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Optimization of Base Oil Recovery from Used Lubricating Oil through Extraction-Adsorption using Response Surface Methodology

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Abstract

Response surface methodology, based on the central composite design, was successfully applied to study the optimum conditions and the statistical effects of the variables on base oil recovery from used lubricating oil using a two-step process for oil recovery: solvent extraction with 1-butanol compared to methyl ethyl ketone, followed by adsorption using activated clay. 1-Butanol was found to be more effective than methyl ethyl ketone for oil extraction and sludge removal. The optimum conditions for oil extraction were determined to be a 1-butanol-to-oil ratio of 3.2:1 at 57ºC and a mixing speed of 600 rpm. The removal of sludge was achieved at 12.08% by weight. Residual contaminant adsorption from the extracted oil was optimized using 55% activated clay by weight, stirred at 200 rpm 130°C. The physical properties of the treated oil were analyzed, revealing a specific gravity of 0.84, a viscosity index of 144, and a color reduction from 8.0 to 1.0. The chemical properties were analyzed by IR spectroscopy, showing reduced nitration, oxidation, sulfation, total base number, and the absence of water. Additionally, the GC-MS composition analysis of the treated oil revealed that 99.64% of the oil was composed of alkanes, with a trace amount of aromatic compounds and no naphthalene or polycyclic aromatic hydrocarbons (PAHs). The elemental content analysis by atomic emission spectroscopy showed that the residue was less than 1 ppm. As a result of the recovery under optimum conditions, the treated oil has chemical and physical characteristics that make it suitable for repurposing as base oil in industrial applications.

Keywords: *adsorption; base oil; central composite design; response surface methodology; solvent extraction; used lubricating oil*

1. Introduction

Lubricating oil is a fluid substance designed to reduce friction, wear, and heat generation between moving mechanical parts while providing several protective functions. Its composition is primarily divided into two main components: base oils and additives. Base oils constitute most of the lubricating oil's composition, typically making up 70-99% of the total volume. Additives make up 5-30% of the composition, depending on the specific lubricating oil and its intended use. Base oils are primarily mixtures of paraffinic, naphthenic, and aromatic hydrocarbons (for mineral oils), refined or synthesized to achieve desired properties such as viscosity, stability, and solubility. During engine operation, used lubricating oil undergoes chemical and physical changes from its fresh state by forming contaminants such as soot, metallic wear particles, adsorbed fuel residues, oxidation products, and polycyclic aromatic hydrocarbons (PAHs). Elements such as zinc (Zn), phosphorus (P), calcium (Ca), and sulfur (S) remain predominant, originating from additives and wear metals. Viscosity typically decreases after use due to thermal and mechanical degradation but may vary depending on contamination and treatment. Used lubricating oil should be properly managed to effectively remove contaminants and to obtain reusable base oils, which will reduce environmental impacts and resource consumption.

Several research studies have investigated various treatment processes for used lubricating oil (ULO), focusing on methods such as solvent extraction, vacuum distillation, acid/clay treatment, and adsorption techniques (Anisuzzaman et al., 2021; Oladimeji et al., 2018). The effects of solvent choice, mixing speed, temperature, and solvent-to-oil ratio on treating ULO using solvent extraction combined with adsorption have been studied (Dinesh et al., 2021; Osman et al., 2018; Pinheiro et al., 2018). Vacuum distillation has been used to treat different types of used lubricating oils, effectively removing water and light hydrocarbons. This method was shown to improve oil quality by separating contaminants and volatile fractions under reduced pressure and controlled temperature to prevent oil deterioration (Saleem & Karim, 2019). A comparative study including solvent extraction, followed by vacuum distillation found that distillation can improve viscosity and other oil properties (Eman & Shoaib, 2013) or used acid/clay treatment to remove impurities, degraded additives (Maneechakr et al., 2023), and heavy metals (Abu-Elella et al., 2015; Danane et al., 2014; Riyanto et al., 2018a; Ugwele et al., 2020). Treatment of used lubricating oil by solvent extraction followed by adsorption to remove remaining impurities is an effective method, which include solvent recovery (Khan et al., 2023; Khudhur & Mohammed, 2020; Riyanto et al., 2018b). These techniques resulted in different quantities and qualities of treated oil and demonstrate that combining physical and chemical methods tailored to specific conditions can efficiently regenerate ULO for reuse.

Several studies have used the central composite design and the response surface principle to increase the extraction process efficiency for base oil recovery from ULO (Araromi et al., 2018; Zgheib & Takache, 2020; Daham et al., 2017; Kamthita & Tiamsri, 2019; Sarkar et al., 2022). The study of solvent types and independent variables affecting the extraction and removal of contaminants has led to the development of more efficient lubricating oil treatments. The selection of an appropriate solvent is an important part of the base oil recovery process, considering the chemical properties of the solvent to dissolve the base oil, separate it from contaminants without reacting with the base oil. As well as the physical properties, such as a low boiling point, which can be recovered by distillation, reducing the impact on the environment, and conserving resources. Therefore, the application of response surface methodology (RSM) is a useful statistical approach to understand, develop, and optimize processes and products. Using this methodology, responses influenced by multiple variables can be modeled, analyzed, and optimized.

