Recovery of Base Oil from Spent Automobile Oil Using Elementary and Binary Solvent Extraction

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Abstract

This research was carried out to evaluate the effect of working parameters such as; type of solvent, solvent ratio, extraction temperature and mixing time on recovery of base oil from used oil by extraction / distillation technique. Bench scale experimental work was performed on spent motor oils collected from different service stations to select the suitable elementary solvents that could extract and regenerate the base oil such as (nbutanol, 2- propanol, and ethanol) and binary mixture of solvents such as (heptane and methyl ethyl ketone) with (acetone). Different ratios of alcohol solvents to used lubricants from 1:1 to 4:1 (wt. /wt.) at atmospheric pressure with different extraction temperatures (30, 40, 50, 60°C) and different mixing time (15, 30, 45, 60min) were investigated. Moreover, three different types of earth fullers (acidic bentonite, basic bentonite and Dead Sea clay) were tested at different weight from 0.1 to 0.5g/20ml to bleach the base oil color which was regenerated at specific conditions of extracted temperature, solvent ratio and mixing time which were 40°C, 3:1 and 30min respectively.

It has been found that the oil recovery and solvent recovery when using alcohol are increased with progressively increasing solvent/oil ratio to a certain limit. In such a manner that, n-butanol gives the highest extraction yield percentage of oil recovery and solvent recovery comparing with 2- propanol and ethanol. Moreover, higher mixing time granted higher percentage of base oil recovery and solvent recovery for 2- propanol and ethanol solvents. While insignificant effect of mixing time beyond 30min. on the recovery of oil and solvent was recorded with n- butanol.

On the other hand, the oil recovery and solvent recovery are considerably increased with increasing binary solvent amount and with increasing the ratio of polar solvent (acetone) in the individual binary mixture. However, the binary solvent mixture of acetone plus methyl ethyl ketone is indicated to achieve highest extraction yield percentage of oil recovery. The final color of regenerated oil is evidently improved when it was treated with acidic bentonite and activated Dead Sea clay. On the

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other hand, there was no enhancement color recorded when it was treated with basic bentonite. **Keywords:** spent oil; used lubricants; base oil recovery; solvent extraction; waste treatment

1. Introduction

Lubricants, either mineral-based or synthetic made, are the products derived from petroleum which is an essential natural resource used as the source of energy and raw material for almost all industries. Lubricating oils, sometimes referred as "Lube", are complex arrangement of straight chains hydrocarbon structure containing from C20 - C70 carbon atoms per molecule and a side chains consisting of five or six rings structures [1]. Many studies have confirmed that used petroleum-based lubricants collected from oilchange are slow in degradation and promote a serious pollution problem. It will contaminate water and earth if discharged to the environment and cause air pollution if burnt as a low-grade fuel [2]. Re-refining technology has advanced from the early processes when used oil was reclaimed by removing water, dirt, sludge, and some volatile compounds, thus the final product must meet the same specifications as unused crankcase oil for the re-refined product to be of value. There are a number of standards and specifications that are used to ensure the quality of lubricating oils. In this standard, several of ASTM methods are used to determine the properties of lubricating oil and whether or not the oil meets specifications [3]. Extraction processes using solvents typically include the separation of paraffin and naphthenic compounds, by solubility difference, from undesirable compounds such as lees, resins and asphaltic constituents. In this process, the waste oil is mixed with a solvent at ratios able to guarantee the highest possible solubility of the base oil in the solvent [4, 5, 6]. Additives and carbon impurities, normally present in waste oils, are not miscible in the solvent; thus, they eventually sediment by gravity, forming lees. The solvent used in this operation can be recovered by distillation and recycled [7].

The main objective of this research is to focus on the investigation of recycling of used motor lubricates by solvent extraction/distillation technique using elementary and binary solvents extractor under lighting of the polarity theories. Moreover, this study aims to spread the culture of recycling the depleted oil in a manner that preserves our natural wealth and reduce the bad influence on the environment.

2. EXPERIMENTAL WORK

2.1 Materials

1. Feed stock

The used lubricating oils were collected directly from different automobiles service stations, with different operating conditions. Table (1) shows the determined properties of the used lubricating oils.

