An Optimal Operating Approach for Efficient Regeneration of Used Lubricating Engine Oil by Solvent Extraction Technique

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Abstract: In this work, Response Surface Methodology for ensuring optimality in solvent extraction process for recycling used lubricating engine oil to obtain base oil was carried out using 2-propanol, 1-butanol and ethanol as solvent. Solvent oil ratio (SOR), temperature and contact time were considered as operating variables while percent sludge removal (PSR) as response. Historical Data Design with one hundred and eight runs was employed for the study. The optimized recycling process yielded maximum PSR (11.27) utilizing 1-butanol at temperature 500 C, SOR 3:1 and contact time of 30 minutes. At temperature 50° C, SOR 3:1 and contact time 20 minutes, PSR (7.97) was the optimum using 2-Propanol. In the case of mixture of 1-butanol and ethanol, the maximum PSR (8.41) was obtained at temperature 500 C, SOR 3:1 and contact time 30 minutes.

Index Terms— Recycling, Used Oil, Contaminant, Solvent, Extraction, Optimization.

INTRODUCTION

Lubricating oil, which is used in locomotive engines to bring about friction reduction, heat removal and reduce wears and tears, is enriched with contaminants in the process of its usage. These contaminants sources include additive breakdown products such as barium and zinc, dust and dirt moisture from the surrounding air; fuel combustion products that are transported into the crankcase and got deposited. Buildup of these pollutants changed the lubricant physical and chemical properties leading to aging and deterioration [1]. The deterioration process yields products such as sludge, lacquer, soluble and insoluble oil products that prevent it from performing its original functions. At this point the efficiency and life of both the lubricant and the device being lubricated are affected. Therefore, the used lubricating engine oils (ULEO) need to be replaced in order to improve the working efficiency [2, 3].

With large amount of engine oils used owing to proliferation of various form of cars, heavy duty automobiles and generators, the disposal of ULEO has become a major problem. Although it is not widely viewed as a significant problem, ULEO that is improperly discarded eventually enters into water runoff and adversely affects the environmental health of receiving water bodies [4]. Hazardous effects posed by ULEO on human and aquatic life can be eliminated through removal of contaminants that preclude it from having unlimited lifetime [4, 1, 5]. Several processes exist for reconditioning, reclamation or recycling of waste or used lubricating oil. Recycling of used oil is to pass it through a cycle of changes by the use of an appropriate selection of physical and chemical methods of treatment to preserve the non-renewable source and prevents its detrimental effects on the environment.

There are several recycling techniques that are available for recovering base oil from used lubricating oil and these include acidclay, vacuum distillation and solvent extraction techniques [6, 7, 5]. Solvent extraction is one of the cheapest and most environmental-friendly techniques for recycling used lubricating oil [8]. A large number of organic solvents have been used to separate suspended deteriorated additives and other impurities from base oil by dissolving or precipitation [9]. Organic sludge that has the potential to serve as burning fuels and raw material for ink production is produced as by-product by this technology [10, 11]. The ability to achieve high sludge removal using minimum volume of solvent is one of the factors that determine selection of the solvent to be used. These criteria can be measured by different methods such as percentage of sludge removal, percentage of ash content removed and percentage of base oil recovered after treatment [7].

Solvent extraction technology has become popular and is widely used due to its efficiency and competitiveness for regenerating ULEO. [5] studied recycling of waste lubricating oil using different composite solvents formed through combination of toluene and butanol with methanol, ethanol and isopropanol, respectively. The performances of these solvents were evaluated and analyzed for SOR (1:1-1:3) and the results obtained confirmed the efficiency of solvent extraction in restoring lubricating oil after usage.

Proficiency of single (1-butanol and 2-propanol) and composite (1-butanol-ethanol) solvents in regenerating ULEO was studied by [12] at different extraction parameters considered. The aim of conserving natural resources and eliminating significant environmental

impact through regenerating ULEO was achievable with 1-butanol displaying the best performance. However, determination of most appropriate value of operating variables under given condition is required in order to minimize utilization of resources. In spite of efforts directed towards enhancing this technique, attention paid to data-driven approaches that can describe processes taking place in this system for purpose of optimization is not much. Various techniques such as deterministic, stochastic and statistical approach can be employed for optimization of the process [13].

Ref [14] carried out research on determination of conditions (acid concentration, temperature and time) necessary for optimum recycling of used lubricating oil using RSM. However, the process presents acid mud in large quantities as waste product that require further treatment and optimization in addition to difficulties in removing contaminants from used oil with high additive content.

