

## Re-refining of used lubricant oil by solvent extraction using central composite design method

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**Abstract**–The primary aim of this study was to recover base oil from used oil using solvent extraction followed by the adsorption method. Many effective variables were examined within the solvent extraction method, including using different solvents, solvent/used oil, temperature and speed of blending. Central composite design (CCD) was applied as the statistical method. Response surface methodology was then used to find the optimum conditions in the process of extraction: ratio of solvent/used oil 2.4 and 3.12 vol/vol, temperature=54 and 18 °C, and speed of mixing=569 and 739 rpm for 1-butanol and methyl ethyl ketone (MEK), respectively. Various flocculation agents were used with the solvent, such as Sodium hydroxide (NaOH), Potassium hydroxide (KOH) and Monoethylamine (MEA); they provided an increase in the separation efficiency. The best result was obtained when using 2 grams of MEA/kg solvent; this amount of MEA increases sludge removal from 12.6% to 14.7%. In the process of clay adsorption, the variables that were tested included the ratio of clay/extract oil, temperature and time of contact. The best conditions in the process of adsorption by activated bentonite were a ratio of clay/extract oil=15 wt/vol%, temperature=120 °C, and time of contact=150 minutes. The recovered base oil was analyzed by Fourier transform infrared spectroscopy (FTIR) and compared to Iraqi specifications of base oils. The recovered base oil specifications were analyzed, including, viscosity @100 °C 8.32, 9.22 cSt, pour point –17.35, –22.23 °C, flash point 210.12, 223.04 °C, total acid number (TAN) 0.25, nill, total base number (TBN) nill, nill, ash 0.031, 0.0019 wt% and color 3.0, 2.5 for two types of base oil recovered using MEK, 1-butanol with activated bentonite, respectively.

Keywords: CCD, Optimization, Re-refining, Used Lubricant Oil

### INTRODUCTION

Within our modern society of advanced technology, the rise in the number of systems pertaining to power generation, for several services, requires an increasing amount of lubricants [1]. When utilized in machines and equipment, the lubricating oils are exposed to severe conditions and excessive temperatures that lead to the formation of a complex mixture called sludge; this usually contains additives causing degradation. The sludge can include heavy metals from engine wear and poly aromatic hydrocarbons (PAHs) from additive degradation, both of which are considered hazardous materials [1,2].

The disposal of used lubricating oil creates an environmental risk because it contains numerous amounts of impurities that have a negative impact on the environment if they are not treated. Furthermore, the used lubricating oil contains a high percentage of the base oil that can be recycled [3]. In general, lubricating oil contains ~71-96 wt% base oil and an additive ~29-4 wt% [4]. The primary reason for re-refining waste is usually to recover the lubricating oil base oil from the waste lubricating oil [5,6].

Solvent extraction is the preferred method in the re-refining of

used lubricating oils since it does not require complex processes, it produces acceptable properties of recovered base oil, does not require large energy, and the expense is less compared to other methods [7].

The solvent extraction process includes constructing a model based on the central composite design (CCD) for simulating the sludge outlet yield of waste lubricating oil, and to find the optimum condition [5,6]. The CCD approach represents a method of investigating the response surface methodology, which includes a simulation of the relationship between quantitative variables as well as the process variables and the response variable locking combination process which offers an optimum expected response [8,9].

The present work describes the structure models based on the statistical and mathematical methods of CCD to simulate the sludge yield during the extraction process of used lubricant oil and optimization of its conditions, such as solvent/used oil ratio, temperature, and speeds of mixing.

### MATERIALS AND METHODS

#### 1. Materials Used

The selected lubricating oil for this study with specifications as shown in Table 1 was obtained from Shell Co. after it was used in the electrical power generator type 350 KVA Perkins for a period of time. The used oil was first filtered and then subjected to a de-

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**Table 1. Characterization of used and fresh lubricant oil**

Properties	Units	Used oil	Fresh oil
1 Viscosity @ 40 °C	cSt	100.20	97.90
2 Viscosity @ 100 °C	cSt	14.31	13.62
3 Viscosity Index		129.60	126.43
4 Flash point	°C	224.3	227.5
5 Pour point	°C	-27.5	-27.6
6 Density	kg/m <sup>3</sup>	887.12	886.24
7 TAN	mg KOH/gm oil	3.13	0.12
8 TBN	mg KOH/gm oil	2.72	11.45
9 Ash content	wt%	0.89	1.12
10 H <sub>2</sub> O content	vol%	Nil	Nil
11 Sulfur content	wt%	0.82	0.96
12 Color value		Black	3

hydration process using vacuum distillation at 200 °C to remove water and any lighter hydrocarbon contents. In our study, analytical grade solvents 1-butanol and MEK supplied by REDAL DE HANE and SIGMA-ALDRICH were selected.

## 2. Experimental Set-up and Methods

### 2-1. Process of Extraction

A mixture of solvent and used lubricating oil was homogenized

to ensure adequate mixing at the different conditions as shown in Table 2. The sludge was permitted to settle by using a centrifuge. Subsequently, the solvent-oil mixture was separated into extract phase and sludge as raffinate phase. The same solvent was used to wash the sludge to remove any base oil remaining in the sludge and was subsequently dried in the oven. The sludge removal percentage (P.S.R) can be calculated using the following equation [1,10].

$$(P.S.R) = (W_{dry}/W_{oil}) \times 100\%.$$

### 2-2. Experimental Design

CCD is a combination of statistical and mathematical techniques used to design experiments, evaluate the effect of variables, find the optimum conditions and obtain a relationship between variables that are useful to model and analyze the problem [11-14].

