REFINING OF USED LUBRICATING OIL BY USING ACID CLAY METHODE

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Abstract*–The lubricating oil is of great interest as it provides a thin film on moving parts to reduce the friction, heat, wear between them. Recently, with the rapid increase in quantities produced of the used lubricating oil, its disposal has become a global environmental issue due to its impact on all life aspects. The aim of this work is the refining of used lubricating oil so it can be used again. This was investigated using the acid-clay process. The most important step in this work is the desludging reaction. The Response Surface Methodology is used to find out the optimum conditions for the desludging reaction, and the selected parameters were time, temperature, and acid to oil ratio. The temperature ranged from 40°C to 60°C, the stoichiometric ratio of acid to waste oil ranged from 10% to 20%, and the reaction time ranged from 60 to 120 minutes. The design is applied and the predicted optimum conditions are the temperature of 40°C, 20% acid to oil ratio, and reaction time of 60 minutes. To predict the results, five runs were performed at the optimum conditions to predicate the standard deviation of 0.739 and the human error which did not exceed 5.7%. The characterization results of the product of the refining at the flash point of 180°C, carbon residue of 0.487, kinematic viscosity at 100°C of 17cSt, the water content of 0.01 %, ash content of 0.02 %, and viscosity index of 110.2996 were found in complies with the Egyptian standards.

Keywords—Used lube oil, Acid clay process, Lube oil refining methods, and techniques of refining used engine oil.

I. INTRODUCTION

Lubricating oil (LO) plays a predominant role in machinery like vehicles, industrial gearboxes, compressors, turbines, and hydraulic systems. It is called engine blood as it produces a thin film between two movable surfaces to form an oily layer to reduce erosion, machinery noise, heat resulting from the mechanical friction in engines, and to remove pollutants and debris [1-4].

Although, spent lubricating oil is considered hazardous waste as it contains toxic contaminants, as well as one gallon of used lubricating oils can destroy two thousand gallons of freshwater. However, the demand for LO increased due to an increase in the rate of establishing factories, vehicles, and public transportations, etc. [5-8].

It has been displayed the global demand of LO from year 2000 up to 2020. It is clear that there is continuous increase of demand on LO due to its use in different applications [9-11].

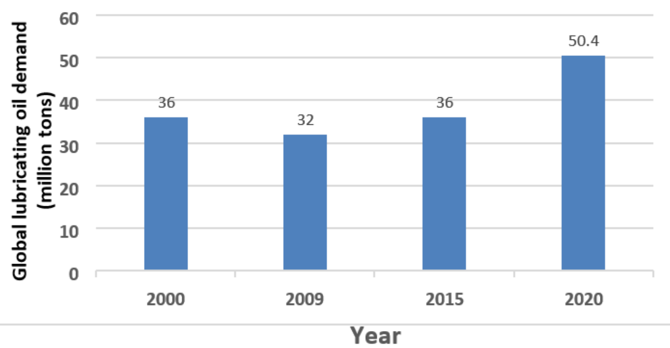


Fig. 1 Global lubricating oil demand volume

Lubricating oil loses its efficiency after a specific number of kilometres according to its type. When using it in an engine the properties of it deteriorate with time due to the high temperature in combustion engine. The additives in lubricating oil give it the ability to hold on for longer time but due to high temperature, oil will be decayed and it should be replaced with fresh one, that lead to increase on consumption of LO, leading to generation of large quantity of used LO. The importance of refining used oil comes from the cost of production of refined used oil is 1/9 of the cost of production from crude oil [4-6].

Egypt uses about 300,000 to 400,000 tons of LO every year, and this amount is produced as used oil, and only 5% of this quantity is refined, and the rest of the quantity is used in incineration furnaces as a source of energy or disposed of in landfills which the disposal quantity become hazard to environment [7,8].

Disposal of used lubricant oil can cause the following problems [12-15]:

- Great damage to public health and ecology.
- Air pollution when it is re-used as fuel in different factories. for example, furnaces in cement factories, this fuel can produce pollutant gases like NOx and Sox these gases have a bad effect on the environment and human health.

- Water pollution when thrown into the sea due to its damage effect on aquatic life drinking water.

The ULO is considered one of the most dangerous chemical substances since after usage it contains hazardous contaminants that if not disposed of in a safe way, can cause great danger to
the environment and living organisms [7,8,16].

The aim of this research is to produce refining lube oil from used one has specification within the range of Egyptian standard.

