Statistical Model for Re-Refining of Used Lubricating Oil by Solvent Extraction and Bentonite Clay Adsorption Method

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Abstract. Lubricating oils are widely using for reducing friction and wear by interposing a film of material between rubbing surfaces, but these oils lose their properties after a certain period of use. For protection from environmental pollution and the economic side, used oil can be recycling and purifying by various methods. Solvent extraction+ clay adsorption technique is one of the cheapest, competitive, and most efficient processes experienced in the recycling of used lubricating oils. After vacuum filtration of the oil is distilled by vacuum distillation and extracted by Methyl Ethyl ketone as a solvent. Bentonite clay (400 gm) is activated by mixing (100 ml) diluted sulfuric acid under 100 °C for 4 hours. The extracted oil is treated by activated clay (Clay/Extracted oil =%15 wt) at (120°C) with mixing speed (500 rpm) for 90 minutes. However, the capability of this process largely depends on the optimum condition of each parameter in the recycling process. For this purpose, the experiments of three variables (Temperature, mixing speed, extracted solvent/ oil ratio) at three levels for the extraction process are designed by Design-Expert software. The optimum condition for the extracted process is (Solvent /used oil=3, Temperature =58 °C, mixing speed =710 rpm) confirmed by ANOVA. The recovered oil with 72% yield is analyzed in the laboratory, found it very close to base oil specifications.

Keywords: Response surface methodology (RSM)- Re-refining used oil- Solvent extraction- Clay adsorption -Central composite design

1. Introduction
Lubricating oil representing a mixture of hydrocarbons containing 18-40 carbon atoms with molecular weight 300-750 in boiling point 300-565 °C [1]. Its composition of two main parts: base oil and additives. After production of virgin base oil, additives are blended to the base oil for production of various types of lubricating oil for achieving the necessary protection of equipment and internal parts of machines.
from wearing which is specifically designed for the specified condition [2,3]. The main purpose of lubrication is besides reducing the friction, lube acts as an insulator layer between the moving part of the machine, which is elongated the age of the machine and lowering the required energy also of cleaning parts in bearing housing [4].

There are several additives inside the lubricant for increasing efficiency and performance features. Typically, a lubricant containing 10-20% of additives, and the rest is base oil [1,4]. For steam turbines that formed from high-quality base oil contain antifoaming, antioxidant, and anti-rust additives. [5]

After a specific period of operation of lubricating oil, as recommended by the manufactured company, lubricating oil should be changed to ensure equipment working efficiently [6,7]. That because the quality degraded by the decomposition of atoms and additives or contamination by metallic particle, dirt, or water after a period of operation [4,7]. These metals and dirty will deposited from worn up surfaces into the lubricating oil and contaminated it. At elevated temperature and after a long time of use lead to a loss in lube properties such as viscosity, viscosity index, and some oil molecules will oxidize and form corrosive organic acids [3]. These deposited metals at high-temperature work as an assistant factor for corrosion reaction.

This waste oil containing a large amount of Magnesium, lead, iron, etc.; which is mostly being environmentally hazardous [8]. The disposal of waste oil to the environment has large damage to human health and plants [9]. The underwater, live organism, and soil are all contaminated by the release of waste oil, because of the presence of PAHs (Polycyclic aromatic hydrocarbon) and metallic in additive which turns them to carcinogenic and toxic [10,11]. There are ways to get rid of this waste oil like burning as fuel after blending it with other constituents for furnaces. Environmental pollution is caused by the incineration of waste oil due to the diffusion of the PM (particulate matter) and gases and impact on the greenhouse effect [12]. It is good to know that the base oil is never spoilt and after removing the impurities and water by re-refining processes it is re-useable [13,4]. Re-refining of used oil shows its benefit not only as environmental preservation but also in the economic side 65-75% could be recovered [14].

Solvent extraction and clay treatment one of the effective methods for re-refining of the used oil [7]. Concentrated H2SO4 is used in this process for removing asphaltic material followed by the clay treatment to remove undesirable compounds such as the high polar compounds such as nitrogen and oxygen and aromatics [1,13]. Achievement of this process with high accuracy at optimum condition for saving energy and recovering material and reduce the cost to a minimum, statistical Response surface method by design expert software is applied [15].