CCD was applied in this study to determine the optimum conditions that give the highest percentage of sludge removal in the process of used oil extraction. The experiments were designed to use three independent variables: solvent/used oil ratio ( $X_1$ ), temperature ( $X_2$ ), and speeds of mixing ( $X_3$ ). CCD matrices are composed of 20 run of experiments involving the full design of the five levels to build second-order models as shown in Table 2. Minitab Release 16 (Minitab Inc., USA) software was used to simulate data in the extraction process. The following equation can represent the CCD model:

**Table 2. The experiments trial when 1- butanol and MEK is used**

Run	Ratio of solvent/Used oil ( $X_1$ )	Extraction temp. (°C) ( $X_2$ )	Speed of mixing (rpm) ( $X_3$ )	P. S. R. % when using (1-butanol)	P. S. R. % (when using MEK)
1	2.5	40	12000	11.1	6.1
2	1	60	5000	6.8	3.5
3	2.5	40	12000	11.24	5.85
4	4	20	5000	6.4	11.4
5	1	20	19000	2.7	7.9
6	4	60	19000	8.8	7
7	2.5	7	12000	1.2	13.3
8	2.5	40	12000	11.19	6.2
9	0.05	40	12000	0	0
10	2.5	40	569	12.6	7.11
11	2.5	40	23431	7.39	9
12	4.9	40	12000	6.8	8.7
13	2.5	72	12000	8.2	5.1
14	2.5	40	12000	11.31	6.51
15	4	20	19000	4.1	12.8
16	2.5	40	12000	10.9	6
17	1	20	5000	2.7	4.9
18	2.5	40	12000	11.17	6.12
19	1	60	19000	5.3	4.7
20	4	60	5000	12.8	8

**Table 3. Analysis of activated bentonite**

	SiO <sub>2</sub>	CaO	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	Surface area m <sup>2</sup> /gm	Pore volume cm <sup>3</sup> /gm
Activated bentonite	74.44	1.5	2.16	1.41	6.79	158.035	0.2144

$$Y = \beta_0 + \sum_{i=1}^n \beta_i x_i + \sum_{i=1}^n \beta_{ii} x_i^2 + \sum_{i=1}^{n-1} \sum_{j=i+1}^n \beta_{ij} x_i x_j + e_i \quad (1)$$

## RESULTS AND DISCUSSION

## 3. Adsorption Process

Activated bentonite was employed as an adsorbent in this process. Analysis of activated bentonite is shown in Table 3. The extract oil was mixed with the activated bentonite in a variable percentage ratio of clay/extracted oil, extraction temperature, and mixing time at a constant speed of ~500 rpm. The next stage involved separating the recovered base oil adsorbent clay using a filter paper type 542 Whatman. The final stage involved an analysis of the produced base oil.

## 1. Analysis of Variance

Data obtained from Table 2 was analyzed using the CCD. The analysis results by ANOVA (95% confidence) show that the model F value was 83.57 and 69.2 as highlighted in Table 4 and Table 5. The model P value was less than 0.05; this means the model has significance, while values larger than 0.1 do not have significance. The coefficient of determination  $R^2$  was controlled by the suitable mode. The model's  $R^2$  98.70% and 98.44% for 1-butanol and MEK were recorded. Also, there was a suitable agreement with the ad-

Table 4. Regression model ANOVA for the extraction 1-butanol

Source	DF	Squares sum	Mean square	F value	P value	Coefficient
Regression	9.0	282.38	31.38	83.58	0.000	+11.10
Linear	3.0	123.50	41.66	109.65	0.000	
$X_1$	1.0	45.41	45.43	120.96	0.000	+3.01
$X_2$	1.0	59.34	59.36	158.09	0.000	+3.45
$X_3$	1.0	18.74	18.75	49.94	0.000	-1.94
Square	3.0	154.73	51.58	137.57	0.000	
$X_{12}$	1.0	87.88	98.77	263.09	0.000	-7.29
$X_{22}$	1.0	65.94	66.70	177.66	0.000	-5.99
$X_{32}$	1.0	0.91	0.91	2.45	0.155	-0.66
Interaction	3.0	4.17	1.39	3.73	0.051	
$X_1, X_2$	1.0	1.92	1.91	5.07	0.049	+1.31
$X_1, X_3$	1.0	1.71	1.72	4.57	0.058	-1.24
$X_2, X_3$	1.0	0.56	0.56	1.48	0.254	-0.08
Error (residual)	10.0	3.757	0.38			
Lack-of-fit	5.0	3.66	0.74	36.30	0.001	
Error (pure)	5.0	0.11	0.03			
Total	19.0	286.15				