Several effective and successful methods have been applied in refining ULO whereby each method would be developed to improve the prior one [9]. The refining of ULO depends upon physical-chemical and chemical processes to remove oil contaminations and excess metals until reaches the standard characterization. The sedimentation and evaporation processes are considered the simplest processes to remove impurities from used lubricating oil. However, the sedimentation process needs a lot of time on the other hand an evaporation process consumes a large amount of heat (energy-intensive) [16-20].

There are currently several refining technologies for spent oil, including acid-clay treatment, solvent extraction, clay vacuum distillation, and hydrogenation finishing. However, all innovations merely serve the purpose of refining lubricating oil. This is achieved in four-step methods which are removal of water, solids and light hydrocarbons, dissolved metals, and degraded additives [20-24].

II. Excremental Work

A. Used Lubricating Oil Characterization

Density, flash point, pour point, ash content, water content, carbon Residue, dynamic viscosity, and viscosity index value are measured.

Density

The sample is placed in a cylinder at ambient temperature. Then an appropriate hydrometer is placed in the sample and left to settle, then the hydrometer is read, and the measurement is recorded as shown in Fig. (2). The cylinder and its contents can be placed in a constant temperature water bath to avoid temperature differences during testing [12,13].

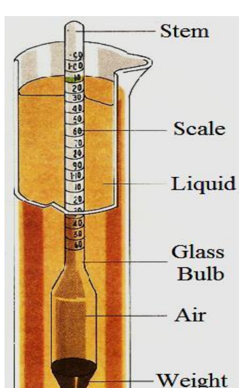


Fig. 2 Hydrometer

Flash Point

Pensky-martens closed cup apparatus (Herzog) consists of a test cup, a stirring device, a heating source, an ignition source device, an air bath, and a hot plate as shown in Fig. (3). The cup is filled with oil to the inner mark and the temperature of the sample is gradually increased at regular intervals, and the ignition source is applied to the steam area in the cup. The sample is considered reached to the flash point when a large flame has occurred and spread over the entire surface of the sample [14].

Fig. 3 Automatic Pensky Martens Flash Point Tester

Pour Point

Pour point test (Petrotest) consists of a test jar, cylindrical of clear glass with the flat bottom disc, 33.2 mm to 34.8 mm outside diameter, and 115 mm to 125 mm in height. A specific amount of oil is placed in a test jar cylinder with a thermometer inside as shown in Fig. (4). The oil is heated in a water bath at 48°C, then cooled at intervals of 3° C and tipped to determine the flow characteristics. The pour point is recorded when the oil stops moving horizontally for 5 seconds [13].

Fig. 4 Manual pour point tester

Ash Content

This method is based on measuring the amount of ash in the oil, which indicates the quality of the product and the adequacy of any application where ash and impurities are considered undesirable substances. The sample is placed in a suitable combustion crucible as shown in Fig. (5), and exposed to high temperatures of 775 ° C for 5 hours until the oil evaporates and only ash remains. Then cool to ambient temperature and weigh the remaining ash. Equation (1) is used to calculate an ash content percentage. The model of used furnace is (LT/ME-271000/M) [15].

$$\% \, Ash = \frac{m_{ash}}{m_{intial}} \times 100 \quad (1)$$

Fig. 5 Furnace

Water Content

This test is used to find out how much water is in a lubricant. This is done by placing a certain amount of sample (100 ml) in an oven at 120 ° C for an hour. The dried sample is then weighed. Equation (2) is used to calculate the [16].

$$\% \, Water = \frac{\mathrm{M_{initial\ sample}} - \mathrm{M_{final}}}{\mathrm{M_{initial\ sample}}} \times 10 \quad (2)$$

Carbon Residue

Carbon residue is determined by weighing the residue after heating the oil to a high temperature in the absence of air. 3 grams of the used oil is put into a crucible and place in a steel tube device, and heat it to 550°C for 30 minutes. Equation (3) is used to calculate remaining residues percentage of the total sample [13].

$$\% \, Carbon\ Residue = \frac{\mathrm{M_{Carbon\ residue}}}{\mathrm{M_{initial\ sample}}} \times 100 \quad (3)$$

Dynamic Viscosity

The spindle is immersed in the test fluid. The viscometer (Brookfield) measures by the additional torque required of the spindle to overcome the viscosity resistance and restore velocity as shown in fig. (6). This value is converted to centipoise (cp) and displayed on the screen. The dynamic viscosity is measured at 40 and 100 ° C. Equation (4) is used to calculate the kinematic viscosity by dividing the dynamic viscosity by the density at each temperature of 40 and 100 °C [14].

$$\text{Kinematic viscosity} = \frac{\text{dynamic viscosity}}{\text{density}} \quad (4)$$

Fig. 6 Brookfield viscometer

Viscosity Index

The viscosity index is a widely used measure of the change in the kinematic viscosity due to the change in oil temperature between 40 and 100 ° C. For oils with similar kinematic viscosity, the higher the viscosity index, the less the temperature effect on the kinematic viscosity [16,17].

$$VI = \frac{L-U}{L-H} \times 100 \quad (5)$$

Where:

"L" is the kinematic viscosity at 40°C of an oil 0 viscosity index having the same kinematic viscosity at 100°C.

