Malaysian Journal of Chemical Engineering & Technology 5(1) (2022) 43–53

Malaysian Journal of Chemical Engineering & Technology

e-ISSN: 2682-8588

Journal Homepage: http://myjms.mohe.gov.my/index.php/mjcet

A comparative study on treatment for recycling of waste lubricating oil

[a] Huey Ling Tan, [a] Nurul Nadhirah Azmi, [a] Harumi Veny *, [a] Ying Pei Lim, [b] Rossi Passarella

[a]School of Chemical Engineering, College of Engineering, Universiti Teknologi MARA, Selangor, Malaysia
[b]Faculty of Computer Science, Universitas Sriwijaya, Jl. Raya Palembang-Prabumilih Km. 32, Indralaya, South Sumatra, Indonesia

*Corresponding email: harumi2244@uitm.edu.my

Abstract

In the present work, the recycling of waste lubricating oil by treatment was investigated using three types of acids such as nitric acid, hydrochloric acid, and sulphuric acid. This research aims to gain a high-quality refined lubricant oil using acid as an alternative to distillation process. The characteristics and quality of the treated lubricating oil were compared with the base oil by several parameters such as pour point, cloud point, dynamic viscosity, kinematic viscosity specific gravity and ash content, following American Society for Testing and Materials (ASTM) standards. Initially, filtration was employed to remove the dirt that may be present in the used lubricant oil, followed by adding gasoline to the used lubricating oil and centrifugation at 1500 rpm for 10 minutes. The atmospheric distillation was conducted for complete removal of water and gasoline. Finally, the lubricating oil was treated with the acids (nitric acid, hydrochloric acid, or sulphuric acid) and neutralised with 6% sodium hydroxide before being separated using settling and centrifugation. Results showed that sulphuric acid exceeds nitric acid and hydrochloric acid in treating the waste lubricating oil. Furthermore, treated waste lube oil using 20% sulphuric acid was comparable to the fresh lubricating oil. The results showed that the waste lube oil after acid treatment can be reused.

Article Info

https://doi.org/10.24191/mjcet.v5i1.15726

Article history:

Received date: 3 October 2021
Accepted date: 18 April 2022
Published date: 30 April 2022

Keywords:

Acid treatment
Recycle
Waste engine oil
Lubricant
Pollution

1.0 Introduction

Lubricant oil is a viscous liquid derived from petroleum that has been used extensively as a lubricating medium for various automobile parts such as engines and gearboxes to reduce the friction by maintaining and creating a lubricating film between the two-moving metal surface (Kannan et al., 2014). Lubricant oil also plays an important role in removing heat from the machines or engines, keeping the machines, engines and equipment clean and minimising the corrosion. Waste lubricating oil posed an environmental hazard as it can give adverse impact on health and environment. The improper handling of waste lubricating oil such as dumping the waste lubricating oil into the drains, streams, rivers, lakes and ocean nearby results in water and soil contamination. Waste lubricating oil is classified as hazardous waste according to the European List of Waste (Commission, 2014) and Commission Regulation (EU) No 1357/2014 (EU, 2014). The waste lubricating oil contains heavy metals such as PCB (poly-cyclic benzenes) and PAH (poly-aromatic hydrocarbons) which are the main contaminants and highly toxic when dumped into environment especially in water as it can cause the blockage of sunlight, obstruction of oxygen from the atmosphere and impair photosynthesis which gives huge impact to aquatic lives (Yang, 2008). Thus, treatments of waste lubricating oil will be of utmost importance and beneficial in reducing engine oil costs besides the significant positive impact on the environment by reducing the municipal solid waste.

Waste lubricating oil can be either recovered into base lubricating oil or recycled as fuel or as feedstock in producing the petroleum-based products. Other commercially valuable products can also be produced through different methods (Isah et al., 2013). Refining waste lubricating oil takes about one-third of the energy needed to refine crude oil to lubricant quality. In addition, one gallon of refined waste lubricating oil produces the same 2.5 quarts of lubricating oil just like 42 gallons of crude oil does (Mekonnen, (2014); Yang, 2008). There are a few conventional practices in treating waste lubricating oil such as pyrolytic distillation, extraction of organic contaminants with liquid propane followed by a cascade of three consecutive distillation stages and solvent extraction (Maceiras et al. 2017; (Botas et al., 2017). A combination of solvent extraction with vacuum distillation using a ternary solvent consisting of 2-propanol, 1-butanol and methyl ethyl ketone (MEK) was also reported for pre-treatment (Zgheib & Takache, 2020). The preliminary pre-treatments for purification of used lubricant oil can also be carried out by adsorption using conducting polymer (Parekh et al., 2022). However, these methods are high cost technologies and may produce harmful by-products. Therefore, a recycling process using non-toxic and cost-effective materials will be a highly sought after solution. In this work, a comparative study to evaluate the different acid treatments of waste lubricating oil was performed. The characteristic of treated waste lubricating oil was then analyzed for their properties (cloud point, pour point, specific gravity, kinematic viscosity, dynamic viscosity, and ash content) and compared with fresh commercial lubricant oil and international standard for motor oil-based (DEF STAN 91-43/8)(Jafari & Hassanpour, 2015). The treated waste lubricating oil properties were found close to the properties of fresh lubricating oil as well as the standard, hence indicating its potential for reuse.

2.0 Methodology

2.1 Material

The waste lubricating oil (WLO) was collected from various workshops nearby Shah Alam, Selangor, Malaysia while the unused lubricating oil was bought from an oil service station in the same town. All the chemicals were purchased from Sigma-Aldrich and used without further purification except when mentioned specifically. Sulfuric acid, hydrochloric acid and nitric acid were used as the washing agents, whereas sodium hydroxide and gasoline were used for acid neutralization and oxidation.

