DETERMINATION OF OPTIMAL MIX FOR ACID RECLAMATION OF USED ENGINE OIL

B. N. Nwankwojike*, O. S. Onwuka, A. A. Lewachi and T. C. Nwadiugwu
Department of Mechanical Engineering, Michael Okpara University of Agriculture, Umudike
*Corresponding author’s e-mail address: jikeobodo@gmail.com

Abstract
The effects of varying ratio of used engine oil and sulphuric acid, with kaolin as contacting agent, on reclaimed oil properties was analyzed in this study using response surface methodology. Results revealed optimal used engine oil-acid ratio of 1: 6.3. The reclaimed oil obtained from this optimal ratio has viscosity at 100°C, viscosity at 40°C, viscosity index, specific gravity, pour point, flash point and total acid number as 12cst, 120cst, 92.87, 0.85, 9°C, 177°C and 0.53mg/KOH respectively. Also, the engine oil reclaimed from this optimal mix gave values of viscosity at 40°C, specific gravity and total acid number so close to that of a standard grade/fresh lube oil.

Keywords: Acid treatment, optimal mix, reclamation, sulphuric acid, used engine oil

1. Introduction
Engine oil is a mixture of lube oil stock (crude oil derivatives) and certain other chemicals (additives). It is used as a lubricant to function as a contaminant cleaning agent, an anti-corrosion, a cooling agent, and for friction/wear reduction in moving parts of engines (Rashid et al., 2013). It is the only petroleum product that does not wear out or get used up during operation but rather gets contaminated (Mithilesh, 2005). Thus recycling of the used oil is preferable to its disposal to help avert environmental pollution and sustain this valuable resource. In addition, less energy is required to produce a gallon of re-refined base stock than a base stock from crude oil (EPA, 2017). During use, engine oil gets contaminated with metal particles (iron, steel, copper, lead, zinc, etc.) from engine wear and other impurities such as barium, sulphur, water, dirt, burnt carbon, and ash from the surrounding air (Udonne and Bakare, 2013). Most of these compounds are highly toxic in nature, and react with the additives in the base oil, thereby deteriorating the qualities desired of the engine oil for effective/specific application. Hence, reclamation/recycling of used engine oil entails the removal of contaminants in it to restore the desired qualities of the oil for its reuse.

Used engine oil can be regenerated from processes like centrifugal separation, magnetic separation, vacuum dehydration/distillation, acid refining and solvent refining. Albeit these methods effectively removed contaminants from used lubricating base oil and returned the oil to a quality essentially equivalent to oils produced by fresh lube oil stocks, acid treatment method is much cheaper to apply (Rashid et al., 2013). According to this author, the main drawbacks of this method is high acid value of sludge, which creates series of environmental problems. This is dependent on the quantity of acid utilized for the reclamation process. In addition, Onwuka et al. (2016) revealed that varying ratio of acid to used engine oil in this reclamation process affects the properties of the reclaimed oil in a way that as one improves another deteriorates. The contacting agent is not considered a factor in the reclamation of engine oil because the effective responsibility of breaking and reclaiming the base stock is achieved by the quantity of acid used and the mixing ratio of engine oil and acid. Contacting agents act as a chemical sump which collects the waste from the reclamation process for discharge as well as bleach the colour of the reclaimed oil (Usman and Kayode-sote, 2011). Thus, the need to determine the optimal ratio of
acid and used oil at which the properties of the reclaimed oil will adequately conform to the set standard. In an empirical optimization problems of this nature where varying input combinations relates to multiple desired levels of product characteristics, response surface methodology is usually applied. This is because the technique involves special experimental designs/optimization tactic with small number of experimental runs to save time and cost (NIST/SEMATECH, 2012). Thus, this study applied response surface methodology to determine optimal ratio of acid to be used in engine oil for effective reclamation.

2. Materials and Methods
The determination of the optimal ratio of acid to engine oil for the reclamation of the used engine oil using kaolin as contacting agent involves: experimental design, data collection, model fitting, model selection, model validation and optimization (NIST/SEMATECH 2006). A response surface design generated with MINITAB 17.0 was used to evaluate the empirical relationship between the factors and responses. The volume of the sulphuric acid and Used Engine Oil (UEO) are the reclamation factors considered while the kinematic viscosity, flash point, pour point, viscosity index, specific gravity and colour clarity of the reclaimed oil constitute the responses. A central composite design comprising of four factorial points (-1, 1), six centre points (0) and four axial points (-α, α) amounting to fourteen (14) experimental runs was used in this study. The high levels (+1) of the UEO and Acid used are 200ml and 100ml respectively while 20ml and 10ml constitute their corresponding low levels (-1). The transformation equations are given by equations (1), (2), and (3).

\[ Coded\ value = \frac{Real\ value - (\text{high\ value}\ +\ \text{low\ value})}{2(\text{high\ value}\ -\ \text{low\ value})} \]  

\[ x_1 = \frac{\text{UAE} - 150}{50} \]  

\[ x_2 = \frac{\text{A} - 15}{5} \]