Note that,  $R^2=98.70\%$ ,  $\text{Adj-}R^2=97.50\%$

Table 5. Regression model ANOVA for MEK extraction

Source	DF	Squares sum	Mean square	F value	P value	Coefficient
Regression	9.0	177.90	19.77	69.22	0.000	+6.12
Linear	3.0	138.65	46.22	161.82	0.000	
$X_1$	1.0	78.77	78.77	275.81	0.000	+3.97
$X_2$	1.0	55.46	55.45	194.17	0.000	-3.33
$X_3$	1.0	4.44	4.44	15.53	0.003	+0.94
Square	3.0	32.60	10.86	38.05	0.000	
$X_{12}$	1.0	7.60	5.14	17.98	0.002	-1.66
$X_{22}$	1.0	17.26	18.85	66.02	0.000	+3.19
$X_{32}$	1.0	7.74	7.74	27.11	0.000	+2.04
Interaction	3.0	6.66	2.22	7.78	0.007	
$X_1, X_2$	1.0	2.65	2.65	9.27	0.013	-1.53
$X_1, X_3$	1.0	1.81	1.81	6.33	0.032	-1.27
$X_2, X_3$	1.0	2.23	2.21	7.71	0.020	-1.40
Error (residual)	10.0	2.85	0.29			
Lack-of-fit	5.0	2.62	0.52	10.64	0.012	
Error (pure)	5.0	0.25	0.05			
Total	19.0	180.75				

justed determination coefficient. Adj- $R^2$  values of 97.50% and 97.1% were found to 1-butanol and MEK, respectively. The values of  $R^2$  and Adj- $R^2$  approximate 1.0; this is high and supports a high relation between the determined values and the values which were expected. This indicates that the regression model offers a perfect explanation of the relations between the independent variables and also the response.

The response model of surface regression achieved for sludge deposition according to Table 2 and Eq. (1) for 1-butanol and MEK is:

$$Y_b \text{ (sludge removal \%)} = 11.11 + 3.01 X_1 + 3.44 X_2 - 1.94 X_3 - 7.30 X_{12} - 6.01 X_{22} - 0.67 X_{32} + 1.32 X_1 X_2 - 1.24 X_1 X_3 - 0.08 X_2 X_3 \quad (2)$$

$$Y_M \text{ (sludge removal \%)} = 6.12 + 3.97 X_1 - 3.34 X_2 + 0.95 X_3 - 1.66 X_{12} + 3.19 X_{22} + 2.05 X_{32} - 1.53 X_1 X_2 - 1.27 X_1 X_3 - 1.41 X_2 X_3 \quad (3)$$

From Eq. (2) it can be concluded that  $X_1$  and  $X_2$  have a positive effect which crosses the percent of sludge removal ( $Y_b$ ) in the extraction process, while  $X_3$  has a negative effect. Also, a positive interaction occurred between  $X_1$  and  $X_2$  due to the solubility change when the temperature changed, when using 1-butanol as a solvent.

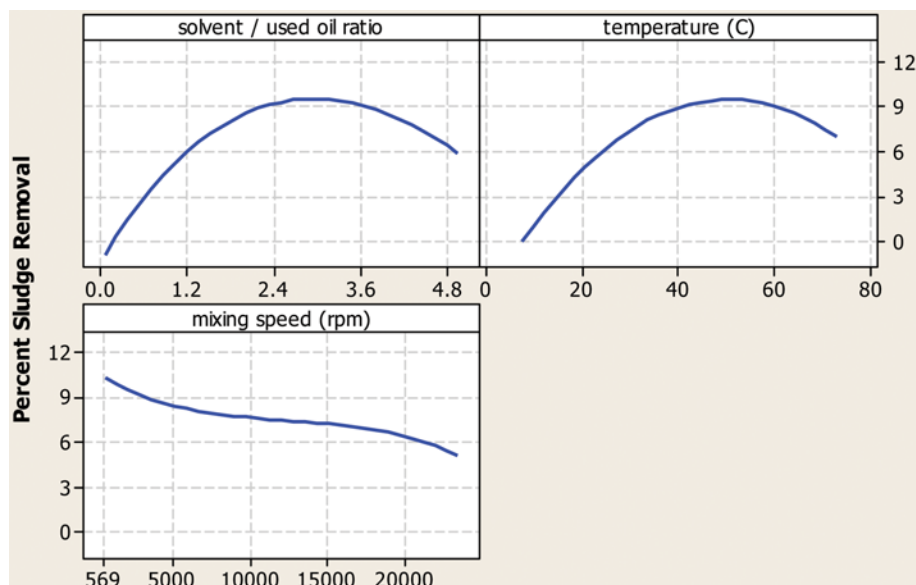


Fig. 1. Effect of extraction variable on the percent of sludge removal for 1-butanol extraction.

