# Article

Optimum Conditions to Maximize Re-Refined Lube Oil Recovery within Specifications via Acid-Clay Treatment

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### Abstract

Waste lubricating oils contain pollutants which are poisonous compounds derived from combustion, abrasion, and chemical reactions. Removal of such harmful impurities allows for the reuse of these oil, in addition to minimizing environmental hazards. Re-refining is still considered as one of the preferred techniques which preserve resources and minimize waste discharge. The effect of re-refining operating conditions on the quality of lube oil re-refining process has been studied previously in many publications. However, adjustment of the operating conditions to meet the quality standards with maximum recovery is still not well satisfied. In this study, empirical equations are introduced to relate produced oil recovery and qualities to the operating conditions in acid-clay re-refining process. The introduced correlations helped in building up an an optimization program through LINGO software, V.17, to predict the optimum operating conditions those maximize the process recovery, while producing lube oil within the required specifications. The present study indicates that the effect of distillation time as well as the temperature is negligible. However, the quality of the produced lube oil from acid-clay treatment is most affected by the catalyst/oil ratio and the distillation column pressure. It is found also that maximum recovery of 61.8% have been achieved at pressure of 432 mmHg and catalyst/oil ratio of 7.94. The re-refined lube oil meets the required standard specifications for flash point (268.65 C), viscosity index (107), Total Acid Number (TAN 0.05 mgKOH/g), ash content (0.4 wt%), and cloud point (-12.8C).

Keywords: Waste oil; Regeneration; Base oil; Re-Refining.

## 1. Introduction

Lubricating oil could be considered as one of the most important petroleum fractions, as it is widely used in service and industry. It is an important mean that promote easier motion of connected parts and protect rubbing surfaces by reducing friction and wear between metallic surfaces in engines, generators, power plants and other appliances and mechanical equipment. Further build-up of temperature, used oil is exposed to normal degradation, and picks up number of contaminants from the working environment. This leads to reduction in properties such as: viscosity, specific gravity, etc. <sup>[1]</sup>. Three basic options to deal with the regarding waste oil in the world <sup>[2]</sup>; a) dumping the waste oil on land, garbage heap and sewerage system, b) regeneration of base-oil from waste oil and c) extracting of heat value of waste oil through combustion process. The problem of environmental pollution, posed by dumping the waste or used lubricating oils, has been addressed by many nations in their countries, <sup>[3]</sup>. Furthermore, the conservation of petroleum resources has been declared national policy for several countries and the benefit of wise resource management are obvious, <sup>[4]</sup>. Gorman <sup>[5]</sup> and El-Fadel and Khoury <sup>[6]</sup>, addressed the different methods to recycle waste lubricating oils.

Acid clay is a traditional technique used for lubricating oil re-refining <sup>[7]</sup>. The effect of different variables on the properties of the generated lubricating oil has been studied in many publications <sup>[8-15]</sup>. Few publications worked on introducing some correlations relating the operating condition to the production <sup>[16-19]</sup>. However, the integration of all the affecting conditions simultaneously is not well introduced till now. In addition, using the integrated relation to predict the maximum recovery and best quality is also not introduced in previous publications. The objective of the present study is to introduce new correlations aims at expecting lube oil properties produced from acid/clay treatment, as well as recovery, depending on the operating conditions. The new correlations have been extracted depending on experimental data introduced by Falah and Hussein <sup>[10]</sup>, and have been validated using sample data from the literature by Rahman *et al.* <sup>[2]</sup>. Multiple regression analysis is used to obtain the new empirical correlations. Optimization of the operating conditions in order to maximize recovery and produced re-refined oil within standard specifications in also presented in this work.

# 2. Experimental data

The experimental data used to extract the correlation is taken from Falah and Hussein <sup>[10]</sup>. Their experimental work was based on waste oil samples collected from different sources and a variety of oils, including lubrication of vehicles and machines in the workshops and places of various industries around the city of Irbid. The different samples are mixed together to introduce a new sample with new specifications. In their work, they studied the impact of different sets of variables, (catalyst-waste oil ratio, the distillation column pressure, distillation time and the temperature), on regenerating the properties of lubricating oil. The catalytic cracking temperature for the first stage was 180°C and in the stimulating waste oil, was under 130°C. Their work was based on conducting four different runs. Data for each run, as indicated in their work, are shown in Table1. Waste and regenerated oil properties are presented in Table 2.

