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Regeneration of Engine Used Oils by Solvent Extraction

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Abstract

The regeneration of used oil is one of the essential processes for economical, industrial and environmental targets. Used oil is rich of hydrocarbons, metals (such as: aluminium, chromium, copper, iron, lead, manganese, nickel, silicon and tin), gasoline, water and antifreeze. Due to the high increasing rate of the number of cars, there is a huge quantity of used oil. In this study, different brands of used oil were involved in extraction and adsorption processes as a regeneration process of these used oils. The optimum conditions were determined such as solvents composition, solvent: oil ratio, KOH concentration and temperature. The solvent mixture of 40% of petroleum ether, 11% of 1-butanol and 4% of 2-propanol has shown the best results in removing sludge. The 3:1 (solvent: oil) ratio may be considered as the best ratio practically and economically.

Keywords: used oil, regeneration, solvent extraction, solvent-oil ratio, extraction.

إعادة تدوير بعض زيوت المحركات المستعملة باستخدام الاستخلاص بالمذيب

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الخلاصة

تعد عملية إعادة تدوير زيوت المحركات المستعملة من العمليات الأساسية في المجال الصناعي والاقتصادي. تحتوي زيوت المحركات المستعملة على كميات كبيرة من الهيدروكربونات وعناصر مثل الألومنيوم والكروم والنحاس والحديد والرصاص والمنغنيز والنيكل والسيليكون والرصاص والكاواويل والماء مع كميات من مضاد التجمد. تم استخدام عدد من الزيوت لشركات مختلفة ذات مواصفات مختلفة واستخدام لقياس كفاءة الاستخلاص وقياسات التشخيص اجهزة طيف الأشعة تحت الحمراء وطيف الامتصاص الذري لتقدير العناصر بالإضافة الى قياسات اللزوجة , مؤشر اللزوجة, معمل الانكسار, نقطة الوميض, نقطة الانسكاب, اجمالي الرقم الحامضي واجمالي الرقم القاعدي. كما تم دراسة الظروف المثلى مثل مكونات ونسب المذيب الى الزيوت وتركيز القاعدة ودرجة الحرارة. وجد أن نسبة المذيب التي تتضمن 40% من الأثير النفطي و 11% من البيوتانول و 4% من البروبانول قد أظهر أفضل كفاءة استخلاص. كما أن النسبة 3:1 من الأثير البترولي الى مزيج الكحولات تعد أفضل النسب المستخدمة عمليا واقتصاديا.

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1. Introduction

Generally, used oil contains wide range of hydrocarbons. It also contains metals such as copper, chromium, nickel, iron, aluminium, silicon, tin, manganese and lead with small amounts of gasoline, water and antifreeze [1, 2]. Extraction and adsorption process is one of the known methods for the regeneration of used oils.

In 2007, Rincon et al [3], treated used lubricating oil with liquid and super critical ethane as a solvent for regeneration of used lubricant oil. In 2009, Kamal and Khan [4] studied the effects of adsorption and extraction on the regeneration of used lubricating oil. A good and cheap solvent was searched for, and the performance of different types of solvents in sludge formation was studied where the ratio of used polar solvents like some alcohols and ketone such as MEK and 2-pentanol to used oil was 3:1 (volume). The adsorbents silica gel, alumina, and magnesite native rock produced an oil with good quality similar to the original oil [5].

In 2009, Singh et al [6], investigated an extraction adsorption combo-process for regeneration of used oil, where N-methylpyrrolidone (NMP) was taken as solvent in continuous phase and used oil distillate as dispersed phase and activated charcoal was used as adsorbent to improve the colour of re-refined oil. In order to attain the stringent limits for sulfur content in re-refined used oil, solvent free raffinate was percolated through charcoal bed to further complement extraction step. Significant improvement in the quality of re-refined used oil in terms of sulfur content, color and odor has been achieved through the combo process.

In 2010, Filho and coworker [7], invented liquid-liquid extraction and adsorption on solid surfaces applied to used lubricant oils recovery, it was possible to the recovery of lubricant base oils with lower content of contaminants.