"H" is the kinematic viscosity at 40°C of an oil 100 viscosity index having the same kinematic viscosity at 100°C.

Firstly, the kinematic viscosity of the oil (U) is measured at 40 ° C. Then from the table shown in appendix A, the corresponding values of "L" and "H" were recorded.

Substituting the values obtained from "U", "L", and "H" in the above equation (5), and yielded the viscosity index.

Characteristics of used lubricating oil can be seen in Table (1).

TABLE 1
CHARACTERISTICS OF USED LUBRICATING OIL

B. Methodology

1. Materials

- A suitable amount of used lubricating oil for our experimental work was collected from different local mechanical centers in Cairo.
- Conc. Sulfuric acid (98%).
- Sodium hydroxide (NaOH).
- Activated bentonite clay.

All chemicals were purchased from El Shark El-Awsat company for chemicals (Giza).

2. Method

The stock of a homogeneous mixture of used lubricating oil allowed for settling for 3 days to settle the coarse particles in used oil [25].

Vacuum distillation was applied for 400ml sample used lubricating oil at temperature 250℃ for 1 hour by using an electrical heater to eliminate water and light hydrocarbons from the used lubricating oil sample [24-27].

The reaction between used oil and conc. sulfuric acid takes place to remove heavy impurities from used oil. The time, temperature, and acid to oil ratio can be changed according to the conditions predicted from statistical analysis, and the speed of stirring is about 250 rpm [24-27].

The main parameters affecting the reaction (temperature, reaction time, and acid/oil ratio by volume) are studied using the statistical design approach which is displayed below. The other parameters are fixed such as the stirring speed was 250 rpm for all runs [24-27].

Response surface methodology (RSM) analysis of problems in which a response is affected by several independent variables to optimize this response and it is used to reduce the time, cost, effort, etc., and prevent the overlapping between the variables, due to its ability to vary all these variables simultaneously according to a specific scheme and determining experimentally the output function on each run [12]. Several techniques are available to that aim which is carried out in case of the presence of three or more independent variables, like Box – Wilson method, Box – Benhken method, and Tagoshi method, etc.

The Box – Benhken model was adopted. Equation (6) used to get the number of required experiments is based on the number of effecting variables (k) which should be three variables or more and repotted experiments at the center of design (Co). The most common for Co is five experiments. Then the number of experiments (N).

Characteristics	Measured Value	Standard Value
Pour Point (ºC)	-30	-3
Flash Point (ºC)	126	185-195
Density at 40 ºC (Cp)	0.875	0.87
Density at 100 ºC (Cp)	0.841	0.83
Kinematic Viscosity @ 40ºC	178.4	120-130
Kinematic Viscosity @100ºC	15.933	9.3-21.9
Viscosity index	91.75	80-120
Water Content (Wt %)	1.3	0.01
Ash Content (Wt %)	2.1	0.04
Carbon Residue (Wt %)	1.7	0.35-0.55

$$N = 2k\,(k-1) + C_o \qquad (6)$$

Table (2) illustrated the experiments obtained from Box-Benhken statistical design in actual value. Viscosity index (VI) was the response.

TABLE 2
THE STATISTICAL ANALYSIS PROGRAM

Run	Factor 1 A:temp oC	Factor 2 B:time min	Factor 3 C:ratio A/O
1	50	90	15
2	50	120	10
3	40	90	20
4	50	60	20
5	60	90	20
6	60	120	15
7	50	90	15
8	40	120	15
9	40	60	15
10	50	60	10
11	60	60	15
12	50	90	15
13	50	90	15
14	60	90	10
15	50	120	20
16	40	90	10
17	50	90	15

After reaction completion, the sample is allowed to settle for 72 hours in a separating funnel, the upper layer represents the oil produced while the lower layer is acidic sludge.
Neutralization is carried out by using NaOH to adjust pH between 6.5-7.

Activated bentonite clay acts as an adsorbent to improve the color, odor, and oxidation stability of the oil. Oil is mixed with 15% by weight of activated bentonite at 110℃ and 450-500 rpm for 1 hour.