RSM (response surface methodology) is a significant branch of design experiments, it is a technology for improving the formulation of a specific product, optimizing its performance, and developing new processes. One of the most advantageous of the RSM method illustrates graphically the relation between the response and independent variables, this graphical perspective led to the term Response Surface Methodology. Thus, the effect path of the input variable on the process is clear and optimization for the process optimal conditions are very straightforward.

One of the highest efficient ways in RSM to design and estimate second-order polynomial [16] is CCD (Central composite design), which is used in this study because it is higher efficient from other methods [17]. CCD is known as experimental design in RSM for building a second order model without needing to use complete three level factorial experiment.

Experiments that are designed by the CCD method exist to three levels or more for each variable to cover more space of possible effects of input factors, therefore it gives enough information on the variable's behavior in the process.
The three distributed levels of points are: axial points, central points, and factorial points as it is obvious is Fig 1. Factorial points are consisting of two levels of points +1 and -1 of factors. Axial points put on the coordinate axes symmetrically with respect to the center point on distance $\alpha$. Central point set on coded level (0,0) and are usually repeating to gain a good evaluation of error in experimental design.

2- Material and Methods

2.1-Material Used

Used lubricating oil was provided from the North Gas company after using in steam turbines 90 days by the recommendation of the manufactured Shell Company. The specification of the lubricant oil was illustrated in Table 1.

| Properties            | Units | Used Oil | Fresh oil |
|-----------------------|-------|----------|-----------|
| Viscosity@ 40 $^\circ$C | cSt   | 71.8     | 61.50     |
| Viscosity @ 100 $^\circ$C | cSt   | 8.6      | 8         |
| Viscosity Index       | -     | 89       | 95        |
| Flash point           | $^\circ$C | 230     | 218       |
| Pour point            | $^\circ$C | -24     | -15       |
| Density               | kg/m$^3$ | 882     | 890       |
| TAN (Total acid number) | mg kOH/gm oil | 0.12 | 0.115  |
| H$_2$O content        | ppm   | 75       | Nil       |
| Color value           | -     | Black    | 5         |
Removing suspended impurities in the oil done by filtration (filter paper Whatman, \( \phi = 150 \text{mm} \)) followed by the vacuum distillation for removing water and light hydrocarbons to 760 mmHg and 75 \(^{\circ}\text{C}\). Three kinds of solvents are used in this study: 1-Butanol, Hexane, and MEK (methyl ethyl ketone). The solvent MEK is shown the highest PSR\% (percentage sludge removal) between other solvents and it is more suitable for refining the steam turbine lubricating oil. Table 2 illustrates the highest PSR\% by the MEK.

### Table 2. Used Solvents

| Solvent | Activity | PSR\% | Type of solvent | Purity | Company          |
|---------|----------|-------|-----------------|--------|------------------|
| 1-Butanol | Weak     | 0.36  | Alcohol         | 99\%   | Sigma Aldrich    |
| Hexane  | Good     | 4.3   | Alkane          | 99\%   | SRLChem.         |
| MEK     | Excellent| 6.11  | Ketone          | 99.5\% | Chem-Lab NV      |

2.2 Experimetal Set-up and Methods

2.2.1. Extraction Process

Extraction by the solvent is the best process for removing the aromatic rings which affect adversely the viscosity of the lube oil. The solvents are blended with the 20 ml of the used sample according to the conditions: quantity of the Solvent /used oil Ratio varied from (2.5-3.5) while the temperature from (40 \(^{\circ}\text{C} – 70 \(^{\circ}\text{C}\)) with the mixing speed (400 rpm – 900 rpm) using Magnetic hot plate Stirring (Cimarec Company). For the study, the effects of each factor in each solvents Design expert software are used for the formation of the experiments as its obvious in Table 3. Each experiment is done in 10 minutes to obtain suitable homogeneity between the solvent and the used oil. Centrifuge (Petro-test) 1000 rpm used to separate and settle the sludge as a raffinate phase while the mixture of used oil-solvent is separated as an extracted phase. The remaining base oil in the sludge is washed by using the same solvent followed by drying in the Furnace (Heraeus-vacutherm: VT6025) half-hour in 130 \(^{\circ}\text{C}\) for evaporating the solvent in sludge. PSR\% can be calculated by the equation:

\[
\text{(P. S. R) } = (\frac{W_{\text{dry}}}{W_{\text{oil}}}) \times 100\% \quad \ldots (1)
\]

\(W_{\text{dry}} = \text{weight of dry Sludge (gm)}\)

