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Optimization of Diesel-Like Fuel Production from Spent Lubricating Oil Using Adsorbent Treatment

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Abstract: *This research was aimed at optimizing an adsorption process used to treat spent engine oil to obtain a product that has properties similar to fresh diesel. In order to achieve this, some kaolin clay was obtained locally and made to undergo some pretreatment steps prior to sulphuric acid activation. Also, the spent engine oil used for the study was pretreated through filtration, acid/base neutralization and drying before the main treatment step. Thereafter, the face centered central composite method of response surface methodology was employed to generate thirteen runs with activated clay dosage and adsorption time as independent factors and three physical properties of the treated oil (density, flash point and viscosity) as the responses. Accordingly, adsorption experiments were carried out and the treated oil was analysed to obtain the aforementioned dependent variables. Regression analyses were carried out to obtain models that would represent the relationship between the independent variables and each of the responses. Results obtained showed that the predictors-density, viscosity and flash point data were best represented by linear, two-factor interaction and quadratic models respectively, based on their p-values which was less than 5% on 95% confidence level. Also, the coefficient of determination value for all the response was fairly close to unity. In addition, analysis of variance results showed that adsorption time and/or adsorbent dosage significantly affected the treatment process as p-value of factorial terms in all the models were significant. Density model being affected by adsorbent dosage alone can be attributed to mass transfer between the adsorbent and the adsorbate. The treated oil with best physical properties (840 kg/m^3, 76 ^{o}C and 5.5 mPa.s of density, flash point and viscosity respectively) was achieved experimentally at 10 wt% adsorbent dosage and 60 min adsorption time. This optimum result was close to predicted one obtained as 842.436 kg/m^3, 76.3818 ^{o}C and 5.4785 mPa.s at 9.86 wt% and 59.76 min from the numerical optimization. The optimum results revealed that the recycled spent oil has properties that are similar to diesel. Since the best properties obtained are within the standard values for diesel oil, the recycled oil can be blended with fresh diesel. The use of treated oil in diesel engine is very promising but further work needs to be done to ensure thermodynamic stability of the oil.*

Keywords: *RSM, activated clay dosage, adsorption time, flash point, density, viscosity.*

1. INTRODUCTION

Lubricating oil is a fluid applied to surfaces of rolling or moving parts to prevent friction and protect the surface from wearing. After some period of use in machines such as vehicle, some physical characteristics (colour, viscosity, density etc.) and chemical compositions of the oil change and the resulting product is referred to as spent lubricating oil. Chemical breakdown of additives when the oil is in use has been identified as the main contamination source in additions to contaminants which emanate from incomplete combustion, environmental condition and dissolution of metals [1]. Thus, spent engine oil normally contains high concentration of metals, asphaltic compounds, and halogenated solvent and aromatic compounds [2]. Due to the fact that engine oil is used in large quantity, handling of the by-product of lubrication has been responsible for certain environmental pollution and hence the effort by the stakeholders to find solution to this problem through recovery the oil and some of its valuable components [1].

Huge quantities of these spent oils from auto workshops in Nigeria are dumped directly into open drainages and land, which contaminates the surrounding groundwater. Due to lack of effective spent oil management and recycle system in the country, the environmental impact from these oils have persisted. Therefore, there is a need to stimulate treatment technology that could promote better spent engine oil management practices in the country [3].

Clays such as montmorillonite, vermiculite, tillite, kaolinite, and bentonite are perceived as potential adsorbent materials due to several economic advantages, and intrinsic characteristics such as excellent textural and surface properties, and physical and chemical stability in aggressive environment [4]. Adsorption is process of using a substance usually referred to as adsorbent to recover targeted material (adsorbate) from the bulk of fluid. Though, adsorption is very popular in reclamation of wastewater and water treatment. The application of the process has also been extended to the recycling of

spent lubricating oil. Researchers have shown that spent lubricating oil can be effectively reclaimed by using adsorbents such as bentonite clay [5], zeolite [6], kaolin [7]; egg shell powder, date shell powder and activated date shell powder [8]. The use of zeolite has been reported to remove up to 50 and 62 % zinc and magnesium ions respectively from recovered waste oil at the optimum temperature and time range of 30-50 ^{o}C and 0-10 min respectively [6]. Also, Riyanto et al. [7] investigated the adsorption of some metals from chemically pre-treated waste lubricating oil using kaolin. The study showed that the treatment of the spent lubricating oil led to significant reduction of the selected metals (calcium: 99.77 %, magnesium: 97.73%, lead: 71.89%, iron: 88.71% and chromium: 31.67%). Besides, the process was observed to be affected by butanol, potassium hydroxide and kaolin dosage. The alcohol and the base have been used in this work to break the water bond and coagulate suspended substances in the oil.

