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Purification of Used Lubricating Oils Using Vacuum Distillation

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Abstract

Engine oil (EO) is produced by mixing base oil derivatives from crude oil with chemical additives to the lubricity of moving parts and reduce the friction inside the engine. Used lubricating oil (ULO) is one of the hazardous materials that consists of pollution harmful to the environment, it needs to be managed properly. In this work, vacuum distillation technique is used to recycle used lubricating oil. Used lubricating oil samples from two different brands of diesel engine oil (20w-50) and gasoline engine oil (10W-30) are used in this study. Various properties of ULO and recycled oil were characterized such as kinematic viscosity, viscosity index, density, pour point, flash point, Sulphur content, and Fourier transform Infrared spectroscopy FTIR. The yield recycles for ULO of gasoline engines, diesel engines, and mix (gasoline and diesel) by vacuum process were 85%, 74%, and 75% respectively, it was discovered that the sulfur component decreased from 9792.3 ppm of ULO to 405 ppm of yield distillates. The pour point results show an increase from -30 °C of used lubricating oil to -18 and -6 for distillates cut for vacuum distillation, compared to the pour point of Iraqi base oil 40 and 60 Stook (SN150 and SN200) -18 °C and -6 °C.

Keywords: Used oil, engine oil, viscosity index, flash point, base oil, vacuum distillation.

تنقية زيوت التزيت المستعملة باستخدام التقطير الفراغي

الخلاصة:

يتم إنتاج زيت المحرك (EO) عن طريق خلط مشتقات الزيت الأساسي من الزيت الخام مع إضافات كيميائية لتزيت الأجزاء المتحركة وتقليل الاحتكاك داخل المحرك. تعتبر زيوت التزيت المستعملة (ULO) من المواد الخطرة التي تتكون من مواد ملوثة ضارة بالبيئة، ويجب إدارتها بشكل صحيح. في هذا العمل، يتم استخدام تقنية التقطير الفراغي لإعادة تدوير زيوت التزيت المستعملة. تم استخدام عينات زيت التزيت المستخدمة من علامتين تجاريتين مختلفتين لزيت محرك الديزل (20w-50) وزيت محرك البنزين (10W-30) في هذه الدراسة. تم تشخيص الخصائص المختلفة لـ ULO والزيت المعاد تدويره مثل اللزوجة الحركية، مؤشر اللزوجة، الكثافة، نقطة الانسكاب، نقطة الوميض، محتوى الكبريت، ومطياف الأشعة تحت الحمراء FTIR. بلغت نسبة إعادة التدوير لـ ULO لمحركات البنزين ومحركات الديزل والمزيج (البنزين والديزل) بعملية التفريغ 85%، 74%، و75% على التوالي، وقد لوحظ أن عنصر الكبريت انخفض من 9792.3 جزء في المليون

من ULO إلى 405 جزء في المليون. من نواتج التقطير العائد، تظهر نتائج نقطة الانسكاب زيادة من -30 درجة مئوية لزيت التزييت المستخدمة إلى -18 و-6 لنواتج التقطير للتقطير الفراغي، مقارنة بنقطة الانسكاب الزيت الأساسي العراقي 40 و60 ستوك (SN150) و (SN200) -18 درجة مئوية و -6 درجة مئوية.

1. Introduction:

The high lubricity of lubricating oil reduces the contact surfaces and lowers wear, friction, and energy losses. Moreover, lubricants are commonly used in engines to minimize friction because lubricants perform essential functions such as lubricating, cooling, and protecting metal surfaces from corrosion damage. Lubricating oils are made up of up to 80% of base oil stocks, which give the lubricating oil its viscosity, stability, pour point and flash point, and additives to enhance these properties. Base oil is commonly defined as oil derived from crude oil refining (mineral base oil) or chemical synthesis oil (synthetic base oil) with a boiling point of 300 °C to 565° C and containing hydrocarbons ranging from C18 to C40. [1]- [4]. The crude oil origin and production process affect the chemical compositions of lubricant oil. Blending base oil with a few per cent of polymers controls the technical properties of the oil. Furthermore, additives are used in lubricating oil to neutralize the acids that produce during the degradation and decrease corrosion [5], [6]. The Lubricating oil molecule is classified into three types: paraffinic, naphthenic, and aromatic. Paraffinic types have mostly straight chains, are waxy, have a high pour point, have good viscosity, and are more temperature stable (high viscosity index), thus leading to improved performance in several applications such as automotive engine oils. Naphthenic oil is polycyclic hydrocarbons with a saturated straight chain. They perform better at low temperatures than paraffinic oil and typically have a low pour point. However, it has less stability than paraffin oil and is more disposed to oxidation. Aromatic oils are components that have an aromatic ring with or without a saturated side chain. They have a high degree of polarity. Their main disadvantage is their lack of stability. Indeed, removing aromatics from paraffinic and naphthenic oil is a portion of the refining process in base oil production. As a result, the aromatic is referred to as aromatic extract [7], [8].