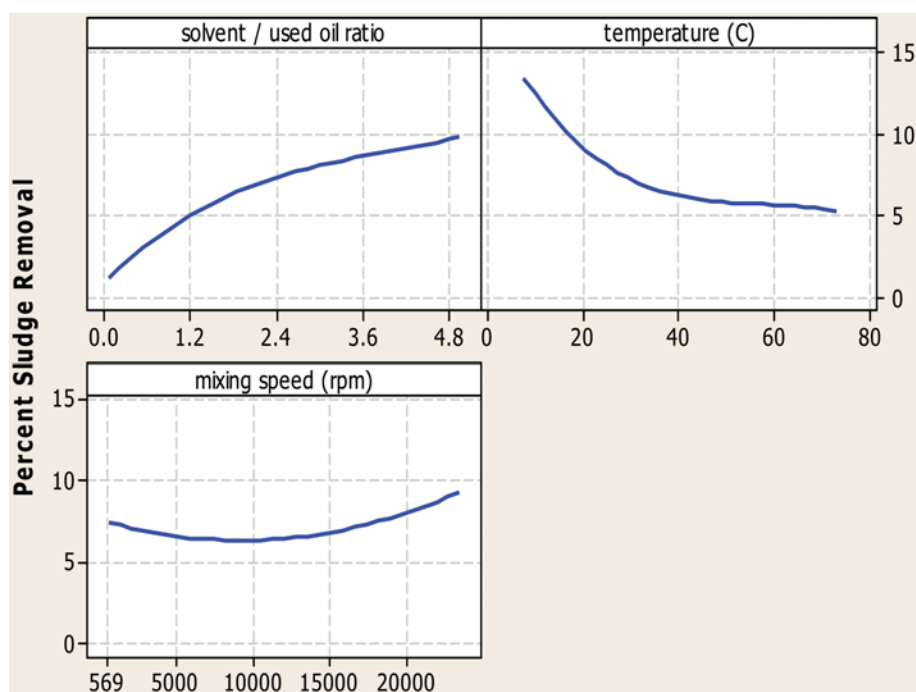


Fig. 2. Effect of extraction variable on the percent of sludge removal for MEK extraction.

From Eq. (3) it is concluded that  $X_1$  and  $X_3$  have a positive effect which crosses the percent of sludge removal in the extraction process, while  $X_2$  has a negative effect for MEK extraction.

## 2. Effect of Extraction Process Variables

The experiments that were made when 1-butanol and MEK were used as a solvent are shown in Table 2. This set of experiments indicate an increase in the percent of sludge removal with increasing solvent/used oil ratio ( $X_1$ ) for 1-butanol and MEK extraction. The percent of sludge removal (P.S.R) increases with increasing temperature ( $X_2$ ) for 1-butanol extraction, while it decreases with increasing temperature for MEK extraction. The P.S.R decreases with increasing mixing speed ( $X_3$ ) for 1-butanol extraction and increase in MEK extraction as shown in Figs. 1 and 2.

The percent of sludge removal is low at a small solvent/used oil ratio because at the smaller ratio, the solvent saturates and does not dissolve all base oil in the used lubricating oil. Subsequently, more base oil will dissolve with increasing solvent ratio until reaching maximum sludge removal at 2.5-3.5 solvent ratio. This is because of the increase in solubility with increasing solvent ratio as shown in Fig. 1 for 1-butanol extraction. This ratio range can also indicate that increase in solvent ratio by more than 3.5:1 leads to a decrease in the percent of sludge removal because the increase in solvent ratio leads to improved solvency power, and this leads to redissolution of sludge for 1-butanol extraction. This is in agreement with the results of Rafie et al. [1].

When using MEK solvent as shown in Fig. 2, the percent of sludge removal increases greatly with increasing solvent/used oil ratio ( $X_1$ ) because of the increase in solubility. This result is in agreement with those obtained by Durrani et al. that showed the sludge removal percentage increases with the ratio of MEK solvent/used oil [8].

The result shown in Fig. 1 indicates that the increase in percent sludge removal with increasing temperature occurs as increasing temperatures lead to decreased viscosity of 1-butanol and base oil, which results in an increase in the amount of base oil dissolved. However, an increase in temperature of more than 40-60 °C leads to increased solubility; therefore, the sludge will dissolve again. These results are in accordance with those of Durrani et al. [8] and Aremu et al. [7], which confirmed that better removal of the sludge occurs at 50 °C when 1-butanol is used as a solvent. Nevertheless, when MEK is used as a solvent, higher sludge removal takes place at a lower temperature ( $X_2$ ) as shown in Fig. 2.

Therefore, this (MEK) solvent can be used effectively at ambient temperature in the winter season. These results are in line with those of Durrani et al. [8] and Kamal et al. [15], which proved that effective sludge removal takes place at low temperatures.

The increasing in mixing speed using 1-butanol as a solvent did not exceed the removal percentage of the sludge as shown in Fig. 1. Nevertheless, when MEK is used as a extraction solvent, increasing mixing speed leads to increasing the percent of sludge removal, especially when mixing more than 15,000 rpm, as shown in Fig. 2. This is due to increasing the distribution of solute molecules with the solution, which creates more effective contact areas for mass transfer between oil and solvent.

## 3. Optimum Condition Specifications

Finding the best operating conditions is the main goal of the

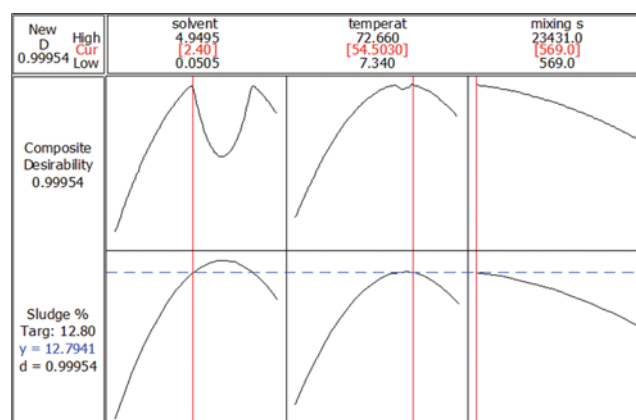


Fig. 3. Best conditions for extraction of 1-butanol.

extraction process that gives the optimum percentage removal of the sludge when response surface methodology is used.

Komaki et al. [16] explained that choosing optimum condition depended on minimum error and high composite desirability.

We have depended on the Minitab program to locate the optimal conditions by composite desirability; in this method we chose 2.4 as optimum solvent/used oil ratio. Note that the choice of this ratio depends on the error percentage of the mathematical model, which must be kept at minimum and high composite desirability.