2.2 Methods

The waste lubricating oil was filtered to remove all the dirt and impurities that may present such as metal chips, sand, dust, particles and micro impurities. Here, a funnel layered with filter paper and a vacuum pump connected to the filtering flask were used. A 450 mL of the lubricating oil was measured and transferred to a beaker. Next, a 150 mL of gasoline was added to the waste lubricating oil and the mixture was stirred thoroughly to promote homogeneity. The lubricating oil mixture was transferred to a centrifuge and centrifuged at 1500 rpm for 10 minutes. It was then left for settling for another 10 minutes before decanting into a beaker. Thereafter, the atmospheric distillation was conducted for the complete removal of water and gasoline. In this stage, the lubricating oil mixture was heated with a heating mantle from 120 to 140°C under atmospheric pressure for an hour. Then the content in the flask was left cooled. The lubricating oil was treated with the acids (nitric acid, hydrochloric acid, or sulphuric acid) and the mixtures were then agitated and allowed to cool. The shaker temperature was fixed at 40 °C and the agitation intensity was 250 rpm. The treated sample was kept undisturbed for 24 hours for de-asphalting and settling of acid sludge. For the neutralisation process, the treated oil was mixed with 6% of sodium hydroxide to neutralise any soluble acid that remained. The neutralisation product (salts) and unreacted sodium hydroxide were left for about 24 hours at room temperature in a shaker with a constant stirring intensity of 300 rpm for an hour. The treated lubricating oil was separated by using settling and centrifuging the supernatant oil to remove remaining suspended solids and salts that were present during the neutralisation process. This step was conducted at 5000 rpm for 45 minutes.

2.2.1 Analysis of quality and characterization of lubricating oil

Cloud point (ASTM D97) and Pour Point (ASTM D97): The cloud Point analysis was carried out by measuring and transferring a 20 mL lubricating oil sample into a container. The lubricating oil sample was chilled and paraffin hydrocarbon (in the form of wax) began to solidify and separated into crystalline form. The pour point procedure was similar to the cloud point. However, further chilling was continued until lubricating oil stop to flow

Specific gravity (ASTM D941-55): Specific gravity is the ratio of the density of the material to the density of an equal volume of water. This was measured using a hydrometer. The density was observed at 60 °F and the value was recorded.

Viscosity (ASTM D445: Viscosity (ASTM D445) was analysed by using an automated viscometer AMVn. The fresh base virgin oil, the waste lubricating oil and the refined waste lubricating oil treated with various types of acid-filled the capillary block of a clean viscometer to the mark while immersing in a thermostat. The temperature was set to 40 °C, and the obtained result was tabulated using a viscometer AMVn software.

Ash content (ASTM D48): A quantity of incombustible material might present in a lubricating oil which can be determined by measuring the amount of ash remaining after combustion of the oil in a furnace. This can be done by measuring amount of waste lubricating oil in a crucible and kept for five hours in the furnace at 800 °C. The mass of the remaining ash was measured and its percentage was calculated by dividing it by the initial mass of the sample as shown in Eq. (1):

$$\% \text{ Ash } = \left(\frac{m_{\text{ash}}}{m_{\text{initial sample}}}\right) \text{x } 100 \quad (1)$$

3.0 Results and discussion

3.1 Colour quality of recycled lubricant

Fig. 1 shows the results of the fresh, refined lubricating oil, and wastes lubricating oils in terms of colours and opacity for (a) fresh lubricating oil (light yellow), (b) refined lubricating oil treated with sulphuric acid (dark yellow), (c) refined lubricating oil treated with nitric acid (reddish black), (d) refined lubricating oil treated with hydrochloric acid (brown), and last, (e) the untreated waste lubricating oil (black). The difference in colours was influenced by the chemical composition and contaminant that may be present in the lubricating oil, the darker the colour, the higher the contaminants. From the results presented, the colour of the lubricating oil treated with sulphuric acid has the lightest colour compared to other lubricating oil that was treated with nitric acid (reddish black) and hydrochloric acid (dark brown). The selectivity sequence can therefore be given in the following order sulphuric acid > hydrochloric acid > nitric acid.

3.2 Characteristics of waste and re-refined lubricating oils: Study of various types of acid

Pretreatment of waste lubricating oil was carried out using three different acids, which are Nitric acid, sulphuric acid and hydrochloric acid. The pretreated waste lubricant was then analyzed for its characteristics and properties, and compared with the fresh commercial lubricant oil and the standard. The analysis result of these three pretreated waste lubricant oil is shown in Table 1. The result shows that all three acid treatments managed to reduce the ash content below 1% as per standard requirements.

3.2.1 Specific gravity

Specific gravity is the proportion of the density of the material to density of the equal volume of water (Abu-Elella et al., 2015). The temperature at which the density

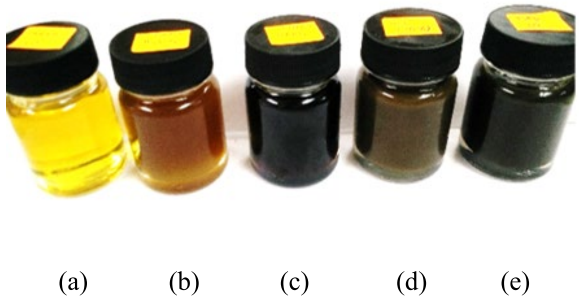

Fig. 1: Colour of different quality lubricating oil: (a) fresh lubricating oil (light yellow), (b) refined lubricating oil treated with sulphuric acid (dark yellow), (c) refined lubricating oil treated with nitric acid (reddish black), (d) refined lubricating oil treated with hydrochloric acid (brown), and (e) the untreated waste lubricating oil (black)

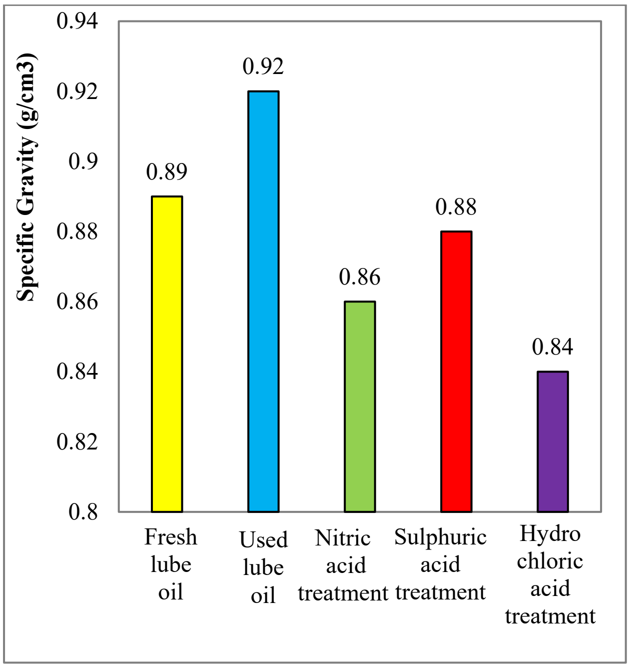

Fig. 2: Specific gravity of different treated lubricating oil

has been measured is essential as the density changes with temperature. The specific gravity for the fresh and untreated waste lubricating oils was 0.88 g/cm^3 and 0.92 g/cm^3, respectively, as shown in Fig. 2. Meanwhile, specific gravity of the lubricating oils treated with acids were 0.86 g/cm^3 (nitric acid), 0.88 g/cm^3 (sulphuric acid), and 0.84 g/cm^3 (hydrochloric acid), which were generally lower compared to the untreated waste lubricating oil. The specific gravity of contaminated oil may be higher or lower depending on the type of the contaminations (Chevron lubricating oil FM ISO 100) and the chemical composition of the lubricating oil. The presence of a higher the number of aromatic compounds in the lubricating oil increases the specific gravity, whereas higher number of saturated compounds results