Engine oil is very important because it is considered one factor that ensures the proper operation of an automobile, which plays such a large role in daily life. The lubricating oil of the automotive engine is produced by adding suitable additives to base oil that is produced from paraffin or mixed-base crude oils. The lubricating oil plays a crucial role

in reducing friction and wear, carrying away heat and removing pollutants such as metals, carbon residue, ash, gums, and varnish. Because most of these pollutants are highly toxic, they must be removed before the engine oil can be reused. Besides, the engine oil acts as a coolant in some of the engine parts, keeping the lubricated components clean by avoiding clogs and sealing possible clearances, and providing corrosion protection [9]-[11]. In the long-term operation of the engine oil after several kilometers inside the engine, a build-up of temperature occurs, leading to oil cracking and losing its characterizations quality, including pour point, flash point, density, viscosity, etc. [12]. Used lubricating oil (ULO) is a toxic component that is dangerous to the environment and human health due to the existence of contaminants, that lead to excessive wear, iron rust, occlusion of oil channels, also the impurities harm oil pumps, dilute the oil, and decrease the viscosity to critical levels [13], [14]. Chemical changes and pollution buildup increase with oil use and are dependent on the base oil origin, refining process, package of additives, application type, operation time, and other factors [15]. Engine oil additives are used to prevent undesired properties. The most common additives of engine oil are anti-oxidation, pour point depressants, coloring agents, anti-corrosion agents etc. Because of the high cost of crude oil, countries are attempting to find alternative fuel production methods.

As a result, there is a great opportunity to produce fuel from ULO, which may aid to decrease the environmental problems, and save ULO removal costs [16] The recycling of ULO is primarily determined by the nature of the oil base stock as well as the nature and number of impurities in the lubricating oil as a result of the operations [17]. Automotive Engine oil should change after a few thousand kilometers of driving as a result of the strain caused by a significant deterioration in service. Annually, approximately 1.7 to 3.5 million tons of lubricating oils are collected in Europe and the United States. This large amount of waste engine oil has a significant economic and environmental impact [18]. Poor management of the improper disposal of ULO can negatively impact the environment. Therefore, recycling ULO is important in this regard; it requires less energy and costs than refining crude oil, and it aids in the reduction of air, land, and water pollution in the environment. Experts believe that reusing used lubricating oil generated by consumers is the best option [19]. Around 60 to 80 % of base

oil can be produced from the ton of used lubricating oil compared to 20% production from the same amount of crude oil [20].

The recycling of ULO to base feedstock or fuel oil (FO) is a suitable path to preserve the environment from pollution waste. For the used lubricating oil recycling, various extraction processes have been described such as re-distilling and re-refining processes, ionization radiation, and solvent extraction treatment methods [14]. In developed countries, the re-distilling procedure is widely used. It includes recycling wasted oil through the distillation process into high-quality lubricants and other petroleum products. Thin-film vacuum distillation or a traditional vacuum column, where atmospheric dehydration is the first step in the vacuum distillation VD and hydrogenation process, which removes the light hydrocarbons and water. The VD process is carried out at a temperature of around 250°C- 350 °C and the last stage is the hydrogenation of ULO to remove the toxic materials such as nitrogen and Sulphur. The VD process residue can be used to make or add to asphalt used for roads and roofs [10], [19], [21]. For a long time, the acid-clay method has been used as a recycling process for ULO, where sulfuric acid is added to remove the asphaltic components, and natural clay that has been activated and calcined is used as an adsorbent in the recycling of used oils to remove the black color and odor of ULO.

This method is no longer recommended because it produces a high amount of hazardous waste, is toxic and difficult to manage and remove asphaltene contaminants, [10], [22], [23]. The solvent extraction method has replaced acid-clay treatment as the dominant way of enhancing base oil oxidative stability, viscosity, and temperature properties. The base oil produced through Solvent Extractions of high quality and contains fewer impurities. Solvent extraction has been done with a variety of solvents, including 2-propanol, 1-butanol, methyl ethyl ketone (MEK), ethanol, toluene, acetone, propane, and others. The solvent selectively dissolves the undesirable aromatic components (extracts), while leaving the desired saturated products, particularly alkanes (raffinates) [24], [25]. The process used of treatment lubricating oil by vacuum distillation/thin wiped film evaporation technique consists of dehydration, gas oil evaporation, thin wiped film evaporation and clay treatment. In this study improved that this technique is a useful process to produce base oils, grade SN-150 and SN-200 from local used lubricating oil

in Iraq, some technique used for characterization of products such as FTIR, viscosity, and sulfur content [26].

Bamiji Z. Adewole et al. (2019) [27] explores the application of solvent extraction technology in recycling used automotive lubricating oils and assesses the viability of using the reclaimed oil again in automotive engines, where examining and comparing the characteristics of the reclaimed oil samples with the SAE specifications. It was observed that the flash point of the reclaimed oil was below the specified values for SAE20, SAE30, and SAE40. Additionally, the viscosity index of the reclaimed oil exceeded the SAE specifications. On the other hand, the pour point of the reclaimed oil was lower than the standard values. Furthermore, the viscosity of the reclaimed oil at 40 and 100 °C was higher than the values set by the society for automotive engineers.

Armioni et al. (2020) [10] analyzed three sets of experimental data from various sources to compare and illustrate the differences between them and their impact on motor oil properties. Each technique used in the experiments has its own advantages and disadvantages. The acid/clay method, although once used, is now discouraged globally due to its production of toxic waste. However, other technologies such as solvent extraction and vacuum distillation are being industrially developed in different countries and are continuously being improved.

The main objective of this work is the recycling of used motor oils in order to protect the environment from the dangers of pollutants that result in improper disposal of these oils and to study the possibility of maximum benefit from them in obtaining oil products through processing them instead of disposing of them in irregular ways. Table (1). show the advantage and disadvantage of ULO techniques.

Table (1): Advantages and disadvantages of ULO techniques [10].

Recycling technique	Advantages	Disadvantages
Acid/clay	Historically proven efficiency; Lower cost of production; Simple process, easy to apply.	It generates acid sludge, which is considered hazardous waste; Causes corrosion of equipment and reduces its life; Low oil recovery
Solvent extraction	Recyclable solvent; Low to minimum waste generated; Good base oils recovery.	Economical only for high capacity plants; It requires highly skilled operators.
Vacuum distillation	Suitable for high capacity plants; Environmentally friendly process; Produces good quality base oils.	High capital investment required; Due to sophisticated equipment, highly skilled operators are necessary.