\(W_{\text{oil}} = \text{weight of used oil (gm)}\)

### Table 3. Experiments of Extraction process for three Solvents

| Runs | Solvent / Oil Ratio | Mixing Speed (RPM) | Temperature(\(^{\circ}\text{C}\)) | PSR\% (MEK) |
|------|---------------------|--------------------|-------------------------------|-------------|
| 1    | 3.7                 | 650                | 55                            | 5.2         |
| 2    | 3                   | 650                | 55                            | 3.8         |
| 3    | 2.5                 | 400                | 40                            | 0.84        |
| 4    | 3                   | 650                | 55                            | 6.11        |
| 5    | 3                   | 650                | 76.21                         | 0.4         |
| 6    | 2.5                 | 900                | 70                            | 0.56        |
| 7    | 3                   | 650                | 55                            | 4.15        |
2.2.2. Experimental Design

RSM is a group of statistical and mathematical techniques suitable for improving, optimizing, developing process [23]. It has many extensive applications especially in manners where some of the input factors potentially impact quality features or performance parameters of a product or process.

The scope of RSM is consisting of experimental tactics discovering the space of independent variables (Solvent/Oil ratio, Temperature, mixing speed) empirical statistical model to develop suitable approximating relation by an equation between the input variable and output product, and various optimization approaches for finding that values or levels of independent variables (inputs) that leading to produce in-demand responses (output)[24]. The experiments are designed according to 15 trails of three variables by RSM methodology for building a second-order model. Design expert software version 10.1 is used for analyzing parameters of extraction.

2.2.3. Adsorption Process

Activated bentonite clay was used as an adsorbent for refining used oil. The clay first washed and dried in a furnace at a temperature of 110 °C for 4 hours. Subsequently, for activation 100 gm of the bentonite clay is blended with the 400 ml diluted H₂SO₄ for 4 hours in 100 °C. Separation of activated clay by Whatman filter paper from the sulfuric acid and washing it distilled water again in the last step.

3. Results

3.1. Analysis of Variance

The data obtained from Table 3 is analyzed by the RSM method. The accuracy of the result's experiments can ensure to be correct from Fig 2, results of actual and predicted are very close to each other.
The model has F value 30.41 that indicates the model of this process is significant. The F-statistic is simply a ratio of two variances. Variances are a measure of dispersion, or how far the data are scattered from the mean. Larger values represent greater dispersion, F value equal to (variation between sample means/variation within the samples).

P value is the probability of obtaining results as extreme as the observed results of a statistical hypothesis test, assuming that the null hypothesis is correct. In case the value of the P value is less than 0.05 this means that that parameter or model had a significant impact on the process. In this study the P value of the Model = 0.0008 less than 0.05, this means that the model is accepted.

The model has a coefficient of determination 98.21% which in agreement with the adjusted determination of coefficient adj-\(R^2\) 94.98%, both values are approximate 1.0. these high values support the relationship between the expected and the determined values. \(R^2\) is a proportion of the variance in the dependent variable (the response) from the independent variables (the predictors) and can be used for model performance assessments, where adj-\(R^2\) also indicates how well terms fit a curve or line, but adjusts for the number of terms in a model.

This point that the ANOVA (Analysis of Variance) shown in table 4, explained perfectly the relations of input variables and in addition to the response, where ANOVA is a collection of statistical models and their associated estimation procedures used to analyze the differences among group means in a sample.
Table 4. ANOVA for Response Surface Quadratic Model

| Source         | Squares Sum | df  | Mean Square | F value | P value | Significance |
|----------------|-------------|-----|-------------|---------|---------|--------------|
| Model          | 48.83       | 9   | 5.43        | 30.41   | 0.0008  | significant  |
| A-Solvent/Oil (Ratio) | 9.42       | 1   | 9.42        | 52.79   | 0.0008  |              |
| rpm            | 0.067       | 1   | 0.067       | 0.38    | 0.5656  |              |
| C-Temperature(C) | 0.033     | 1   | 0.033       | 0.19    | 0.6849  |              |
| AB             | 0.085       | 1   | 0.085       | 0.48    | 0.5213  |              |
| AC             | 0.048       | 1   | 0.048       | 0.27    | 0.6263  |              |
| BC             | 5.20        | 1   | 5.20        | 29.13   | 0.0029  |              |
| A2             | 0.64        | 1   | 0.64        | 3.59    | 0.1166  |              |
| B2             | 18.81       | 1   | 18.81       | 105.47  | 0.0002  |              |
| C2             | 21.51       | 1   | 21.51       | 120.56  | 0.0001  |              |
| Residual       | 0.89        | 5   | 0.18        |         |         |              |
| Lack of Fit    | 0.81        | 1   | 0.81        | 38.33   | 0.0035  |              |
| Pure Error     | 0.064       | 4   | 0.021       |         |         |              |
| Cor Total      | 49.72       | 14  |             |         |         |              |