Adsorption is a surface phenomenon which is usually affected by morphology and chemical composition of adsorbent in addition to operating conditions such as adsorbent dosage, adsorption time, initial concentration of the adsorbate and temperature [9]. Thus, optimization of the operating conditions is necessary in order to achieve the maximum efficiency of the process.

Response surface methodology is a statistical tool employed for optimization of multifactor dependent system. It usually proceeds through design of experiment followed by statistical modelling and numerical or graphical step of finding the optimum operating conditions that give the desired goal of the responses [10].

In this work, an attempt has been made to make use of Jos-South kaolin clay which is found in the Local Government Area in abundance at no cost to treat spent engine oil obtained from the Plateau state capital of Nigeria with the aim of recycling the pollutant to curb its environmental impact. Based on the available literature, many of the works on waste lubricating oil treatment have focused on conventional one-factor at a time optimization method. Therefore, in this study, adsorption experiments were carried out according to a central composite design of response surface methodology and optimum conditions to achieve the set goals of optimization were achieved. The efficiency of treatment method was evaluated using physical properties of spent, treated and fresh engine oil.

2. MATERIALS AND METHODS

2.1 Sampling Techniques

Samples collection of used engine oils was done using systematic sampling technique covering various districts in Jos-North from used oil dumps of car mechanics in various mechanic workshops around Jos North metropolis. A total of ten (10) litres of the sample was collected in a plastic container from twenty (20) different locations.

2.2 Preparation of spent oil and Characterization

The methods used in the treatment of the spent engine oils include filtration to remove impurities, acid treatment, alkali treatment (to neutralize any excess acid), and acid/clay treatment [11].

The spent engine oil was filtered to remove impurities such as metal chips, sand, dust, particles, micro impurities that are contained in the lube base oil. This was done using a funnel with a filter paper placed in it, then a vacuum pump was connected to the filtering flask to which the funnel was fixed with the aid of a rubber stopper. The sample was made moisture free by first carrying out atmospheric distillation. Mild heating was gently applied to remove the dissolved gases.

Spent engine oil obtained was placed on a regulator hot plate. The temperature of the spent engine oil was maintained between 40 and 45 ^{o}C. At this temperature, concentrated Sulphuric acid was introduced into the spent engine oil with simultaneous stirring of the mixture for ten minutes.

The density of the treated oil was determined with aid of density hygrometer by using 100 ml of the sample. In order to measure the flash point of the treated oil, 10 ml of the oil sample was measured in a 100 ml separate beaker, the sample was placed on a Bunsen burner and the oil was stirred. A flame source was then brought at intervals to determine the temperature at which a flash would appear on the surface while the oil in the beakers is being heated at a specific rate.

Viscometer was used to measure the viscosities of treated oil. The viscometer set up consisted of silver line test vessel with an agate orifice placed in a casing. 50 ml of the sample was placed in the test vessel and until it attained a required temperature. It was then allowed to flow by gravity and the time of flow was noted. Generally, viscosity is related to the time of flow of a fixed volume of the test sample through a given capillary viscometer.

2.3 Preparation of Activated Clay

The beneficiated kaolin clay was collected and grounded and made into slurry with distilled water. Impurities such as sand and stone settled at the bottom. The slurry was then decanted. The slurry was kept in an oven at temperature of 110^{o}C to be dried up. The dried clay was then grounded into very fine particles and sieved to a mesh of 0.5 mm using test sieve on a mechanical shaker. 200 g of clay (after dirt, sand and stone have been removed) was weighed and made into slurry with distilled water of about 80 cm^3. 50-60cm^3 of Sulphuric acid in 0.35 mole/cm^3 concentration was measured and added to the slurry made.

The slurry was then poured into aluminium pan and left for one hour at temperature between 90 and 100^{o}C. After the time duration, the mixture was washed with distilled water in order to remove any excess acid. The pH of washing water was monitored until it was found to be neutral. The washed clay mixture was dried in an oven for one hour and grounded into powdery form [12].