The regeneration process may involve the removing water and fuel contaminants using solvent extraction and vacuum distillation [8-11]. In 2019, Santos et al [12], developed a method for recovering base oil from the used motor oil by using different organic polar solvents and then recognized by chemical, physical and thermal analysis, and turns out from their study of the solvents used to extract the base oil that the best product was obtained from 1-butanol and methyl-ethyl-ketone.

In 2020, Jose and co-worker [13], developed a method to remove the sludge and regeneration of used oil by solvent extraction. They selected two polar and nonpolar solvents with the waste oil and the solvents selected were methanol with toluene and methyl isobutyl ketone according to the miscibility with waste oil and their parameter of solubility values. In 2020 Ali and Zakariya [14] proposed regeneration of used lubricant oil by solvent extraction and clay adsorption technique. The oil is distilled by vacuum distillation and extracted by Methyl Ethyl ketone as a solvent. In 2021, Nancy Zgheib et al [15] investigated the regeneration of used oil by extraction through the use of three solvents which include methyl ethyl ketone, 1-butanol, and isopropanol is an excellent mixture for reducing pollutants in the oil with good removing of sludge.

In 2021, Azhari et al [16] investigated regeneration of used oils by using one solvent like isopropyl alcohol or a mixtures of toluene, ethanol and isopropyl alcohol. The researchers note that best results were by using isopropyl alcohol as a solvent at 40°C of the extraction. In the current study, the optimum solvents composition of ternary solvent (petroleum ether 60-80°C), 1-butanol and 2-propanol) was investigated. The mixture of used oils with different

ratios of the solvents and the added quantity of potassium hydroxide was investigated. In addition, the best time and good temperatures used for the solvent extraction are elucidated.

Experimental Methods

1- Instruments and chemicals: The following instruments were used in this study: Fourier Transformers infrared analyzer (FTIR) Spectrophotometer of the type fts3000, Biorade/made in USA. Atomic Absorption Spectrophotometer of the type Pyunicam SP9, Philips/ made in England. Cloud and pour point instrument of the type (3305), petrotest/ made in Germany. Cleveland Flash point meter open cup of the type (F1055), market Azmayesh Abzar/ made in Iran. Viscometer of the type SCHOTT-GERATE, Hofhheim/ made in Germany. Viscosity bath of the type Haake/ made in Germany. UV/Visible digital double beam spectrophotometer/ made in England. Virgin oils of SAE 50 of (Fuchs, from Saudi Arabia, Super Rakib, from Iran and German oil, from UAE). And all chemicals were obtained from BDH, Fluka, Merck companies. The samples of three types of Nissan pickup cars of 2006 model collected from Faisal and Hawes service station in Arbil city having same grade of SAE 50, by mixing equal fraction of used lubricating oil to produce mixture.

2- Procedure:

Dehydration: The samples of Fuch oil, Super Rakib oil, German oil and used oil mix (1-4) were dehydrated by heating to 150°C under atmospheric pressure to remove water and volatile compound from the used oils.

Percentage of Sludge Removal: The present amount of dry sludge which was isolate from the used engine oil after mingling with the solvents according to the procedures with filtering, which is called Percentage of Sludge Removal (PSR). The residual phase was heated for 1 hour at 100°C, then, washed to take out the impurities. The wet sludge re-scatted by adding 30ml of n-hexane and then, destabilized recurring by the addition of 70 mL 2-propanol. Next, leaving the mixture to settle for 24 hours. The liquid was ignored by vacuum filtration and then, the sludge was stocked. The wet sludge was heated at 100°C for half an hour then cooled down and the dry sludge was then weighted as shown in Tables 1 and 4, and then, the PSR was calculated.

Percentage of Oil Losing: The Percentage of oil losing (POL) is the loss of oil through the extraction process that has no dissolved in the solvent phase, but remains with the sludge from 100g of waste oil. The results are shown in Table 4.

