Statistical Optimization of Biolubricant Production from Jatropha Curcas Oil using Trimethylolpropane as a Polyol

Musa Umaru, Mohammed Ibrahim Aris, Sadiq Muhammad Munnir, Aliyu Musa Aliyu,
Folorunsho Aberuagba, Adekunle Joseph Isaac

ABSTRACT - This paper presents the optimization and modeling of transesterification of Jatropha curcas methyl ester (JME) with trimethylolpropane (TMP) as a polyol in the presence of potassium hydroxide catalyst. The optimization of the transesterification reaction was carried out using 2^3 response surface methodology experimental design studying the effect of temperature (109.77-160.23 ^oC), reaction time (20 minutes to 3.68 hours) and catalyst concentration (0.73 to 1.73 % w/w) at a constant mole ratio of JME to TMP of 3.9: 1 under vacuum condition. The results of effect of process variables revealed that an optimum condition for the biolubricant synthesis was at a temperature of 120 ^oC, reaction time of 3 hours and catalyst concentration of 0.8 % w/w optimum was obtained. The percentage yield of biolubricant was 96.95 % as against 98.09 % predicted by the model. The statistical analyses of the data lead to development of the second order quadratic polynomial regression model which establishes the relationship between biolubricant yield and the process variables. The model was able adequately to predict the biolubricant yield with a co efficient of determination (R) of 0.9068.

1. INTRODUCTION

Lubricants are used for a number of applications in automobile engines, refrigeration systems and compressor (Musa *et al*., 2015). Lubricants are commonly used in these applications to minimize friction, (Mobarak *et al*., 2014). It also serves as anti-wear, cooling medium and corrosion prevention agents (Bilal *et al*., 2013). Commonly used lubricants worldwide are principally derived from petroleum based products. There is a growing concern about the continuous usage of mineral oil lubricants that are non-renewable, toxic and recalcitrant thereby harmful to the eco- system (Salimon and Salih, 2010). The high dependence of the industrial and automobile sectors on mineral based lubricants whose feedstock are not only toxic but subject to depletion in a human time scale has stimulate the need to search for sustainable alternatives in order to facilitate economic development and a sustainable green environment (NNPC, 2013).

Vegetable oil have distinguishable properties compared to mineral oils as feedstock for lubricants production, this is as a result of their unique chemical structure: such as a higher lubricating capacity, higher viscosity indices and better anticorrosion properties which provide greater affinity for metal surfaces (Salimon *et al.*, 2010). However, the low oxidative and thermal oxidative stability of the vegetable oil poses a setback to its use as lubricant; this draw back can be overcome by chemical modification of the vegetable oil (Shalini *et al.,* 2012). Amidst all the chemical modification techniques available transesterification of vegetable oil have been a more probable possibility for the production of lubricant with better temperature performance and appreciable fluidity. This techniques help to effectively replace the hydrogen atom on the β-carbon structure of the oil. This improvement brought about by change in the structure of the oil by conversion into a new ester called the polyol ester (PE). Common polyhydric alcohols used in the transesterification of fatty acids methyl ester are neopentyl glycol (NPG), pentaerythritol (PT) and trimethylolpropane (TMP). TMP is however the most popular alcohol for polyol ester synthesis because the resulting ester are characterized with superior lubricating properties. TMP is known for it high melting point and branched structure which are vital features for biolubricant synthesis (Kamil and Yusup, 2010). Recently, products like vegetable oil biolubricants are fast gaining recognitions and acceptance as a base oil for lubricant production due to its excellent biodegradability, low toxicity and environmental friendliness (IENICA, 2004; Siti *et al.*,2007), low volatility, high solubility power for polar contaminants (Salimon and Salih, 2010). Biolubricants are esters of heavy alcohols with alkyl chain usually higher than a C_5 unit, it is usually derived from vegetable oil- based feedstock and it

The authors are with Department of Chemical Engineering Federal University of Technology, P.M.B 65, Gidan Kwano Campus, Minna, Niger State, Nigeria.