Source	p-value	
Model	0.0003	Significant
A	0.0033	Significant
B	0.3114	Not- Significant
C	0.5734	Not- Significant
AB	0.0006	Significant
AC	0.0096	Significant
BC	0.0003	Significant

The separation of oil from clay is carried out by using a centrifuge (Hettich - Universal 16A) at 5000 rpm for 45 minutes.

III. Results And discussion

Table (3) illustrated in actual value results of the desludging reaction. Where A is temperature (ºC) , B is time (min), and C is acid to oil ratio (A/O), and the response is viscosity index (VI).

Run	Temperature	Time	A/O ratio	VI
1	50	90	15	100.984
2	50	120	10	108.208
3	40	90	20	107.595
4	50	60	20	106.108
5	60	90	20	96.9352
6	60	120	15	102.281
7	50	90	15	100.054
8	40	120	15	98.9303
9	40	60	15	110.64
10	50	60	10	98.1994
11	60	60	15	94.9455
12	50	90	15	101.727
13	50	90	15	102.203
14	60	90	10	101.922
15	50	120	20	94.5851
16	40	90	10	100.108
17	50	90	15	96.558

TABLE 3
ACTUAL EXPERIMENTAL RESULTS WITH DIFFERENT PARAMETERS

The suggested model was found to be highly significant, its determination coefficient R^2 being (0.88).

Table (4) shows the analysis of variance (ANOVA) for the two-factor interaction (2FI) model which used to know the coded and actual equation by knowing P-values**.**

TABLE 4
ANOVA FOR 2FI MODEL

In case of P-values are less than 0.05 indicate model terms are significant, in this case, A, AB, AC, and BC are significant model terms. However, in case of P-values are greater than 0.05 indicate the model terms are not significant. So, B and C are not significant model terms.

Equations (7) and (8) shown the correlation between viscosity index and the three studied parameters was obtained in coded and actual equations respectively which include the significant model terms only.

$$VI = (101.29) - (2.65*A) + (4.76*AB) - (3.12*AC) - (5.38*BC) \quad (7)$$

$$VI = (101.29) - (2.65*temperature) + (4.76 *temperature*time) – (3.12*temperature*acid\ to\ oil\ ratio) - (5.38 * time *acid\ to\ oil\ ratio) \quad (8)$$

Interaction Between Selected Parameters

2-D and 3-D plots can be drawn for a different variation of parameters which exhibit the trend of response varying within the selected range of input parameters and also the effect of each parameter over the other parameters. With the aid of statistical design analysis, the interaction between the three studied parameters namely Temperature "A", Time "B" and acid to oil ratio "C" can be studied using the obtained model graph contours and 3D cubic model. The contours are at which each factor is studied at its minimum, average, and maximum value concerning the other two parameters, while in the 3D cubic model all the parameters are varied from their maximum and minimum values indicating the value of viscosity index at each edge of the cubic model.

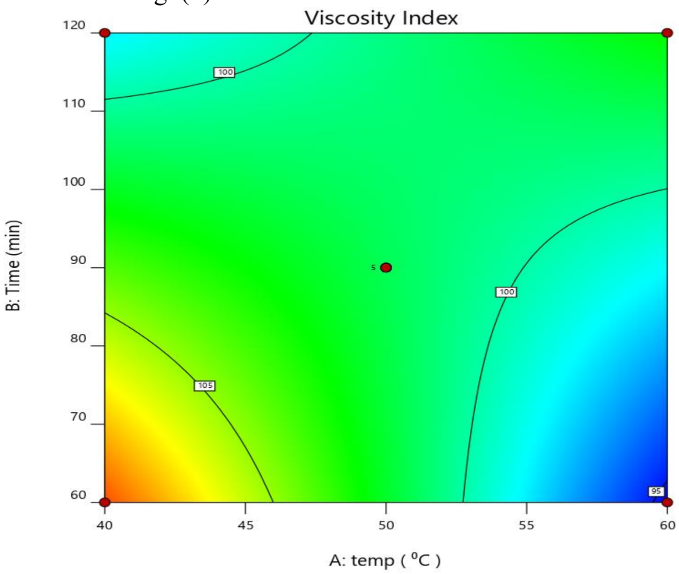
a) Effect of the Reaction Time and Temperature on the Oil's Viscosity Index

The temperature "A" on X-axis and reaction time "B" on Y-axis, these factors were studied while varying the value of the acid to oil ratio "C" to its minimum, maximum, and average values to study its effect on the lubricating oil's viscosity index.

At a minimum value of acid to oil ratio (C=10). The viscosity index will decrease when the temperature increases. However, the viscosity index will increase when time increases, especially when temperature and time increase simultaneously, the viscosity index also will increase as shown in fig. (7).