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2.4 Experimental Procedure (Adsorption Experiment)

This spent oil stream was treated by weighing and applying variable quantity of activated clay to the oil and mixing the oil stream with clay. The amount of activated clay was varied at 2 and 10% w/w of the oil and mixed thoroughly while the time was varied between 0 to 60 minutes. A total of thirteen adsorption experiments was carried according to a design matrix generated using central composite design of response surface methodology with the aid of Design Expert 7.0.0 [13]. The variation of percentage addition is to establish the ideal adsorbent quantity for the best result. After each experiment, the adsorbent was recovered by filtration while the treated oil was still warm. Shortly after adsorption, the resulting mixture obtained was filtered through a Buchner funnel connected to a vacuum pump to remove the adsorbent.
The pH of the filtered oil was adjusted to a neutral value by introducing some amount of caustic soda with continuous stirring for ten minutes. At the end of this exercise the oil was allowed to sediment in a beaker for four hours and was subsequently decanted into a beaker while the residue at the bottom of the beaker was discarded.

2.5 Statistical Analysis of Experimental Data and Optimization

After carrying out adsorption experiments, the responses were entered appropriately to their columns and entire data in the design matrix were modelled using regressional method available in the software. Density and Flash point data were fitted using linear and quadratic model as suggested by Fit Summary. Also, using the same criterion, two factor interaction model was used for Viscosity as this gave the best fit summary that maximized both the predicted and adjusted R-squared while minimizing PRESS. For the optimization aspect, the numerical approach of Design Expert 7.0.0 was used and the criteria were set based on the standard requirements of the responses as "in range" for both factors and all the responses except Viscosity which was minimized. The optimum conditions were validated by comparing predicted results to those of experimental ones at approximate conditions.

3. RESULTS AND DISCUSSION

3.1 Characterization and Adsorption Experiments

The characterization carried out on the spent engine oil revealed that the oil had density flash point and viscosity of 882 kg/m^3, 102 ^{o}C and 19 mPa.s respectively. All the values of the properties investigated were obviously high when compared to a typical standard of a heavy oil like diesel. Thus, a justification for treating the oil so as to reduce the pollutant load.

Table 1: Experimental design matrix

Runs	Percentage of Adsorbent (%w/w)	Time (min)	Density of Treated diesel-grade fuel (kg/m^3)	Flash point of Treated diesel-grade fuel (^{o}C)	Viscosity of Treated diesel-grade fuel (mPa.s)
1	6.00	60.00	850	79	7.0
2	10.00	60.00	840	76	5.5
3	6.00	40.00	853	78	8.3
4	2.00	40.00	854	78	8.4
5	6.00	40.00	853	78	8.3
6	2.00	60.00	853	78	8.3
7	2.00	20.00	855	81	8.6
8	6.00	40.00	853	78	8.3
9	6.00	40.00	853	78	8.3
10	6.00	20.00	854	81	8.8
11	10.00	20.00	850	79	8.5
12	6.00	40.00	845	77	8.3
13	10.00	40.00	840	76	5.7

The results obtained at various adsorption conditions were as given in Table 5. The densities of treated oil obtained all fell within in the range of 820-870 kg/m^3 reported by Adeyanju & Manohar [14] as being the required speciation for diesel fuel. As found in a report by Seo [15] diesel fuel is safe for use if its flash point is in the range of 52 ^{o}C and 88 ^{o}C. As it can be seen from Table 1, the flash points obtained for all the experimental runs fall within this limit. Also, the minimum and maximum values of viscosity of the treated oil were 5.5 and 8.8 mPa.s respectively.

Moreover, going the maximum factorial point (10, 60) yield a product with density of 840 kg/m^3, flash point of 76 ^{o}C and viscosity of 5.5 mPa.s. and minimum factorial point (2 w/w%, 20 min) yielded 855 kg/m^3, 81 ^{o}C and 8.6 mPa.s respectively. This is an indication that variation of the factors significantly affected those responses. As it can be noticed from the table, variation of these factors along the axial direction also had effect on the responses by comparing runs 1 and 10; and runs 4 and 13.

3.2 Statistical Evaluation and Optimization

As earlier stated, the modelling was done in accordance with the suggestions of the Fit Summary and the results of analysis of variance together with Fit Summary obtained for each model are given Tables 2, 3 and 4 respectively for Density, Flash Point and Viscosity respectively.