2. Material and Methods

1.1 Used lubricating oil

Three types of used lubricating oil were collected from vehicles after 2500 and 3000 kilometers of use for diesel engine oil (20w-50) and gasoline engine oil (10W-30), respectively. The main properties of used lubricating oil samples as shown in Table (2).

Table (2): Properties of used oil

Properties of used oil	Sample1 (Gasoline engine oil)	Sample 2 (Diesel engine oil)	Sample 3 (Mixed)
Viscosity @40 °C (cSt)	91.157	203	121.83
Viscosity @100 °C (cSt)	13.611	19.65	15.183
Viscosity index	151.3	110.7	129.2
Pour Pint (°C)	-36	-30	-33
Flash Point (°C)	225	227	230
Density (g/cm ³)	0.892	0.907	0.885
Sulfur Content (ppm)	9792.3	7850.7	4049
Oxidation (A/cm)	12.5	11.5	11.2

3. Method

In this method, the used lubricating oil was collected from different vehicles (cars, buses) and transferred to an appropriate tank. The first step is to filter used lubricating oil using a funnel with filter paper inside, which was then connected to a vacuum pump; this process was used to remove solids from the used oil, and then dehydrated at a temperature of 100 °C under atmospheric pressure to eliminate the water. The following steps are involved in the re-refining of used lubricating oil (ULO). Then 2.25 L was subjected to a vacuum distillation device (ASTM D 5236) as shown in Figure (1). The light gasoil was obtained at a temperature range of 300-350 °C while base oil cuts were distillate at a temperature range of 400-550 °C vacuum pressure 1-0.1 torr. as shown in Figure (2), and the bottom product (residue) was collected at a temperature above 570 °C. Table (3) shows the temperature and yields of the distillates for each cut.

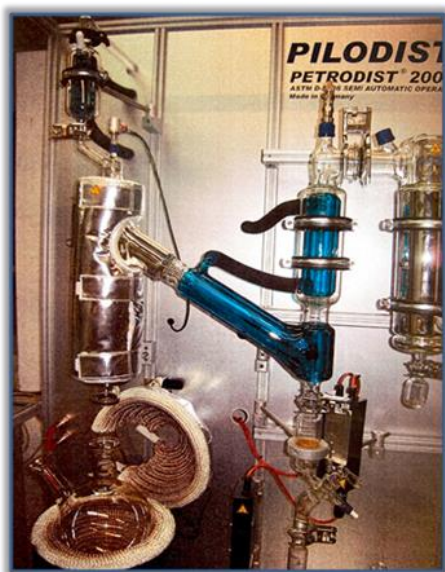


Fig. (1): Vacuum distillation unit

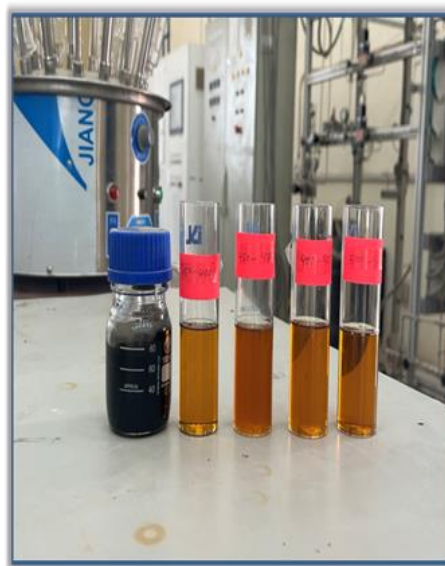


Fig. (2): Pictures of ULO and distillates cut

Table (3): Temperatures and yields of the vacuum distillate cuts

Temp ULO	300-350	350-400	400-425	425-475	475-500	500-525	525-550	550-575	Residue
Gasoline Engine Used lubricating oil yield %	0.24	0.79	2.22	23.47	32.89	19.11	7.56	-	13.33
Diesel Engine Used lubricating oil yield %	-	-	2.54	5.8	8.48	11.61	19.2	25.89	25.89
Mix (Gasoline + Diesel engine used oil) yield %	-	1.053	3.89	8.383	19.101	23.95	18.56	-	23.9521

4. Results and Discussion

4.1 Specific gravity

After treated used lubricating oil (UO) samples, changes in the specific gravity values were observed as shown in Figure (3).

Depending on what was obtained from the experimental results, it was noticed that the specific gravity of the used lubricating oil (UO) has increased compared to the new and base oil. The specific gravity of the used oil was (0.892, 0.907) g/cm³ for gasoline, and diesel engine respectively. It can be said that the reasons behind these results are that the increase in the number of aromatic compounds in the oil results in an increase the specific gravity, while an increase the saturated compounds results in a decrease in the specific gravity also the used lubricating oil contained solids materials, which led to a high density [28]. The other reason for the high density is due to the presence of the sulfur compound, whose percentage increases significantly in the used lubricating oil compared to the new oil as well as the treated oil [29].