Fig. 7 AB contour with respect to the ratio " C" minimum

At an average value of acid to oil ratio (C=15). The viscosity index will decrease when the temperature increases and also the viscosity index will decrease when time increases, especially when temperature and time increase simultaneously, the viscosity index also will decrease as shown in fig. (8).



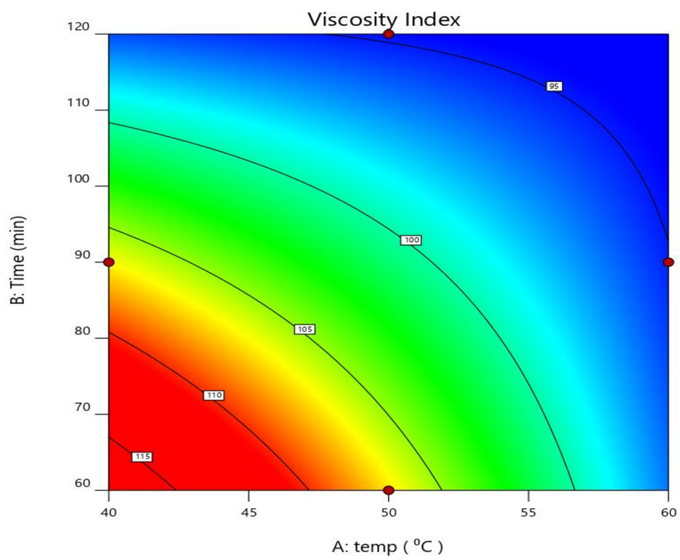
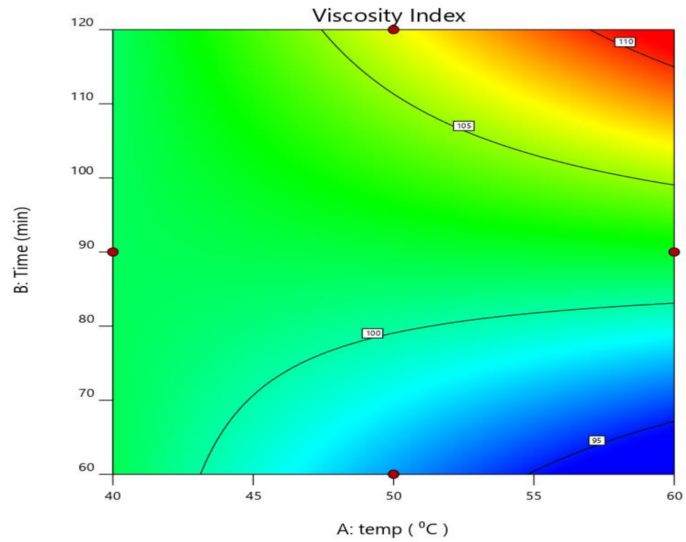


Fig. 8 AB contour with respect to the ratio " C" average

At a maximum value of acid to oil ratio (C=20). The viscosity



index will decrease when the temperature and time increase, especially when temperature and time increase simultaneously, the viscosity index also will decrease as shown in fig. (9).

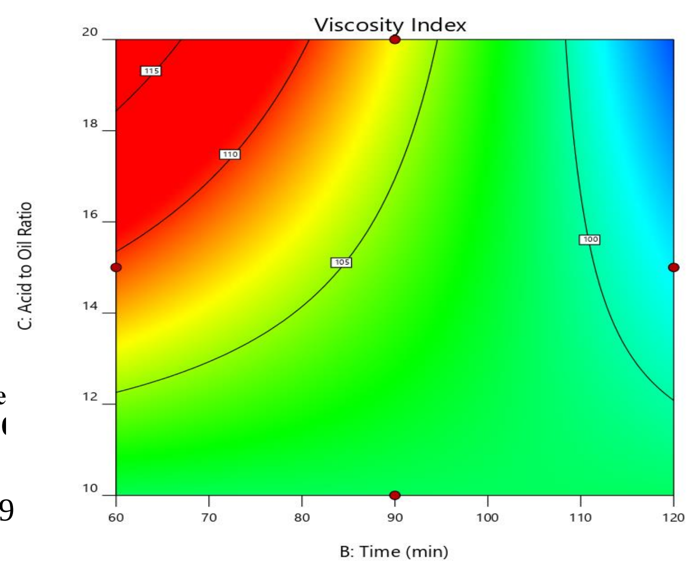
Fig. 9 AB contour with respect to the ratio " C" maximum

b) *Effect of the Reaction Time and Acid to Oil Ratio on the Oil's Viscosity Index*

The temperature "A" and acid to oil ratio "C" factors were studied while varying the value of the reaction temperature "A" to its minimum, maximum, and average values to study its effect on the lubricating oil's viscosity index.