2. Objectives

This research studied the recovery of base oil from used lubricating oil by solvent extraction followed by adsorption. In the solvent-extraction process of base oil, a comparative study was conducted between 1-butanol which contains a hydroxyl group and methyl ethyl ketone with a carbonyl functional group. Each solvent has the same number of 4 carbon atoms but different molecular structures and properties. Then, adsorption with activated clay was performed to remove the remaining impurities in the oil and improve the oil's color. Optimization of base oil recovery from used lubricating oil through extraction and adsorption was investigated using central composite design (CCD) and response surface methodology (RSM). A second-order polynomial regression model was applied to analyze the results. The effects of independent variables on the oil extraction and adsorption processes were analyzed statistically.

3. Materials and Methods

3.1 Materials

Used lubricating oil (synthetic formula 5W-30 used in gasoline engines) was collected from car service centers. The used oil was filtered to separate large sediments and stored in bottles at room temperature. The solvents used for oil extraction were 1-butanol ($C_4H_{10}O$, AR grade >99.4% from RCI Labscan) and methyl ethyl ketone (C_4H_8O, AR grade >99.5% from RCI Labscan) with properties as shown in Table 1. Potassium hydroxide (KOH, AR grade from Ajax Finechem) was used as a flocculant. Activated clay ($H_2Al_2(SiO_3)_4{\bullet}nH_2O$, Guangxi Long'an Ruifeng Industrial) with a surface area ≥ 130 m^2/g and particle size ≥ 90 wt% passing a 0.075 mm screen mesh was used as an adsorbent.

Table 1 Physicochemical properties of 1-butanol and methyl ethyl ketone

Physicochemical properties	1-butanol (NBA)	methyl ethyl ketone (MEK)
Functional groups in molecular structure	Hydroxyl group (–OH group)	Carbonyl group (C=O group)
Boiling point	117.7°C	79.6°C
Dipole moment at 25°C	1.75 Debye	2.76 Debye
Polarity index	3.9	4.7

(Source: https://macro.lsu.edu/Howto/solvents.htm)

Table 2 Independent variables and their levels used for ULO extraction

Independent variables	Symbols	Variable levels				
		–1.682	**–1**	**0**	**+1**	**+1.682**
Solvent-to-used oil ratio (wt/wt)	R	2.66:1	3:1	3.5:1	4:1	4.34:1
Extraction temperature (°C)	T	33	40	50	60	67
Mixing speed (rpm)	S	64	200	400	600	736

3.2 Optimization for Recovering Base Oil from Used Lubricating Oil

3.2.1 Experimental Design for ULO Extraction Process

In this work, a comparative study of the used lubricating oil extraction process using solvents between NBA and MEK was developed to investigate the effects of the main operating variables (Pinheiro et al., 2018). A CCD was used to study the effects of three independent variables on sludge removal as the response: solvent-to-oil ratio (R), extraction temperature (T), and mixing speed (S). The experiments were conducted using a 5-level, 3-parameter design, with a total of 20 experiments. The independent variables and their levels are shown in Table 2. The experimental data were analyzed using response surface methodology, a statistical technique that used Minitab regression analysis and SigmaPlot. A second-order polynomial regression model, Equation (1), was used to examine and compare the solvents used in the extraction process at the optimal and most efficient conditions for sludge removal.

$$Y = \beta_o + \sum_{i=1}^{n} \beta_i X_i + \sum_{i=1}^{n} \sum_{j=i+1}^{n} \beta_{ij} X_i X_j + \sum_{i=1}^{n} \beta_{ii} X_i^2 \quad (1)$$

where Y is the predicted response, X_i , X_j are the uncoded independent variables, β_o is the intercept, β_i, β_{ii}, and β_{ij} are the model coefficients; n is the number of independent variables.

3.2.2 ULO Extraction Procedure

The experimental method started with dissolving KOH in the solvent at a ratio of 0.3 g per 100 g of solvent. The mixture was poured into ULO, using the specified solvent-to-oil ratio, mixing speed, and extraction temperature for 1 hour. The mixture was allowed to settle at room temperature for 24 h, then centrifuged at 2,000 rpm for 1 h. The solvent-oil mixture was separated from the sludge by filtration and solvent distillation to obtain the EO. The sludge was dried in an oven at 80°C for 3 h, the weight was recorded, and the percentage of sludge removal (PSR) was calculated according to Equation (2).

$$\text{PSR (\%)} = \frac{W_{\text{dry sludge}}}{W_{\text{ULO}}} \text{ x } 100 \quad (2)$$

where W_{ULO} is the weight of used oil and W_{Sludge} is the weight of dry sludge.

3.2.3 Experimental Design for EO Adsorption Process

The next step was to adsorb the EO under the optimum conditions with activated clay to remove any remaining contaminants and improve the color of the oil. In this work, the adsorption process was developed to investigate the effects of the main operating variables (Sarkar et al., 2022; Zgheib et al., 2020). A central composite design was used to study the effects of three independent variables: the amount of activated clay (A), the adsorption temperature (T), and the agitation speed (S). The response was measured based on the color of the oil. The experiments were conducted according to a 5-level-3-parameter, and the total number of experiments was 20. The independent variables and their levels are shown in Table 3. The experimental data was analyzed using response surface methodology, a statistical technique that used Minitab regression analysis and SigmaPlot. Equation (1), a second-order polynomial regression model, was utilized to study the optimum and efficient conditions for effective adsorption of residual contaminants.

