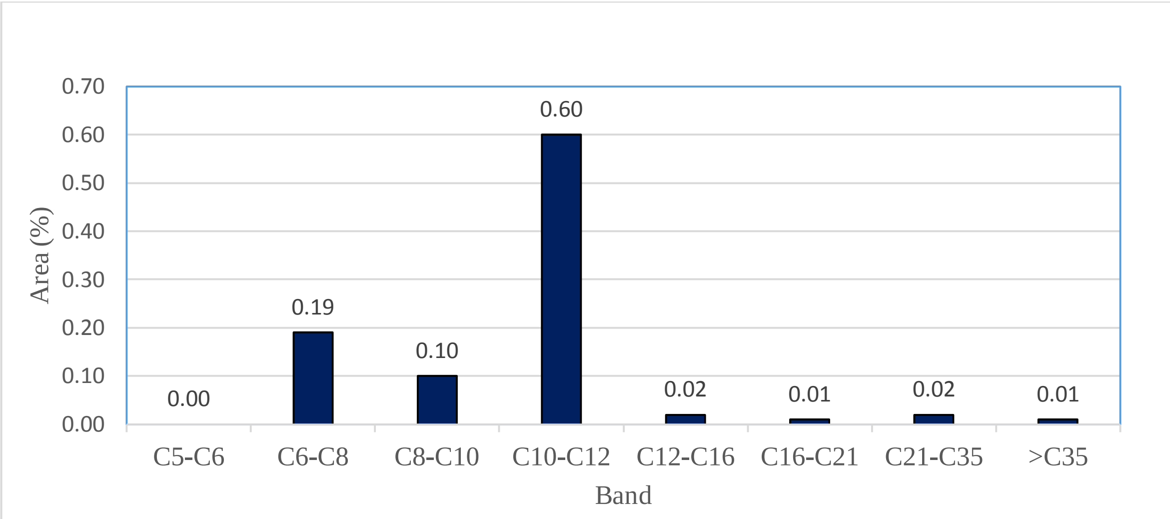

Figure 2: Chemical Composition of Spent Lubricating Oil. Modified from (Rodriguez-Hernandez et al., 2015)

1.3 Environmental impact of spent lubricating oil

The indiscriminate disposal of spent lubricating oil in waste water systems, as well as its dumping on the ground, depletes soil fertility and pollutes surface and ground water. According to Nwachukwu et al. (2012), one litre of spent lubricating oil is enough to contaminate one million litres of water. Inhalation, ingestion, and skin contact with impurities or contaminants in spent lubricating oil can cause a variety of human diseases (Salem et al., 2015). In fact, only a small percentage of petroleum products degrade naturally through oxidation, photochemical reactions, and biodegradation, nevertheless, when burned, the majority of them pollute the soil, water, and air (Mynin et al., 2004). As reported by UN (2016), nearly six million fatalities are attributed to air pollution yearly.

According to Emetere (2017), burning spent lubricating oil produces aerosol and greenhouse gases in the environment that can travel at speeds of 10-12 m/s. The implication of this report is that air pollution caused by burning spent lubricating oil is not limited to the source of pollution, but can travel to other locations over time (Oladimeji et al., 2018). Waste lubricating oils are disposed of in most countries into the soil, water bodies, or utilized directly as fuel, resulting in major health ailments and environmental hazards (Merai, 2013). The removal of contaminants, particularly metal components, from waste lube oils is a critical step in reducing toxicity, simplifying processing, and increasing the potential reuse of spent lube oils, all of which contribute to waste minimization.

1.4 Spent Lubricating Oil Management

Various regeneration procedures have been developed in response to social responsibility for environmental preservation to contain the environmental impacts associated with indiscriminate dumping of spent lubricating oil. Reprocessing, reclaiming, and regenerating (re-refining) are the three types of spent lube oil recycling processes (Pelitli et al., 2017). Reprocessing involves eliminating impurities from spent lube oils using a variety of procedures such as heat treatment, precipitation, filtering, dehydration, and centrifugation (Speight and Exall, 2014).). Reclaiming is the process of separating solids and water from waste lube oils using physical treatment to produce a product that is similar to the original but still contains metal contamination (Speight and Exall, 2014). Regeneration is a procedure that produces base oils with the highest degree of contaminant removal, which may then be used to make fresh lubricants.