At a minimum value of temperature (40 °C). The viscosity index will decrease when time increases. However, the viscosity index will increase when acid to oil ratio increases, especially when time and acid ratio increase simultaneously, the viscosity index also will decrease as shown in fig (10).

Fig. 10 BC contour with respect to the temp." A " minimum



At an average value of temperature (50 °C). The viscosity index will increase when time and acid ratio increase, especially when time and acid to oil ratio increase simultaneously, the viscosity index will decrease as shown in fig. (11).

Fig. 11 BC contour with respect to the temp.” A " average

At a maximum value of temperature (60 °C). The viscosity index will increase when time increases. However, the viscosity index will decrease when acid to oil ratio increases, especially when time and acid to oil ratio increase simultaneously, the viscosity index also will increase as shown in fig. (12).

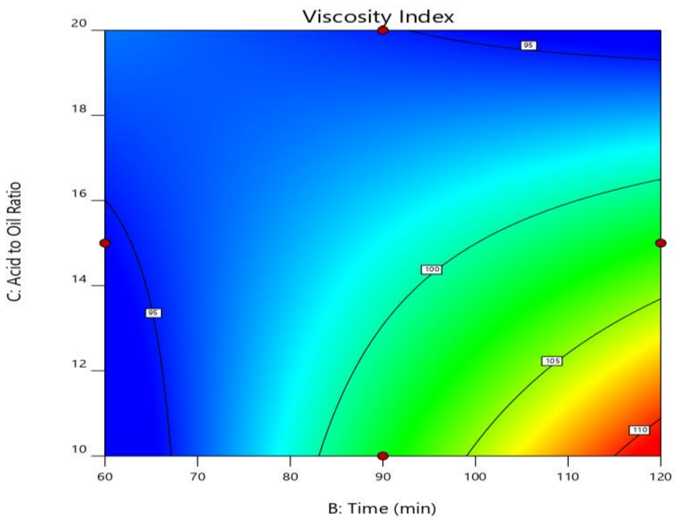


Fig. 12 BC contour with respect to the temp.” A " maximum

c) *Effect of the Reaction Temperature and Acid to Oil Ratio on the Oil’s Viscosity Index*

The temperature “A” on X-axis and acid to oil ratio “C” on Y-axis, these factors were studied while varying the value of the reaction time “B” to its minimum, maximum, and average values to study its effect on the lubricating oil’s viscosity index.

zAt a minimum value of time (60 min), the viscosity index will decrease when temperature increases. However, viscosity index will increase when acid to oil ratio increases, especially when temperature and time increase simultaneously, the viscosity index also will decrease as shown in fig. (13).

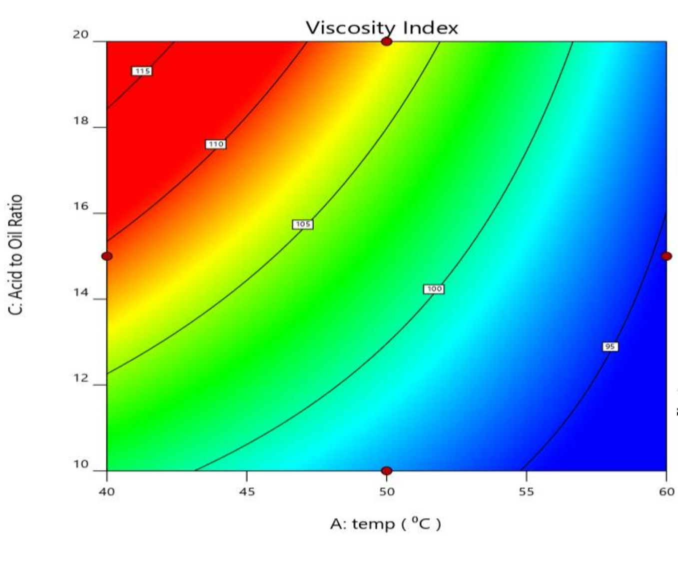
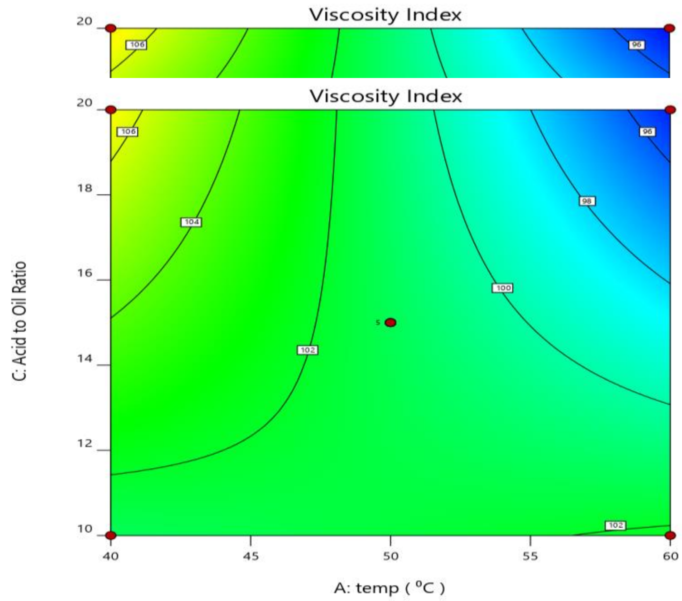