Musa Umaru is the corresponding author (email: umar.musa@futminna.edu.ng)

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have lubricating properties similar to mineral oil based lubricants (Shalini *et al.,* 2012).

Jatropha curcas oil is potential non edible for bio based synthesis due to toxic nature and high oleic acid (43 %) in the oil makes it a good feedstock for biolubricant production. The bio-lubricant produced from Jatropha curcas oil have a more preferable cooling characteristics than that from palm oil, better viscosity than that from castor oil and higher oxidative stability and lower acidity than that from soybean oil (Arbian and Salimon, 2011). The production of biolubricants by transesterification process involves the transesterification of methyl ester with polyol in the presence of base catalyst to produce the desired biolubricant (Phani, 2012). The reaction requires the use of polyol such as neopentylglycol, trimethylolpropane (TMP), and pentaerythritol. The low melting point and the branching structure of TMP make it a commonly used polyol for the biolubricants production (Arbian and Salimon, 2011).

A number of studies have been reported on transesterification of Jatropha curcas oil for biolubricant synthesis (Arbian and Salimon, 2011; Ghazi *et al.,* 2010; Mohammed *et al.,* 2011; Gunam Resul *et al.,*2012; Bilal *et al.,*2013). But to the best of the author knowledge there is are relatively few documented work on the optimization of the effect of process variables on the synthesis bio-based lubricant from these non-edible oil. Most studies are limited to the use of classical experiment design involving the variation of one variable (parameter) at a time. This method is known to be time wasting, cannot give true optimal condition and do not show the significant interaction between the variables under investigation (Mohammed *et al.,* 2014).

Response surface methodology is a collection of statistical and mathematical techniques useful to develop, improve and optimize processes and products. The technique is largely applied in industry, particularly in the situations where several input variables influence some process performances or quality characteristics. In the case of a chemical reaction the dependence between the response variable yield and the two inputs, process or independent variable time and temperature can be represented. It consists on experimental strategy for exploring the process space or independent variables, empirical statistical modeling to establish an adequate approximate relation between response and process variables. The method allows the determination of optimum set of experimental conditions which minimize or maximize the response and the changes in response surfaces produced by variation of independent variables (Leca *et al.,* 2010). This statistical technique has been applied in research for complex variable systems. It has advantage of limited number of experimental runs required to generate adequate information for statistically acceptable results. It is an effective tool for process optimization (Mohammed *et al.,* 2014). Chowdhury *et al* (2013) reported the use of statistical tool for the optimization of esterification of *Jatropha curcas oil* for biolubricant production. Esterification reaction requires longer reaction time, higher operating temperature and acid employed are corrosive to the equipment.

This paper studied the response surface optimization (RSM) of *Jatropha curcas* biolubricant studying the effect of temperature, reaction time and catalyst concentration.

II. MATERIALS AND METHODS

2.1 Materials

Potassium Hydroxide Pellet from Burgoyne & Co, Mumbai (India); Trimethylolpropane from Oxford Laboratory (INDIA); Orto-phosphoric acid from May and Bakers and Distilled water from Biochemistry Department Federal University of Technology Minna, Niger State, Nigeria.

2.2 Transesterification of Jatropha Curcas Oil

A known mass of JME was measured into two neck flask equipped with thermometer, magnetic stirrer and condenser for the collection of methanol as by-product. The JME was heated to 60 ^{o}C after which TMP was measured (mole ratio of JME to TMP is 3.9:1) into the reactor. KOH catalyst was measured based on the percentage of each experimental run into the reactor, temperature of reaction was raised to that of each experimental run and the reaction time was maintained based on each experimental run (Jieyu, 2012). Constant vacuum condition was applied at each experimental run in order to reduce excessive foam formation as a result of methanol loss. The reaction product was cooled and washed with distilled water containing 10 volume % of O-phosphoric acid (35 %) in separating funnel (Phani, 2012). Continuous water washing was done until the pH of the solution was 7. The percentage production yield was determined according to (1) while the percentage yield of biolubricant was determined according to (2).