Fig. 3 is divided into two parts, the second part from Fig. 3 shows that the percent sludge removal increases by increasing solvent ratio until reaching 3.5 and then decreases, but the Minitab program choice was 2.4 as the optimum solvent ratio. The first part from Fig. 3 explained why 2.4 was the choice as optimum because at this solvent ratio the model error was a little and high composite desirability was around 0.99954, but when increasing solvent ratio, the model error increased as shown in part 1 from Fig. 3.

Aremu et al. [7] and Hussein et al. [12], found by doing experiments that the best solvent/used oil ratio was 4:1 when using 1-butanol as a solvent.

Katiyar et al. [17] found that the best solvent/used oil ratio was 8:1 when using MEK as a solvent.

In our research the optimum conditions were: solvent/used lubri-

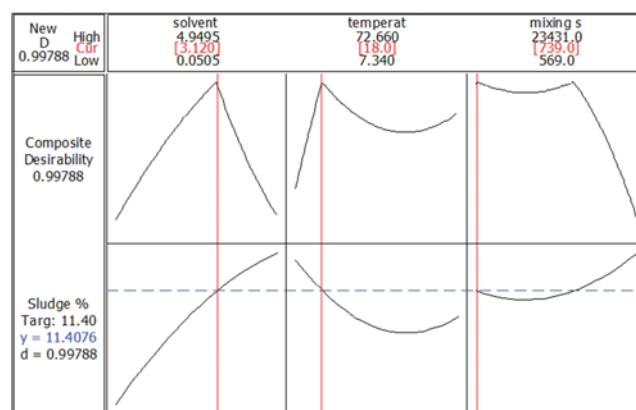


Fig. 4. Best conditions for extraction of MEK.

cating oil=2.4 and 3.1, temperature=54.5 and 18 °C, and mixing speed=569 and 739 rpm for 1-butanol and MEK extraction as illustrated in Figs. 3 and 4.

Experiments were conducted at the optimum conditions of solvent/used oil ratio, temperature, and mixing speed as shown in Figs. 3 and 4. The optimal percentage of sludge removal was 2.6% when using solvent butanol, and 11.15% for MEK solvent.

#### 4. Flocculation Agent

In this group of experiments, various amounts of different flocculation agents (KOH, NaOH, MEA) were added to the alcohol solvent (1-butanol only) at the best conditions. When the solvent (1-butanol) is mixed with used lubricating oil, it dissolves with the base oil, but some contaminants do not dissolve due to an electrical repulsion. Therefore, to control this electrical repulsion, a flocculation agent was added to the solvent to separate the small particles of flakes, coagulation, and precipitate [18]. Fig. 5 illustrates the sludge percentage removal when using a flocculation agent with 1-butanol solvent at its optimum condition. MEA was shown to be the best flocculation agent used at a concentration of 2.5 gm/kg solvent; it produced an increase in the percentage of sludge removed from 12.6% to 14.7%. Tables 6 and 7 show the properties of the base oil produced when the MEA was used as a flocculant with the solvent in the process of extraction.

#### 5. Adsorption Process

Adsorption process was the next step. The aim of the process is to separate any remaining contamination metal and then to enhance the color of the extract base oil, and choose the conditions

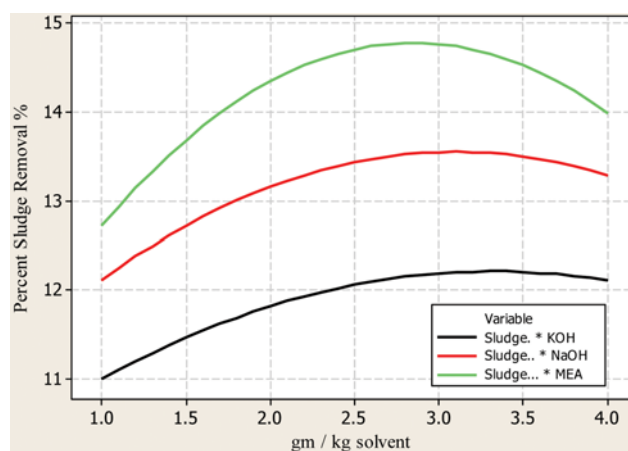


Fig. 5. Influence of flocculent agents on the percentage removal of sludge in extraction process by 1-butanol solvent.

that give or reach Iraqi specifications for base oils. The content of ash content was adopted as a response in the adsorption process.

#### 6. Variables Effect

These experiments were conducted using activated bentonite as an adsorbent for two types of extract oil by 1-butanol and MEK, as shown in Figs. 6 and 7. An improvement in the removal efficiency of ash was observed in the experiments by the increase in the temperature of adsorption from 30 to 120 °C, as shown in Figs. 6 and 7. This is due to an increase in adsorbate mobility in solu-

Table 6. Effect of adding MEA on the properties of extracting oil

No.	Property	Units	Used oil	Extraction by 1-butanol	Extraction by 1- butanol+MEA	Extraction by MEK
1	Viscosity @ 40 °C	Cst	100.20	8.35	8.58	7.73
2	Viscosity @ 100 °C	Cst	14.28	61.70	64.68	49.92
3	V. I		129.58	100.25	100.04	
4	Density	kg/m <sup>3</sup>	887.16	882.34	880.23	881.45
5	Flash point	°C	224.50	220.50	220.47	190.67
6	Pour point	°C	-27.25	-15.45	-19.26	-18.11
7	TAN	mg KOH/gm oil	3.14	1.72	0.11	1.93
8	TBN	mg KOH/gm oil	2.73	1.27	0.65	0.93
9	Ash	wt%	0.89	0.19	0.13	0.16
10	Sulfur content	wt%	0.82	0.69	0.60	0.18
11	Color		Black	8	6	7