Table 3 Independent variables and their levels used for EO adsorption

Independent variables	Symbols	Variable levels				
		–1.682	**-1**	**0**	**+1**	**+1.682**
Amount of activated clay (%wt)	A	28.2	35	45	55	61.8
Adsorption temperature (°C)	T	76	90	110	130	144
Agitation speed (rpm)	S	164	300	500	700	836

3.2.4 Extracted Oil Adsorption Procedure

The extracted oil was heated, then activated clay was added and stirred with the oil according to the specified conditions as shown in Table 3. The mixture was left at room temperature for 24 h and centrifuged at 2,000 rpm for 30 min. The mixture was filtered to separate the treated oil (TO) from the adsorbent. The weight was recorded, and the percentage yield of treated oil (% yield) was calculated according to Equation (3).

$$\%\text{Yield} = \frac{W_{TO}}{W_{ULO}} \text{ x } 100 \qquad (3)$$

where W_{ULO} is the weight of used lubricating oil and W_{TO} is the weight of treated oil.

3.2.5 Characteristics of Used Lubricating Oil and Treated Oil

ULO, EO, TO, and fresh lubricating oil (FLO) were examined for their physicochemical characteristics, including: 1) oil color, ASTM D1500, using a UV-visible spectrophotometer at 485 nm, which provides a visual and numerical scale for color measurement, ranging from 0.5 (light yellow) to 8.0 (deep red); 2) specific gravity, ASTM D1298; 3) kinematic viscosity, ASTM D445 and viscosity index, ASTM D2270; 4) nitration, oxidation, sulfation, total base number (TBN), and water content, using IR spectroscopy, ASTM D7889 reference ASTM E2412; 5) elements content, using atomic emission spectroscopy, ASTM D6595; and 6) organic components, using Gas chromatography-Mass spectroscopy.

4. Results and Discussion

4.1 Statistical Analysis for ULO Extraction

The comparative study results of the used lubricant oil (ULO) extraction using solvents between NBA and MEK were carried out according to the CCD as shown in Table 4. The RSM was applied to study the effects of three variables: solvent-to-oil ratio (R), extraction temperature (T), and mixing speed (S), on the percentage of sludge removal (PSR). The second-order polynomial regression models were performed using analysis of variance (ANOVA) at the 95% confidence level. A 95% confidence level was considered statistically acceptable. Terms are considered statistically significant if their associated p-values are less than 0.05 (Ugwele et al., 2020). In the regression analysis output, Significance F refers to the p-value associated with the F-statistics, indicating the overall significance of the regression model. As shown in Table 5, the Significance F-values were < 0.0001 for both NBA and MEK, indicating that the regression models are statistically significant and explain a substantial portion of the variance in the dependent variable. The coefficient of determination (R^2) for NBA and MEK was 0.9497 and 0.9411, respectively, indicating that the responses for both solvents were highly correlated with the independent variables. The significance of each variable was analyzed in terms of its probability value (p-value). For both solvents, the square terms R^2, T^2, and S^2 had p-values < 0.01, indicating highly significant quadratic effects on the response. The linear terms R and T exhibited statistically significant effects, whereas S did not. All interaction terms R*T, R*S, and T*S had p-values > 0.05, indicating no statistically significant interactions; thus, each variable independently influenced the response. However, the absence of significant interactions suggests that the model may not fully capture the underlying relationships for all factor combinations. A lack-of-fit test was performed to evaluate whether the model adequately explained the relationship between independent and dependent variables. For both solvents, the lack-of-fit test yielded a p-value <0.05, indicating that the models exhibited statistically significant lack-of-fit. These results indicate that the models did not fully represent the true relationships between the variables, as systematic patterns remained in the residuals that were not captured. Overall, the p-values of the main variables (R and T) were <0.05, confirming their statistical significance. Therefore, the second-order polynomial regression models were applied to relate all variables to the oil extraction responses using NBA and MEK as solvents, as shown in Equations (4) and (5), respectively.

Table 4 Experimental design and results in ULO extraction using NBA and MEK

Run No.	**R (wt./wt.)**	**T (°C)**	**S (rpm)**	**NBA**	**MEK**	**Run No.**	**R (wt./wt.)**	**T (°C)**	**S (rpm)**	**NBA**	**MEK**
				PSR (%)	**PSR (%)**					**PSR (%)**	**PSR (%)**
1	3.0:1	40	200	8.55	5.89	11	3.5:1	33	400	7.72	5.68
2	4.0:1	40	200	7.46	6.85	12	3.5:1	67	400	10.85	8.02
3	3.0:1	60	200	9.05	6.68	13	3.5:1	50	64	6.24	4.55
4	4.0:1	60	200	8.11	7.98	14	3.5:1	50	736	11.55	8.86
5	3.0:1	40	600	10.63	6.55	15	3.5:1	50	400	11.02	10.04
6	4.0:1	40	600	10.01	8.34	16	3.5:1	50	400	10.98	9.90
7	3.0:1	60	600	11.85	8.35	17	3.5:1	50	400	11.25	10.10
8	4.0:1	60	600	10.45	10.74	18	3.5:1	50	400	11.12	9.95
9	2.66:1	50	400	9.98	5.75	19	3.5:1	50	400	10.95	10.08
10	4.34:1	50	400	8.55	8.36	20	3.5:1	50	400	11.04	10.12