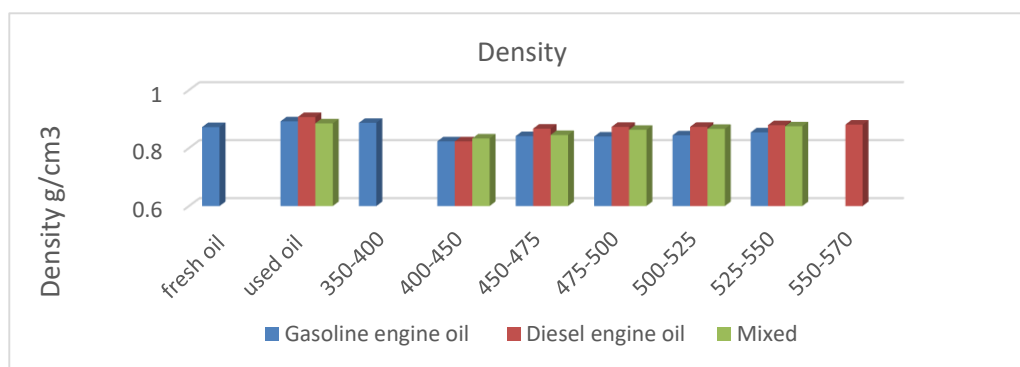


Fig. (3): The density of fresh oil, used oil, and distillates cut

4.2 Kinematic viscosity and viscosity index

Viscosity is an important characteristic and one of the first determinations of type of engine oil. It is considered the most noticeable characteristic of the used oil. Figures (4 and 5) show the obtained viscosity results at (40 and 100) °C for different distilled cuts, the viscosity values increased with the increase in distillation temperature. The decrease or increase in the viscosity of the used lubricating oil depends on different reasons. The reason behind the increasing in kinematic viscosity is due to oxidation or contamination, this is clear in the samples of used oils for diesel engines [11], [26], while the values of kinematic viscosity for gasoline engines used oils are decreasing, the reason for this decreasing it also may be due to dilution with light fuels. The values of kinematic viscosity for used lubricating oil was (72.836, and 12.355) cSt, (203, and 19.65) cSt at (40, and 100) °C for gasoline, and diesel engine oil respectively.

In general, the results of viscosity at (40, and 100) °C for three treated samples are visibly changed as it decreased its value compared to used oil. After treatment, it can be observing changed in kinematic viscosity values, it is become more closely from values of base oil, and this change can be considered an improvement in this specification. The treatment of used lubricating oil led to the reduced of the causes related to the breakdown of viscosity, which have a direct impact, such as oxidation, sulfur compounds, and metals. These components were removed or reduced.

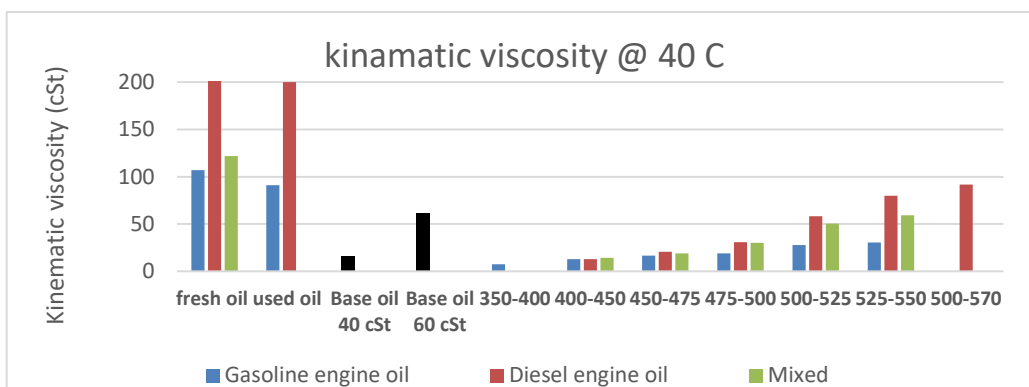


Fig. (4): Kinematic viscosity at 40 °C of fresh oil, used oil, and distillates cut.

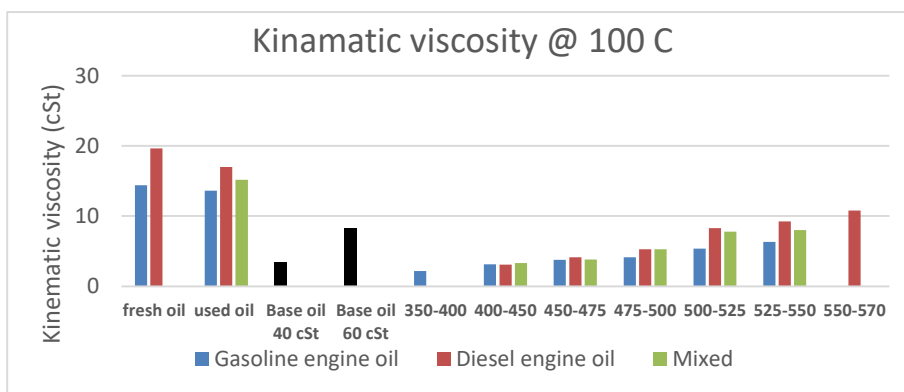


Fig. (5): Kinematic viscosity at 100 °C of fresh oil, used oil, and distillates cut

The change in the viscosity index was evident when comparing the results obtained before and after treatment for used oil. The values for viscosity index for used lubricating oil before treatment were (72.836 and 110.7) for gasoline and diesel engine oil, after treated oil these values were improved and became closely to their values of base oil for some cuts.

The improvement in kinematic viscosity and viscosity index are due to of the improvement in the other specifications mentioned above, in addition the removing some impurities were improving this property. Figure (6). show the change in the viscosity index.