Table 2: Analysis of variance result for density model

Source	Sum of Squares	Df	Mean Square	F Value	p-value	Remark
Model	213.33	2	106.67	9.44	0.005	significant
A-Dosage	170.67	1	170.67	15.11	0.003	significant
B-Time	42.67	1	42.67	3.78	0.0806	not significant
Residual	112.97	10	11.3			
Lack of Fit	61.77	6	10.3	0.8	0.6142	not significant
Pure Error	51.2	4	12.8			
Cor Total	326.31	12				
			Fit Summary			
Std. Dev.	3.36	R-Squared	0.6538			
Mean	850.23	Adj R-Squared	0.5845			
C.V. %	0.4	Pred R-Squared	0.4232			
PRESS	188.21	Adeq Precision	9.909			

As it can be seen in Table 2, the linear model developed for density of the treated oil was significant with probability of error value of 0.005 since this value is less than 0.05 which is the maximum allowable p-value at 95% confidence interval. Also, on the same basis, the only significant term of this model was adsorbent dosage with p-value of 0.003. This implied that adding adsorbent to the oil statistically affected the density of the oil. The dosage significance may be as a result of mass transfer that normally occurs during adsorption process in which adsorbate moves from the fluid to the surface of adsorbent. As shown in the sign of the coefficient of the dosage (d) in the developed empirical model (Equation 1), density of the treated oil increased with reduced amount of activated clay dosage. This is in line with adsorption mechanism as increasing the dosage normally increases the rate of transfer of adsorbate to the adsorbent prior to equilibrium and this in turn reduces that amount of substances present in the fluid phase (in this case spent engine oil). Though not statistically significant, reducing the stirring time (t) also led to increase in the density of the treated oil as indicated by the negative value of t coefficient. Figures 1 and 2 also corroborate these observations on density –dosage, time relationship respectively.

$$\rho = +863.5641 - 1 \cdot 3333 * d - 0 \cdot 13333 * t \quad (1)$$

Also, the results in Table 2 show that the model had insignificant lack of fit which mean that the developed equation was a good representation of the experimental data. The Fit goodness was moreover evident in the values of coefficient of determination (R^2), adjusted R^2 and predicted R^2 which were obtained to be 65.38%, 58.45% and 42.32% respectively. All the obtained R^2 were in good agreement though not very high.

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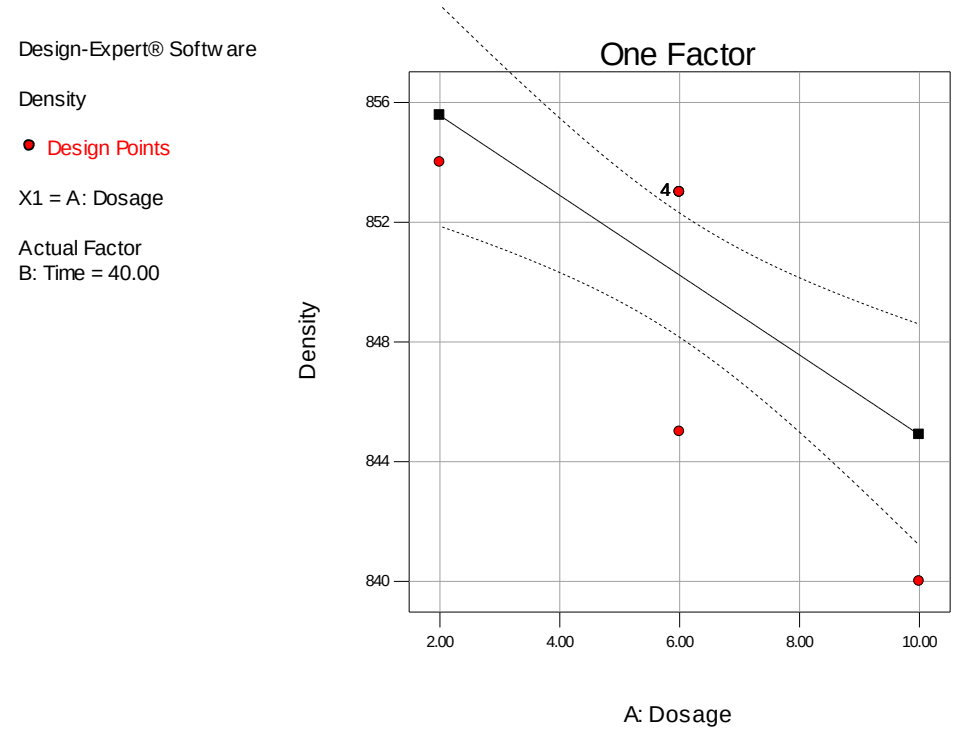