Where UAE and A are the real values of used engine oil and acid respectively, while \( x_1 \) and \( x_2 \) are their corresponding coded values. The Flash Point (F.P), Pour Point (P.P), Viscosity Index (V.I), viscosities at 40°C and 100°C (\( V_{40} \) and \( V_{100} \)), Total Acid Number (TAN) and Specific Gravity (S.G) of the recovered oil were analyzed using Cleveland Open Cup Tester ASTM D 92, Pour Point ASTM D 97, Viscosity Index ASTM D 2270, Total acid number (TAN) ASTM D 664 and Density (Specific Gravity) ASTM D 1298 respectively (Onwuka et al., 2016). The UEO was collected from garages within umuahia metropolis, while sulphuric acid, kaolin, magnet bar, filter paper, buchner funnel, vacuum pump, thermometer, centrifuge, laboratory glasswares and electric hot plate were some other materials and equipment used in this study. The response surface models were developed with MINITAB 17.0 from the developed design. Analyses of variance, lack of fit test, residual analyses were used to check the adequacy of the estimated models to approximate the measured data at 95% confidence interval (NIST/SEMATECH 2006). Validation of the models were carried out by comparing the actual and predicted results. Desirability function approach was used to determine the optimal settings of the factors. Thereafter, the results were tested experimentally to check the success of the prediction and the optimization results.
3. Results and Discussion
Table 1 shows the experimental design in both coded and natural variables as well as their corresponding responses for viscosity at 100°C and 40°C, viscosity index, specific gravity, flash point, pour point and total acid number.

Table 1: Response surface experimental layout

<table>
<thead>
<tr>
<th>Run Order</th>
<th>FACTORS</th>
<th>RESPONSES</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Coded Values</td>
<td>Natural Values</td>
</tr>
<tr>
<td>1</td>
<td>-0.70711</td>
<td>114.6</td>
</tr>
<tr>
<td>2</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>3</td>
<td>0</td>
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<tr>
<td>4</td>
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<tr>
<td>5</td>
<td>0.707107</td>
<td>185.4</td>
</tr>
<tr>
<td>6</td>
<td>0.707107</td>
<td>185.4</td>
</tr>
<tr>
<td>7</td>
<td>-0.70711</td>
<td>114.6</td>
</tr>
<tr>
<td>8</td>
<td>1</td>
<td>0</td>
</tr>
<tr>
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<td>0</td>
</tr>
<tr>
<td>14</td>
<td>-1</td>
<td>0</td>
</tr>
</tbody>
</table>

The developed response functions of the reclaimed oil expressed in natural values are shown in the equations below:

\[ V_{100} = 4.3 - 0.0679E + 1.164A + 2.13e^{-4}E^2 - 0.0407A^2 \]  
\[ V_{40} = 2.7 + 14.43A - 0.4332A^2 \]  
\[ VI = 25.73 + 0.391E + 4.883A - 0.001293E^2 - 0.1492A^2 + 8e^{-5}EA \]  
\[ S.G = 0.493 + 0.045A - 0.0014A^2 \]  
\[ P.P = 4.0 - 0.019E + 0.71A - 4.4e^{-4}E^2 - 0.064A^2 + 0.01049E \]  
\[ TAN = 0.687 - 0.00128E - 0.0012A - 7e^{-7}E^2 - 9.2e^{-4}A^2 + 2.02e^{-4}EA \]  
\[ F.P = -148 - 0.12E + 39.6A + 0.00472E^2 - 0.81A^2 - 0.0807EA \]  

The confirmatory test graphs and residual plots which include the normal probability plot, histogram, residual versus fitted value and residual versus observation order are shown in figures 1-7. Smaller outliers in the normal probability plot and the formation of “S” shape along the straight line of the residuals indicate high adequacy of the fitted models. The figures also show that the percentage errors between the actual and predicted values of the responses lie within ±5%. This further confirms that the developed models statistically fit the responses and therefore can be used to establish the optimal acid to oil ratio required for reclaiming used engine oil. The desirability function optimization plot of the models (figure 8) when converted to natural values using the transformation equations (equations 2 and 3) revealed 12cst, 120cst, 92.87, 0.85, 9°C, 177°C and 0.53mg/KOH as the optimal viscosity at 100°C, viscosity at 40°C, viscosity index, specific gravity, pour point, flash point and total acid number of the reclaimed oil respectively. This is for an optimal ratio of UEO to acid (in coded input variables) of -0.47:0.96. In natural values, the optimal ratio of UEO to acid is again obtained by substituting the value of the coded...
optimal input variables in the transformation equations. The approximate optimal values of the UEO and acid are obtained as 126ml and 20ml respectively.

Figure 1: Confirmatory/residual plots of the viscosity (100°C) model

Figure 2: Confirmatory/residual plots of the viscosity (40°C) model

Figure 3: Confirmatory/residual plots of the viscosity index model
Figure 4: Confirmatory/residual plots of the flash point model

Figure 5: Confirmatory/residual plots of the pour point model

Figure 6: Confirmatory/residual plots of the total acid number model
Comparatively, the reclaimed engine oil from this optimal ratio gave values of viscosity at 40°C, specific gravity and total acid number so close to that of a standard grade/fresh lube oil which are 100.62cst, 0.9 and 0.52 mg/KOH respectively (Onwuka et al., 2016). The outstanding contribution of the quantity of acid to the thermo-chemical properties of the reclaimed engine oil is shown from the models and it is practically expected since used engine oil contains aromatic compounds which readily react with sulphuric acid. The effective breakdown of the aromatic compounds present in the used engine oil by the acid constitutes the rate at which the used engine oil is reclaimed. However, this cannot be effectively achieved without the bleaching action of the kaolin, thus the substantial contribution of kaolin to the reclamation of used engine oil.
4. Conclusion
The study revealed 126ml and 20ml as the optimal ratio of Used Engine Oil (UEO) and sulphuric acid required for the reclamation of used engine oil. The reclaimed oil obtained at these optimal ratio has viscosity at 100°C, viscosity at 40°C, viscosity index, specific gravity, pour point, flash point and total acid number as 12cst, 120cst, 92.87, 0.85, 9°C, 177°C and 0.53mg/KOH respectively. Also, the reclaimed engine oil from this optimal ratio gave values of viscosity at 40°C, specific gravity and total acid number so close to that of a standard grade/fresh lube oil.

References