Decolorization/Adsorption process: The extracted oil (100 ml) was mixed with bentonite clay, 20gm and the mixture was heated to 150°C for 1 hour with stirring, after that, it was left at laboratory temperature for 24 hours to allow settling down and filtrated. The higher portions of impurities were removed. The remained portion of the impurities was diminished from base oil with clay treatment to formulate new lubricant. The absorption of regenerated oil was recorded at maximum wave length (λ_{\max}) of 371 nm. Each oil sample was diluted 10-times with xylene. Decolorization process was studied for the recovering base oil at 150°C and the adsorption of colored materials was decreased until 20g of clay and settling time of 24 hours.

The Samples Analysis: The quality of oil samples before and after the solvent extraction process indicates the degree of regeneration. Infrared spectra of oil samples were taken by FTIR spectrophotometer; the result are listed in the Table 2.

For the analysis of metallic content, used oil samples were diluted to 1/20 (w/v) with an organic solvent (kerosene or iso-butylmethyl ketone), then, a comparison with a known set of organometallic standards of 4-cyclohexylbutric acid salts of Zn, Ca, Mg, Cu, Fe and Pb was performed. The standard solutions were introduced to the atomic absorption spectrophotometer to get the responses from the calibration curve,

Kinematic Viscosity [17], Viscosity Index [18], Refractive index [19], Specific Gravity [20-21], Flash point [22], Pour point [23], total acid number [24] and total base number [25] of the virgin oils, the used oils and the base oils of all types were measured and the results of all analysis are shown in Table 3.

Water content [26]: To determine the amount of water content, 100 ml of used oil was heated under reflux with 100 ml of xylene, then, co-distillate with the water in the samples. Then, the water and the condensed solvent were continuously separated in trap till no water is left. The water content of samples was in the range of 0 - 1.5%.

Results and Discussion:

Optimum solvents composition: good extraction solvent of the base oil should be miscible with oil and should be able to remove the additives and other impurities. As shown in Table 1, by increasing the percent of 2-propanol at a constant percent of petroleum ether, the sludge removal increases. However, increasing petroleum ether percent reduces the sludge removal at a constant percent of 2-propanol. Solvent mixture number 4 (40% of Petroleum ether (60-80°C), 12% of n-butanol and 48% of 2-propanol) has shown the best results for removing sludge.

Solvent to oil ratio: The effect of solvents:oil ratio on sludge removal and oil loss was studied in 2:1, 3:1, 4:1, 5:1 and 6:1 ratios. The maximum percent sludge removal (PSR) with minimum percent oil loss (POL) are achieved using 6:1 ratio; which shows better oil recovery than 2:1 and 3:1 ratio. However, 2:1 ratio has shown higher wet sludge removal. 3:1 ratio may be considered as the best ratio practically and economically; as the solvent to oil ratio is less than 4:1 which produced viscous mixture during separation. In addition, ratios greater than 3:1 are not suitable for two reasons; first, because enormous solvents are needed and second because of the dissolution of some contaminants in the solvent phase. Generally, when a high ratio of solvent is used compared to the ratio of used oil mix sludge, a high ratio of oil extracted and an oil-free sludge can be obtained, while when the ratio of the solvent is low, the solvent become saturated with the oil that is obtained, which in turn, leads to less amount of production, the results are listed in Figures 1 and 2.

Table 1: Effect of Solvent mixtures composition on sludge removal and base oil viscosity and viscosity index

No.	Solvent Mixture Component			Wet Sludge Removal %	Kinematic Viscosity of base oil (cSt) at 100°C [16]	Viscosity Index [17]
	Petroleum ether (60-80°C) %	1-butanol %	2-propanol %			
1	25	30	45	14.7	11.24	98
2	25	25	50	15.6	10.65	92
3	40	12	48	12.5	11.1	92
4	42	10	48	11.8	10.4	89