Table 7. Effect of adding MEA on the metal analysis of extracting oil

No.	Metal	Used oil	Extraction by 1-butanol	Extraction by 1-butanol+MEA	Extraction by MEK
1	Cu	15.87	6.48	0	7.8
2	Mg	28.81	17.11	9.73	13.9
3	Zn	632.48	98.32	23	158.3
4	Fe	7.84	5.75	0	3.9
5	Al	64.04	18.55	0.81	2.2
6	Ca	3903.54	328.84	62.73	86.1
7	Pb	23.48	11.83	3.28	13.8
8	Mo	8.73	0	0	0
9	Mn	0.15	0	0	0

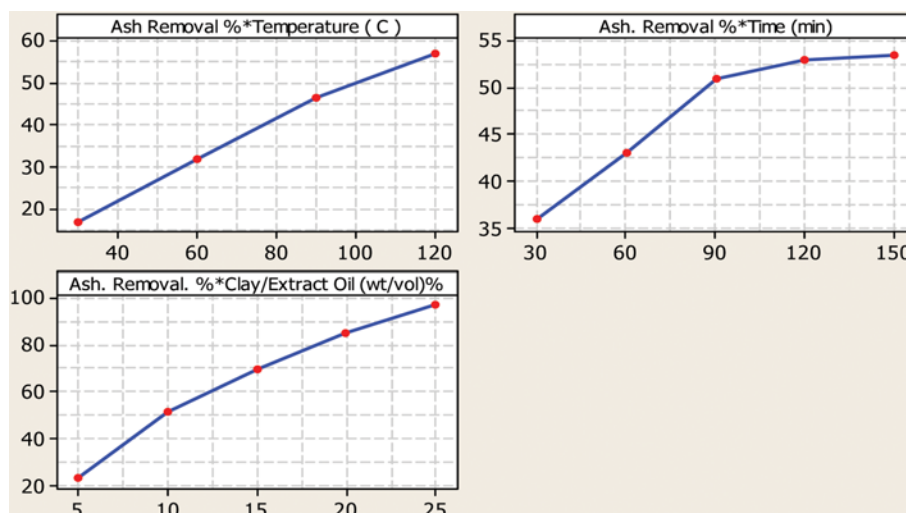


Fig. 6. Effect of adsorption variable on the percent of ash removal from the extracted oil by 1-butanol.

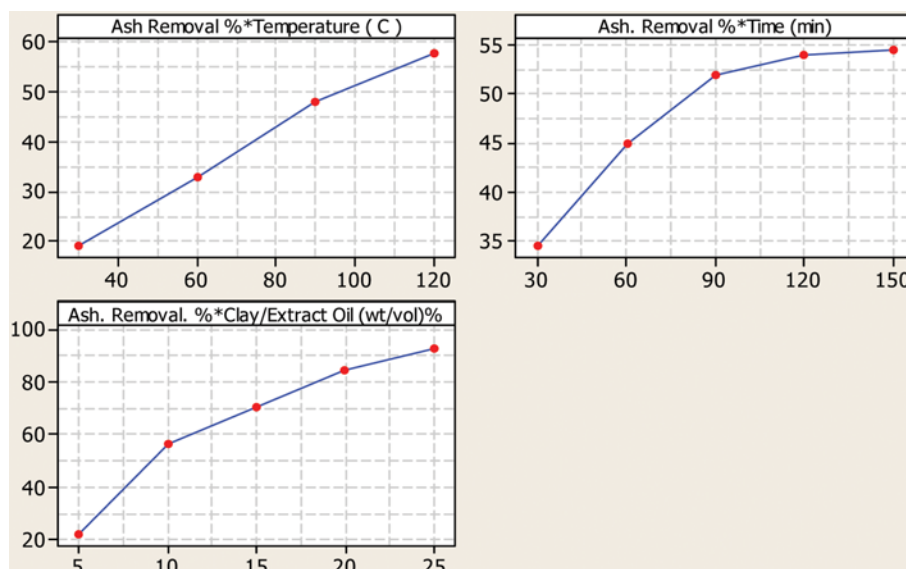


Fig. 7. Effect of adsorption variable on the percent of ash removal from the extracted oil by MEK.

tion within the sorbent structure overcoming the activation energy barrier [19]. Also, the rate of adsorption increased with the temperature due to the diffusion of adsorbate molecules to the adsorbent; moreover, the solubility and adsorption are inversely related as temperature affects the extent of adsorption [20]. Increasing the temperature more than 120 °C was non-economically viable, because at the best conditions we achieved 99% percent ash removal at 120 °C. This result is in accordance with Jodeh et al. [21].

The results in Figs. 6 and 7 indicate that the removal of ash grew very fast during the first 120 minutes of treatment and slowly reached equilibrium at 150 minutes when activated bentonite was used for two types of extract oil (extract oil by MEA+1-butanol and extract oil by MEK). This is because the areas of surface and pore volume are high for activated bentonite, creating more active sites [22].

This results were compared with Abdel-Jabbar et al. [23], who

found the contact time will reach equilibrium and give the best ash removal after 240 minutes when using activated bentonite as an adsorbent.