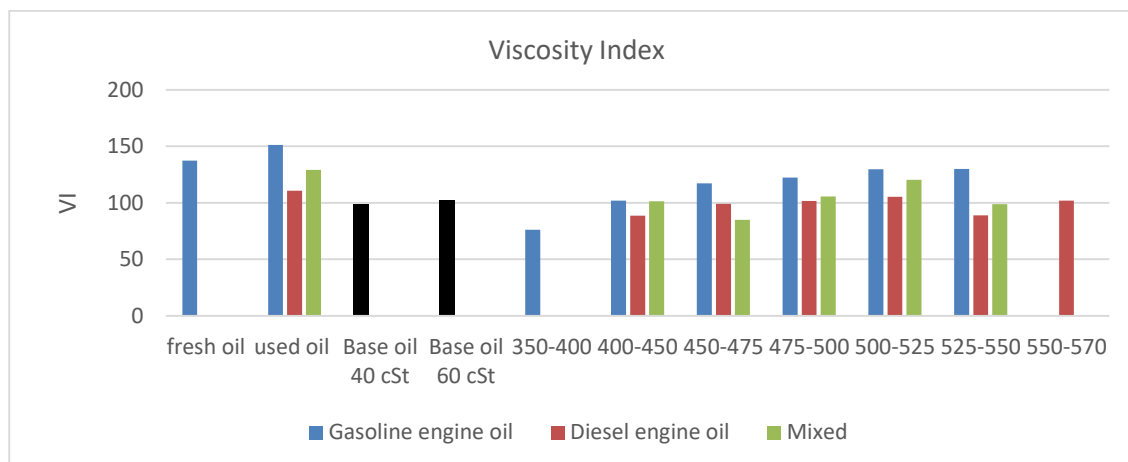


Fig. (6): Kinematic viscosity at 100 °C of fresh oil, used oil, and distillates cut

4.3 Flash point

The lubricating oil flash point is the lowest temperature sufficient amount of vapour exists to result in a flash vapor/ air mixture under specific conditions. A high flash point indicates that base oil content has low volatility components while a drop in flash point means that base oil includes high volatility with more contamination [14], [30]. Figure (7), shows the results obtained in the laboratory regarding the flash point. The values for new oil were (232, 234) °C for gasoline, and diesel engine oil respectively, we note that there is a clear decrease to (225, 227, and 230) °C for used lubricating oil for gasoline, diesel engine oil, and mixed sample respectively compared to the new oil, as there are special additives to it that raise the flash point for new oil. The decrease in results can be attributed to the consumption of these additives and the most important reason is the presence of light cuts in the used lubricating oil (fuel dilution), such as gasoline [26], we notice that the flash point at the lowest temperature was less than at other, then begins to increase especially results of the vacuum distillation method, the flash point increases with increasing molecular mass [28]. The results of flash point value for gasoline were (220, 223, 241, 242, and 258) °C, and (220, 220, 230, 252, 268, and 278) °C for diesel engine oil is support that was discovered from the density results. The results of flash point are also related to oxygen materials, which have a higher percentage in used lubricating oil due to the presence of aldehydes and ketones. (Whose locations were determined from the FTIR) that these substances reduce the flash point [4], [31].

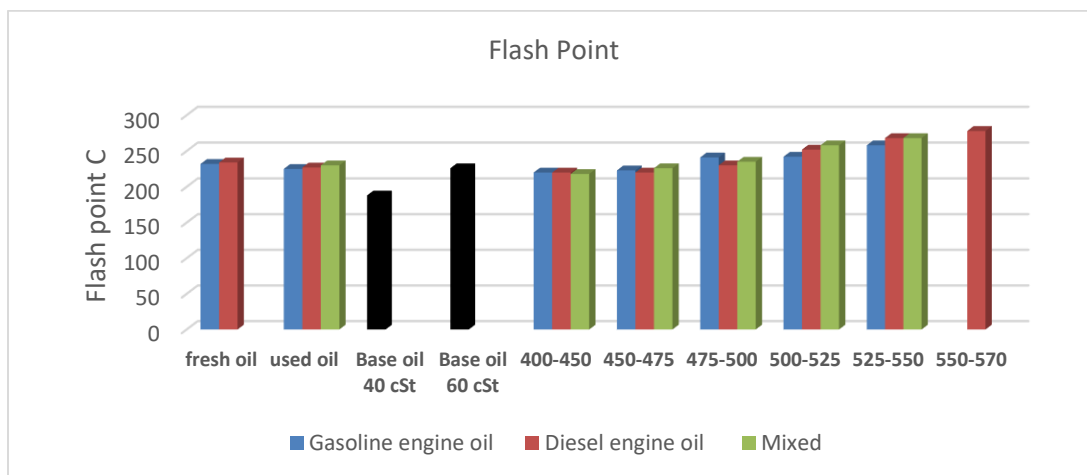


Fig. (7): The flashpoint of fresh oil, used oil, and distillates cut.

4.4 Fourier transform infrared (FT-IR)

Fourier transform infrared (FT-IR) spectroscopy is a versatile tool used to detect common contaminants, lube degradation byproduct and additives within lubricating oils. The ability to express these results in more quantitative terms is compromised by the impracticality of having to rely on the availability of reference oil relative to which changes can be compared [28]. From the first moment and when comparing the Figures (8, 9, and 10) of the samples (new oil, used lubricating oil Gasoline Engine, and Diesel Engine before treatment) with the cuts resulting from vacuum distillation treatment at range of temperatures (500-552) as shown in Figure (11), the difference in the general form can be distinguished, with regard to the intensity and size of the peaks, as this signal is one of the clear indicators of change in the substance. In Figure (8). for new, oil it is observed very sharp and clear peaks. New peaks were appearing in FT-IR spectra of used lubricating oil Figures (9 and 10) such as peaks at around 3174 cm^{-1} , 3608 cm^{-1} for stretching vibration of ($=\text{C-H}$) on aromatic, also display bending vibration of ($=\text{CH}$) at about 765 cm^{-1} , 720 cm^{-1} [32]. In addition, the peaks for aromatic compounds also appear in 814 cm^{-1} , and 88 cm^{-1} these peaks indicated for present of fuel residue in the used oil [28]. Peaks in 1039 cm^{-1} , 1053 cm^{-1} , 2025 cm^{-1} (C- O in carboxylic acid), and 2397 cm^{-1} (H-O in carboxylic acid) this peaks attributed to, oxidation products at high temperatures. Carbonyl-containing degradation products also presence in used oil, these have been identified as ester, ketones, and salt, and the peaks that it is attributed for it can observe 1738 cm^{-1} , 1709 cm^{-1} . Also The peak at 1629 cm^{-1} appear in used lubricating oil while not in new, and disappear in refined oil, this peak attributed to carbonyl group [28], [32]. In new sample there is peak in range (1150-1155) cm^{-1} while in all samples of oil