Table 1. Process parameters

Process variable	1st Run	2nd Run	3rd Run	4th Run
Catalyst-waste oil ratio (g/L)	7.94	7.94	11.62	16.88
Cracking temperature (°C)	180	130	130	130
Distillation time (h)	3	2	1.5	1
Distillation column pressure (mm Hg)	580	500	450	350

Table 2. Waste and regenerated oil properties

Parameter	Standards	Waste Oil	1st Run	2nd Run	3rd Run	4th Run
Viscosity index	ASTM D2270	97	102	105	106	107
Pour point (°C)	ASTM D97	-9	-12	-12	-11	-12
Cloud point (°C)	ASTM D5773	-10	-14	-13	-13	-14
Light fraction from distillation column as % of feed	-	-	0.55	1.23	1.30	1.20
Flash point(°C)		-	225	265	269	270
Specific gravity @ 40(°C)	ASTM D92	0.834	0.865	0.870	0.868	0.874
Total acid number (mg KOH/g oil)	ASTM D1298	2.1	0.110	0.116	0.185	0.210
Recovery (%)	ASTM D664	-	66	63	62	62

## 3. Results and discussion

In this section, the correlation representing the effect of independent variables (catalystwaste oil ratio, distillation time, and distillation column pressure) on the viscosity index, flash point, ash content, Total Acid Number (TAN), specific gravity, cloud point and recovery were studied. As mentioned in the abstract, the distillation time has negligible effect on most of the above properties and recovery, while the most affecting variables are the distillation column pressure (P) and catalyst to waste oil ratio (cat/oil). It is found that distillation time (Dist time) has an observed effect only on the ash content. As the variation in temperature is not obvious, it was not introduced as a parameter in correlation deriving. The regression analysis for experimental data generated the following equations:

Viccosity index 10E 2722 + 0		$* C^2$ (1)
VISCOSILY INDEX= $105.2725 \pm 0$	0.03277 * P - 6.5*10 <sup>-5</sup> * P <sup>2</sup> - 0.00622 <sup>·</sup>	$* C^2$ (1)
Flash point=190.6492 + 0.38	$2 * P - 0.00047 * P^2 + 0.011013 * C^2$	(2)

Total acid number =  $-2.25242 + 0.008507 * P - 7.9 * 10^{-6} * P^2 + 0.001608 * C^2$  (3) Specific gravity =  $1.345344 - 4.7 * 10-6 * P^2 + 5.73 * 10-9 * P^3 - 2.9 * 10-5 * C^3$  (4) Ash content =  $.14058 + 0.01879 * C + 0.1023 * T - 2.9 * 10^{-5} * P$  (5) Cloud point =  $-25.712 + 0.058751 * P - 6.6 * 10^{-5} * P^2 - 0.0027 * C^2$  (6) Recovery=82.9 -  $0.10647 * P + 0.000133 * P^2 + 0.000117 * C^2$  (7)

where P, C and T are corresponding to pressure (mm Hg), catalyst to oil ratio (g/L) and distillation time (h) respectively.

The  $R^2$  statistical test was used to evaluate how well the experimental data were represented by the correlations. This value for all the introduced correlations was found to be within the range of 0.97 to 1.

The crossplots are used to present how the predicted data matches the experimental data. Figures 1-7 are representing the predicted vs. experimental values for various mentioned properties and recovery.



275 270 E 2 265 Flash 260 ð 255 250 250 270 275 255 260 265 **Exp Flash Point** Flas Point Exp. flash point calc - Linear (Flas Point Exp) —— Linear (flash point calc)

Figure1. Crossplot for viscosity index

0.57

**Vertex** 0.54 0.51

0.48

0.45





Figure 3. Crossplot for ash content







Figure 4. Crossplot for total acid number



Figure 6. Crossplot for cloud point



Figure 7. Crossplot for recovery

The plotted data points obtained by the new correlations are quite close to the perfect correlations of the 45° line. This shows that the correlation is able to learn and predict the properties of lube oil after acid-clay treatment, depending on the operating conditions.