Table 1:	The measured	properties	of the used	
	lubricati	ng oils.		

	-	
No.	Specification	ASTM
1	Specific gravity	0.9
2	Pour point °C	-20
3	Viscosity at 100 °C	15.3cp
4	Total acid	3.3
4	number(TAN) mg/g.	
5	Ash wt.%	0.93
6	Carbon Res. wt.%	2.3
8	Flash point °C	155
9	Color meter	Too dark

2. Base Oil

The base oil (stock 60) from Durah Refinery was used as a measuring reference to evaluate the final product. The measured properties of base oil were evaluated at the Research Center of Chemical and Petrochemical Laboratories/Ministry of Industry and the results were shown in table (2).

Table 2: The measured properties of the baseoil (stock 60) supplied from Durah Refinery.

No.	Properties	ASTM
1	Specific gravity	0.8
2	Pour point °C	-10
3	Viscosity at 100 °C	11.7 ср
4	Total acid number (TAN)	1.2
5	Ash wt.%	Nil
6	Carbon Res. wt.%	0.3
8	Flash point °C	162
9	Color meter	2.4

3. Solvents

Two types of organic solvent systems were used in this work, solitary and binary systems. The solitary solvents were mainly analytical grade alcohols that obtained from Durah Refinery are nbutanol (BDH, England), 2- propanol (Riedel, Germany) and ethanol (Aldrich, Germany).

The binary solvent systems consist of polar and non-polar solvent. The acetone was selected as the most effective polar solvent whiles the heptane and methyl ethyl ketone as non-polar solvents. These solvents were purchased from (Hi. Media, India), (Thomas Baker, India) and (GCC, England) respectively [8]. The properties of solvents were shown in table (3).

Table 3: The properties of the organic

solvents [8].

	Properties	1	2	Ethanol	Non polar		Polar
No.		I- Butanol	2- ropanol		Heptane	MEK	Aceton e
1	Molecular Weight g/mole	74.12	60.10	436.07	100.20	72.11	58.08
2	Specific gravity	0.810	0.812	0.785	0.794	0.805	0.791
3	Boiling point, °C	117	82	78.4	98.7	80	56.29
4	Freezing point, °C	-79.9	-78	-130	-43	-780	-94.7
5	Purity %	99.8	98	99.5	95	99.5	99.9
6	Vapor pressure, kPa, at 25 °C	0.930	5.82	7.83	5.33	9.5	30.59

4. Earth Fullers

Three types of earth fullers were suggested to test in this research as belching agents. Acidic, basic activated bentonite supplied from Durah Refinery was used and Dead Sea salt after activated with sulfuric acid was used to improve color and remove impurities for base oil.

3. Experimental Procedure

The used lubricants which collected from different service stations was vacuum filtrated using ceramic micro-filter in order to remove dirt, gums, fine metals from crankcase and any undesired suspended. It was dehydrated by heating the oil at150°C for 1h with continuous mixing in order to remove associated water. The dehydrated oil was allowed to cool at room temperature then added solvents at different ratios to extract the base oil from used lubricants. Several organic solvents were selected such as (n-butanol, 2- propanol, and ethanol) and as binary mixture of solvents such as (heptane and methyl ethyl ketone) with (acetone).

Different ratios of alcohol solvents to used lubricants from 1:1 to 5:1 (wt. /wt.) at atmospheric pressure with different extraction temperatures (30, 40, 50, 60 °C) and different mixing time (15, 30, 45, 60min) were investigated. The mixture was left for 24 hours at room temperature to be separated in to two phases. The upper liquid phase was the extraction solvent and base oil, and the bottom liquid phase was heavier material (contaminants). After steady state indicated by constant interface level, the two phases were separated. For recovering the solvent and extraction the oil, the up layer was put in a vacuum distillation.