(BLO, ULO, and treated samples TUO) band at (1150-1154) Cm^{-1} is still appear, this bands attributed to sulfur compounds that are present [33].

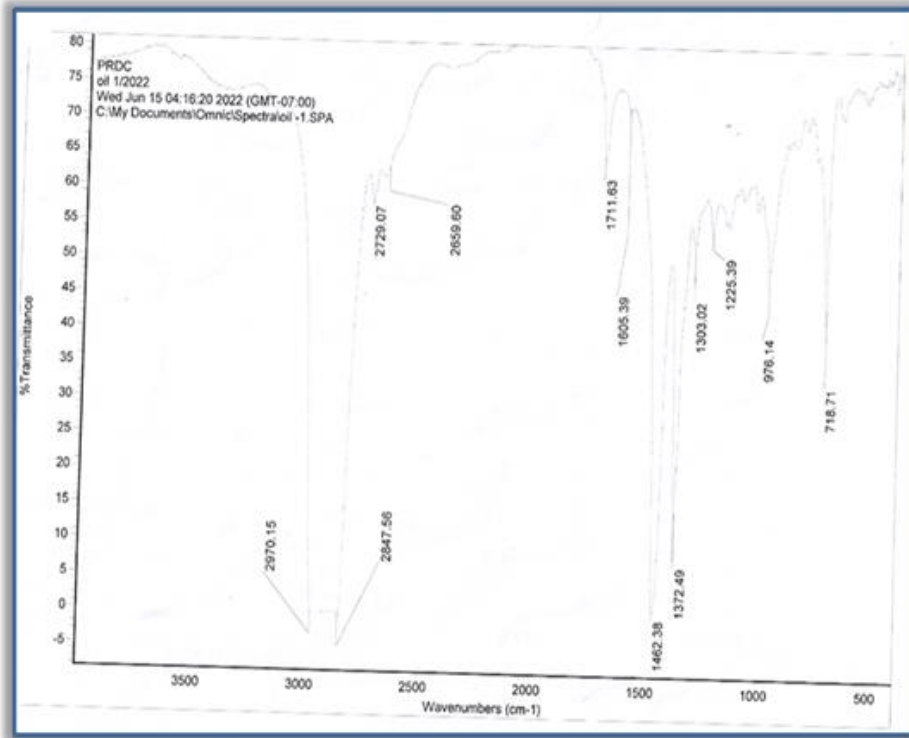


Fig. (8): FTIR of new EO (Gasoline Engine)

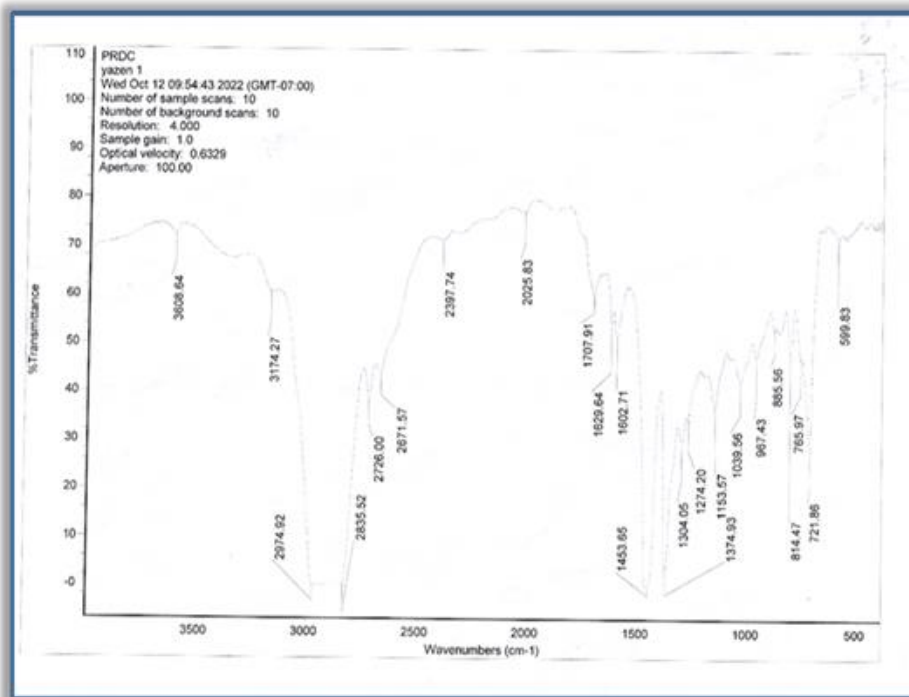


Fig. (9): FTIR of ULO (Gasoline Engine)