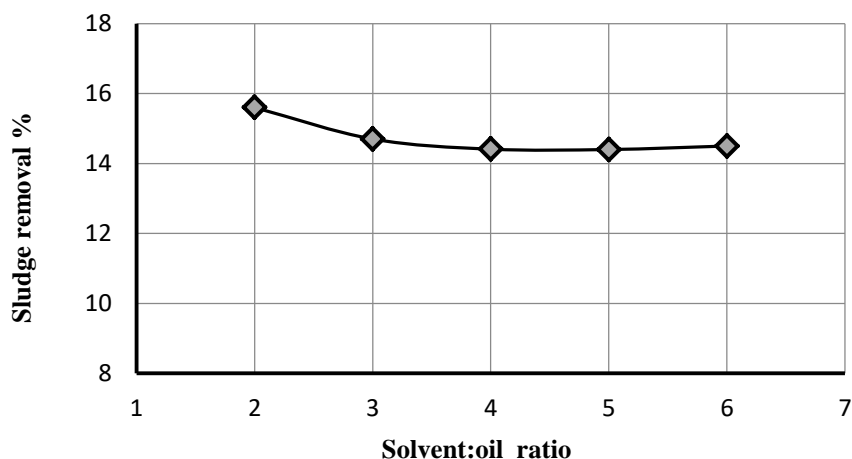


Figure 1: Effect of changing the solvent: oil Ratio on the percent of the sludge removal

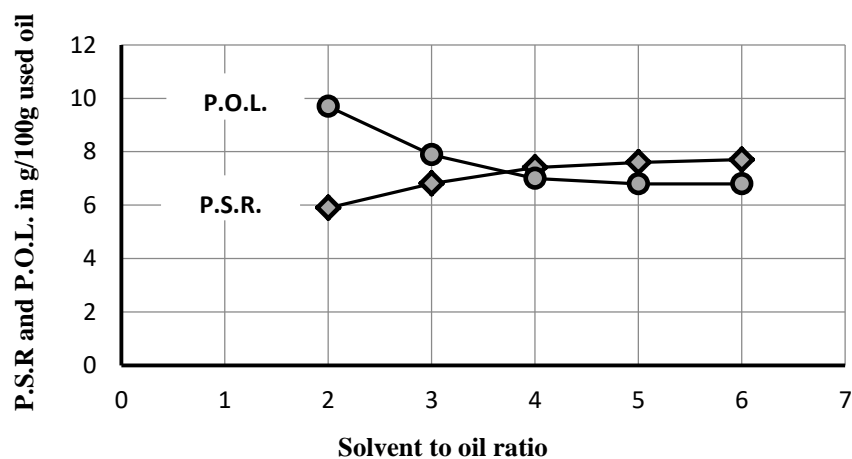


Figure 2: Effect of changing the solvent: oil ratio on the percent of the sludge removal and the percent of oil loss in gm/100gm of used oil

Effect of KOH:

The effect of KOH concentration on sludge removing was studied with solvent to oil ratio of 3:1. With increasing the addition of KOH to destabilize the impurity particles in the solution, the sludge removal increase with increasing KOH. The best added weight of KOH was 3g/L, as having higher sludge removal, (maximum PSR and minimum POL) From Figure 3, it is shown that the increase in the addition of KOH leads to increase in the PSR to up to 3g/L KOH, and after that, decrease in POL up to 3g/L KOH and after that increase.

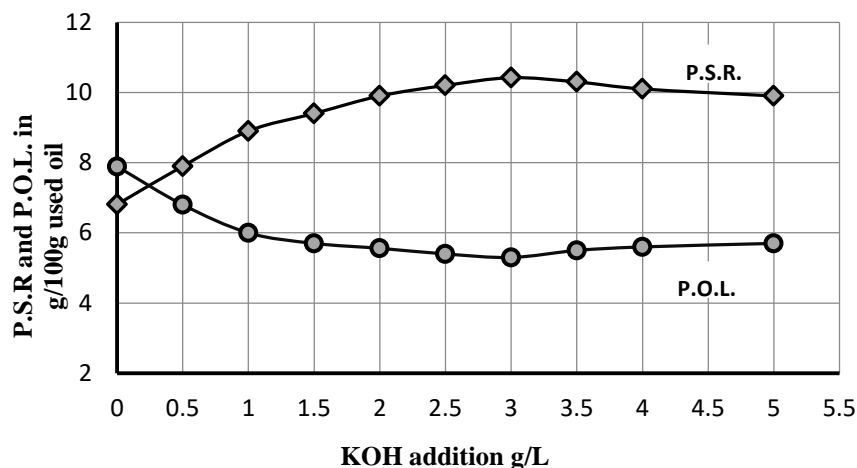


Figure 3: Effect of addition of KOH g/L on percent of sludge removal and percent of oil losing in gm/100gm used oil