Table 5 Summary output from data analysis in ULO extraction

Solvent	**NBA**		**MEK**	
Regression statistics				
	R-Square	R-Square (adj.)	R-Square	R-Square (adj.)
	0.9497	0.9044	0.9411	0.8881
ANOVA				
	F-value	p-value	F-value	p-value
Regression	20.98	0.0000	17.75	0.0000
Term:	Coefficients	p-value	Coefficients	p-value
Intercept	–35.3	0.0056	–58.9	0.0009
Linear; R	14.84	0.0047	23.19	0.0011
T	0.620	0.0063	0.837	0.0041
S	0.01855	0.0416	0.0090	0.3904
Square; R*R	–2.144	0.0020	–3.391	0.0004
T*T	–0.00519	0.0022	–0.00903	0.0002
S*S	–0.000017	0.0004	–0.000024	0.0001
Interaction; R*T	–0.0158	0.6602	0.0235	0.6029
R*S	0.00001	0.9944	0.00240	0.2983
T*S	0.000032	0.7215	0.000143	0.2219
Error:	F-value	p-value	F-value	p-value
Lack-of-Fit	39.16	0.001	97.47	0.000

4.2 Optimization of ULO Extraction

The optimization of three variables: solvent-to-oil ratio, extraction temperature, and mixing speed, on the percentage of sludge removal was studied using the second-order polynomial regression model of oil extraction with NBA and MEK as solvents in Equation (4) and Equation (5), respectively. The RSM are illustrated in Figures 1 and 2 at the midpoints of each variable. Under optimal conditions using NBA, a solvent-to-oil ratio of 3.2:1, extraction temperature of 57°C, and mixing speed of 600 rpm, the maximum sludge removal achieved was 12.08%. Similarly, for MEK extraction, the optimal conditions, a solvent-to-oil ratio of 3.8:1, extraction temperature of 56°C, and mixing speed of 540 rpm, achieved a maximum sludge removal of 10.78%. To validate the regression models, experiments were performed under the predicted optimal conditions in triplicate. The observed sludge removal efficiencies were 12.01% ± 0.58 for NBA and 10.53% ± 0.45 for MEK, which closely matched the predicted values. Therefore, NBA was identified as the more efficient solvent for used oil extraction under optimal conditions.

NBA solvent extraction:

$$\begin{aligned} \text{PSR (\%)} = &-35.3 + 14.84\,R + 0.620\,T + 0.01855\,S \\ &- 2.144\,R{*}R - 0.00519\,T{*}T - 0.00001668\,S{*}S \\ &- 0.0158\,R{*}T + 0.00001\,R{*}S + 0.0000319\,T{*}S \end{aligned} \quad (4)$$

MEK solvent extraction:

$$PSR (\%) = -58.9 + 23.19\, R + 0.837\, T + 0.0090\, S - 3.391\, R*R - 0.00903\, T*T - 0.00002429\, S*S + 0.0235\, R*T + 0.00240\, R*S + 0.000143\, T*S \quad (5)$$

4.3 Effects of Variables on Solvent Extraction

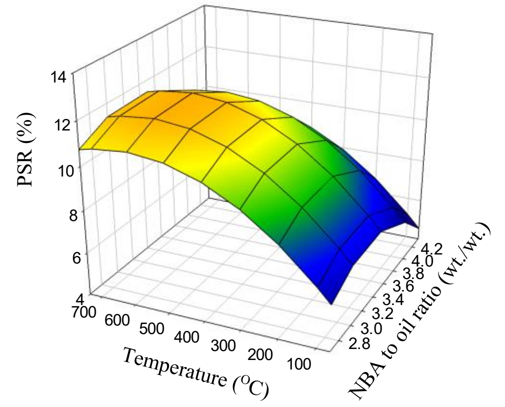
The solvent-to-oil ratio had a significant influence on extraction efficiency. Higher solvent-to-oil ratios improved oil recovery by enhancing solvent contact with ULO; however, beyond the optimal ratio, sludge separation efficiency decreased (Pinheiro et al., 2018). At a constant extraction temperature of 50°C (Figure 1a), NBA solvent extraction showed increased PSR at solvent-to-oil ratios between 2.7:1 and 3.2:1, whereas further increasing the ratio beyond 3.3:1 caused a decline in PSR. Since NBA has a solubility equilibrium with non-polar hydrocarbon components in oil, the NBA-to-oil ratio of 3.2:1 was considered the optimum ratio to achieve a high percentage of sludge removal. For MEK extraction at a constant temperature of 50°C (Figure 2a), PSR increased between solvent-to-oil ratios of 2.7:1 and 3.8:1 but decreased when the ratio exceeded 3.8:1. Overall, NBA demonstrated greater sludge removal efficiency than MEK under similar conditions. NBA contains a hydroxyl (-OH) group, enabling strong hydrogen bonding with polar compounds, while its amphiphilic structure, comprising a polar -OH group and a nonpolar hydrocarbon chain, allows partial miscibility with nonpolar oils. This dual solubility enhances its ability to separate polar contaminants (e.g., metals, oxidation products) from oil while maintaining oil recovery. MEK contains a carbonyl group (C=O) that engages in dipole-dipole interactions but lacks hydrogen-bond donors, which limits its ability to effectively isolate contaminants despite dissolving both polar and nonpolar substances. MEK-rich solvents may dissolve more oil but retain contaminants, reducing purity. Therefore, the hydroxyl group in NBA enhances oil solubility and extraction efficiency through hydrogen bonding and amphiphilic interactions, which the carbonyl group in MEK cannot provide.