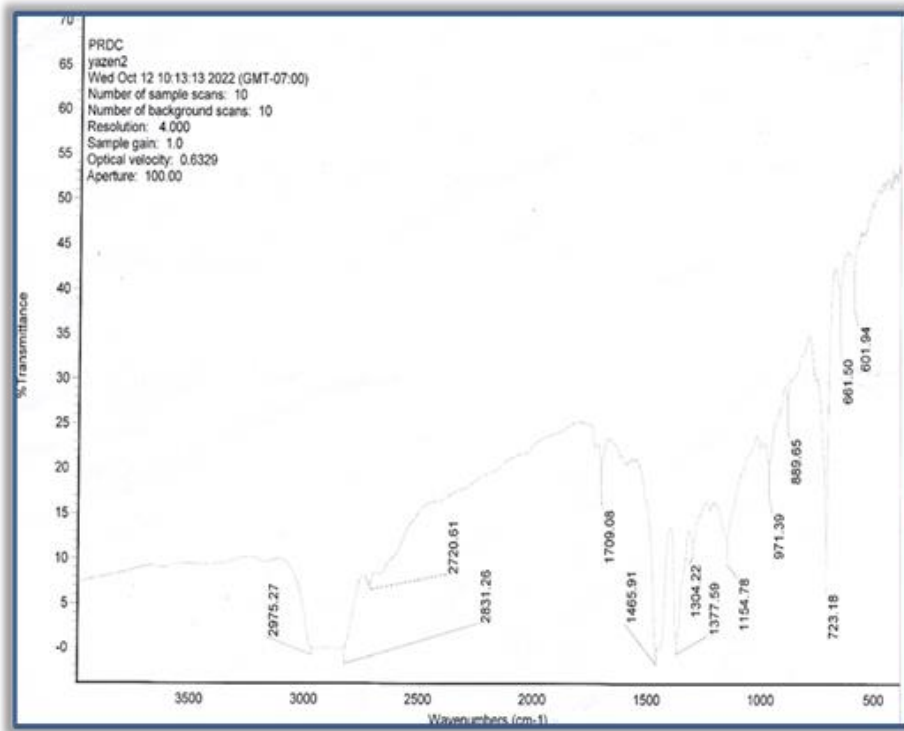


Fig. (10): FTIR of ULO (Diesel Engine)

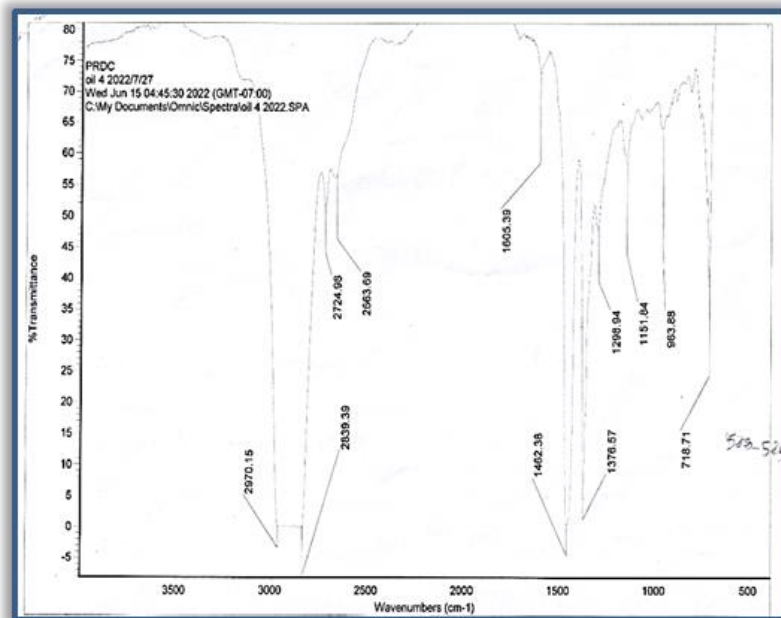


Fig. (11): FTIR of distillate (500 -525) °C

4.5 Sulfur content

There may be various causes for the presence of sulfur in the oil. In mineral oils, sulfur is usually a component of the base oil, but sulfur can also be added to the oil as an anti-wear additive. And

in some cases, it represents contamination. High percentage of sulfur in the base oil of lubricants can cause acid formation, among other things, which causes non-ferrous metal corrosion. To avoid this, the starting product must be desulfurized. However, it is not possible to determine how much "active" sulfur, i.e., sulfur added by additives and thus having a positive effect, is present in an oil sample or how much "bound" sulfur originates from the base oil. Therefore, the process of reducing the percentage of sulfur compounds in used oil is a necessary process to prevent or reduce the possibility of the presence of spent and harmful sulfur compounds. The used lubricating oil samples showed high levels of sulfur content, which reached to (9700, 7850.7, and 4356.4) ppm in (gasoline, diesel, and mix sample) respectively. After the treatment of these samples using vacuum distillation. The results of sulfur content obtained by vacuum distillation rote were (3694, 850, 405.1, 549, 687.2) ppm for gasoline, (2821.4, 2105.4, 2277.3, 2440.5, and 3210) ppm for diesel, and for a sample of mixed-used lubricating oil the results were (2836, 1810.4, 1657.3, 2087, 2674.6) ppm. The improvement in sulfur content was also observed in used lubricating oil, this means that the treatment process was efficient in reducing these compounds [26], [29].

When comparing the concentration of sulfur in the different distilled cuts, noticed an increase in the ratio with the increase in temperature at the last cuts. This increase is considered logical, because there are different sulfur compounds present in the petroleum cuts, and the more difficult sulfur compounds to remove are concentrated in the heavier cuts. Figure (12). shows the changes of in the concentrations of sulfur compounds present in the samples that were worked on before and after treatment by vacuum distillation.