On the other hand, binary mixture of heptane – acetone and methyl ethyl ketone – acetone with

ratios from 20 - 80 % (wt. /wt.) were added to spent lubricant at ratios from 1:1 to 5:1 (wt. /wt.) at atmospheric pressure and room temperature to evaluate the efficiency of base oil recovery with same procedure. The extraction solvents will be separated from the base oil and recycled back using vacuum distillation.

1. Vacuum Distillation

The vacuum separation process was adjusted at vacuum pressure of (- 0.2 bar) and 60°C water bath temperature with 300 rpm mixing speed. The recycled solvent was collected at the 500ml conical flask and the treated base oil was left at the round flask as perversely shown in figure (1).



Figure 1: The vacuum distillation unit and flow diagram of the vacuum distillation unit

2. Bleaching

Five different weights, from 0.1 to 0.5 g, for each three types of clay were added to 20g of two types of base oil obtained from ethanol extraction process as solitary solvent, and from the mixture of methyl ethyl ketone – acetone extraction process as a binary solvent. The suspended mixture of oil and clay was continuously stirred for 30min. and 40°C using hotplate magnetic stirrer. Then, the clay will be separated from the base oil by vacuum filter. The color number of the filtrate was measured using colorimetric. The suspended mixture was continuously stirred for 30min. and 50°C and then left for soaking to 24hr. The clay sample was filtered and washed with distilled water for several time to remove any trace of acid. In the final step, the clay was burned at 500°C using electrical oven.

4. Results & Discussion

4.1 Solitary Solvents Extraction System.

1. Effect of Temperature

The experimental results indicated, increasing in percent of oil recovery with increasing of solvent to used oil ratio as shown in Figs. (2-4). The ratio of solvent at specified temperature. The values of oil recovery are generally between (20 -93.7)%, (17.5 - 88)% and (12.5 - 83)% for nbutanol, 2-propanol and ethanol respectively. On the other hand, it is obvious noted from figures that the significant increment of oil recovery is obtained when the solvent/oil ratio is increased from 1:1 to 4:1 and beyond that the oil recovery seems to be insignificantly effected by solvent increment. These phenomena may be explained as the solvent is extracting all the oil available to recover and the excess amount of solvent will be ineffective.

Moreover, it is clearly seen from aforementioned plots that the oil recovery percentage is progressively increased with increasing the extracted temperature from (30 - 40) °C and dramatically decreased when the temperature increased above 40 °C i.e. when the temperature reached (50 and 60) °C. These results are quite agreed with other workers [9]



Figure 2: The effect of solvent/used oil ratio on percent oil recovery at different extraction temperatures for n- butanol extraction solvent.



Figure 3: The effects of solvent/used oil ratio on percent oil recovery at different extraction temperatures for 2- propanol extraction solvent.



Figure 4: The effect of solvent/used oil ratio on percent oil recovery at different extraction temperatures for ethanol extraction solvent.

While figures 5, 6 and7 represent the variation of solvent recovery percentage with different solvent/used oil ratios under diverse extracted temperatures for n- butanol, 2- propanol and ethanol respectively. It was shown that solvent recovery percent is progressively increased with increasing solvent ratio, when the solvent/oil ratio ranged from 1:1 to 4:1. Where, the values of solvent recovery are generally between (20 - 96.2) %, (17.5 - 90) % and (15 - 86) % for n- butanol, 2- propanol and ethanol respectively. While, the insignificant increment of solvent recovery percentage beyond certain limits referred to the fixed temperature used in the vacuum distillation step to recover the solvent and make the solvent percentage recovery almost stable at certain amount. This type of trained was verified by pervious researchers [9, 10].



Figure 5: The effect of solvent/used oil ratio on percent solvent recovery at different extraction temperatures for n- butanol extraction solvent.



Figure 6: The effect of solvent/used oil ratio on percent solvent recovery at different extraction temperatures for 2- propanol extraction solvent.



Figure 7: The effect of solvent/used oil ratio on percent solvent recovery at different extraction temperatures for ethanol extraction solvent.