Spent lube oil is treated in a regeneration process in various steps, including water removal, pollutants and dirt separation, acid treatment, solvent extraction, clay treatment, hydrogenation, or a combination of these processes (Diphare et al., 2013). Re-refining consists of four stages as presented in Figure 3: pre-treatment (i.e., removal of water and solid particles), regeneration (elimination of degradation products), fractionation of the recovered base oil (i.e., separation of light hydrocarbons), and finishing (i.e., improvement of colour and smell of the regenerated base oil) (Widodo et al., 2018).

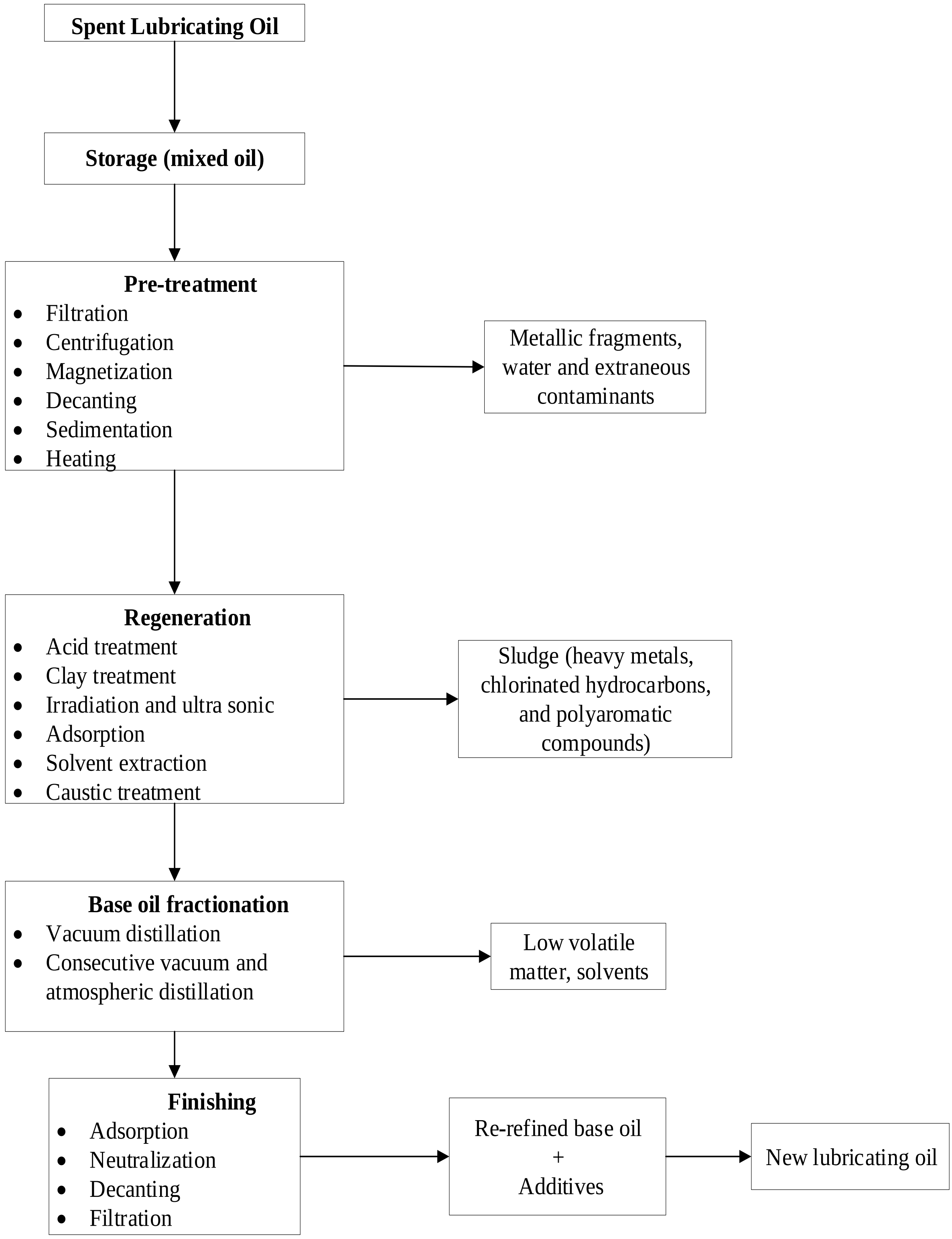

Figure 3: Main Stages of Conventional Technologies for the Re-refining of Used Oils. Adapted from (Sánchez-Alvarracín et al., 2021; Widodo et al., 2018)

Details on each technology and the corresponding advantages and disadvantages of each re-refining method are presented in Table 1

Table 1: Technologies Employed at an Industrial Scale for Re-refining Spent Lubricating Oils. Adapted from (Sánchez-Alvarracín et al., 2021)

Re-refining Method	Process description	Advantages	Disadvantages	Ref.
Acid clay	• Preliminary distillation at low temperatures to remove water, solvents, and antifreeze • Addition of sulphuric acid for reverse reaction to form sludge • Adsorption with clay to improve colour	Cost effective. Does not require qualified operators. Suitable for small capacities. Low capital investment Low energy consumption.	Low yield Acidic sludge generation Corrosion of equipment Not compatible with environmental regulations	(Jonidi and Hassanpour, 2015; Nwachukwu *et al.*, 2012)
Solvent Extraction	• Dehydration at (120°C – 130°C) • Solvent extraction to produces insoluble and suspended substances such as asphalt, metal compounds, and resin. • Sludge removed as non-hazardous waste. • Distillation to recover solvents • Adsorption with clay to reduce odour and colour	Solvents are recyclable. Does not cause contamination. Functional recovery of base oils. Produces good quality base oils. Operates at room temperature	Economical only for high plant capacity. Requires operating systems and qualified personnel. May involve operating losses of solvent.	(Jonidi and Hassanpour, 2015; Nwachukwu et al., 2012; Oladimeji, Sonibare, Omoleye, Emetere, et al., 2018)
Hydro-treatment	• Dehydration at (120°C – 130°C) • Vacuum distillation • Treated with hydrogen • Adsorption with clay	Improves the colour and smell of the oil. Removes sulphur, nitrogen, metals, or unsaturated hydrocarbons	Expensive. Unsafe. Not suitable at small scale. High energy consumption. Operates at high temperature and pressure	(Jonidi and Hassanpour, 2015; Kupareva et al., 2013; Maghsoodi et al., 2018)

The steps for each re-refining technology and the yields that can be recovered from the process are presented in Figure 4.