Increasing temperature and agitation speed improved removal efficiency for both solvents under constant solvent-to-oil ratios and constant mixing conditions, as shown in Figures 1(b), 2(b), 1(c), and 2(c). However, removal efficiency decreased and oil quality deteriorated when the temperature exceeded 57°C. Additionally, removal efficiency decreased when mixing speed exceeded 600 rpm because excessive agitation caused unidirectional vortex formation, reducing solvent–oil contact and limiting contaminant separation. This is because the agitation at higher speeds resulted in unidirectional vortex movement, which made it harder for the solvent to dissolve the used oil and separate it from the contaminants.

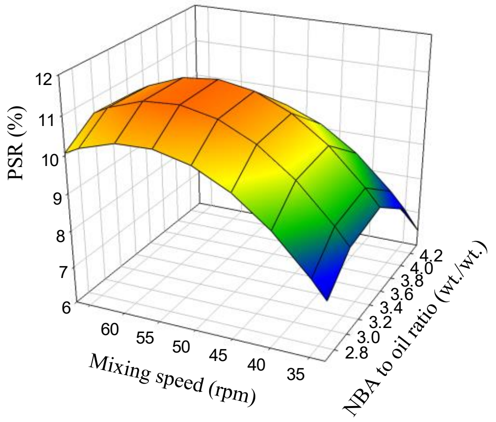
4.4 Statistical Analysis for EO Adsorption

The used oil had an initial color of 8.0, but after sludge was removed through extraction, the color dropped to 6.5, indicating that contaminants were still present. In this study, acid-activated clay was selected as the adsorbent to eliminate any remaining contaminants and lower the oil color because of its high surface area and enhanced porosity. This provides more active sites for adsorption, allowing it to trap contaminants such as oxidation products, degraded additives, color bodies, sludge, and heavy metals. Therefore, the optimal conditions for efficient adsorption of oil contaminants using activated clay were investigated. Table 6 shows the adsorption results of EO based on CCD with three independent variables: agitation speed (S), adsorption temperature (T), and activated clay dosage (A). The response is expressed as oil color. Analysis of variance (ANOVA) was used to create a regression model with a 95% confidence level. In the regression analysis output, Significance F refers to the p-value associated with the F-statistics, indicating the overall significance of the regression model. As shown in Table 7, the significance F was less than 0.05 (< 0.0001). The model was statistically significant, meaning it explains a significant portion of the variance in the dependent variable. The p-value for each variable was used to analyze its significance. The response was significantly correlated with the independent variables, as indicated by the coefficient of determination (R^2) of 0.9478. Temperature was the most significant variable in the adsorption process, as evidenced by the fact that its p-value was the lowest p-value among the variables. The linear terms A and T also showed significant effects, except for S. The square term variables A^2 and T^2, with p-values less than 0.05, showed highly significant quadratic relationships with the response. Furthermore, the p-values for the interaction term A*T were less than 0.05, suggesting significant interaction effects. A lack-of-fit test was performed to assess whether the model could explain the relationship between independent and dependent variables. The lack-of-fit test yielded a p-value below 0.05, signifying a statistically significant lack-of-fit. The model did not adequately describe the true relationships between the variables; there were systematic patterns in the residuals that the model may not capture. Collectively, the p-values of the main variables (A, T) were below 0.05, signifying statistical significance as predictors. Therefore, a second-order polynomial regression model was used to relate all variables to the adsorption response using activated clay, as indicated in Equation (6).

(a) Constant temperature of 50^{O}C



(b) Constant mixing speed of 400 rpm



(c) Constant NBA to oil ratio of 3.5:1

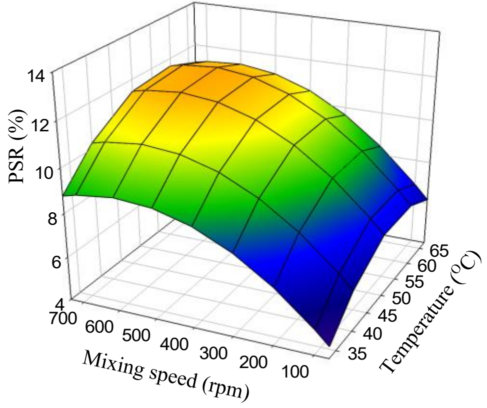
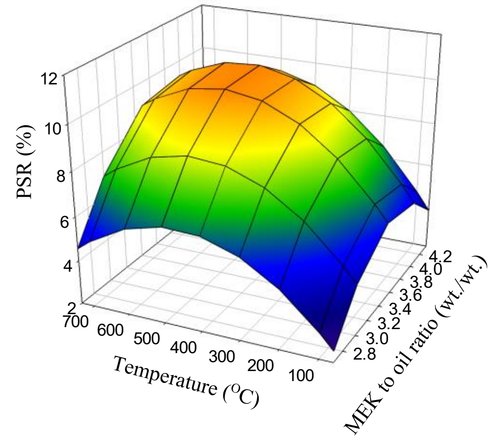
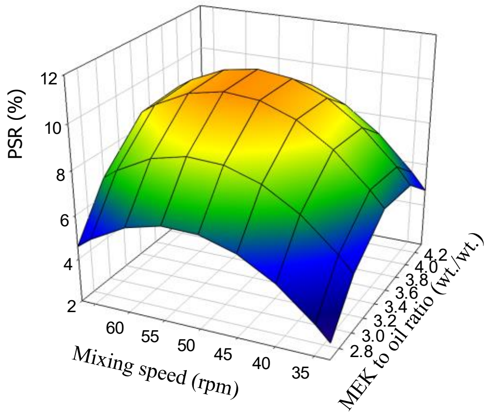


Figure 1 Response surface plots on PSR in ULO extraction process by 1-butanol.