2. Effect of Mixing Time

The effect of solvent/used oil ratio on the oil recovery and solvent recovery percentage for different solvents are properly illustrated in Figures (8, 9, 10 and 11) which show the data obtained using n-butanol as an extraction solvent where the percentage of oil recovery and solvent recovery are progressively increased with increasing solvent ratio at different mixing times. Whereas, the percentage of oil recovery and solvent recovery are stabilized on (89.7wt. %) and (91.5wt. %) respectively and both at solvent/oil ratio of 3:1.

It is also realized from these two figures that increasing mixing time has very limited effect on the percentage of oil and solvent recovery , i.e. when the mixing time increased from 15 to 60 min, the oil and solvent recovery will be insignificantly improved specially when the solvent / oil ratio at 3:1 and above.

On the other hand, figures (10 to 13) show slightly different behavior of oil and solvent recovery when using 2- propanol and ethanol. It is obvious noted from those figures that increasing mixing time will give enhancement in both oil and solvent recovery. Where, the oil recovery will be promoted almost 17% at 3:1 solvent/oil ratio when using 2- propanol as solvent with increasing the mixing time from 15 to 60 min.



Figure 8: The effect of solvent/used oil ratio on percentage oil recovery at different mixing times for n- butanol as an extraction solvent.



Figure 9: The effect of solvent/used oil ratio on percent solvent recovery at different mixing times for n- butanol as an extraction solvent.



Figure 10: The effect of solvent/used oil ratio on percent oil recovery at different mixing times for 2- propanol as an extraction solvent.



Figure 11: The effect of solvent/used oil ratio on percent oil recovery at different mixing times for ethanol as an extraction solvent.



Figure 12: The effect of solvent/used oil ratio on percent solvent recovery at extraction mixing times for 2- propanol as an extraction solvent.



Figure 13: The effect of solvent/used oil ratio on percent solvent recovery at different mixing times for ethanol as an extraction solvent.

4.2 Binary Solvent Extraction

The effect of solvent/used oil ratio on the oil recovery and solvent recovery percentage for different solvents is properly illustrated in Figures 14, 15, 16 and 17 respectively at conditions of temperature (40° C) and mixing time of (30 min). The data collected and represented in the above mentioned figures indicated that the oil recovery was evidently increased with increasing binary mixture solvent/used oil ratio. In average the increment of base oil recovery was noted to be between (35 - 45) percent when the binary mixture of solvent raised four times.

It is also noted from the above mentioned figures that the oil recovery and solvent recovery were increased when the binary solvent composition varies from (M1 – M4), where M1 indicated that the solvent mixture consists of 20/80 wt. /wt. polar solvent (acetone) / non-polar solvent (methyl ethyl ketone or Heptane). Whereas, M4 represents solvent composition of 80/20 wt. /wt. acetone/non-polar solvent. The values of oil recovery and solvent recovery are generally between (10 - 69) %, (15 - 71) % and (9 - 61) %, (13 - 70) and (acetone & heptane) respectively

















In general, the oil recovery through the extraction process when using the acetone plus methyl ethyl ketone as a binary mixture gave higher yield percent comparing with other mixture of acetone plus heptane at the same working conditions.

4.3 Bleaching

The measured data of color number for final product of base oil which is extracted using ethanol and mixture of methyl ethyl ketone and acetone with various type and weight of bleaching agents at specified working conditions from temperature, solvent ratio and mixing time at 40°C, 4:1 and 30min. Various weight of clay from (0.1- 0.5) g were tested to modify the color of base oil extracted using ethanol as solitary solvent and methyl ethyl ketone plus acetone as binary solvent at selected working conditions. These will be discussed below.

1. Bleaching of Base Oil Extracted by Solitary Solvent

The variation of base oil color tone with weight of bleaching agent at different types of bleaching clays are shown in Figure (18). This figure indicates that color number is almost unaffected by increasing the clay weight of base bentonite. While, there is a significant decreasing of color number with increasing the weight of bleaching agent i.e. the color transform from dark brown to light yellow when the weight of clay increases from 0.1 to 0.5 grams for both acid bentonite and dead sea clay. The acid bentonite gave better result than Dead Sea clay where it modified the color by 1.4 degrees on color number scale comparing with 0.5degree achieved with the same amount used from Dead Sea clay. This effect could be attributed to the difference in the mineralogical compositions of two clay samples. Also, the method of activating the Dead Sea clay needs more study to obtain the best morphological surface of the clay particles and remove the impurities constituent from the clay such as quartz and feldspar [11].