Table1: Factors and their levels for historical design for regeneration of ULEO

Variable	Symbol	Coded level .					
		-3	-2	-1	1	2	3
Temperature	A	-	35	45	50	-	-
Contact time	В	-	20	25	30	-	-
Solvent type	C	-	1-Butanol	2-Propanol	1-Butanol + Ethanol	_	_
Solvent to Oil	D	1:1	2:1	3:1	4:1	5:1	6:1
Ratio							

The main objective of this study is to optimize the key factors like SOR, temperature and contact time of solvent extraction technique for regenerating ULEO. The work of [7] was investigated and the results obtained for percent sludge removal were used as data for this study.

II. Experimental Design

The optimization of process parameters that have effects on regeneration of used lubricating oil was carried out using Response Surface Methodology (RSM). The tool was used to maximize base oil regeneration and sludge removal by evaluating the combine effects of different variables [15, 13]. Historical Data Design (HDD) with one hundred and eight experimental runs was employed for this study and the experimental results for predicting values for percent sludge removal were sourced from the work of [12]. The factors investigated were temperature, contact time, solvent type and SOR as independent variables while percent sludge removal was dependent variable. The experimental design was generated by employing version 6.0.8 design expert with coded and uncoded factors (A, B, C and D) and levels used are shown in Table 1

III. Statistical Analysis of Regeneration

Response surface methodology was used to analyze the data obtained in the experiment in order to fit the quadratic polynomial equation generated. Analysis of variance (ANOVA) was employed to evaluate the quality of the fit of the models. The notation for the fitted quartic response models is:

$$Y = B_0 + B_1 X_1 + B_2 X_2 + B_3 X_3 + B_4 X_4 + B_{12} X_1 X_2 + B_{13} X_1 X_3 + B_{14} X_1 X_4 + B_{23} X_2 X_3 + B_{24} X_2 X_3 + B_{34} X_3 X_4 + B_{123} X_1 X_2 X_3 + B_{1234} X_1 X_2 X_3 X_4 + E$$
(1)

Y denotes responses, B_0 represents the overall mean, B_1 represents the independent effect of the first factor (X_1) , B_2 represents the independent effect of the second factor (X_2) , B_3 represents the independent effect of third factor (X_3) , and B_4 represent the independent effect of the fourth factor (X_4) . The B_{12} , B_{13} , B_{14} , B_{23} , B_{24} , B_{34} , B_{123} and B_{1234} represents the effect of the interaction X_1X_2 , X_1X_3 , X_1X_4 , X_2X_3 , X_2X_4 , X_3X_4 , $X_1X_2X_3$ and $X_1X_2X_3X_4$ out of other interaction effects, respectively with E denoting the random error.

The Response Surface Plots

In order to visualize the variation in responses as a function of processing variables and the effects that these parameters have on regeneration process, series of three-dimensional response surface were drawn using the design expert software. One variable was fixed so as to obtain the effect of other variables on the responses [14]. The response surfaces were plotted utilizing the relationships between dependent and independent variables.

IV. RESULTS AND DISCUSSION

A. Data Analysis for percent sludge removal

The ANOVA analysis results for the response surface models are presented in Table 2 while Table 3 shows the post ANOVA

statistics. The F-value of 94.29 and p-value <0.0001 shown in Table 2 indicates that the model is significant. The p-values (<0.05) for the model indicates that terms A, B, C and D (linear coefficients); AC (cross product) and A2 (quadratic coefficient) are significant. In Table 3 the coefficient of determination (R2 = 0.98) indicates that the model is highly significant and sufficient to describe the correlation between percent PSR and the variables [16]. The significance of the model is supported by high value of adjusted determination coefficient (Adj. R2 = 0.96) [17]. The "Pred R-Squared" of 0.95 is in reasonable agreement with the "Adj R-Squared" of 0.96. The adequate precision (38.22, a measure of signal to noise ratio) greater than four provides adequate model discrimination. The second-order polynomial obtained for the historical data response surface quadratic model is shown in equation 2

 $Y = 7.37 + 0.34A + 0.32B + 1.43C_1 - 0.68C_2 - 4.67D_1 - 1.13D_2 + 0.66D_3 + 1.40D_4 + 1.78D_5 + 0.49A^2 - 0.049AB + 0.37AC_1 - 0.16AC_2 - 0.046AD_1 - 0.11AD_2 + 0.32AD_3 - 3.492E - 005 * AD_4 - 0.087AD_5 - 0.032BC_1 + 0.057BC_2 + 0.033BD_1 - 0.098BD_2 - 0.059BD_3 - 0.048BD_4 + 0.067BD_5 - 0.39C_1D_1 + 0.19C_2D_1 - 0.092C_1D_2 + 0.25C_2D_2 + 0.10C_1D_3 - 0.11C_2D_3 - 0.015C_1D_4 - 0.042C_2D_4 + 0.17C_1D_5 - 0.24C_2D_5$ (2)