Figure 1: Effect of activated clay dosage on density of the treated oil

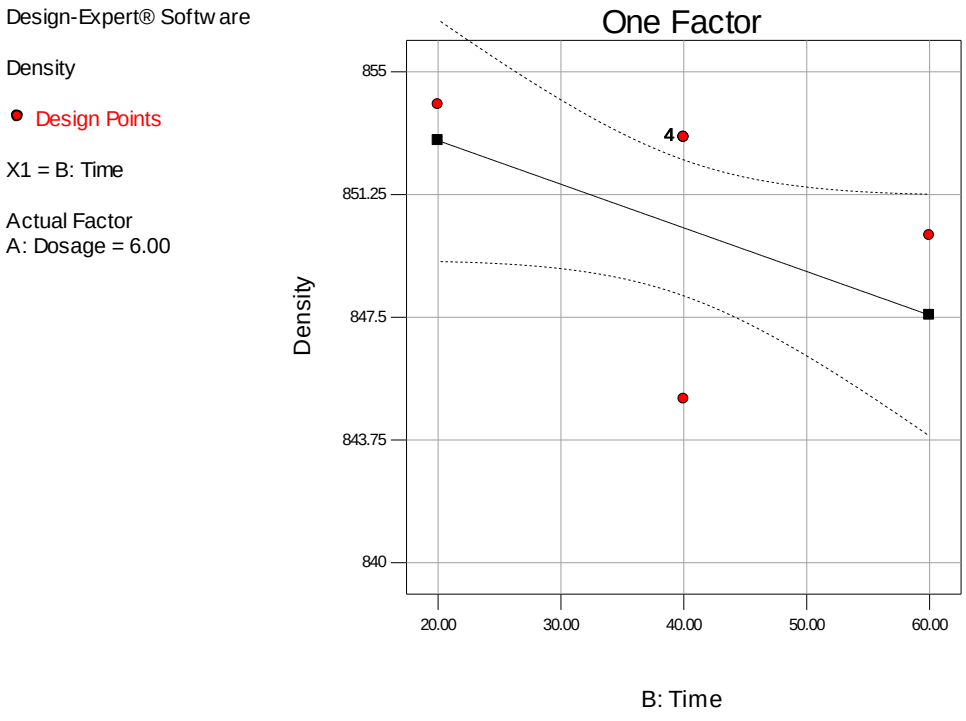

Figure 2: Effect of stirring time on density of the treated oil

The second-order polynomial developed for Flash Point (FP) data is given in Equation 2 and its results of analysis of variance are given in Table 3. As it can be seen in the remark column of this table, the model was significant and importantly affected by dosage and time linearly and quadratically. The lack of fit for the model was insignificant which meant that the model adequately represented the experimental data. Also, adequate precision of the model was evident

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from the small value of Coefficient of variation obtained to be 0.0059 fell within the range of acceptable value [16,17]. The coefficient of determination value of 94.8% and closeness of adjusted and predicted values all confirm the fit goodness.

$$FP = +86.95115 + 0 \cdot 60345 * d - 0 \cdot 43908 * t + 0 \cdot 000000 * dt + 0 \cdot 071121 * d^2 + 4 \cdot 65517E - 003 \quad (2)$$

Though, the variation of the synergic term is insignificant as shown in Table 3, it can be noticed from Figure 3 that flash point could increase with simultaneous decrease in dosage and time. The statistical insignificance of this term can also be noticed from the flash point model (Equation 2) in which the coefficient of interaction factor "dt" was obtained to 0.000000.