$$\text{Production yield (\%)} = \frac{mass\ of\ unwashed\ biolubricant\ X 100}{mass\ of\ methyl\ ester\ uesd} \quad (1)$$

$$\%\ \text{yield of biolubricant} = \frac{mass\ of\ dried\ biolubricant X\ 100}{mass\ of\ methyl\ ester uesd} \quad (2)$$

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2.3 Factorial experimental design and optimization of parameters for biolubricant production

A five-level, three-factorial central composite design (CCD) was applied between the transeterification of methyl ester and TMP. 20 (2^k+2k+6) experiments were conducted including the 2^3 factorial experiments, 6 axial points and 6 center points, k represent the number of independent variables (Goyal *et al.*, 2012) which are temperature, time and catalyst concentration. ± α= $2^{n/3}$ gives the distance from the axial point from the center point, n is the number of factors (α= 1.62). The temperature was varied between 120 ^{0}C and 150 ^{0}C, reaction time between 1 and 3 hours and catalyst loading between 0.8 and 1% w/w, these were based on literatures. Table 1 present Codes, ranges and levels of independent variables of temperature (T), time (t) and catalyst concentration (C) in RSM design while the catalyst percentage, the reaction temperature and the reaction time for each experimental run are shown in Table 2.

III. RESULTS AND DISCUSSION

3.1. Second-Order Quadratic Polynomial Regression Model and Statistical Analysis

Regression and graphical analysis of the data was carried out using Design-Expert 7.0. The maximum values of JC biolubricant yield were taken as the response of the design experiment. The experimental data obtained by the above procedure was analyzed by the response surface regression using the following second -order polynomial equation (4.1):

$$y = \beta_o + \sum_{i-1}^{k} \beta ix1 + \sum_{i-1}^{k} \beta_{ii} x_i^2 + \sum_{1i>j}^{k} \sum_{j}^{k} \beta_{ij} x_i x_j \quad (4)$$

where; y is the predicted, x_i and x_j are the uncoded independent variables, i and j are the linear and quadratic coefficients respectively, βo is the regression co-efficient, k is the number of factors studied and optimized in the experiment. Statistical analysis of the model equation and evaluation of the analysis of variance (ANOVA) was carried out (Goyal *et al.*, 2012).

A quadratic regression model in equation (4.2) (based on the coded factors) was generated based on the central composite design and of the experimental data:

$$Y = 86.27 + 5.24\ A + 1.04\ B - 8.73\ C - 3.78\ AB + 7.12\ A\ C + 0.63\ BC + 1.48\ A^2 - 0.043B^2 - 4.41C^2 \quad (5)$$

Where Y was the biolubricant yield (%), A is the temperature (^{o}C), B is the reaction time (hours) and C is the catalyst concentration (%), respectively, these are clearly shown in Table 4.4.

Final Equation in Terms of Actual Factors:

Yield = + 399.47725 - 5.20259 * Temperature + 29.6193 * Time + 50.9896 * catalyst concentration - 0.25325 * Temperature * Time + 4.74250 * Temperature * catalyst concentration + 6.36250 * Time * catalyst concentration + 6.62885E-003* Temperature^2 - 0.028789 * Time^2 - 439.51737 * catalyst concentration^2

The statistical significance of the model equation was evaluated by the F-value for analysis of variance (ANOVA), which showed that the regression is statistically significant at 95% confidence level. The model F-value of 10.81 for biolubricant production implied that the model was statistically significant (Table 3 and Table 4). There is only a 0.05% chance that a "Model F-Value" this large could occur due to noise.Values of "Prob > F" less than 0.0500 indicate model terms are significant (Table 4). In this case A, C, AC, C^2 are significant model terms while B, AB,BC and B^2 are the insignificant model terms.Values greater than 0.1000 indicate the model terms are not significant, however model reduction may be applied to insignificant model terms in order to improve the model. The value of regression coefficient R^2 for the model is 0.9068, this indicate that the model adequately represents the experimental results.

3.2. Effect of process variables on JC Biolubricant yield (%)

Temperature, reaction time and catalyst concentration were choosen as the process parameters in order to determine their effect on the biolubricant yield.