Table 1: Acid treatment analysis results

Parameter	Waste lubricating oil	Fresh lubricating oil	Nitric acid treatment	Sulphuric acid treatment	Hydrochloric acid treatment	Standard range (Jafari & Hassanpour, 2015)
Colour	Black	Yellow	Reddish black	Yellow	Brown	-
Specific gravity (g/cm^3)	0.92	0.89	0.86	0.88	0.84	0.88 -0.89
Kinematic viscosity (Mm^2/s)	61.60	76.66	70.39	73.99	70.69	-
Dynamic viscosity (mPa·s)	56.68	68.23	60.53	65.11	60.79	
Pour point (°C)	−34	−8	−13	−10.5	−16	-
Cloud point (°C)	−37	−13.5	−17.5	−15	−19.5	-
Ash content (%)	2.3	0.12	0.32	0.26	0.41	<1.0%

in a decrease in the specific gravity. The specific gravity of the untreated waste lubricating oils were the highest, indicating the presence of a high amount of contaminants in the waste engine oil. Meanwhile, the specific gravity of sulphuric acid-treated lubricating oil was close to the fresh lubricating oil,and also within the range of acceptable standards for lubricating oil (Jafari & Hassanpour, 2015). This result is comparable to the specific gravity of treated used oil by Solvent extraction and alumina treatment as reported by Osman et al. (2018).

3.2.2 Dynamic viscosity

Dynamic viscosity increases or decreases depending on the level of oxidation and contamination present with insoluble matter in the lubricating oil. From the results obtained, used and acid treated lubricating oil were observed with lower dynamic viscosity due to the contaminant in the form of slush. In general, lubricating oil is considered unsuitable for use if the original viscosity increases or decreases to the next SAE number code for specifying the viscosity of lubricating oil established by the Society of Automotive Engineering (Udonne, 2011). Increasing viscosity is owing to oxidation or contamination while decreasing viscosity is due to dilution with light fuel (Scapin et al., 2007). Fig. 3 shows the dynamic viscosity of the fresh and waste lubricating oils are 68.23 and 56.68 mPa·s, respectively, while the acid treated lube oils were 60.53 (nitric acid), 65.11 (sulphuric acid), and 60.79 mPa·s (hydrochloric acid). It shows that the refined lubricating oil has higher dynamic viscosity comparing the waste lubricating oil, indicating that the acid treatment gas restored most of its viscosity.

This is most prominent for the lubricating oil refined using sulphuric acid, which can be attributed to the possible conversion of contaminants by the acid. Lubricating oil with viscosity above 60 is necessary to act as an effective coolant or heat transfer medium. All the acid treated lubricating oil meet this specification, but sulphuric acid shows advantage over others with the viscosity nearest to the fresh lubricating oil.

3.2.3 Kinematic viscosity

In the lubrication industry, the viscosity is usually expressed as kinematic viscosity (Pinheiro et al., 2018). The kinematic viscosity of lubricating oil is depending on the temperature, pressure and density. When the temperature of the lubricating oil decreases the viscosity increases and vice versa (Abu-Elella et al., 2015). The viscosity testing can assess the presence of contaminants in the waste lubricating oil, where increasing viscosity is due to the oxidised and polymerised products dissolved and suspended in the lubricating oil; while decreasing viscosity may occur due to fuel contamination (Udonne, 2011). Oxidation of lubricating oils happens in the engine environment that produces contaminants such as corrosive oxidised products, deposits, and varnishes which lead to the increased viscosity. The kinematic viscosity of the samples was measured by using the falling ball viscometer and the viscosity can be calculated using the following equation:

$$\mu = gD_p^2(\rho_p - \rho)t_p / 18L \qquad (2)$$

where t_p is the time taken for a sphere to fall a distance, L, ρ_p is the density of solid sphere, ρ is the density of fluid, D_p is the diameter of the solid sphere and g is the gravitational acceleration equivalent to 9.8 m/s^2.

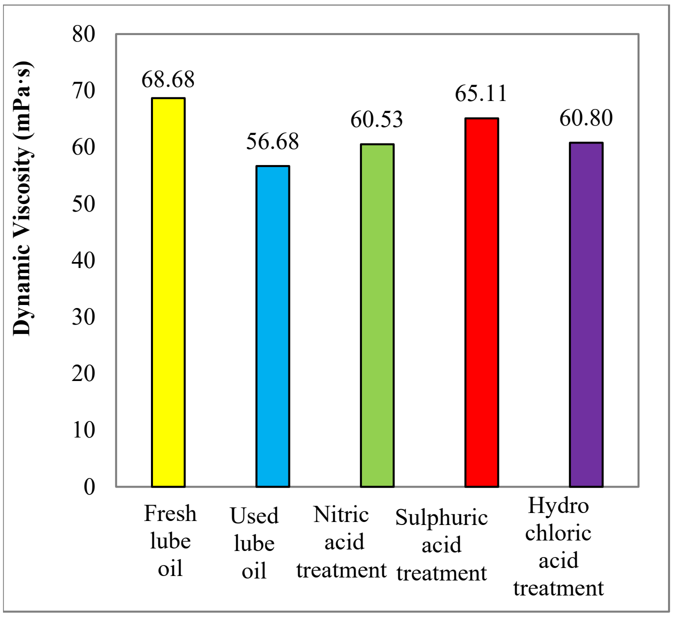

Fig. 3: Dynamic viscosity of fresh, used and different acid treated lubricating oil

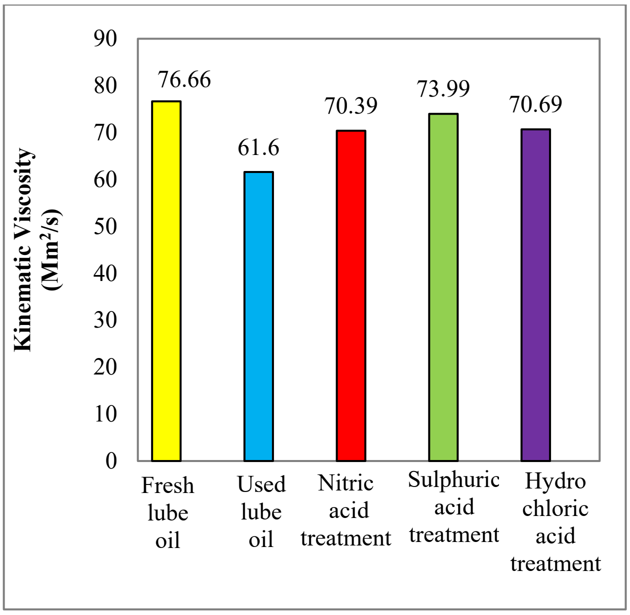

Fig. 4: Kinematic viscosity of the fresh used and treated lubricating oil.