Figure 18: The effect of weight of bleaching agent on color number at different types of clay for base oil extracted with solitary solvent at 40°C, 4:1 solvent/used oil ratio and 30min mixing time.

2. Bleaching of Base Oil Extracted by Binary Solvents

Generally, the trained of improving color of base oil which is extracted using binary solvent (methyl ethyl ketone plus Acetone) is the same as that observed from using solitary solvent. Figure (19) shows the effect of beaching agent weight on the color number of base oil extracted by mixture of solvents at particular conditions. It is clearly shown in this figure that the basic bentonite has very limited effect on improving color of base oil and the variation of color number with increasing solid weight is constant. On the other hand, the activated Dead Sea salt gave better result on improving the color of base oil which is almost the same that obtained from acidic bentonite where the color was improved as 1.5 degree for the both clays. This may be regarded due to acid effect which is able to dissolve the impurities from the clay and raise the Si/ (Al ³⁺ +Mg ²⁺ +Fe ^{2+/3+}) ratio. These results were confirmed by the results published by Foletto et al [11]. Where approved that the increase of acid concentration cause a greater attack on the clay structure and consequently, improves the bleaching capacity.





While, table (4) shows the final comparison between base oil (stock 60), base oil extracted by solitary solvent and binary solvent to the properties of ASTM. It is obviously noted from the above mentioned table that the base oil extracted using solitary solvent gave more close measurements than that extracted by binary solvent. While, oil seems to be more light than (stock 60) oil referring to the specific gravity measurements. That could be explained as that solvent technique just extract light compounds

and make the oil less specific gravity. Moreover, it clearly seen that both types of base oil still have little amount of ash comparing with slandered sample and that may attributed to presence of some impurities with oil. That could be regarding to the inaccuracy when the separation of extract / raffineate step take place. Regardless those points, the base oil produced by extraction process could be match or very close to that produced from mineral oil in Durah Refinery.

Table 4: '	The measured p	roperties of th	ne base oil	extracted b	oy solitary	solvent and	base oil
	extrac	cted by binary	v solvent v	vith stock 6	0 base oil		

No.	Properties	Base oil extracted by solitary solvent	Base oil extracted by binary solvent	stock 60 base oil ASTM*
1	Specific gravity	0.75	0.74	0.8
2	Pour point °C	-12	-13	-10
3	Viscosity at 100 °C in cp	10	9.5	11.7
4	Total acid number(TAN)	0.8	0.75	1.2
5	Ash wt.%	0.55	0.58	Nil
6	Carbon Res. wt.%	0.28	0.26	0.3
8	Flash point °C	160	158	162
9	Color meter	1.3	1.5	2.4

*means the measured properties of the base oil (stock 60) supplied from Durah Refinery, before mentioned, table(2)

5. Conclusion

Results presented in this work permit us to conclude that it was possible to establish a set of technical steps that allow the recovery of fresh base oil from used lubricant oils. These steps included recycling of used lubricant oil by using solvent extraction and vacuum distillation facilities. Therefore, the following conclusions were obtained.

- 1- The solitary solvents n- butanol, 2- propanol and ethanol were employed for the extraction of the base oil. The most efficient solvent was n- butanol, followed by 2- propanol, and ethanol. Confirming the similarity of these compounds with petroleum aromatic fractions. Therefore, in this study, the optimum system for recovery 1- butanol gave the best result of oil recovery (93.7%), solvent recovery (96.2%) at 40°C (4:1wt. /wt.) solvent to used oil ratio. While at different mixing time 1butanol gave the best result of oil recovery (89.7%), solvent recovery (91%) at 60 min (5:1wt. /wt.).
- 2- The binary solvents were (acetone &methyl ethyl ketone) gave oil recovery (69%), solvent recovery (71%) and (acetone &heptane) gave oil recovery (61%), solvent recovery (70) at 40 0C ,30 min and ratio solvent to used oil ratio (5:1 wt./wt.).
- 3- The solitary and binary solvents give better result when treated with acidic bentonite and activated Dead Sea clay was(2.2and 2.5) color no at 0.5 weight of bleaching while on the other hand the color there is no enhancement recorded when it is treated with basic bentonite.