Also, Figs. 6 and 7 indicate that the percentage of ash removal depends on the percent of clay added. Salem et al. [24] explained the enhancement of oil specifications (due to contaminant removal) when an increased clay amount was used.

Tables 8 and 9 show the results obtained when using activated bentonite as an adsorbent at optimum conditions namely: 120 °C, 150 minutes, and 15 (wt/vol%) clay/extract oil ratio for two kinds of extract oil by 1-butanol+MEA and by MEK.

## 7. FTIR Analysis

From the FTIR analysis as shown in Figs. 8 to 11, there are two peak types: 1) main peaks like 2,953, 2,921, 2,852, 1,459, 1,376, and 721  $\text{cm}^{-1}$ , which are similar to the main elements found in the paraffin of base oils; and 2) secondary or undesirable peaks for

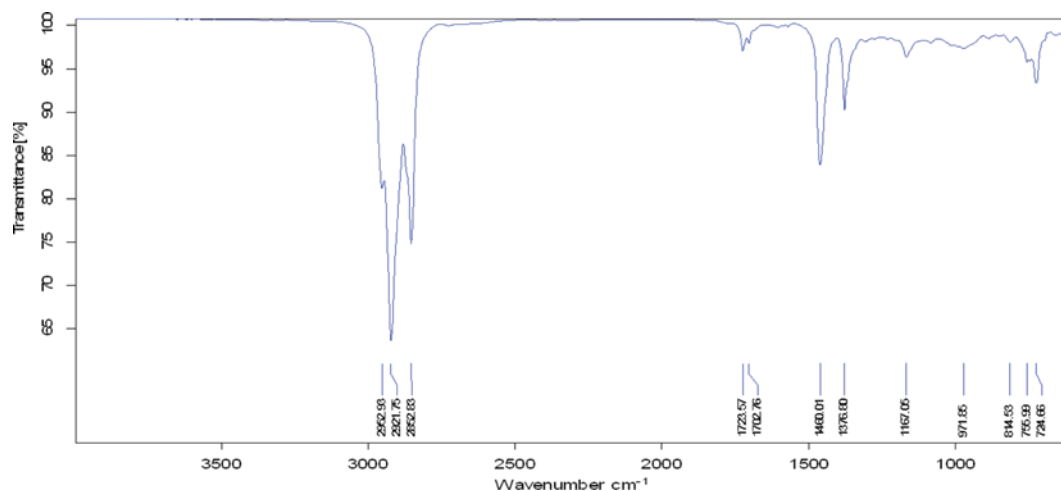


**Table 8. The recovered base oil characterization**

No.	Property	Units	Recovered base oil by 1-butanol+MEA and activated bentonite	Recovered base oil by MEK and activated bentonite
1	Viscosity @ 40 °C	Cst	70.12	53.03
2	Viscosity @ 100 °C	Cst	9.22	8.32
3	V. I		100.06	122.07
4	Pour point	°C	−22.23	−17.35
5	Flash point	°C	223.04	210.12
6	TAN	mg KOH/gm oil	Nil	0.25
7	TBN	mg KOH/gm oil	Nil	Nil
8	Density	kg/m <sup>3</sup>	875.45	868.34
9	Ash	wt%	0.0019	0.031
10	Sulfur content	wt%	0.47	0.61
11	Colour		2.5	3

**Table 9. Effect of adding MEA on the metal analysis of recovered base oil by 1-butanol+MEA and activated bentonite**

No.	Metal	Used oil	Recovered base oil by 1-butanol+MEA and activated bentonite	Recovered base oil by MEK and activated bentonite
1	Cu	15.75	0	0.1
2	Mg	28.80	0.21	0.34
3	Zn	632.51	0	3.22
4	Fe	7.87	0	0
5	Al	64.06	0	0
6	Ca	3903	3.87	41
7	Pb	23.60	0	0
8	Mo	8.73	0	0
9	Mn	0.17	0	0

**Fig. 8. FTIR analysis of used lubricant oil.**

metal contaminants: 2,725, 2,366, 2,323, 1,723, 1,704, 1,158, and 972 cm<sup>−1</sup>. Figs. 10 and 11 illustrate that the 1-butanol and MEK extraction followed by activated bentonite adsorption leads to the removal of all contaminants, leaving the base oil components like saturated alkane hydrocarbons as represented in 722, 1,456, 1,376, 2,953 cm<sup>−1</sup> peaks. The reason for the reduced TAN and TBN values is due to the elimination of all carboxylic acids as represented

in 951, 1,723, 1,702 cm<sup>−1</sup> peaks. Also, the decrease in the percentage of ash is due to the removal of oxidation products as represented in 2,366, 2,323 cm<sup>−1</sup> peaks. Furthermore, the improvement of the flash point is due to the elimination of aromatics and alkenes as represented in the 1,601, 1,567, 1,304, 971, 814 cm<sup>−1</sup> peaks. The lower viscosity and viscosity index improved are due to the separation of various components, such as aldehydes, alkyl halides, as