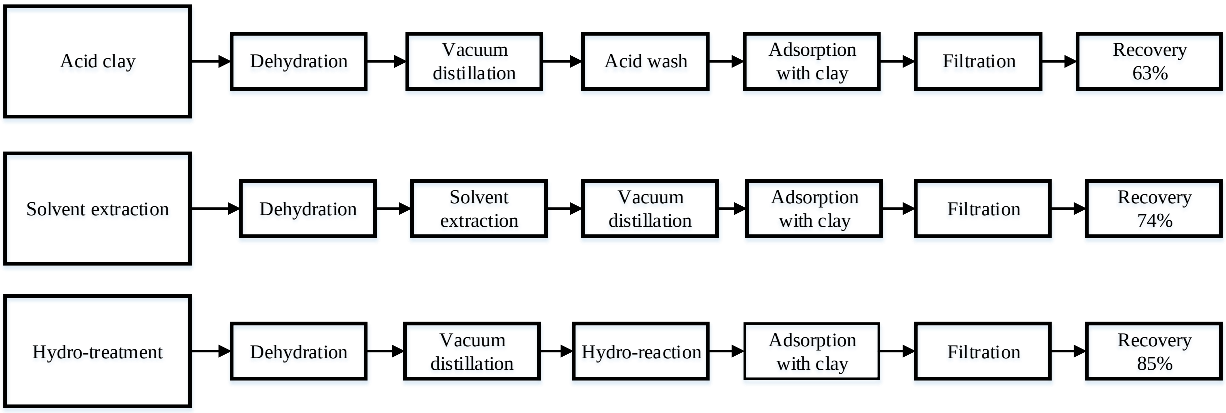

Figure 4. Summary of the Conventional Technologies Employed for Re-refining of Used Lubricant Oil. Adapted from (Kupareva et al., 2013; Maghsoodi et al., 2018).

In contrast to hydro-treatment and acid clay treatment, solvent extraction produces good quality base oil and lower pollution rate (Aremu et al., 2015) as presented in Table 1. According to Sánchez-Alvarracín et al. (2021), the selection of a specific process for re-refining of spent lubricating oil involves three criteria; technical and sustainability aspects, health safety and environmental impacts, and lastly economic considerations. Osman et al. (2017) confirmed solvent extraction and adsorption to be a more effective process in regenerating spent lubricating oil. Solvents chosen must have high solubility parameter for base oil recovery (Durrani et al., 2012; Emam and Shoaib, 2013; Kamal and Khan, 2009) and less soluble in additive and carbonaceous compounds (Durrani et al., 2012).

II. AN INSIGHT INTO SOLVENT EXTRACTION PROCESS

Solvent extraction is a process where the spent lubricating oil is mixed with a solvent that will be able to extract the base oil and flocculate the impurities. The solvent after extraction is recovered using distillation technique and reused. Re-refining of spent lubricating oil by solvent extraction-flocculation process is mainly associated with two objectives; base oil recovery and segregation of solid contaminants (Hussein and Abdulkarim, 2017). The effects of process parameters and solvent type on the extraction process have been studied by several researchers.

Filho et al. (2010) evaluated the recovery of waste lubricating oil by solvent extraction/adsorption using 1-butanol, tert-butanol, 2-propanol and ethanol followed by adsorption using activated carbon and powdered silica. From their findings, 1-butanol was the most efficient with values of kinematic viscosity @40^{o}C (15.25 cSt) and water content 0.0023% all in close conformity to ASTM standards while acidity index (1.079) was found to be above the standard value (0.05). Activated carbon proved to be effective in removing polyaromatic hydrocarbons (PAH's) base on the PAH's absorbance curves presented in the research work. Optimum condition was 1:3 solvent to oil ratio which gave a yield of 86% utilizing 1-butanol and activated carbon.

Sterpu et al. (2012) used a composite solvent (2-propanol, 1-butanol and butanone) to investigate the effect of the solvent to oil ratio on the regeneration of base oil from spent lubricating oil. They discovered that increasing the solvent to oil ratio beyond 6:1 was not cost-effective. The best operating conditions were found to be 4:1 solvent to oil ratio, which resulted in a 49% ash reduction and 92% oil recovery.

Mohammed et al. (2013) combined solvent extraction and adsorption for recovering base oils. It was found that 1-butanol achieved the best performance with maximum percentage sludge removal followed by n-hexane, petroleum ether, 1-hexanol, carbon tetrachloride and acetone. For the adsorption process, it was found that acid activated clay was able to produce high quality product with 84% yield, density (896 kg/m^3), kinematic viscosity @40^{o}C (85 cSt), ash content (0.483%) and total acid number (2.9 mg KOH/ g oil), followed by almond shell powder with a yield recovery of 74% , density (898 kg m^{-3}), kinematic viscosity @40^{o}C (75 cSt), ash content (0.505%) and total acid number (3.6 mg KOH/g oil).