Fig. 4 shows that the kinematic viscosity of the fresh and waste lubricating oils were 76.66 and 61.60 Mm^2/s, respectively, while those treated lube oils were 70.39 Mm^2/s (nitric acid), 73.99 Mm^2/s (sulphuric acid), and 70.98 Mm^2/s (hydrochloric acid). The decrease in kinematic viscosity of the waste lubricating oil is due to contamination in the form of sludge in the waste lubricating oil.

However, treatment with the acids has restored most of its viscosity, with sulphuric acid showing the best performance followed by nitric acid and hydrochloric acid. This can be attributed to the possible conversion of possible contaminants by the acid and removal by the filtration from the lubricating oil (Mohammed et al., 2013).

3.2.4 Pour point

Pour point is important when considering oil under cold weather or condition. A higher pour point of waste lubricating oil is caused by the degradation of additives. Results shown in Fig. 5 indicate that the refined lubricating oil retained good pour point and has the potential for reuse. From the Fig. 5, the results for pour point of fresh and waste lubricating oils are −8 °C and −34 °C, respectively, while those treated oils obtained by the treatments with acid were −13 °C for treating with nitric acid, −10.5 °C for sulphuric acid treatment, and −16 °C for hydrochloric acid treatment.

From the results obtained for the waste oil, pour point for the waste lubricating oil is higher as compared to others, this might due to the degradation of additive in the lubricating oil. Pour points will vary widely depending on the base, the source of the lubricating oil and the method of refining, especially if dewaxing has been done (Awaja & Pavel, 2006; *Chevron Lubricating Oil*, 2008). Here, the treatment of various acid treatments proved good when compared with the fresh lubricating oil.

3.2.5 Cloud point

When the lubricating oil is cooled slowly, the temperature at which it turns cloudy is called its cloud point meanwhile the temperature at which the lubricant oil ceases to flow or pour is called its pour point. Cloud and pour points indicate the suitability of lubricant oil in cold conditions (Udonne, 2011). Lubricant oil waste in a working environment at low temperatures should possess a low pour point, otherwise solidification of lubricating oil could cause jamming of machine or engine. (Abu-Elella et al., 2015) The presence of waxes in the lubricating oil could increase pour point. Fig. 6 shows the results for cloud point of fresh and waste lubricating oils are −13.5 °C and −37 °C, respectively, while those treated oils obtained by the acid treatments were −17.5 °C for treating with nitric acid, −15 °C for Sulphuric acid treatment, and −19.5°C for hydrochloric acid treatment.

From the results obtained, pour point for the lubricating oil treated with acid is lower as compared to the waste lubricating oil however slightly higher as compared to fresh lubricating oil. The higher waste lubricating oil is due to the degradation of additives in the lubricating oil. However, the decrease in cloud point for the acid that is treated with acid shows the lubricating

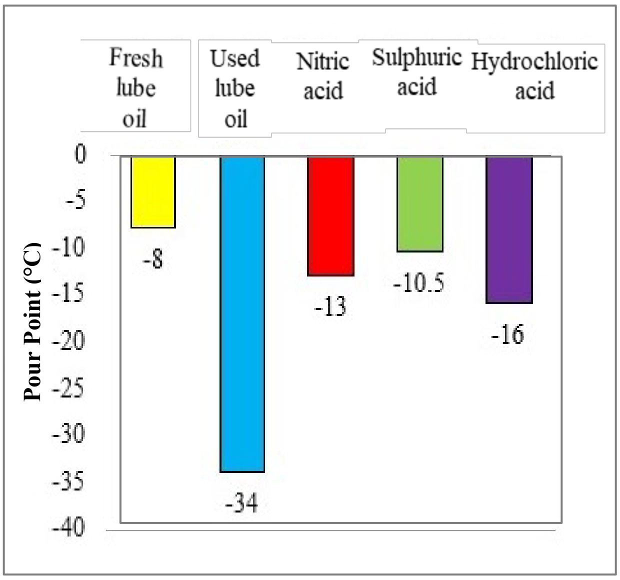

Fig. 5: Effect of refining of waste lubricating oil on pour point

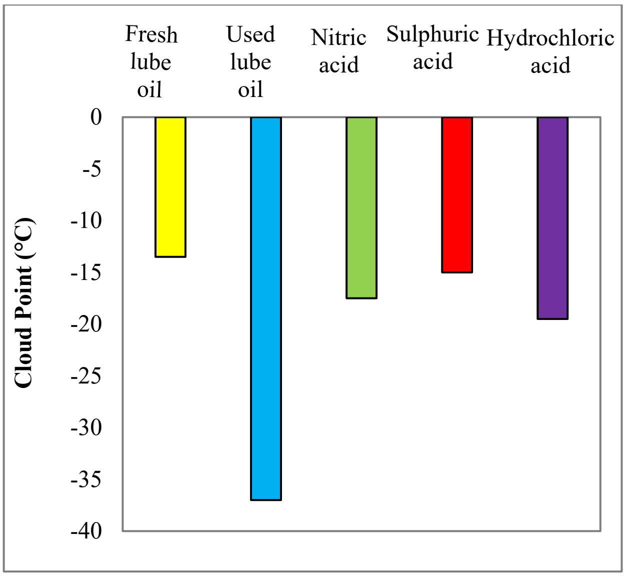

Fig. 6: Effect of refining of waste lubricating oil on cloud point

oil is almost back to its quality and the amount of degradation of additives that are present in the waste lubricating oil is reduced.

3.2.6 Ash content

The purpose of doing the sulfated ash testing is to show the concentration of known metal-containing additives. The increase in the percentage of sulfated ash is due to the presence of contaminants and the degradation of additives. These additives characteristically contain one or more of the following metals such as sodium, barium, magnesium, calcium, zinc, potassium, and tin figure 7, the results for ash content of fresh and waste lubricating oils are 0.12% and 2.3% respectively, while those treated oils obtained by the acid treatments were 0.32% for treating with Nitric acid, 0.26% for Sulphuric acid treatment, and 0.41% for hydrochloric acid treatment.