The effect of settling time on solvent extraction:

The investigated times were 1, 1.5, 2, 2.5 and 3h, where the amount of removal sludge in used oil increases by increasing the time of residence of used oil in the tanks for sedimentation. The speed of settling is achieved after 2h from the addition of KOH solution. Figures 4 and 5 show the effect of more contact time between waste oil and solvent; which plays an important role in separating unwanted substances for two reasons: first, the contact time must be long time to allow the solvent to dissolve the base oil present in the oil to be treated, and second, the time must be sufficient to remove all undesirable substance such as impurities and additives from the solution, by collecting it with particles of large sizes, allowing it to separate from the solution.

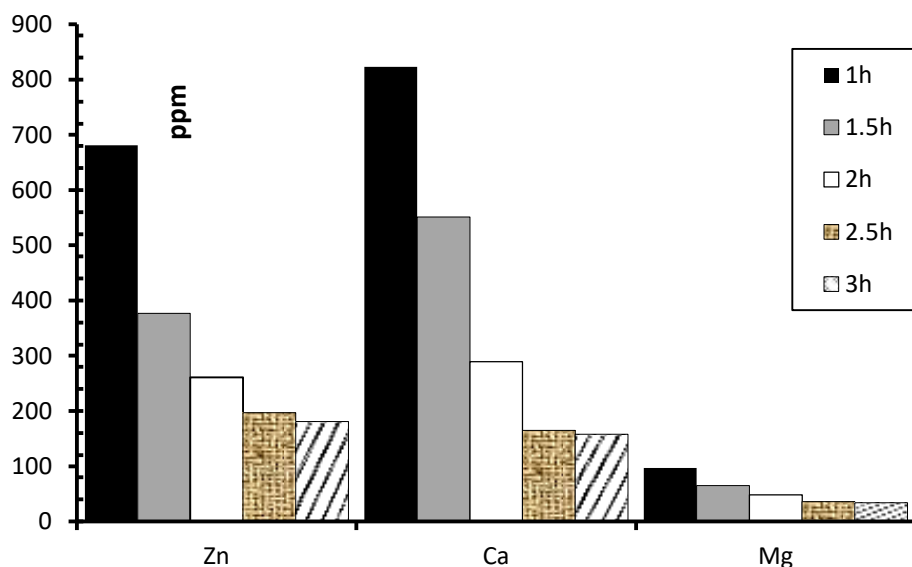


Figure 4: Influence of settling time on some element concentration in extracted oil

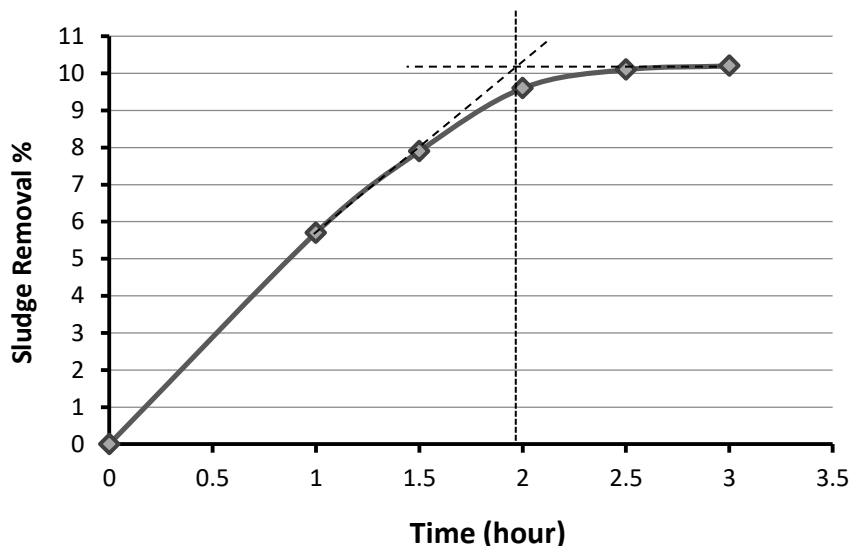


Figure 5: The Influence of time on the percent removal of sludge.