Table 2: ANOVA for respons	se surface quadratic model	l for response 1 (% sludge removal)
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	Sum of		Mean	F	
Source	Squares	DF	Square	Value	Prob > F
Model	753.83	35	21.54	94.29	< 0.0001
A	6.45	1	6.45	28.25	< 0.0001
В	10.45	1	10.45	45.76	< 0.0001
C	115.89	2	57.94	253.67	< 0.0001
D	596.63	2 5	119.33	522.40	< 0.0001
A^2	4.36	1	4.36	19.11	< 0.0001
\mathbf{B}^2	0.00	0		_	
AB	0.18	1	0.18	0.79	0.3781
AC	5.13	2	2.57	11.23	< 0.0001
AD	1.57	5	0.31	1.38	0.2426
BC	0.17	2	0.09	0.38	0.6847
BD	0.55	5	0.11	0.49	0.7858
CD	3.04	10	0.30	1.33	0.2307
Residual	16.45	72	0.23		
Lack of Fit	14.60	70	0.21	0.23	0.9845
Pure Error	1.85	2	0.92		
Correction					
Total	770.27	107			

Table 3: Post ANOVA statistics for percent sludge removal

Std. Dev.	0.4779	R-Squared	0.9786
Mean	7.6979	Adj R-Squared	0.9683
C.V.	6.2085	Pred R-Squared	0.9526
PRESS	36.4853	Adeq Precision	38.2190

B. Diagnostic Test for the Responses

Fig. 1 is a plot of experimental response against predicted response. The plot maintains diagonal position with some distortions. The diagonal position is an indication that the model has been able to capture the effect of operating conditions on the response (PSR). Fig. 2 shows the normal plot of residuals for PSR. The diagnostic plots are tested for the response in order to verify the adequacy of the model. The normal plot of residuals, residual versus run, predicted value, residuals versus factor, studentized residuals, box cox plot, outlier T, cook's distance, leverage and predicted versus actual were considered. Fig. 2 shows how the response was modeled and it is clearly shown that all the points line up normally. The deviation of points from normality for the response is insignificant. Therefore, the responses are significant.

C. Response Surface Plots Analysis for Optimization of Regeneration of ULEO

The present study consists of two numerical and two categorical factors. The value of one variable is fixed in order to see the effect of other variables on the responses. Fig. 3 shows the response surface plot of the effect of temperature (A) on percent sludge removal.

Figure 4 shows the response surface plot of the effect of contact time (B) on percent sludge removal and Figure 5 shows the response surface plot of the effect of SOR (D) on PSR with solvent type (C) held constant. In Figure 3, at extraction temperature 450 C and 500 C for the same SOR (5:1) and contact time of 30 minutes, the PSR were 11.4 and 11.9, respectively using 1-butanol as solvent. At extraction temperature 350 C and 450 C; SOR (1:1) for 20 min of contact time, 7.1 and 7.4 were the PSR, respectively. The PSR increases slightly with increase in temperature. Therefore it can be deduced that the effect of temperature is not too significant as factor in the PSR which is in agreement with [18] and [12]. In Figure 4, at extraction temperature 350 C for contact time of 20 and 30 min, the lowest PSR were 3.21 and 4.0, respectively at the same SOR (1:1). At extraction temperature 500 C, SOR 6:1; contact time 20 and 30 min, 11.57 and 12.31 were the PSR, respectively. The PSR increases slightly with increase in contact time. The effect of contact time is not too significant in the PSR. This is in close agreement with [12]. In Figure 5, at SOR 1:1, contact time 20 minutes, 3.21, 3.8 and 4.6 PSR were obtained respectively at 35o C, 45o C and 50o C. At the same temperature and SOR; contact time 30 min, the yield were 4, 4.1 and 5.17 respectively. The PSR (8.76), (8.9); (10.9) at 350 C, 450 C and 500 C; SOR 3:1 for contact time 20 min and (10.39), (10.9) and (11.37) for contact time 30 min show that the effect of D (SOR) is significant on PSR. Similar trends were found for other solvents at different extraction temperature, SOR and contact time. When C (solvent type) was 2-propanol, the lowest yield was 2.11 and 9.64 as highest at 350 C with 1:1 SOR. The lowest and highest PSR for the mixture of 1butanol and ethanol are 2.18 and 9.29, respectively. This shows that the effect of solvent type is too significant on PSR. The effect of solvent type is stronger on PSR follow by solvent to oil ratio. These results are in agreement with the result of ANOVA that was previously observed.