* Note that, R²=98.21%, Adj-R²=94.98%

3.2. Optimum condition

The terms (A, BC, B² and C²) had significant effect on the process. The P value of (B, C, AB and AC) was higher than 0.1, this means these terms had no significant effect on the process and can remove from the equation of model. The model equation of the extraction process by RSM method is:

\[
PSR\% = -27.56 + 7.92* A + 0.00429* B + 0.479* C - 1.170* A2 - 0.000025 B2 - 0.007419*C2 + 0.00165 A*B + 0.0206 A*C + 0.000432 B*C 
\]

3.2.1 Temperature

The temperature had its effect on the extraction process between used oil and MEK solvent due to an increase from solvent efficiency by increasing molecular movement and kinetic fluency which help more hydrocarbons will solve in the MEK solvent. However, the dissolving of hydrocarbons will decrease with increasing temperature due to the volatility of solvents in which the amount of solvent in the mixture will decrease at high temperature, so the temperature should be done in an acceptable range.
In this research the effect of the temperatures ranges from (40 °C – 70 °C) was studied, as MEK volatile solvent its volatility was noticed during experiments above 60 °C. The temperature effect begins adversary in opposite direction increasing it above 60 °C in experiments and it is shown in Fig 3. Many previous researcher's results from studies illustrated that the temperature participation in the extraction process specifically when it is above 45 °C. [18,19].

3.2.2 Mixing speed

Mixing speed is another factor impacted on extraction by MEK solvent and its effect obvious as it is shown in Fig 3. This increase in speed leads to a homogeneous distribution of solvent in used lube oil where the surface contact area of both two liquids used oil and solvent are increased that enhancing the extraction process. The P value of factors BC is less than 0.05, which means strong interaction of both factors towards the extraction process as it is depicted in Fig.4c. The mixing speed of the process effectively had a positive impact on the extraction process and 710.68 rpm is the optimum value as it is obvious in Fig.3. it is effluent begins to change and reducing the PSR% after further in increasing mixing speed, this is due to the reduction of the overall mass transfer coefficient [20].

![Figure 3. Optimum Recycling Condition of used oil.](image)

3.2.3 Solvent to oil ratio

Equation 3 shows S/O ration coefficient is 7.92 and the positive towered extraction process has higher effects among other factors. The increasing ratio of solvent causes a reduction in oil viscosity and this is because the solvent extracted the sludge in used oil which contains aromatic compounds that had high molecular weight. Decreasing the viscosity enhanced from the velocity of the mixture molecule during the extraction better homogeneity obtain which leading more transfer rate and contact area. Paraffinic compounds remain in lube with lower viscosity in compari son with its aromatic counterpart's removal leading to lower viscosity [21]. The value of viscosity of used oil in table 71.8 cSt at 40 °C compared to extracted oil by MEK shows its viscosity reduced to 57.2 cSt at 40 °C and this is in agreement with (Temitayo et al .2018) [22]. PSR% was increased with the increasing solvent as it is shown in Fig.6.
Figure 4. a. 3D interaction (AB)

Figure 4. b. 3D interaction (AC)

Figure 4. c. 3D interaction (BC)
3.3. Effect of Process Variables

According to the Eq.3, the coefficient of Solvent/oil ratio(A) is +7.92, this value recorded the highest impact of these parameters. Increasing the MEK solvent to used oil ratio increasing from the sludge removal (PSR) as it is obvious is Fig.3.

The sludge removal percent is low at a low ratio of Solvent/oil ratio, that because at a low quantity of solvent doesn’t dissolve all the base oil, the higher ratio of MEK solvent more sludge removal could obtain at optimum ratio 3.7:1.

Higher mixing speed contributes effectively to the removal of the sludge and improvement from the extraction process specifically a speed about 710 rpm, as shown in Fig.3.