Diphare *et al*. (2013) used solvent extraction and degradation-flocculation process to assess the oil recovery efficiency. They found that the optimum KOH concentration as a flocculant due to the presence of the OH functional group was 30%, and that the optimum solvent to oil ratio using hexane was 7:1 with base oil recovery of 74.2 % and a solvent recovery of 88%. While Kamal and Khan (2009) reported that lower solvent to oil ratios can lead to base oil saturation in the extract phase, resulting in reduced oil recovery. With greater solvent to oil ratio, oil free sludge can be achieved with maximum oil recovery. In their study, sludge formation remained constant (2.3%) with increasing temperature (20^oC to 60^oC) when the solvent to oil ratio was greater than 3:1 with increasing temperature from (20^oC to 60^oC).

Yang *et al*. (2013) used butanol as an extractant and monoethanolamine (MEA) as a flocculating agent to find the ideal regeneration conditions for separating base oil from waste lubricating oil while avoiding co-extraction of contaminants. Temperature, extraction time, solvent to oil ratio, and flocculant concentration all had an impact on the regeneration process. The best regeneration conditions were found to be 30°C refining temperature, 20 minute mixing time, 5 g/g solvent to oil ratio, and 2 g kg^{-1} flocculant concentration. MEA not only flocculates the used engine oil contaminants, but it also minimizes the amount of solvent required to achieve equilibrium, according to the study.

Abro *et al*. (2013) compared the regeneration of used lubricating oil utilizing single solvent extraction, composite solvent extraction, and acid treatment, examining several physicochemical parameters of the re-refined engine oil. According to their findings, iron contamination was reduced by 74% for composite solvent (butanol, propane, and butanone), 40% for single solvent (propane), and 70% for acid treatment. They came to the conclusion that, based on the qualities of the regenerated base oil, extraction by composite solvent produced the greatest results, albeit at a high cost.

Emam and Shoaib (2013) investigated the recovery of base oil from spent lubricating oil in local crankcases. They investigated the influence of solvent to oil ratio and settling time on the percentage yield recovered using solvent extraction followed by hydro-treating, and discovered that the yield and ash content removal were unaffected by solvent to oil ratios greater than 5:1. According to their findings, the best quality base oil was obtained using methyl ethyl ketone at a solvent to oil ratio of 5:1 and a settling time of 24 hours, with a composition of 84% saturates, 4% aromatics, and 2% residue in the regenerated base oil.

Kannan *et al*. (2014) investigated the use of methyl ethyl ketone in lubricating oil regeneration. After adding additives, the physicochemical properties of the regenerated base oil, density (884 kg/m^3), specific gravity (0.884), viscosity (266 cp), viscosity index (81), flash point (276^oC), fire point (313^oC), pour point (-22^oC), cloud point (105^oC), aniline point (11^oC), and total acid number (0.53 mg/g) all in conformity with SAE standards. They suggested that the regenerated base be used in engines.

Aremu *et al*. (2015) investigated the effects of three solvents on percentage sludge removal and percentage oil loss: 1-butanol, 2-propanol, and a mixture of 1-butanol and ethanol. 1-butanol produced the best extraction performance with regards to percentage sludge removal as also confirmed by Araromi *et al*. (2016) and Hussein *et al*. (2014). Varying contact time from 20 min to 30 min had little or no effect on percentage sludge removal, and varying the temperature from 35^oC to 50^oC also had little or no effect on improving the process performance efficiency. According to their findings, they discovered that a solvent-to-oil ratio of 3:1 resulted in the best percentage sludge removal (12.4%).

Ani *et al*. (2015) conducted a study on optimization of base oil regeneration using a composite solvent (2-propanol, 1-butanol and butanone). In line with the findings of Kamal and Khan (2009) and Sterpu *et al*. (2012), the results of their study indicated that an increase in the solvent to oil ratio from 3:1 to 5.41:1 increased yield from 22% to 37%. On the other hand, an increase in mixing time from 30 minute to 42.07 minute had no influence on yield. Conversely, increasing the mixing time from 30 minute to 40 minute led to an increase in ash content by 150%, while an increase in solvent to oil ratio from 2.59:1 to 5:1 decreased the ash content by 66.6%. They discovered that for optimum regenerative capacity, the ratio of solvent to oil should be 5:1, with mixing time of 30 minutes at room temperature.