Fig. 13 AC contour with respect to the time “B " minimum

At an average value of time (90 min). The viscosity index will decrease when temperature increases. However, the viscosity index will increase when acid to oil ratio increases, especially



when temperature and acid to oil ratio increase simultaneously, the viscosity index also will decrease as shown in fig. (14).

Fig. 14 AC contour with respect to the time “B " average

At a maximum value of time (120 min). The viscosity index will increase when temperature increases. However, the viscosity index will decrease when acid to oil ratio increases, especially when temperature and acid to oil ratio increase simultaneously, the viscosity index will decrease as shown in fig. (15).

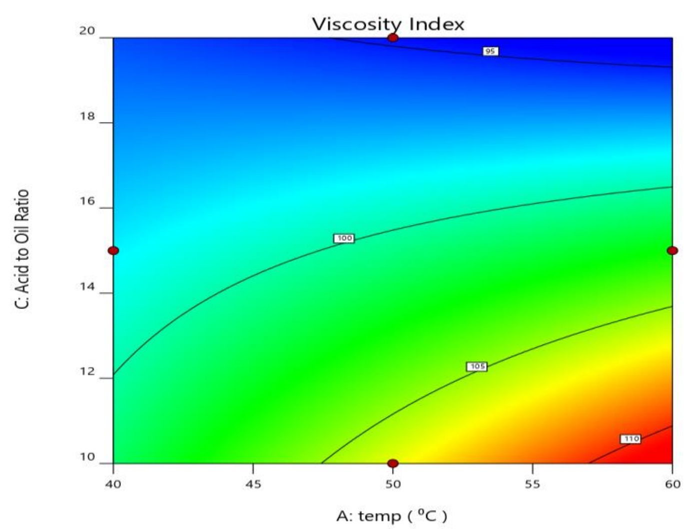


Fig. 15 AC contour with respect to the time “B " maximum

Contour Statistical Model Graphs

A contour graph is the projection of a 3D graph model on a 2D plot, it indicates the maximum, minimum points as well as the effect of two parameters on a certain response on a 2D plot. Contour is a 2D model in which two parameters change while the third remains constant at its minimum, average, and maximum value to determine the maximum viscosity index that can be obtained at these values. Changing the color from blue to red indicates increasing in the viscosity index, as getting closer to red-colored regions indicates a higher viscosity index, and getting closer to blue regions indicates minimum viscosity index obtained at the corresponding conditions to each region. Each line shows the viscosity index obtained at any condition corresponding to the two other parameters through the line. Contour plots are useful for establishing the response values and operating conditions as required.

All experimental results are plotted on a 3D cube. It can be noticed that the Box-Behnken design is a spherical design with all points on its edges. Also, the Box – Behnken design does not contain any point at the vertices of the cubic region created by the upper and lower limits for each variable.

The minimum and maximum values of the three parameters affecting the viscosity index namely: temperature "A", reaction time "B" and the stoichiometric ratio between the acid to oil "C", all the previous parameters lies on X, Y, and Z axis respectively, the cubic model indicates that the maximum viscosity index can be obtained with the value of 117.538 at lowest reaction time and highest stoichiometric ratio used 60 minutes and 20:1 acid to oil ratio and temperature at 40°C, as shown in the lower right rear edge of the cubic model as shown in fig.(16).

The minimum viscosity index of 92.7568 can be obtained at the conditions of high temperature of 60°C, with the high reaction time of 60 minutes and 10% v/v acid to oil ratio, as shown in the upper right front edge of the cubic model.

A predicted maximum viscosity index of 117.538 was obtained using a 20% v/v acid to oil ratio, a reaction temperature of 40°C, and a reaction time of 60 min. To assess the validity of these findings, 5 runs were performed at the aforementioned conditions.