The Influence of temperature:

The Influence of temperature was studied in various degrees of temperature of 20, 25, 35, 45 to 55°C. In order not to evaporate the solvent, we did not conduct the test at high temperatures, and it is certain that the amount of extracted oil production increases with the increase in the reactor temperature due to the combination of the solubility of the base oil in the organic solvent. The obtain results are shown in Figures 6 and 7. The amount of sludge residue decrease with increasing temperature

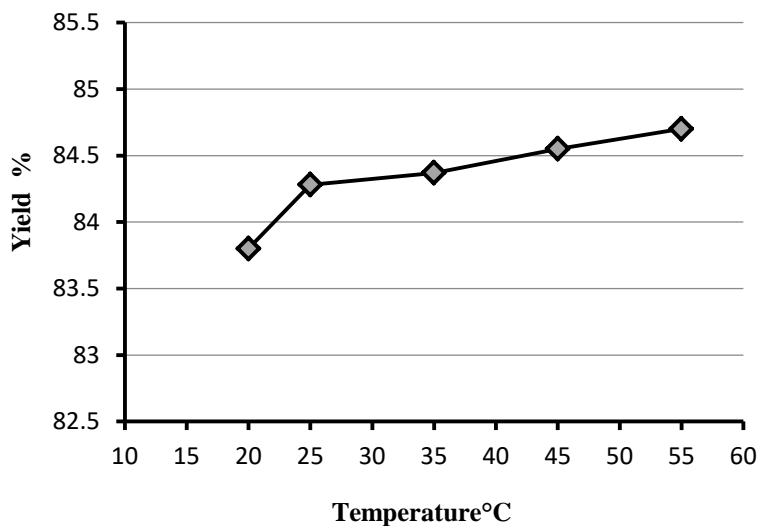


Figure 6: Effect of temperature on the extraction yields.

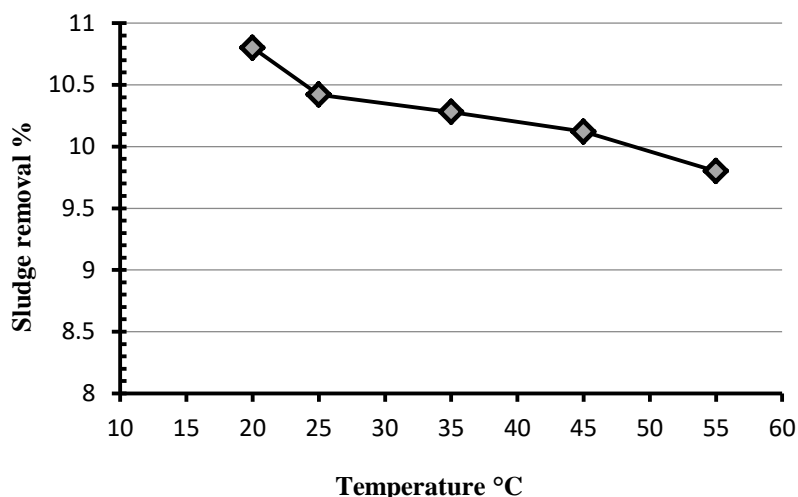


Figure 7: Influence of temperature on the percent of removal sludge.

Oil recovery, Percentage of removal sludge and Percentage of oil losing:

The oil recoveries of samples are in the range of 84.4 - 85%. The relation between oil recovery and sludge removal is in reverse, this is listed in Table 4.

Decolorization Process: The higher portions of impurities were removed, and the remained portion of the impurities must be diminished from base oil with clay treatment to formulate new lubricant. The adsorption of colored materials decreased until approaching 20g of clay and settling time of 24 hours.