Abdulaziz and Mahmood (2016) investigated the extraction/distillation technology for regenerating base oil from discarded lubricating oil. Various solvents, including n-butanol, 2-propanol, ethanol, and binary solvent blends (heptane and methyl ethyl ketone) with acetone, were examined. Base on their findings, 1-butanol produced the best oil recovery (93.7%) and solvent recovery (96.2%) at the optimum conditions of 4:1 solvent to oil ratio and 40^oC extraction temperature, as also reported by Araromi *et al*. (2016).

Araromi *et al*. (2016) studied the effects of temperature, solvent type and solvent to oil ratio on percentage oil yield. When utilizing ethanol as a solvent, the researchers discovered that increasing the solvent to oil ratio beyond 4:1 reduced the percentage oil output at all temperatures studied. When utilizing 1-butanol, a higher solvent to oil ratio (above 5:1) resulted in a drop in percentage oil yield by 11.9%. They found that butanol has the ability to precipitate impurities in spent lubricating oil, with 1-butanol producing the best extraction performance with values of kinematic viscosity at 40^oC (98.7 cSt), kinematic viscosity at 100^oC (11.2 cSt), viscosity index (97) and flash point (210^oC) all in conformity to ASTM standards as reported by Hussein *et al.* (2014) under optimum conditions of 50^oC and a solvent to oil ratio of 5:1, followed by a mixture of 1-butanol and ethanol.

Hussein and Abdulkarim (2016) investigated the ability of binary solvents to enhance spent lubricating oil extraction-flocculation regeneration process. They employed (heavy naphtha and n-butanol) and (heavy naphtha and iso-butanol) as solvents. They found that iso-butanol and heavy naphtha increased the performance, including reduced viscosity index (118.2), ash content (0.5767%), TAN (2.1786 mg KOH/g oil), and higher yield (88.88%). Similarly, Hussein and Abdulkarim (2017) examined the percentage sludge removal with a solvent extraction-flocculation method while varying operating variables. Three binary solvents were used in the research, (n-butanol and heavy naphtha), (heavy naphtha and methyl ethyl ketone), and (heavy naphtha and iso-butanol). Based on the coefficient of determination (R^2) for the multivariate process correlation, binary solvents (heavy naphtha and n-butanol) offered the maximum percentage sludge removal at optimum conditions of 45 minute mixing time, 700 rpm agitation, and extraction temperature of 35^{o}C.

Raza *et al*. (2016) tested the potential of three solvents, methyl ethyl ketone, 1-butanol, and 2-propanol, to regenerate spent lubricating oil. The percentage oil loss increased as the solvent to oil ratio exceeded 3:1, indicating that as the mutual solubility of the mixture increases, the solvent to oil ratio decreases the oil yield. According to their findings, methyl ethyl ketone was considered to be more effective because it showed the least amount of oil loss at a solvent to oil ratio of 3:1 and an extraction temperature of 50^{o}C.

Pai *et al*. (2016) used a composite solvent approach consisting of 1-butanol, 2-propanol, and methyl ethyl ketone to develop a batch process for the regeneration of used motor oil. With a yield recovery of 75.6% and a solvent recovery of 95% at an optimum solvent to oil ratio of 3:1, the process was determined to be cost effective and efficient.

Osman *et al*. (2017) investigated the efficiency of solvent blends (toluene, butanol and methanol; toluene, ethanol and butanol; and toluene, isopropanol and butanol) and activated alumina to regenerate spent lubricating oil. Their results confirmed that solvent mixture (toluene, butanol and methanol) gave the best result with highest percentage sludge removal (52%) which was attributed to the high solubility (23.2 j/m^3) of the solvent blend. The regeneration process significantly improved the physicochemical properties of the regenerated base oil as evidenced by 81.15% reduction in total acid number, 89.5% reduction in ash content and 21.95% reduction in sulphur content. Their findings indicated that high solubility and dielectric constant parameters are excellent indicators of solvent behaviour.