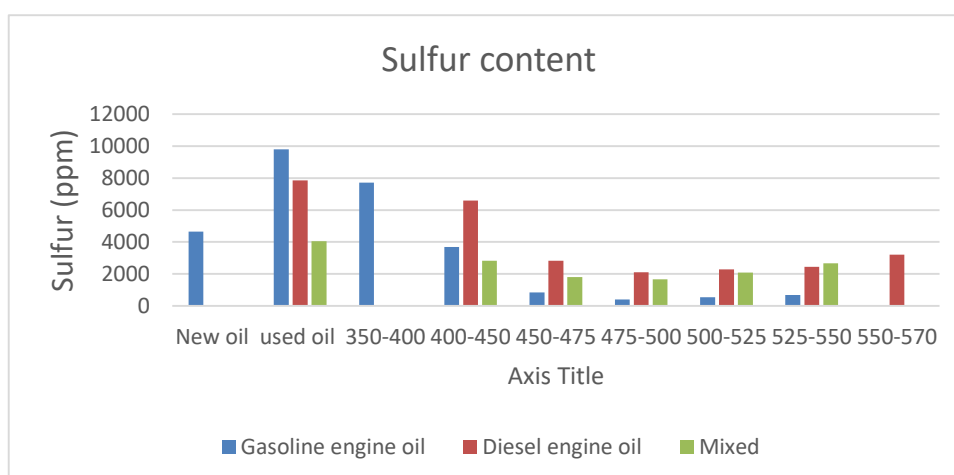


Fig. (12): Sulfur content of fresh oil, used oil, and distillates cut

4.6 Pour point

New engine oils have low pour point values compared with the base oil, some additives were added to improve this characteristic, which is indicative of their quality[2]. Figure (13). shows the results of Pour point for new samples used in this work (-27, -21) for gasoline, and diesel oil respectively, while base oil has a value nearby (-12, -6) for 40 cSt, and 60 cSt. This characteristic begins to change with the consumption of oils due to the depletion of additives' efficiency. Oxygen compounds increase in used oils as a result of the formation of aldehydes and ketones, which appear as by-products of the consumption of additives in the new oil.

From reviewing the results obtained in the laboratory, we find a clear decrease in the pour point values compared to the new oils, pour point for ULO of gasoline, diesel, and mix sample engines were (-36,-30,-33) respectively, while the oils treated by vacuum distillation method showed an improvement in the values as the pour point increased to be close to the base oils such as for cuts of gasoline engine oil (-21,-18,-21,-18,-18) for distilled temperatures are (400, 450, 475, 500, 525, and 550) °C respectively while for extraction method the results were (-12, and -6). This improvement in results indicates the efficiency of the treatment process.

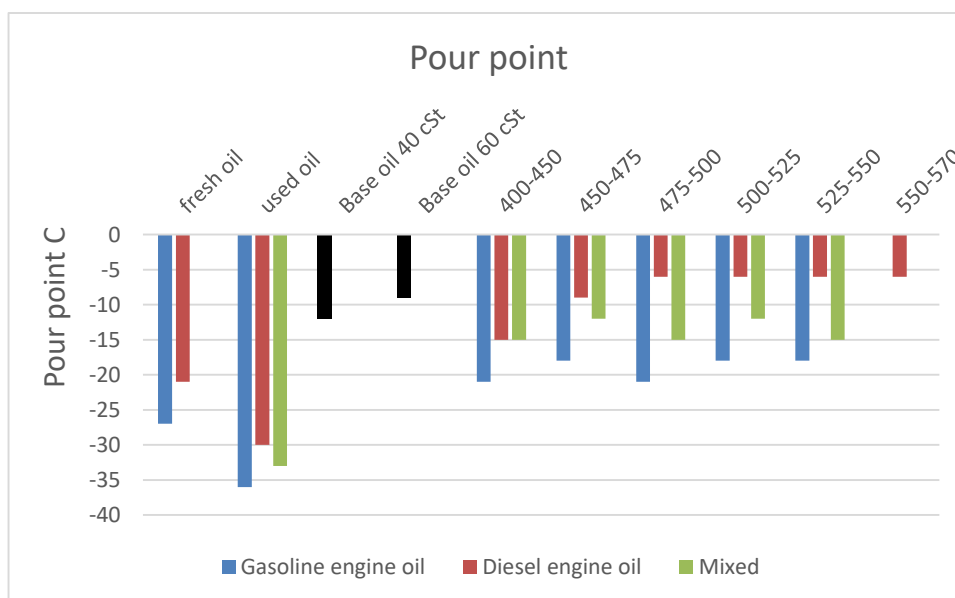


Fig. (13): Pour point of fresh oil, used oil, and distillates cut

5. Conclusions

In conclusion, this study investigated the treatment of three types of used lubricating oil using vacuum distillation. This technique was found to be a valuable process for producing base oils, specifically SN-150 and SN-200, from locally sourced used lubricating oil in Iraq. The results obtained from the distilled cuts demonstrated a significant improvement in the properties of the treated lubricating oil. The treated oil's specifications were comparable to those of the base oil. The yield obtained after vacuum distillation treatment ranged between 74% and 85% depending on the type of sample used. Additionally, the residual percentages of gasoline, diesel, and mixed oil were determined to be 13.3%, 25.8%, and 23.9%, respectively, through the use of vacuum distillation. These findings highlight the effectiveness of vacuum distillation as a viable method for treating used lubricating oil and transforming it into high-quality base oils, thereby contributing to the recycling and reutilization of lubricating oil resources in an environmentally sustainable manner.

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