The ash content for the lubricating oil treated with acid is lower as compared to the waste lubricating oil however slightly higher as compared to fresh lubricating oil. The higher waste lubricating oil is due to the degradation of additives in the lubricating oil. It has been considerably decreased as compared to the waste lubricating oil sample (2. 3%). All the treated lubricating oil is within the range even though it slightly differs from the fresh lubricating oil. The percentages of ash content of lubricating oil treated with sulphuric acid are the lowest as compared to the lubricating oil treated with Hydrochloric acid and Nitric acid, this shows the lubricating oil treated with sulphuric acid contains less concentration of metal containing additives and contains less contaminant and degradation of additives. This can be attributed to the possible conversion of possible contaminants by the acid and removal by the filtration from the lubricating oil degradation of additives. This can be attributed to the possible conversion of possible contaminants by the acid and removal by the filtration from the lubricating oil

3.3 Characteristics of waste and re-refined lubricating oils: the effect of various ratios of sulphuric acid

Various ratios of sulphuric acid were used to treat the waste lubricating oil. Similar to the previous study, several properties were analyzed such as specific gravity, kinematic viscosity, ash content, etc.The result is shown in Table 2.

3.3.1 Effect of different sulphuric acid ratios toward colour of waste lubricant oil

Fig. 8 shows the results for the waste lubricating oil treated with various sulphuric acid ratios ranging from 10 to 20%, from left: (a) lubricating oil treated with 10% of sulphuric acid, (b) lubricating oil treated with 15% of sulphuric acid and (c) lubricating oil treated with 20% of sulphuric acid. The colour difference was influenced by chemical composition and contaminant that may be present in the lubricating oil, the darker the colour, the higher the contaminant. Lubricating oil treated with 20 % sulphuric acid showed light yellow, which was the lightest compared to lubricating oil that was treated with 10% or 15% of sulphuric acid. This shows the higher the concentration of acid, the higher the contaminant can be eliminated.

Table 2: Sulphuric acid treatment analysis results

Parameter	Waste lubricating oil	Fresh lubricating oil	10% sulphuric acid	15% sulphuric acid	20% sulphuric acid	Standard range
Colour	Black	Light yellow	Yellow	Yellow	Light yellow	-
Specific gravity (g/cm^3)	0.92	0.89	0.88	0.87	0.87	0.86–0.89
Kinematic viscosity (Mm^2/s)	61.60	76.66	73.99	74.03	74.57	-
Dynamic viscosity (mPa·s)	56.68	68.23	65.11	63.67	64.87	-
Pour point (°C)	−34	−8	−11.5	−11	−10	-
Cloud point (°C)	−37	−13.5	−16	−17	−15	-
Ash content (%)	2.3	0.12	0.26	0.24	0.21	<1.0%

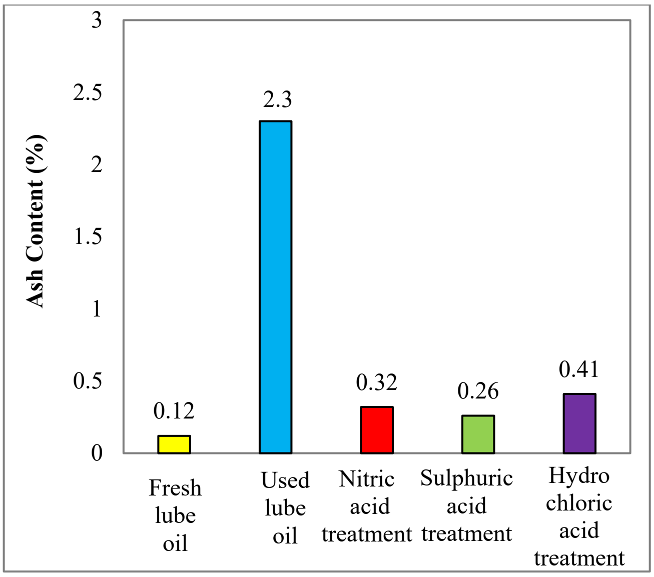

Fig. 7: Effect of refining of waste lubricating oil on ash content.

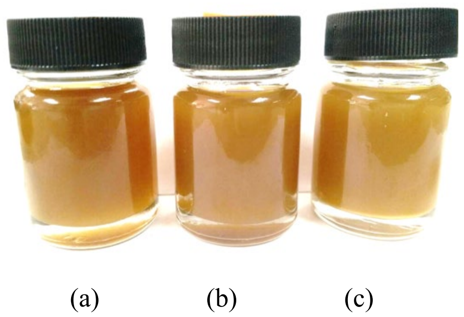

Fig. 8: Effect of sulphuric acid on the colour of the refined waste lubricating oil. (a) lubricating oil treated with 10% of sulphuric acid, (b) lubricating oil treated with 15% of sulphuric acid and (c) lubricating oil treated with 20% of sulphuric acid.

3.3.2 Effect of different sulphuric acid ratios toward Specific gravity

Specific gravity is one of important properties of lubricating oil to be analyzed. This is because it is influenced by the chemical composition of the oil. As for used lubricant oil, the specific gravity is depending on the type of its contaminated (Mohammed et al., 2013). The effect of treatment of waste/used lubricating oil specific gravity by acids is shown in Fig. 9.

Fresh and treated waste lubricating oil using various sulphuric ratios showed similar specific gravity, with sulphuric treated waste lubricating oil showing lower the specific gravity (Fig. 9), suggesting a higher number of saturated compounds present in the treated lubricating oil (Chevron Lubricating Oil, 2008;Diphare et al., 2013; Abu-Elella et al., 2015). Results showed that the lubricating oil treated with 10% of sulphuric acid had the closest specific gravity to the fresh lubricating oil, indicating that the lubricating oil contained less aromatic compound compared to other ratios of sulphuric acid.

3.3.3 Effect of different sulphuric acid ratios toward Dynamic viscosity

Dynamic viscosity of the treated waste lubricating oil was 65.11 mPa.s (10% sulphuric acid), 64.41 mPa.s (15% sulphuric acid), and 64.87 mPa.s (20% sulphuric acid), which were close to the fresh lubricating oil of 68.23 mPa.s as depicted in Fig. 10. Results showed that the waste lubricating oil has lost most of its viscosity due to contamination. However, treating the waste lubricating oil with acids has brought back most of its viscosity with 10% sulphuric acid treatment giving the highest viscosity. This can be attributed to the possible conversion of contaminants by the acid.