(a) Constant temperature of 50^{O}C



(b) Constant mixing speed of 400 rpm



(c) Constant MEK to oil ratio of 3.5:1

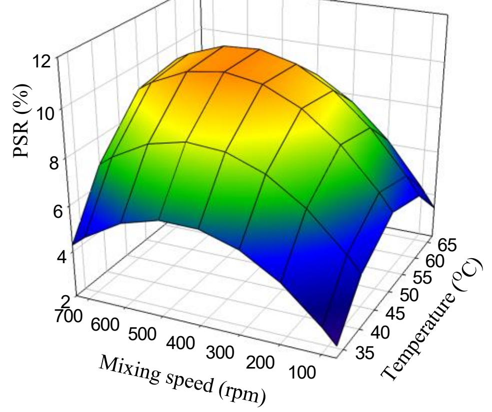


Figure 2 Response surface plots on PSR in ULO extraction process by MEK.

4.5 Optimization of EO Adsorption

The second-order polynomial regression model was used to determine the optimum conditions for adsorption of the extracted oil by activated clay, as shown in Equation (6). Figure 3 displays the response surface plots at the midpoints of each variable. The results showed that the optimum conditions for achieving the lightest oil color were 55 %wt. activated clay, an adsorption temperature of 130°C, and an agitation speed of 200 rpm, with a contact time of 90 min. As a result of adsorption under optimum conditions, the oil color decreased from 6.5 to 0.61. To verify the validity of the regression model, adsorption tests under optimal conditions were performed in triplicate. The experimental results showed an oil color of 1.01 ± 0.03, which was reasonably close to the model prediction.

$$\begin{aligned}\text{Oil color} = &-13.29 + 0.255A + 0.2733T - 0.00003S \\ &- 0.001875\, A{*}A - 0.001074\, T{*}T - 0.00000660\, S{*}S \\ &- 0.001844\, A{*}T + 0.0000731\, A{*}S + 0.0000322\, T{*}S \end{aligned} \quad (6)$$

4.6 Effects of Variables on Adsorption Process

Activated clay effectively removes color-causing compounds from used oil through adsorption. The highly porous structure and large surface area of activated clay allow it to bind with large molecular weight compounds, aromatic hydrocarbons and their oxidation products, metal complexes, carbonaceous particles, and sludge compounds that give used oil its dark, degraded appearance (Daham et al., 2017). Furthermore, the pore structure acts as a molecular sieve, allowing smaller beneficial oil molecules to pass while retaining larger contaminant molecules.

The response surface plots shown in Figures 3(a) and 3(b) indicate that the adsorption of the extracted oil was significantly influenced by temperature. Higher temperature reduces oil viscosity, improves diffusion of contaminants into clay pores, and increases adsorption efficiency. As the temperature increased, more contaminants were adsorbed, causing the oil color to decrease. Additionally, high temperatures help eliminate air trapped in the pores of the adsorbent material. These air bubbles prevent full utilization of the adsorbent's surface area. Removing the bubbles increases the adsorption capacity of the surface. However, excessive temperature should not be used to avoid the thermal degradation of the oil.

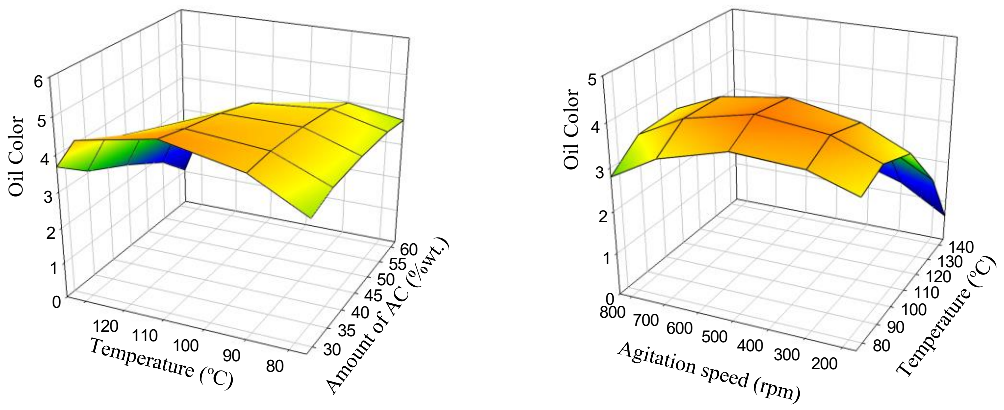
In addition to temperature, the amount of activated clay (AC) significantly affected the color of the oil. This can be observed from the response surface plots, as shown in Figure 3(a) and 3(c). By increasing the amount of activated clay, the color of the oil can be better reduced because the adsorption capacity increases with the surface area of the adsorbent, decreasing contaminants in the oil. However, this study used a high oil-to-clay ratio for highly degraded oil, so the percentage of TO was impacted by excessive activated clay. Due to saturation of the clay's adsorption sites by these contaminants, a significantly greater quantity of clay was required. In addition, the agitation speed should be sufficient to achieve good contact. Adequate agitation helps the system reach adsorption equilibrium faster by ensuring uniform distribution of clay particles throughout the oil and consistent exposure of all clay surfaces to contaminants. The results showed that agitation speed was related to the adsorption temperature and clay content, which influenced adsorption and oil color. If a low agitation speed was used, the temperature and amount of clay should be increased to achieve effective adsorption of contaminants on the clay surface. Therefore, higher temperatures are suitable for lower agitation speeds, since reduced oil viscosity improves mixing efficiency and faster diffusion kinetics require less mechanical mixing.