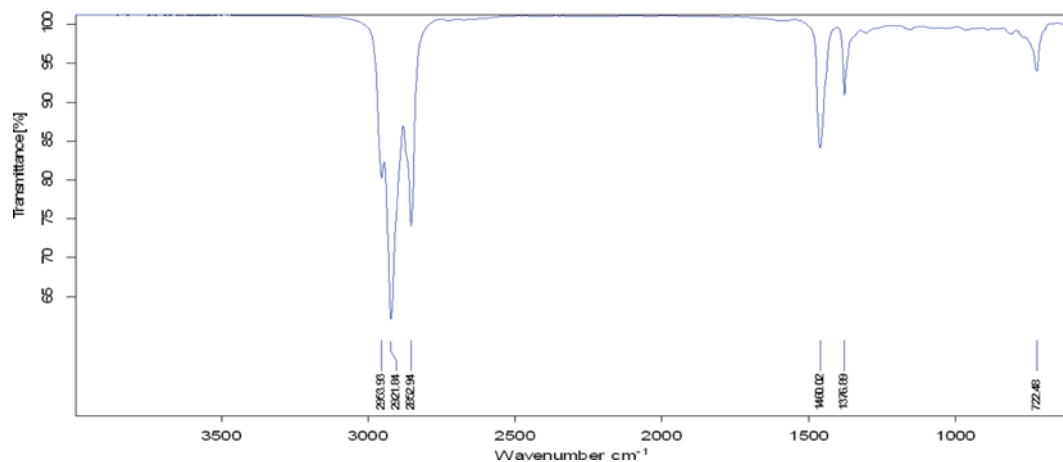


Fig. 9. FTIR analysis of fresh base oil.

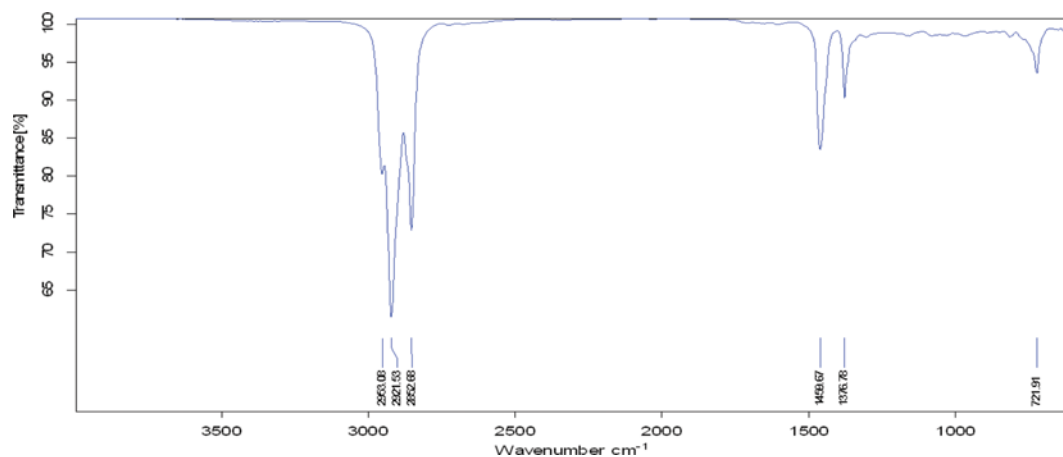


Fig. 10. FTIR analysis of recovered base oil by using 1-butanol+MEA+bentonite.

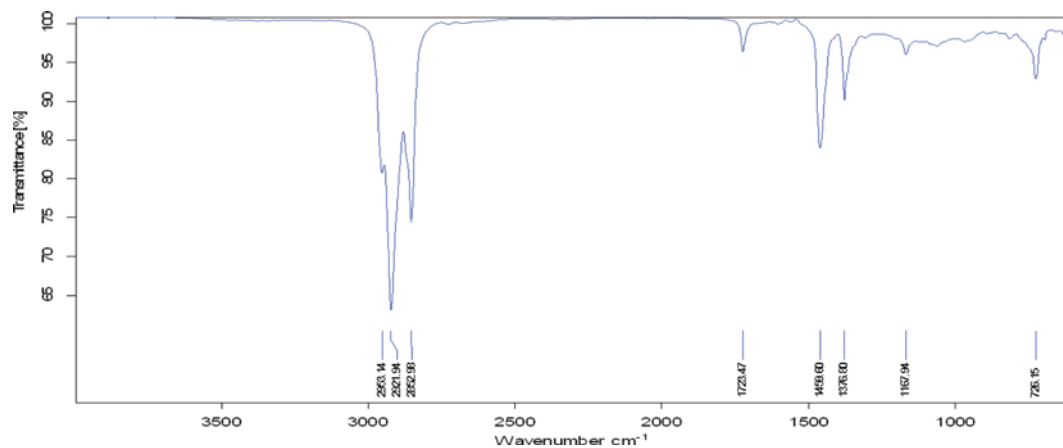


Fig. 11. FTIR analysis of recovered base oil by using MEK+bentonite.

represented in 1,245, 847, 746  $\text{cm}^{-1}$  peaks.

### CONCLUSIONS

1. Response surface methodology (RSM) was used to enhance

process variables for re-refining used lubricant oil.

2. This re-refining method consumes low energy, as high temperatures and low pressure are not needed.

3. 1-Butanol is better than MEK in the extraction process.

4. Optimum conditions in the process of extraction of solvent

were: solvent/used oil ratio=(2.4 vol/vol), temperature=(54 °C), and mixing speed=(569 rpm) for 1-butanol.

5. Adding MEA, KOH, and NaOH as the flocculant agent improves the ability of the butanol solvent as it removes the sludge in the process of extraction.

6. Adding 2 gm/kg of MEA to the solvent causes an increase in the percent sludge removal from 12.6% to 14.7%.

7. Best activated bentonite adsorption conditions were at bentonite/oil ratio 15 wt/vol%, temperature 120 °C, and contact time 90 min.

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