Because of the attraction of the lubricating oil to act as a coolant or heat transfer medium, it must be able to retain adequate body at elevated film temperature, and a viscosity above 80 ensured suitable fluidity elsewhere in the system (Udonne, 2010). Oil that is treated with different concentrations of acids meets this specification, with the ratio of 10% sulphuric acid showing an advantage over others since it has the nearest viscosity to the fresh lubricating oil.

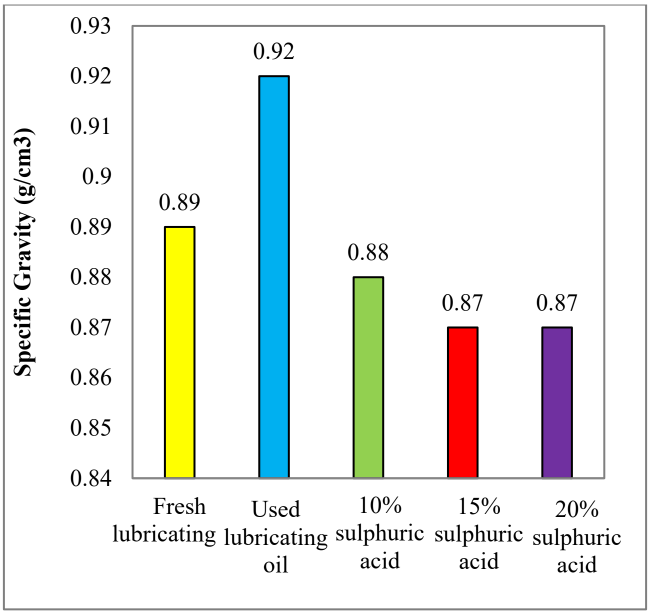

Fig. 9: Specific gravity of treated waste lubricating oil using sulphuric acid in different ratios.

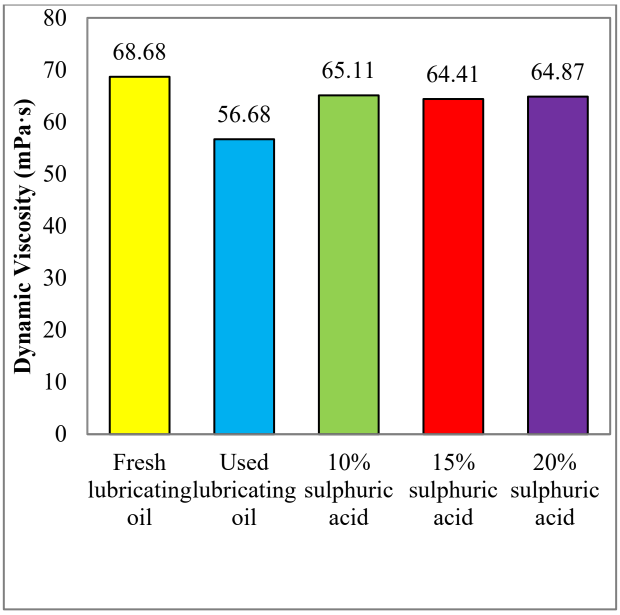

Fig. 10: Dynamic viscosity of the fresh, used and refined waste lubricating oil treated by different sulphuric acid concentrations

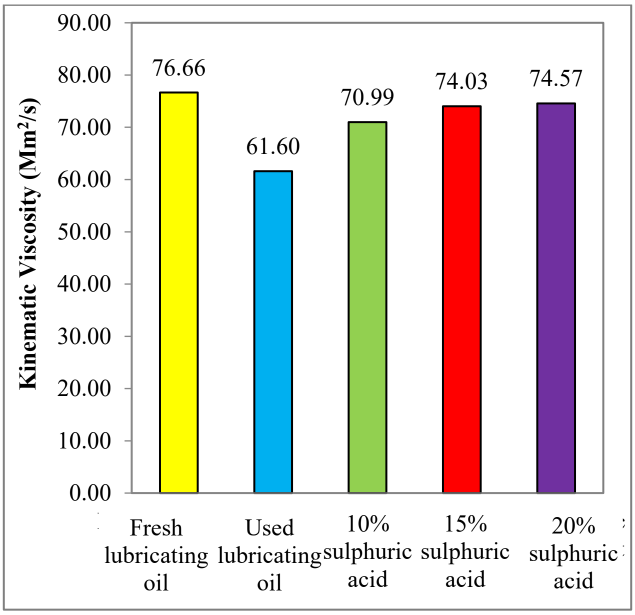

Fig. 11: Kinematic viscosity of fresh, used and refined waste lubricating oil treated by sulphuric acid

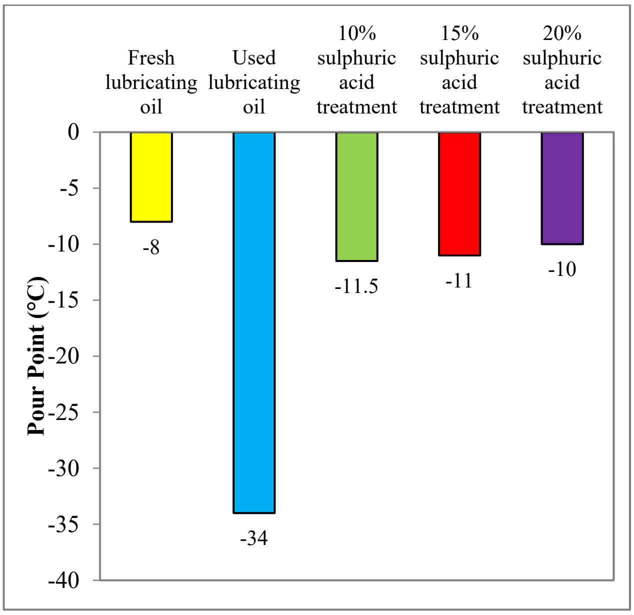

Fig. 12: Effect of different ratios of sulphuric acid on pour point of refined waste lubricating oil

3.3.4 *Effect of different sulphuric acid ratios on Kinematic viscosity*

The study on this effect was carryout using sulphuric acid ratios of 10 to 20% from the waste lubricant oil. Treated lubricating oils using various ratios of sulphuric acid were 70.99 Mm^2/s (10% sulphuric acid), 74.03 Mm^2/s (15% sulphuric acid), and 74.57 Mm^2/s (20% sulphuric acid), that is about 3 to 7% lower than the fresh lubricating oil as shown in Fig. 11. A decrease in kinematic viscosity of the waste lubricating oil was due to the contamination in the form of sludge in the waste oil. The result showed that refining using 20% of sulphuric acid gave the highest viscosity followed by 15% and 10% concentrations. This can be attributed to the possible conversion of possible contaminants by the acid and removal by the filtration from the lubricating oil.