Table 3: Statistical analysis results for flash point model

Source	Sum of Squares	Df	Mean Square	F Value	p-value Prob > F	Remark
Model	26.84	5	5.37	25.54	0.0002	Significant
d-Dosage	6	1	6	28.55	0.0011	Significant
t-Time	10.67	1	10.67	50.75	0.0002	Significant
Dt	0	1	0	0	1	not significant
d^2	3.58	1	3.58	17.02	0.0044	Significant
t^2	9.58	1	9.58	45.56	0.0003	Significant
Residual	1.47	7	0.21			
Lack of Fit	0.67	3	0.22	1.12	0.4405	not significant
Pure Error	0.8	4	0.2			
Cor Total	28.31	12				
		Fit Summary				
Std. Dev.	0.46	R-Squared	0.948			
Mean	78.23	Adj R-Squared	0.9109			
C.V. %	0.59	Pred R-Squared	0.7575			
PRESS	6.87	Adeq Precision	17.124			

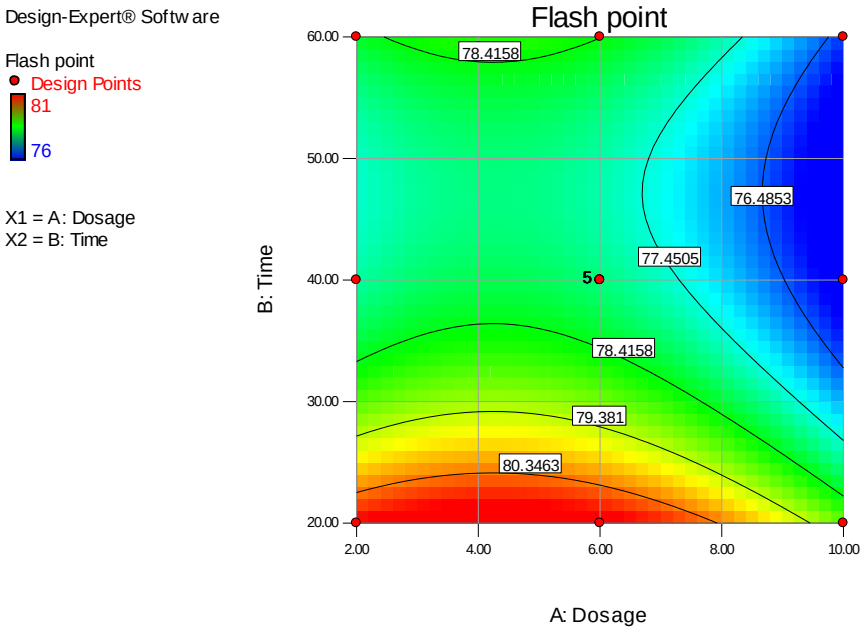

Figure 3: Effect of dosage and time on flash point

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The empirical two-factor interaction model obtained for viscosity (Equation 3) was not only significant but also significantly affected by all its terms as shown in Table 4. The R-squared value obtained (0.7979) was close to unity and the predicted and adjusted R-squared were in reasonable agreement with a difference of 0.1721. The effects of each of the term on the model can be easily understood by studying Figures 4-6. As it can be observed from these figures, viscosity model had inverse relationship with all its terms that the response decreased with increase in adsorbent dosage, adsorption time and their combination. This observation can also be attributed to mass transfer effect.

$$\mu = +8.94423 + 0 \cdot 10417 * d + 8 \cdot 12500E - 003 * t + 8 \cdot 43750 * dt \quad . \tag{3}$$

Table 4: ANOVA results for viscosity model

Source	Sum of Squares	Df	Mean Square	F Value	p-value Prob > F	Remark
Model	11.38	3	3.79	11.84	0.0018	Significant
d-Dosage	5.23	1	5.23	16.31	0.0029	Significant
t-Time	4.34	1	4.34	13.53	0.0051	Significant
Dt	1.82	1	1.82	5.69	0.0409	Significant
Residual	2.88	9	0.32			
Lack of Fit	2.88	5	0.58			
Pure Error	0	4	0			
Cor Total	14.27	12				
Std. Dev.	0.57	R-Squared	0.7979			
Mean	7.87	Adj R-Squared	0.7305			
C.V. %	7.19	Pred R-Squared	0.5584			
PRESS	6.3	Adeq Precision	11.36			