The accuracy and ability of each mentioned correlation for predicting waste oil properties and recovery after acid-clay treatment was checked with four other experimental data given in Rahman *et al.* <sup>[2]</sup>. Tables 3 and 4 show a comparison between the predicted and experimental results.

Pressure Cat/oil Viscosity index		Flash p	oint, °C	Ash content, %wt.			
mmHg	g/L	а	b	а	b	а	b
580	7.49	102.0	103	254.7	225	0.5882	0.61
510	7.49	104.7	105	263.8	267	0.4858	0.48
450	11.26	106.1	108	268.8	273	0.5055	0.5
335	16.88	107.2	107	269.0	270	0.5066	0.54

Table 4. Continued comparison between predicted and observed values given by Rahman et al. <sup>[2]</sup>

Table 3. Comparison between predicted and observed values given by Rahman et al. [2]

Pres- sure, mm Hg	Cat/oig/L	Total acid mgKOH		Specific gravity @40°C		Recovery, %		Cloud point, °C	
		а	b	а	b	а	b	а	b
580	7.49	0.114	0.109	0.864	0.863	65.895	66	-14	-13.99
510	7.49	0.121	0.106	0.866	0.869	63.200	63	-14	-13.06
450	11.26	0.179	0.18	0.871	0.874	61.936	62	-13	-12.98
335	16.88	0.169	0.21	0.892	0.874	62.192	62	-14	-14.20

Tables 3 and 4 confirm the ability of correlations for nearly accurate prediction of waste oil properties as well as recovery after acid-clay treatment, depending on the operating conditions. This could lead the operator to change the operating conditions based on the required specification and recovery. This also leads to reducing time and effort in changing run conditions to obtain the best lube oil quality.

The introduced correlations are used in LINGO optimization software (V.17) to predict the optimum conditions which get maximum recovery in addition to achieving the required standard specifications. The following mathematical model is used to achieve that objective: Maximize recovery,

With the following operating conditions constraints:

$$\begin{array}{l} C^{\min} \leq C \geq C^{\max} \\ P^{\min} \leq p \geq p^{\max} \\ D^{\min} \leq D \geq D^{\max} \end{array}$$

The following constraints are added to adjust the quality of the re-refined lubricating oil.

Flash\_Point>= Flash\_Point<sup>min</sup>;

Total\_Acid\_Number <= Total\_Acid\_Number<sup>max</sup>;

The limits for different operating conditions are presented in Table 5.

Table 5. Lower and upper limits for operating conditions

Process variable	min	max
Catalyst-waste oil ratio (g/L)	7.94	16.88
Distillation time (h)	1	3
Distillation column pressure (mm Hg)	350	580

Table 6 is representing the results compared to limits for the required lube oil quality according to Egyptian Organization for standardization &Quality (EOS), 2005.

Table 6. Egyptian Standard regeneration lubricating oils characteristics

Process variable	unit	Max	Result
Cleveland open-cup	°C	200	268.6
flash point by (min)			
TAN	mgKOH/g oil	0.05	0.05
Ash content	wt %	0.04	0.04
Viscosity index (min)	-	100	107

Optimization of the specified conditions resulted that the maximum recovery is 61.7%. it is achieved when running the distillation process at catalyst/oil ratio of 7.94 g/L, distillation time of 1.2 h and pressure of 432 mmHg. As shown in Table 6, the quality of the produced rerefined oil lies within the limits of Egyptian Organization for standardization &Quality (EOS), 2005. The cloud point of the produced oil is -12.8 °C, with a specific gravity of 0.9, as resulted from running the optimization program.

### 4. Conclusion

In this work, new correlations were developed to predict viscosity index, flash point, ash content, total acid number (TAN), specific gravity, cloud point and recovery after acid-clay treatment, depending on the operating conditions. It is shown that the most affecting parameters are the pressure and catalyst to oil ratio. The introduced correlation have been applied to different experimental data given in literature and shown to be practical. The resulted correlations helped in building up an optimization model, which is solved to find the optimum operating conditions for achieving the lube oil desired qualities with maximum recovery.

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