3.3.5 *Effect of different sulphuric acid ratios toward Pourpoint*

The higher pour point of waste lubricating oil was caused by the degradation of additives. Fig. 12 showed that the cloud points of the fresh and waste lubricating oils were −8 °C and −34 °C, respectively, while those treated lubricating oils were −11.5 °C (10% sulphuric acid), −11°C (20% sulphuric acid), and −10 °C (20% sulphuric acid). The refined lubricating oil showed a

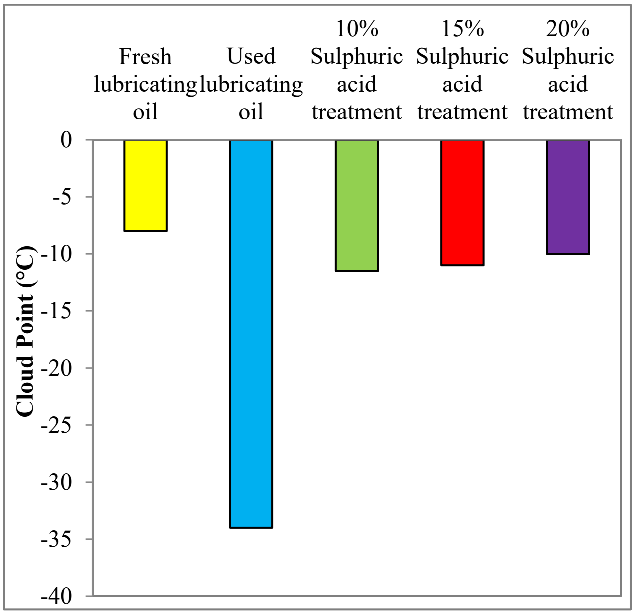

Fig.13: Effect of sulphuric acid concentration on cloud point of used lubricating oil.

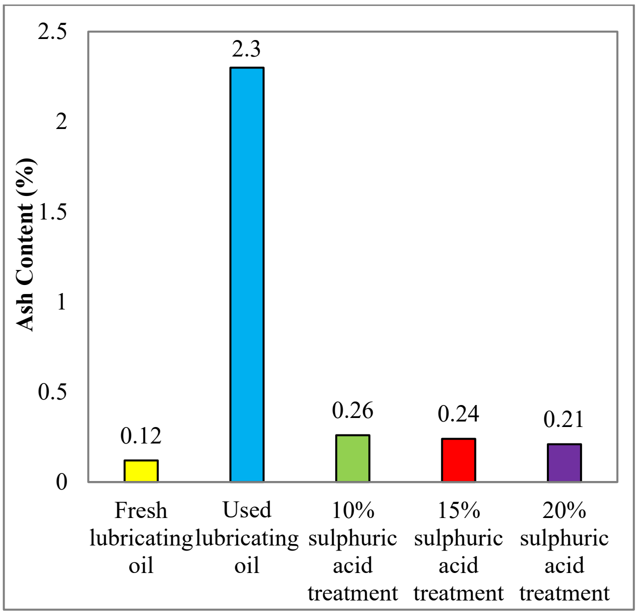

Fig. 14: Effect of refining of different ratios of sulphuric acid on ash content of used lubricating o

good pour point that can still be reused while used lube oil showed a high pour point due to the degradation of additives. Pour point is of special interest when an oil must be under relatively cold conditions. Pour point varies widely depending on the base, the source of the lubricating oil and the method of refining, especially if dewaxing has been done (Awaja & Pavel, 2006; Chevron Lubricating Oil, 2008).

3.3.6 Effect of different sulphuric acid ratios toward Cloud point

When the lubricating oil is cooled slowly, the temperature it turns cloudy is called its cloud point. Cloud and pour points indicate the suitability of lubricant oil in cold conditions. Lubricant oil in a working environment at low temperatures should possess low pour point; otherwise solidification of lubricating oil could cause jamming of machine or engine. The presence of waxes in the lubricating oil also contributes to an increase in pour points.

Fig. 13 shows the cloud point of fresh and waste lubricating oils were −8 and −34 °C, respectively, while those treated lubricating oils were −11.5 °C (10% sulphuric acid), −11 °C (20% sulphuric acid), and −10 °C (20% sulphuric acid). Treated used lubricating oils showed significantly lower pour points compared to the waste lubricating oil, indicating lesser amount of degraded additives in the treated waste lubricating oil and may have similar performance as the fresh lube oil.

3.3.7 Effect of different sulphuric acid ratios on Ash content

Ash content in lube oil can be from oil or water-soluble metallic compounds or extraneous solids such as dirt and rust (Mohammed et al., 2013). In this study The ash content of waste lubricating oil was analyzed and compared with fresh lubricant oil.

The ash content of fresh and waste lubricating oils was found at 0.12% and 2.3%, respectively, while those acid treated lubricating oils were 0.26% (10% sulphuric acid), 0.24% (15% sulphuric acid), and 0.21% (20% sulphuric acid) as shown in Fig. 14. All the treated lubricating oil showed significantly lower ash content compared to the untreated used lubricating oil with 20% sulphuric acid showing the lowest, indicating that the waste lubricating oil treated with 20% sulphuric acid contained less amount of degraded metal containing additives and other contaminants. This can be attributed to the possible conversion of contaminants by the acid and removal by the filtration during the treatment process. Sulfur and chlorine do not affect, however when phosphorus is co-exist with metals, it remains incompletely or completely in the sulfated ash as metal phosphates. On the other hand, the increase in the percentage of sulfated ash is due to the presence of contaminants and the degradation of additives. These additives characteristically contain one or more of the following metals such as sodium, barium, magnesium, calcium, zinc, potassium, and tin. In addition, the elements such as phosphorus, sulfur, and chlorine might also be present in a combined form (Mekonnen, 2014).

4.0 Conclusions

The conducted study showed that treatment using sulphuric acid, nitric acid and hydrochloric acid were effective in removing the slug from the waste lubricating oil and returning the oil to its quality form. Overall, sulphuric acid outperformed nitric acid and hydrochloric acid in refining the waste lubricating oil. Optimum ratio of sulphuric acid for recycling used lubricating oil was 20%. The treated lube oil was of comparable quality to the fresh lube oil, indicating efficacy in reducing contaminants in the waste lubricating oil. Successful recycling of waste lubricating oil can minimise pollution and reduce environmental and health risks associated with the irresponsible disposal of the used lube oil.