The reason behind that the high speed helps to get a better distribution of both solvent and used oil with more contact area of mass transfer which provides a suitable environment for the extraction process.

Exposure to the mixture to higher mixing speed led to lower sludge removal, thus appropriate speed saves energy addition to the best results.

PSR increases by increasing the temperature of the blend to a certain limit because the heat provides higher kinetic energy of the molecules and increases the motion of the blend, but after the 58.2 °C shows an adverse influence on the process as obvious in Fig.3.

The interaction coefficient S/O% with the mixing speed (AB) +0.00165 shows that both parameters interact positively towards the sludge removal from used oil.

Fig 4.a illustrates the interaction of the (A and B), although this relation has a positive effect on the sludge removal with the increasing of the mixing speed (B) the (PSR) are reduced at more high speed.

According to Equation3 Temperature and S/O% had the highest interaction over other recording coefficients 0.0206 for sludge removal shown in Fig.4. b.

Fig.4. c. illustrates the interaction by the Temperature and the mixing speed, the two variables are preferred to be used in the restricted region to continue the process path at the lowest cost and in a better manner.

4. Discussion of recovered base oil properties

Lubricating oil quality tests are performed after each step during the purification and recycling process. Importance of each extraction by solvent and activated bentonite are illustrated in Table 5. (Code No.4008, ISO Grade:68)

| Properties          | Unit     | Extraction (MEK) | Recovered (MEK+Activated clay) |
|---------------------|----------|------------------|---------------------------------|
| Viscosity@ 40 °C    | cSt      | 57.2             | 66.20                           |
| Viscosity @ 100 °C  | cSt      | 7.6              | 8.4                             |
| Viscosity Index     | -        | 94               | 95.5                            |
| Flash point         | °C       | 215              | 220                             |
| Pour point          | °C       | -19              | -17                             |
| Density             | kg/m3    | 860              | 900                             |
| TAN                 | mg KOH/gm oil | 0.011           | 0.020                           |
| H2O content         | ppm      | 0                | Nil                             |
| Color value         | -        | Dark             | 4                               |
**Viscosity**: denotes one of the most important of the performance test for lubricating oil. Lubricating oil is designing for providing viscosity to be an adequate minimum for starting engine lubricating in cold weather and high enough for supplying a thick layer in hot weather.

The increase of viscosity of used oil is an indication of the presence deposits, oxidizing products, deterioration of some additive that added to oil for enhancement its properties [25]. The presence of contaminants is reducing the viscosity; in our study, the viscosity is changed from 61.5 cSt to 71.8 cSt at 40 °C indicates the presence of impurities in the used oil.

Treatment by the MEK extraction can remove the aromatic hydrocarbon and waxes, so its viscosity reduced to 57.2 cSt. Adsorption by activated clay includes removing polar compounds that improve the chemical, thermal and color recovery led to viscosity 66.2 cSt.

**VI**: for fresh oil is 95 and for used oil is 89, because the increase of viscosity at 40°C but this value is reduced to 94 after MEK extraction. Bentonite Clay adsorption increased viscosity at 40°C which is led to increased VI to 95.5 as shown in Table 5.

**The Density** of the oil before using is 890 kg/m³ changed to 882 kg/m³ and this changeset depends on the nature and kinds of contaminants deposited in lubricating oil, the recovered base oil is 900 kg/m³ which is acceptable.

**The Flashpoint**: of the fresh oil 195 °C is increased to 230 °C after using because of saturation of the oil by contaminants. Wax removal by Extraction helps to enhance flashpoint to 215°C.

**TAN**: for fresh oil is 0.115 mg kOH/gm oil while for used oil is 0.12 mg kOH/gm, this increase because of the formation of carboxylic acid and other acids in the used oil through the oxidization process during using. The color and the all recovered properties are in the permitted range which is appropriate to use as in Fig.5.

5. **Conclusions**

1. MEK solvent is more active for removing sludge from used oil Turbine oil 4008.
2. RSM with R²=98.21% is enhanced effectively and recovered base oil suitable for re-use.
3. The optimum condition of the process: Solvent to oil ratio 3.7, mixing speed 710.68 rpm, and the temperature is 58.2 °C, shown in Fig.3.
4. mixing speed and temperature had adversely affected when they are crossing optimum value as it shown in Fig 6.
Figure 6. Influence of Extraction factors.

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