FTIR-measurement: IR spectrum of the used oil models is a set of spectra of all components in the oils model. It consists of many overlapping peaks that are hard to be resolved. The oil samples used is mixture of compounds derived from the base oil, its additives and the pollutants present in it, in addition to the results of the decomposition of the used oil. After it has been used, the virgin oil had degraded and contaminated, as such, some new peaks were observed in the used oil spectrum. All spectra were recorded from 4000 to 400 cm^{-1} . Carbonyl group such as lactones, esters, aldehydes, ketones and carboxylic acids also showed some bands. Nitrogen, sulphates and all unwanted materials have been removed from the processes. The essential FTIR values are shown in Table 2.

Table 2: Infrared group absorbance (cm^{-1}) for oils

No.	Oil type	C=O _{str}	C-H _{str}	C-H _{Bend in}		C-H _{Bend out}	Aromatic C=C _{str}
				scissoring	Rocking		
1	Virgin oil	1708	2846, 2950.9	1452.6	721.9	1376	1600
	Used oil	1704	2946, 2963	1452	721.8	1377	1602
	Base oil	Not detected	2856, 2953.9	1462	722	1377	1603
2	Virgin oil	1716	2841, 2969.	1452.7	721.8	1376.6	1604.36
	Used oil	1713	2844, 2953.5	1459	721.9	1377	1603.8
	Base oil	Not detected	2852, 2958.4	1461.6	721.8	1377	1603.1
3	Virgin oil	Not detected	2844.9, 2971	1457	721.7	1376	1603.8
	Used oil	1704	2846.7, 2959	1453.6	721.8	1376.6	1603.5
	Base oil	Not detected	2856, 2953	1461	721.9	1377.3	1603.22
4	Used oil	1707	2848, 2965	1453.5	721.7	1377.4	1604.7
	Base oil	1707	2863, 2859	1461	721.9	1376.6	1603.2

Reducing Metallic Content: The composition of machinery and automobile oil has changed chemically and physically from the virgin oil due to the high temperature in those engines during operation. Relatively, higher levels of metals such as Fe, Pb, Cr, Zn, Ca, and Mg are found. Using the calibration which is obtained from the standard solution, the metallic content was estimated. An atomic absorber is used to analyze samples of extracted oils to clarify the differences between the regenerated and virgin oil [27]. The result obtained are useful for differentiation purposes and for giving information about the concentrations of minerals present. All result of elemental of virgin oil, used and base oils are listed in the Table 3.

Table 3: Elemental analysis of virgin, used and base oils which we obtained by using Atomic Absorption

No	Type of oils	Zn (ppm)	Ca (ppm)	Mg (ppm)	Fe (ppm)	Cu (ppm)	Pb (ppm)
1	Fuchs	1200	1100	113.16	0.0	0.0	0.0
	Used oil	1436	1315	127	58	3.0	850
	Base oil	0.07 2	0.0	0.04	1.0	0.0	5.0
2	Super Rakib	950	750	32.8	0.0	0.0	0.0
	Used oil	1110	850	58	67	5.0	400
	Base oil	0.144	0.0	0.07	1.5	0.5	4.16
3	German oil	726	950	47	0.0	0.0	0.0
	Used oil	935	1080	80.34	59	5.5	1608
	Base oil	0.794	6.2	0.058	1.0	0.0	5.83
4	Used oil Sample mixture	1020	1328	166.4	57	3.5	683.3
	Extracted oil	176	173	33	7.0	0.0	122
	Base oil	0.1443	0.277	0.06	1.0	0.0	2.66

Physical and Chemical Properties:

1- Kinematic Viscosity, Viscosity Index, and Refractive index [17-19]: For all virgin oil samples, the kinematic viscosity was found to be greater than the base oil samples at the same temperatures of 40°C and 100°C. This is because when the viscosity raised then the oil additives would be subjected to a high stress, as such, the polymer breaks to lower molecular weight crumbs. The viscosity index is used to show the changes and effects that occur at different temperature, as well as, the relation between the temperature and the viscosity. The viscosity index of all virgin oils were greater than the base oils because the viscosity modifiers were added to the virgin oil. Also after removing the additives by the solvent extraction, the viscosity and viscosity index were decreased for the recovered base oil. This showed that the process was successful. The refractive Index of virgin oil was greater than the regenerated base oil sample because the additives were introduced to the virgin oil, hence, after removing the additives, the refractive index decreased for the base oil. The result is listed in Table 4.