Table 6 Experimental design and results in EO adsorption using activated clay

Run No.	A (%wt.)	T (°C)	S (rpm)	Oil color	Run No.	A (%wt.)	T (°C)	S (rpm)	Oil color
1	35	90	300	4.51	11	45	76	500	3.57
2	55	90	300	3.53	12	45	144	500	1.52
3	35	130	300	4.06	13	45	110	164	3.09
4	55	130	300	1.09	14	45	110	836	2.98
5	35	90	700	4.40	15	45	110	500	4.03
6	55	90	700	3.49	16	45	110	500	4.10
7	35	130	700	3.95	17	45	110	500	4.12
8	55	130	700	2.08	18	45	110	500	4.08
9	28.2	110	500	4.51	19	45	110	500	4.05
10	61.8	110	500	1.99	20	45	110	500	4.11

Table 7 Summary output from data analysis in EO adsorption process

Regression statistics	R-Square	R-Square (adj)
	0.9479	0.9009
ANOVA		
Regression	F-value	p-value
	20.98	0.0000
Term:	Coefficients	p-value
Intercept	–13.29	0.019
Linear; A	0.255	0.033
T	0.2733	0.001
S	–0.00003	0.994
Square; A*A	–0.001875	0.051
T*T	–0.001074	0.000
S*S	–0.000007	0.011
Interaction; A*T	–0.001844	0.009
A*S	0.000073	0.227
T*S	0.000032	0.284
Error:	F-value	p-value
Lack-of-Fit	39.16	0.001



(a) Constant agitation speed of 500 rpm

(b) Constant amount of AC of 45%wt.

(c) Constant temperature of 110°C

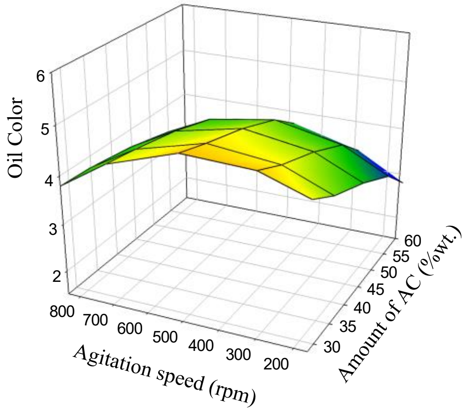


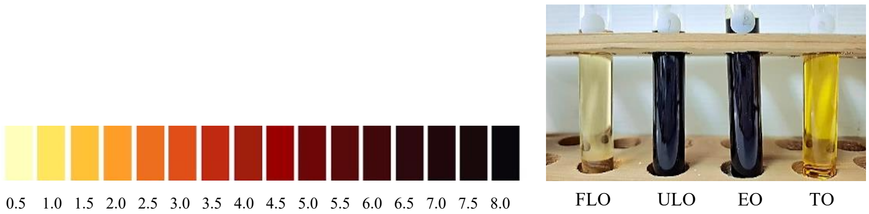
Figure 3 Response surface plots on Oil Color in EO adsorption process by AC

4.7 Characteristic of Oil Treated from Used Lubricating Oil

After lubricating oil is used in machinery or vehicles, it undergoes significant changes caused by contamination and degradation. In this work, used oil was treated by the extraction using NBA and the residual contaminants were removed via adsorption by activated clay under the optimal conditions. It was found that several contaminants, including oxidation products, water, soot, dust, wear metal debris, and degraded additives, were eliminated, resulting in treated oil (base oil) with improved properties. The yield of treated oil was 45%. The physical and chemical properties, including chemical composition, were evaluated for FLO, ULO, EO, and TO, as shown in Figure 4 and Tables 8–10.

The color of the oil decreased sequentially from an initial ULO value of 8.0 to 6.5 for EO and finally to 1.0 for TO. The specific gravity of lubricating oils typically ranges from 0.70 to 0.95, depending on the type of base oil and the refining or synthetic process used. The specific gravity of fresh oil was 0.84, which increased to 0.86 due to contaminants from use; after treatment, it returned to 0.84. The viscosity index of the used oil decreased due to thermal stress and degradation. This treatment under optimal conditions yielded base oil with lower kinematic viscosity at 40°C and 100°C, while maintaining a high viscosity index of 144. These changes in oil properties before and after treatment were consistent with the chemical composition results obtained from GC-MS analysis, as shown in Table 9. After use, several base oil hydrocarbons remained detectable, including alkanes (e.g., C10-C29 linear and branched), aromatic hydrocarbons (benzene derivatives), PAHs (polycyclic aromatic hydrocarbons), including degradation products because of high temperature and oxidation: alcohols, aldehydes, esters, and ketones.