Acknowledgment

Authors would like to acknowledge all parties who have contributed to this work. This research was granted by the Universiti Teknologi MARA through grants GIP (600-IRMI 5/3/GIP (070/2019)) and GPK (600-IRMI/GPK 5/3 (153/2020)) provided by the Universiti Teknologi MARA.

References

Abu-Elella, R., Ossman, M. E., Farouq, R., & Abd-Elfatah, M. (2015). Used motor oil treatment: turning waste oil into valuable products. *International Journal of Chemical and Biochemical Sciences, 7*, 57–67.

Awaja, F., & Pavel, D. (2006). *Design Aspects of Used Lubricating Oil Re-Refining - Firas Awaja, Dumitru Pavel.* https://books.google.com.my/books?hl=en&lr=&id=QNqnVyKYeVAC&oi=fnd&pg=PP1&dq=Firas+and+Dumitru,+2006&ots=ulOxvzuwzK&sig=vDjpcp4L1Z322eyDOKCrC_GQbMI#v=onepage&q=Firas and Dumitru%2C 2006&f=false

Botas, J. A., Moreno, J., Espada, J. J., Serrano, D. P., & Dufour, J. (2017). Recycling of used lubricating oil: Evaluation of environmental and energy performance by LCA. *Resources, Conservation and Recycling*, *125*, 315–323. https://doi.org/https://doi.org/10.1016/j.resconrec.2017.07.010

Yang, C. (PhD). (December,2008). The Feasibility Studies on Sonochemical Processes for Treating Used Oil: Toxin Reduction for Eliminating Recycle Interference. [California, University of Southern California]. https://doi.org/10.25549/usctheses-m1917

Chevron Lubricating Oil. (2008). https://whlubricants.com/files/products/lubeoil-fm.pdf

Commission, E. (2014). Commission Decision of 18 December 2014 amending Decision 2000/532/EC on the list of waste pursuant to Directive 2008/98/EC of the European Parliament and of the Council. *Official Journal of the European Union*, *7*(440), 44–86. https://www.sepa.org.uk/media/162640/decision-2014-955-eu-list-of-waste.pdf%5Cnhttp://eur-lex.europa.eu/legal-content/EN/TXT/PDF/?uri=CELEX:32014D0955&from=EN%5Cnhttp://www.ecolex.org/details/legislation/commission-decision-2014955eu-amending-decision-200

Diphare, M. J., Muzenda, E., Pilusa, T. J., & Mollagee, M. (2013). *A Comparison of Waste Lubricating Oil Treatment Techniques*. *2028*, 106–109.

Eman A. Emam & Abeer M. Shoaib. (2012). Re-refining of used lubricating oil, ii- by solvent/clay and acid/clay-percolation processes. *ARPN Journal of Science and Technology,* 1034–1041

EU. (2014). Commission regulation No 1357/2014 of 18 December 2014 replacing Annex III to Directive 2008/98/EC of the European Parliament and of the Council on waste and repealing certain Directives. *Official Journal of the European Union*, *L 365*(89).

Isah, A. G., Abdulkadir, M., Onifade, K. R., Musa, U., Garba, M. U., Bawa, A. A., & Sani, Y. (2013, July 35–5). *Regeneration of used engine oil.* [Conference presentation]. The World Congress on Engineering (WCE), London, United Kingdom.

Jafari, A. J., & Hassanpour, M. (2015). Analysis and comparison of used lubricants, regenerative technologies in the world. *Resources, Conservation & Recycling*, *103*, 179–191. https://doi.org/10.1016/j.resconrec.2015.07.026

Maceiras, R., Alfonsín, V., & Morales, F. J. (2017). Recycling of waste engine oil for diesel production. *Waste Management*, *60*, 351–356. https://doi.org/https://doi.org/10.1016/j.wasman.2016.08.009

Mekonnen, H. A. (2014). Recycling of used lubricating oil using acid-clay treatment process recycling of used lubricating oil using acid-clay treatment process. [Addis Ababa University].

Mohammed, R. R., Ibrahim, I. A. R., Taha, A. H., & McKay, G. (2013). Waste lubricating oil treatment by extraction and adsorption. *Chemical Engineering Journal*, *220*, 343–351. https://doi.org/10.1016/J.CEJ.2012.12.076

Osman, D. I., Attia, S. K., & Taman, A. R. (2018). Recycling of used engine oil by different solvent. *Egyptian Journal of Petroleum*, *27*, 221–225. https://doi.org/10.1016/j.ejpe.2017.05.010

Parekh, K., Shahabuddin, S., Gaur, R., & Dave, N. (2022). Prospects of conducting polymer as an adsorbent for used lubricant oil reclamation. *Materials Today: Proceedings Journal*. *Article in Press.* https://doi.org/10.1016/j.matpr.2022.01.130

Pinheiro, C. T., Pais, R. F., Ferreira, A. G. M., Quina, M. J., & Gando-Ferreira, L. M. (2018). Measurement and correlation of thermophysical properties of waste lubricant oil. *The Journal of Chemical Thermodynamics*, *116*, 137–146. https://doi.org/10.1016/j.jct.2017.08.039

Scapin, M. A., Duarte, C., Sampa, M. H. O., & Sato, I. M. (2007). Recycling of the used automotive lubricating oil by ionizing radiation process. *Radiation Physics and Chemistry*, *76*(11–12), 1899–1902. https://doi.org/10.1016/J.RADPHYSCHEM.2007.03.017 .

Shri Kannan C, Mohan Kumar KS, Sakeer Hussain M, Deepa Priya N and Saravanan K (2014). Studies on reuse of re-refined used automotive lubricating oil. *Research Journal of Engineering Sciences, 3*(6), 8–14.

Udonne, J. D. (2011). A comparative study of recycling of used lubrication Oils using distillation , acid and activated charcoal with clay methods. *Journal of Petroleum and Gas Engineering, 2* (2), 12–19. https://doi.org/10.5897/JPGE.9000001

Zgheib, N., & Takache, H. (2020). Recycling of used lubricating oil by solvent extraction: experimental results, Aspen Plus simulation and feasibility study. *Clean Technologies and Environmental Policy 2020 23:1*, *23*(1), 65–76. https://doi.org/10.1007/S10098-020-01893-0