3.2.1 Single Effect of Temperature, Reaction Time and Catalyst Concentration on Biolubricant Yield.

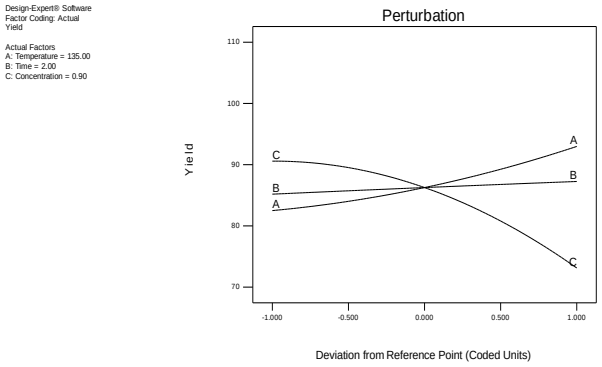

Figure 1: Individual Effect of Temperature, TIME and Catalyst Concentration on JC Biolubricant Yield (%)

Effect of temperature, catalyst concentration and reaction time on the yield of biolubricant is shown in Figure 1. It can be seen that the yield of biolubricant

increases steadily with increase temperature, this shown a positive effect on the yield. This could be due to the fact that the speed of a reaction is greatly influenced by temperature thereby resulting to higher convertion of ester. The optimized temperaturem is 120 ^{o}C, this is in agreement with the optimum temperature of 120 ^{o}C reported by Siti *et al* (2007) and Gunam Resul *et al* (2008). JC biolubricant yield decreases significantly with increases in catalyst concentration. This might be due to soap formation as a result of reaction between the excess alkali catalyst and triglyceride, thereby lower amount of biolubricant that can be separated out from reaction mixture. The optimized catalyst concentration is 0.8 % (w/w). The reaction time does not have significant effect on the biolubricant yield. Thus the yield of biolubricant decreases with increase in catalyst concentration while it increases with temperature of the reaction. The optimized reaction time is 3 hours. The predicted optimum yield is 98.09 %, this is in agreement with the 98 % optimum yield reported by Robiah *et al* (2003) however there is a slight difference to the optimum yield of 97.3, 97.66, 99.7 and 100 % for diferent oils reported by Jieyu (2012).

3.2.2 Interactive Effect of Variables on Biolubricant Yield

The 3 dimensional (3D) surface plot of the second order model helps to understand better the interactive effect of the variables on the yield of biolubricant yield.

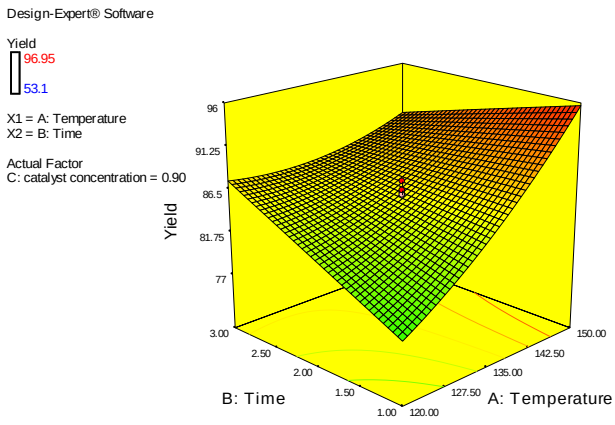

Figure 2: Response surface contour for interaction between Time and Temperature on Biolubricant Yield.

Figure 2 shows the interative effect of temperature and time on biolubricant yield. It can be seen that the biolubricant yield increases as tempeature increases which might be due to increase in the reaction speed resulting to higher convertion of the ester. The increasing effect of temperature shows that 87.3 % yield was recorded at temperature of 120 ^{o}C, reaction time of 3 hours and 0.9 % catalyst concentration while 90.1717 % yield was recorded at temperature of 150 ^{o}C, reaction time of 3 hours and catalyst concentration of 0.9 %w/w. It is also clearly shown that the biolubricant yield increases with incease in the reaction time. The increasing effect of time shows that 77.62 % yield was recorded at tempearture of 120 ^{o}C, reaction time of 1 hour and 0.9 % catalyst concentration. While 96.69 % yield was recorded at 150 ^{o}C, 1 hour reaction time and 0.9 catalyst concentration. Figure 3 shows a clearer view of the interaction of time and temperature on biolubricant yield.