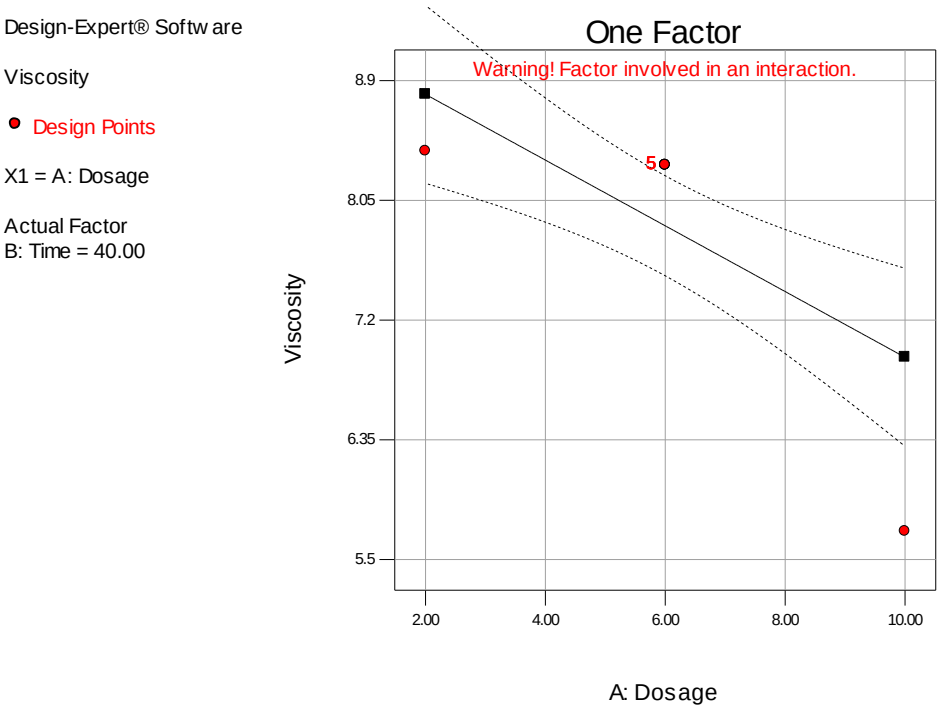

Figure 4: Effect of adsorbent dosage on viscosity

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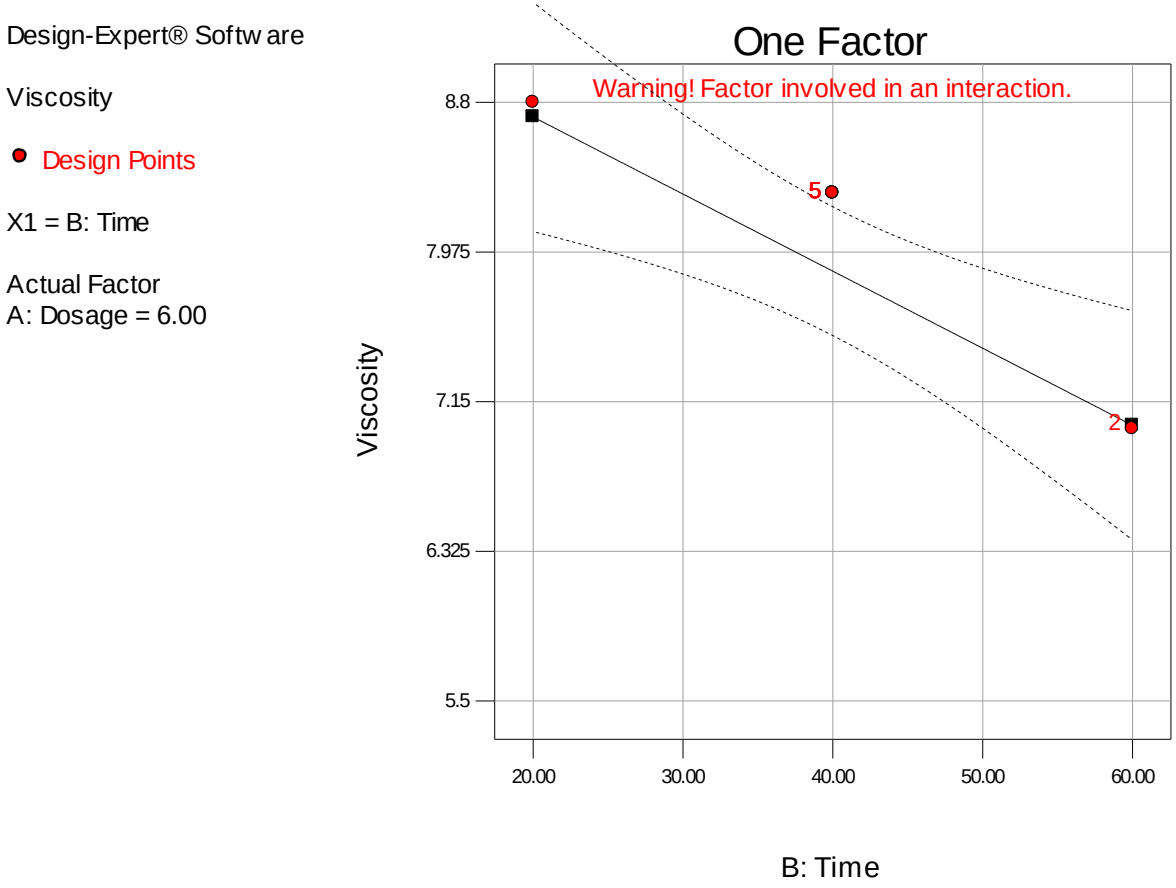