2- Specific Gravity, Flash point, Pour point [20-23]: The high Specific Gravity indicates that the oil has a high percentage of heavy component and water. As well as, it has high metal and high ash content. So, the increase of the specific gravity of the used oil can be observed. For Fuchs, Super Rakib and German oil types, the Specific Gravity of virgin oil was lower than that of its used oil, this is at 20°C, because after driving for a distance, the oxidation product, soot, water and heavy metals were entered to the used oil. After removing the spent additives and impurities, the specific gravity was decreased for the regenerated base oil. Finally, the specific gravity for the used oil mixture sample 4 was found to be 0.8986 at 20°C, while for the regenerated base oil decreased to 0.889.

A low flash point occurs because the oil contains few traces of fuel. The high flash point indicates that the extracted oil is free of solvent and fuel. The flash point of virgin oil is always higher because of the contentious oxidation of oil which occurs as the distance of driving increased. The degradation of oil produces volatile compound. Due to the dilution of oil by fuel after the dehydration of used oil and solvent extraction process, the flash point value of base oil was decreased for regenerated base oil.

Because of introducing the additives to the virgin oil, the pour point of (virgin oil) was lower than that for the used oil, but for regenerated (base oil) was increased. The results are listed in Table 4.

3- Total Acid Number (TAN) and Total Base Number (TBN) [24-25]:

The total acid number is a scale of focusing acid ingredients in oil. An increase in TAN is an oxidative indicator as well as increased oil degradation. Along with oxidation comes an increase in viscosity. For the virgin oils, the total base number was estimated because the virgin oil contains basic compounds which were added to the lubricating oil as basic detergents (to neutralize acidic compounds as soon as formed during the use). But for the used oil samples, TAN were estimated because the used oil contains acidic compound coming from the oxidation of product. The using of KOH with the solvent for the extraction has affected the removed samples. The TAN was decreased to 0.0 of oil sample. The results were listed in Table 4.

The total base number is an important parameter of petroleum products; and its value varies according to the number of milligrams of potassium hydroxide needed to neutralize each gram of oil.

Table 4: Kinematic viscosity, viscosity Index, refractive index, specific gravity, flash point, pour point, total acid number, total base number, oil recovery, sludge remove. and oil loss of oil types.

	Oil Type	Fuch	Super Rakib Oil	German Oil	Used Oil (Sample. Mixt)
Virgin Oil	Kinematic Viscosity at 100 °C	15.84	16.36	18.19	17.89
	Viscosity index	86	114	114	100
	Refractive index	1.489	1.486	1.487	1.485
Base Oil	Kinematic Viscosity at 100 °C	14.62	10.1	10.54	10.91
	Viscosity index	81	94	87	88
	Refractive index	1.486	1.486	1.485	1.484
Specific gravity	Virgin Oil	0.890	0.888	0.889	0.889
	Used Oil	0.908	0.894	0.901	0.898
	Base Oil	0.881	0.887	0.888	0.880
Flash Point	Virgin Oil	225	215	207	225
	Used Oil	175	172	158	160
	Base Oil	208	198	197	211
Total Acid	Virgin Oil	-10	-6	-3	-10
	Used Oil	-5	-4	0	-9
	Base Oil	0	-1	0	-3
	Virgin Oil

Total Base number	Used Oil	1.23	1.36	2.3	1.4
	Base Oil
	Virgin Oil	1.78	0.42	0.97
	Used Oil
	Base Oil
	Oil recovery(wt %)	84.5	85	84.4	84.6
Sludge Removal%	9.5	10.3	10.0	10.1	
Oil Loss%	6.0	4.7	5.6	5.3	

Conclusions

In this study, twelve different brands of used oil were selected for regeneration process. The regeneration process involves extraction and adsorption process. The optimum conditions were determined such as solvents composition, solvent to oil ratio, KOH concentration and temperature. 3:1 (solvent: oil) ratio may be considered as the best ratio practically and economically, and the achieved settling time was two hours and the solvent mixture of 40% of Petroleum ether, 12% of 1-butanol and 48% of 2-propanol has shown the best results in removing sludge.

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