Oladimeji *et al*. (2018) studied the effects of mixing speed, solvent type, temperature, and solvent to oil ratio on the solvent extraction/adsorption process. In this study, methyl ethyl ketone and propan-2-ol were used as solvents. At the optimum solvent to oil ratio of 4:1, methyl ethyl ketone provided the most favourable physicochemical attributes of the regenerated base oil. They also discovered that as the solvent to oil ratio and mixing speed increased, the percentage oil yield increased until it reached an optimum temperature of 50^{o}C, after which the oil quality deteriorated. They came to the conclusion that solvent to oil ratio has a greater impact on the quality of regenerated base oil produced.

Riyanto *et al*. (2018) conducted a research on the chemical and adsorption treatment of spent lubricating oil using butanol, kaolin, and KOH as a coagulant. Their findings revealed that the metal content of Ca^{2+}, Mg^{2+}, Pb^{2+}, and Cr^{6+} had been significantly reduced by 99.98%, 97.31%, 79.59%, and 33.33% respectively while Fe^{2+} content increased by 112.78%. The optimum treatment conditions, according to their findings were 3:1 solvent to oil ratio, 1.5 g of kaolin, and 2.0 g of KOH. They concluded that increasing the concentrations of butanol and KOH had a greater effect on metal content reduction.

Santos *et al*. (2019) studied the thermal decomposition profile and oxidative decomposition profile of regenerated mineral oils using 1-butanol, methyl ethyl ketone and 2-propanol. They posited that the presence of functional groups in solvents played a significant role in the solvent's solubility as methyl ethyl ketone had higher yield due to the presence of carbonyl functional group. Base on the TG/DTG/DTA curves obtained, base oil regenerated by methyl ethyl ketone showed the best thermal stability.

Adewole *et al*. (2019) assessed the properties of regenerated base oil obtained through composite solvent extraction and determined its suitability for reuse. The research used a composite solvent mixture of hexane and butanol, as well as KOH as a flocculating agent. The flash point (222^{o}C) of the regenerated base oil was found to be lower than the standard specification; SAE20 (224^{o}C), SAE30 (226^{o}C) and SAE40 (268^{o}C) while the kinematic viscosity @40 of the reclaimed oil (138.92) was found to be above the standard; SAE20 (37), SAE30 (88) and SAE40 (110). The researchers suggested that the viscosity and flash point of regenerated oil be improved for better lubrication in automotive engines.

Nour *et al*. (2021) conducted extensive research on the recycling of used engine oil. They assessed the performance of single solvent (isopropyl alcohol) and composite solvents (ethanol, isopropyl alcohol and toluene). Based on the results of the experiments, both single and composite solvents significantly reduced calcium and alkaline contamination from degraded detergent additives, as well as zinc contamination from anti-wear additives. However, at 40^{o}C extraction temperature, the results for viscosity, viscosity index, and FTIR revealed that isopropyl alcohol performed better than the composite solvent. They further proposed the use of zeolite as an adsorbent to remove the heavy metal ions.

III. CONCLUSION

Improper disposal and utilization of spent lubricating oil causes environmental degradation and a loss of economic incentives. Because of the health, economic, and environmental implications of spent lubricating oil, the search for the most efficient and cost-effective technology to reduce environmental degradation while promoting socioeconomic incentives has generated research interest. Solvent extraction has gained attraction in the research community as an alternative to traditional acid clay treatment and hydro-treatment techniques. The goal of this process is to counteract the shortfalls of conventional procedures currently in use. This application is based on unique qualities such as high-quality base oil, low pollution rates, solvent reusability, and process conformity with environmental standards. The progress that has been made thus far has been reviewed in order to provide a blueprint and roadmap for future research initiatives.

Temperature, solvent-to-oil ratio, mixing speed, mixing time, and solvent type were all shown to have an effect on the process. These provide important insights into the interaction between these process variables and output variables, which is critical for improving process performance. Despite tremendous success, the issue of cost remains a significant gap in spent lubricating oil regeneration. In light of this, more research into designing a systematic solvent screening process that takes into account the economic and environmental aspects of the regeneration process is recommended.

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