Figure 5: Effect of adsorption time on viscosity

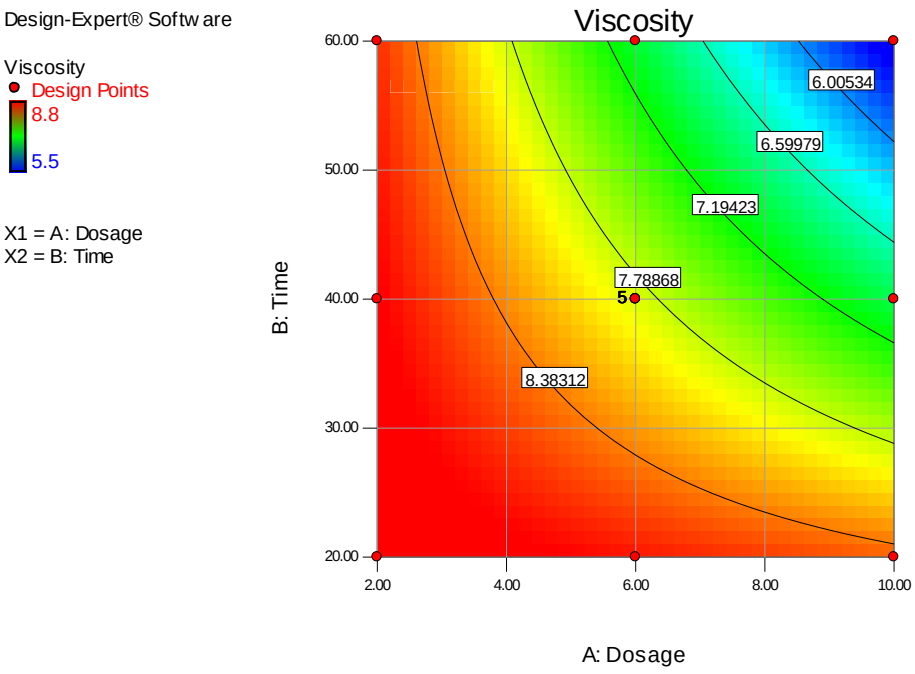

Figure 6: Effect of adsorbent dosage and adsorption time on viscosity

The results of optimization carried out numerically with goal of in range for density and flash point since all the obtained values fell within the standard and minimize for viscosity because its value must not exceed 5.5 mPa.s, revealed that best operating conditions to achieve these goals were 9.86 w% and 59.96 min of the activated kaolin dosage and adsorption time respectively. At these optimum values of the independent variables the achievable density, flash point and viscosity values were predicted to be 842.436 kg/m^3, 76.3818 ^{o}C and 5.4785 mPa.s respectively which compared well with experimentally found of 840 kg/m^3, 76 ^{o}C and 5.5 mPa.s at 10 w/w% adsorbent dosage and 60 min adsorption time. These experimental conditions are approximately the same as the predicted ones.

4. CONCLUSION

In this work, optimization of activated clay adsorbent treatment of spent engine oil to produce diesel-like oil was investigated. The physical test carried out revealed that the waste oil had high density (882 kg/m^3), viscosity (22 mm^2/s i.e. 19 mPa.s) and flash point (102 ^{o}C), thus, required treatment for the purpose of recycling. The optimum dosage of adsorbent used in treating the spent engine oil to obtain a product with a similar characteristic as diesel was established to be 10%w/w and an adsorption time of 60 minutes. The treated oil had density, flash point and viscosity of 840 kg/m^3, 76 ^{o}C and 5.5 mPa.s respectively at the best operating conditions.

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