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تحسين الزيت الاساسي من زيت السياره المستعمل باستخدام مذيب الاستخلاص المفرد والخليط

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الخلاصة:

تم إجراء هذه الدراسة لتقيم تأثير المتغيرات العملية مثل؛ نوع المذيب , نسبة المذيب ,درجة حرارة الاستخلاص, و كذلك الزمن اللازم لاستخلاص كمية الزيت الأساس من الزيت المستنفذ بواسطة تقنيات الاستخلاص و التقطير.

تم إجراء مجموعة من التجارب العملية على مستوى مختبري على زيت المحركات المستهلك و الذي تم جمعة من محطات مختلفة لتبديل الزيت بهدف اختيار مذيبات أحادية مناسبة قادرة على استخلاص و إعادة تتشيط الزيت الأساس مثل (1- بيتانول, 2- بروبانول, ايثانول) و كذلك الخليط الثنائية من المذيبات مثل (الهبتان والمثيل اثيل كيتون) مع (أسيتون). حيث تم دراسة إضافة نسب مختلفة من المذيبات الكحولية إلى زيوت السيارات المستهلكة و كانت تتراوح مِنْ 1:1 إلى 1:5 (وزن إلى وزن) في الضغط الجوي و بدرجات حرارية مختلفة (30, 40, 50, 60) درجه مئوية و زمن خَلُّط مختلف النسب من الزيوت المستهلكة دقيقه. في حين تم اختبار تراكيب وزنيه مختلفة من المذيبات الثنائية مع مختلف الخليط المتيات عند درجة حرارة استخلاص ثابتة في 30 درجة مئوية .

علاوة على ذلك، ثلاثة أنواع مختلفة مِنْ أطيان قصر اللون (البنتونايت ألحامضي والقاعدي وأطيان البحر الميت) تم اختبارها بأوزان مختلفة مِنْ (0.1 إلى 0.5) غرام لكل 20 مل من الزيت الأساس المستخلص لمعرفة قدرتها على قصر اللون للزيت الأساس و ذلك في ظرف استخلاص معينة من درجة حرارة عند 40 درجه مئوية ونسبه مذيب إلى زيت4 :1 ووقت خلط 30 دقيقه.

إن النتائج المستحصلة الخاصة باستخلاص الزيت و استرجاع المذيب عند استخدام مذيبات كحولية لوحظ إنها تزداد مع زيادة نسبة المذيب/الزيت المستهلك و إلى حد معين. حيث إن 1- بيوتانول أعطى أعلى نسبة استرجاع للزيت الأساس ثم 2- بروبانول بعدها الايثانول.

علاوة على ذلك وجد إن زمن الخلط يعطي زيادة في نسبة الزيت الأساس المسترجع و الكمية المذيب المسترجع لمذيبان 2 – بروبانول و الايثانول , في حين لوحظ عدم تأثر نسبة استخلاص الزيت و استرجاع المذيب عند استخدام 1 – بيوتانول بزمن الخلط بعد 30 دقيقة .

من ناحية أخرى، وجد إن كمية الزيت الأساس المستخلص و الكمية المذيب المسترجع تزداد بشكل ملحوظ مع زيادة كمية المذيب الثنائي المستخدم و كذلك نسبة المذيب القطبي (الأستون) في الخليط الثنائي. من الناحية الأخرى إن الخليط الثنائي للأسيتون مع المثيل اثيل كيتون له تحسن كبير بالنسبة إلى المسترجع من الزيت مقارنه مع خليط الأسيتون مع الهبتان.

إنّ اللونَ النهائيَ للزيت تم تحسينه عن طريق البنتونايت ألحامضي وملح البحر الميت المنشط لكن لم يلاحظ إي تحسن عند أضافه البنتونايت القاعدي.