Extraction of the used oil with 1-butanol, under optimal conditions, resulted in a significant reduction of aromatic hydrocarbons and PAHs. Subsequent adsorption using activated clay under controlled conditions, effectively removed residual contaminants, including degradation byproducts. Additionally, elemental analysis using IR spectroscopy confirmed that the extraction and adsorption processes removed elements originating from additive degradation and wear-metal debris, as shown in Table 10. Analysis reveals that the recovered base oil comprised 99.64% alkanes, 0.30% aromatic compounds, and no detectable naphthalene or polycyclic aromatic hydrocarbons (PAHs).



ASTM Color Scale

Used Oil before and after treatment

Figure 4 Oil color comparison using the ASTM color scale before and after treatment

Table 8 Physical and chemical properties of oils before and after treatment

ASTM E2412	FLO	ULO	EO	TO
Specific gravity at 25°C	0.84	0.86	0.85	0.84
Viscosity at 40°C (cSt)	56.80	49.49	23.33	25.64
Viscosity at 100°C (cSt)	11.37	10.03	5.63	5.29
Viscosity Index (VI)	199	195	197	144
Nitration (abs/cm)	0.0	4.5	0.0	0.7
Oxidation (abs/0.1m)	7.9	16.0	8.1	4.1
Sulfation (abs/0.1m)	17.6	25.9	18.0	14.5
Soot (%wt)	0.36	0.42	0.31	0.28
TBN (mg KOH/g)	8.4	4.2	4.1	4.3
Water (ppm)	60	1311	1194	0

Table 9 Chemical composition of each type of oil analyzed by GC-MS

Classification of substances	Components by NIST11	FLO (area%)	ULO (area%)	EO (area%)	TO (area%)
Base oils	Alkanes (C10-C29 linear and branch)	99.07	94.68	96.70	99.64
	Alkenes	0.11	-	-	0.01
	Cycloalkanes (Naphthene)	0.10	-	0.03	-
Additives	Alcohols	0.44	-	-	-
	Thiols	0.26	-	-	-
	Ethers	0.44	-	-	-
Combustion byproducts	Aromatics Hydrocarbons (Benzene derivatives)	-	3.32	1.30	0.30
	PAHs (Polycyclic Aromatic Hydrocarbons)	-	1.02	0.78	-
Degradation products (Oxidation, Nitration, and Thermal)	Alcohols	-	0.02	0.50	-
	Aldehydes	-	0.02	-	-
	Amines	-	0.11	-	-
	Esters	-	-	0.38	-
	Ethers	-	0.16	0.22	-
	Ketones	-	-	0.02	-
Contaminants	Other	0.04	0.67	0.09	0.05

"–" indicates values below the detection limit (not detected).

Table 10 Element content in lubricating oil before and after treatment

ASTM D6595-00			FLO	ULO	EO	TO
Wear metals	Iron	ppm	<1	11	<1	<1
	Chrome	ppm	<1	<1	<1	<1
	Nickel	ppm	<1	1	<1	<1
	Molybdenum	ppm	142	113	1	1
	Aluminum	ppm	<1	2	<1	<1
	Lead	ppm	<1	<1	<1	<1
	Copper	ppm	<1	12	3	<1
	Tin	ppm	<1	<1	<1	<1
	Silver	ppm	<1	<1	<1	<1
	Titanium	ppm	<1	<1	<1	<1
Contaminants	Silicon	ppm	4	15	13	2
	Sodium	ppm	<1	5	<1	<1
	Vanadium	ppm	2	2	<1	<1
Additives	Calcium	ppm	973	1116	<1	<1
	Magnesium	ppm	318	167	<1	<1
	Phosphorus	ppm	712	632	346	38
	Zinc	ppm	945	623	19	<1
	Barium	ppm	<1	1	<1	<1
	Boron	ppm	70	81	30	<1

5. Conclusion

The treatment of used lubricating oil was optimized using response surface methodology (RSM) based on a central composite design (CCD). Under optimal conditions, 1-butanol solvent-to-oil ratio of 3.2:1, extraction temperature of 57°C, and mixing speed of 600 rpm, a maximum sludge removal of 12.08% was achieved. Subsequent adsorption using 55 wt% activated clay, an adsorption temperature of 130°C, an agitation speed of 200 rpm, and a 90 min contact time resulted in significant improvement of oil quality, reducing the oil color to 1.0 on the ASTM scale. The recovered base oil exhibited high quality and purity, with a viscosity index of 144, chemical composition of 99.64% alkanes, 0.30% aromatic compounds, and no detectable PAHs or naphthalene.

These findings demonstrate that combining solvent extraction with adsorption is an effective strategy for recovering reusable base oil from used lubricating oil. Future work should explore the application of recovered base oil in industrial lubricants, fuel blending, or advanced refining to enhance its commercial value.

6. Abbreviations

A	amount of activated clay
AC	activated clay
CCD	central composite design
EO	extracted oil
FLO	fresh lubricating oil
MEK	methyl ethyl ketone
NBA	1-butanol
PAHs	polycyclic aromatic hydrocarbons
PSR(%)	percentage of sludge removal
R	solvent-to-used oil ratio
RSM	response surface methodology
S	speed
T	temperature
TBN	total base number
TO	treated oil
ULO	used lubricating oil
W_{Sludge}	weight of dry sludge
W_{TO}	weight of treated oil
W_{ULO}	weight of used oil

7. CRediT Statement

Patcharee Kamthita: Conceptualization, methodology, resources, writing – original draft, writing – review & editing, supervision.

Budsaba Leelasinlatham: Investigation, laboratory experiments, validation, data curation, formal analysis, visualization.

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