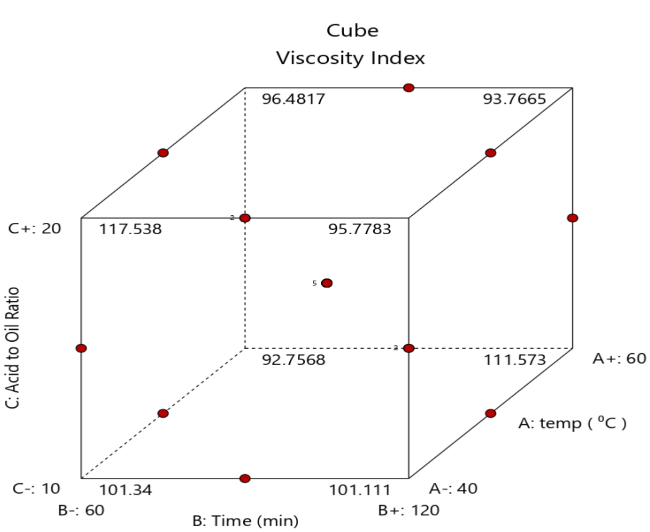


Fig. 16 3D plot for the result of recovered lubricating oil viscosity index

FTIR analysis

Fig. (17) shows the result of Fourier transform infrared spectroscopy (FTIR) for optimum sample which is very close to the standard sample.

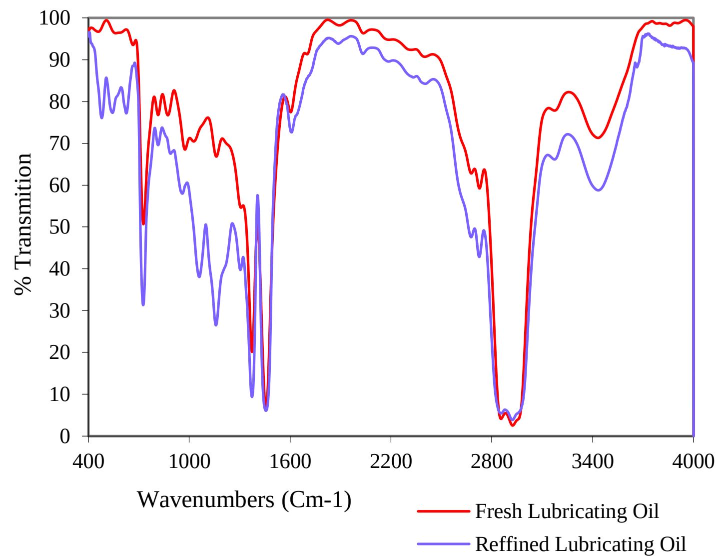


Fig. 17 FTIR for fresh and refined lubricating oil

Table (5) shows the characteristics of refined lubricating oil (RLO) which measured in Egypt Petroleum Company.

All properties of refined lubricating oil are accepted according to the Egyptian standards of refining oil.

TABLE 5
CHARACTERISTICS OF REFINED LUBRICATING OIL

Property (unit)	Measured Value	Egyptian Standard
Pour Point (°C)	-5	> -3
Flash Point (°C)	190	185-195
Density at 40 °C (g/ml)	0.8644	0.87
Density at 100 °C (g/ml)	0.8366	0.83
Kinematic Viscosity @ 40°C	128.44	120-130
Kinematic Viscosity @100°C	17	9.3-21.9
Water Content (Wt %)	0.01	0.01
Ash Content (Wt %)	0.02	0.04
Carbon Residue (Wt %)	0.487	0.35-0.55
Viscosity Index	110.2996	80-120

IV. CONCLUSION

The refining of used lubricating oil has many methods such as acid clay, solvent extraction, and vacuum distillation method. The acid clay process is the most common method for refining used lubricating oil because it produces refined lubricating oil with optimum properties and more economical method. Response surface methodology (box-benhken design) is used to predict the optimum conditions affecting on desludging reaction. The selected parameters for desludging reaction were time (from 60 to 120 min), temperature (from 40 to 60 ° C), and acid to oil ratio (from 10 to 20 v/v). The optimum conditions were time (60 min), temperature (40 ° C), and acid to oil ratio (20 v/v). The average value of viscosity index was 110.3 and the standard deviation was 0.739. Characteristics of product refined lubricating oil were examined which included viscosity index, flash point, pour point, density, dynamic viscosity, ash content, and water content and they were within the standard limits.

V. ACKNOWLEDGMENT

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