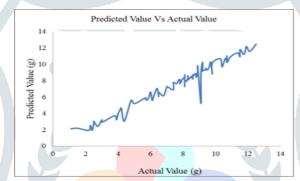


Fig. 1: Experimental results vs. the predicted values for PSR

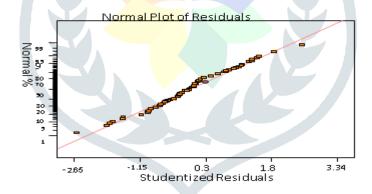


Fig. 2: Normal plot of residuals for percent sludge removal

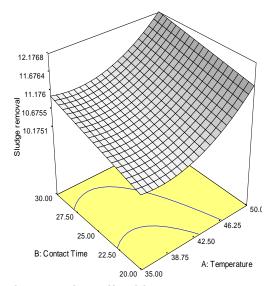


Fig. 3: Response surface plot of the effect of temperature on percent sludge removal

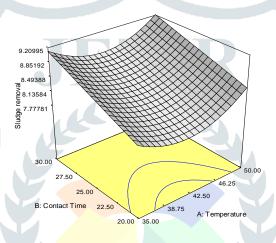


Fig. 4: Response surface plot of the effect of contact time on percent sludge removal

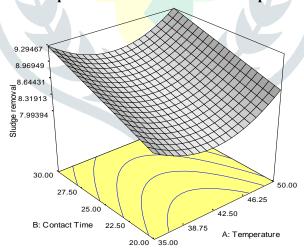


Fig. 5: Response surface plot of the effect of solvent type on percent sludge removal

D. Optimization of PSR

The increase in solvent-oil ratio and extraction temperature resulted to increase in PSR. [18] and [19] presented that the optimum PSR does not necessarily indicate the highest solvent-oil ratio. All the response surface plots for PSR show that there is sharp increase from 1:1 to 1.9:1 and it is observed that there is a linear relationship between PSR and SOR. There exist a gradual increase in PSR between 2:1 and 3:1, while a retreating effect was observed from 3:1 to 6:1 with slight increase. Figures 3, 4 and 5 showed that from 1:1 to 2:1 increase in PSR is linear with SOR. The increase from 2:1 to 3:1 is gradual. Conversely, from 3:1 to 6:1 a retreating effect is observed with very slight decrease. These results revealed that at higher ratio there is a retreating effect instead of further increase in regeneration of ULEO, low PSR is achieved as SOR is increasing. At these sections, low energy cost, low contact time, low SOR and low temperature are able to produce high sludge removal. These sections revealed the region to obtain

the maximum percent sludge removal at a specified extraction temperature and contact time with minimum solvent to oil ratio. Combining all these results revealed that the optimum for PSR lie between 2.8:1 and 3.3:1 for this study. The maximum PSR (11.27) was obtained as optimum for the regeneration of ULEO utilizing 1-butanol at temperature 500 C, SOR 3:1 and contact time of 30 minutes. At temperature 500 C, SOR 3:1 and contact time 20 minutes, PSR (7.97), was the optimum using 2-Propanol. In the case of mixture of 1-butanol and ethanol, the maximum PSR (8.41) was obtained at temperature 500 C, SOR 3:1 and contact time 30 minutes. This will definitely have economic advantages on oil loss and high oil yield. This was in agreement with the works of [20, 21, 12]

CONCLUSION

The investigation revealed that the solvent 1-butanol produced the best extraction performance with respect to sludge removal. The Optimization experiments was successfully designed and analyzed. Data were properly generated and statistically validated. RSM was employed to optimize the parameters since it is an indispensable tool for Process Optimization. The study clearly indicates that there is a potential to optimize regeneration of used lubricating engine oil by optimizing the extraction process parameters. The maximum PSR (11.2692) was obtained as optimum for the regeneration of ULEO at temperature 50oC, SOR 3:1 and contact time 30 minutes. This is advantageous since minimum solvent-oil ratio, low temperature, low contact time and hence low energy cost is required. It was found that there is strong agreement between the values of CCR (Critical Clarifying Ratio) of [12] and optimization results obtained.

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