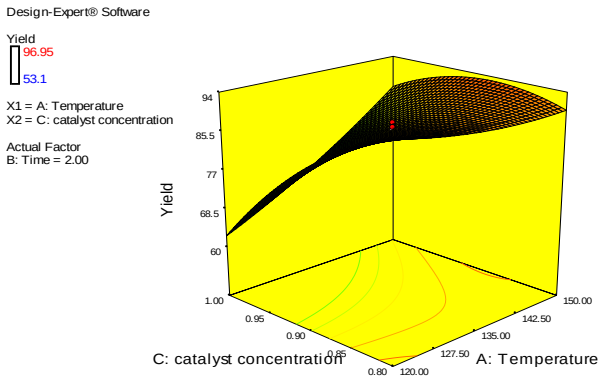

Figure 3: Response Surface interaction of catalyst concentration and Temperature of Biolubricant Yield.

Figure 3 presents the interactive effect of catalyst concentration and temperature on the yield of biolubricant yield. The biolubricant yield decreases with increase catalyst concentration; this is due to saponification reaction leading to soap formation and therefore result into less conversion of the ester into biolubricant. The decreasing catalyst's effect shows that yield of 93.927 % was recorded at the reaction condition; temperature of 120 ^{o}C, catalyst concentration of 0.8 % w/w and reaction time of 2 hours while the yield was 90.1666 % at the reaction condition; temperature of 150 ^{o}C, reaction time of 2 hours and catalyst concentration of 0.8 % w/w. Temperature increase leads to increase yield of the biolubricant, when temperatures increase, the reaction rates are obviously higher because molecules have more energy, but the saponification reaction rate speeds up, therefore the transesterification reaction yield decreases (Silva *et al.*, 2010). The increasing effect of temperature indicated that 60.2624 % yield was recorded at temperature of 120 ^{o}C, reaction time of 2 hours and catalyst concentration of 1 %w/w while 88.9583 % yield was recorded at temperature of 150 ^{o}C, catalyst concentration of 1 %w/w and reaction time of 2 hours.

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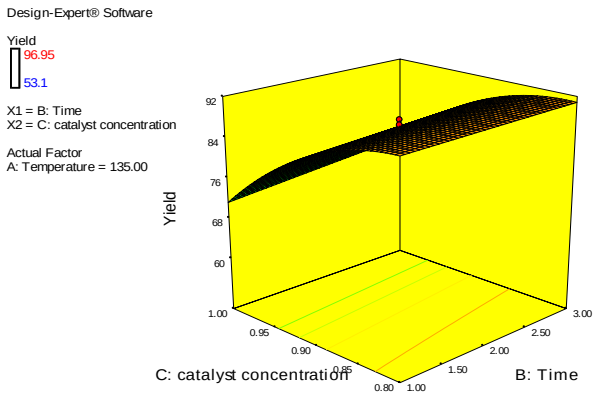

Figure 4: Response Surface of Biolubricant Yield versus catalyst concentration and Time

Figure 4 presents the interactive effect of catalyst concentration and time on the yield of biolubricant. There was no significant effect of the increase in catalyst concentration on the yield, this is due to the saponification reaction leading to high soap formation. 90.12 % yield was recorded at temperature of 135 ^{o}C, catalyst concentration of 0.8 % w/w and reaction time of 1 hour while 90.9313 % yield was recorded at reaction time of 3 hours, catalyst concentration of 0.8 %w/w and temperature of 135 ^{o}C. The biolubricant yield has a linear relationship with the reaction time, the yield increases from 71.41 % to 74.77 % the reaction time increases from 1 to 3 hours.

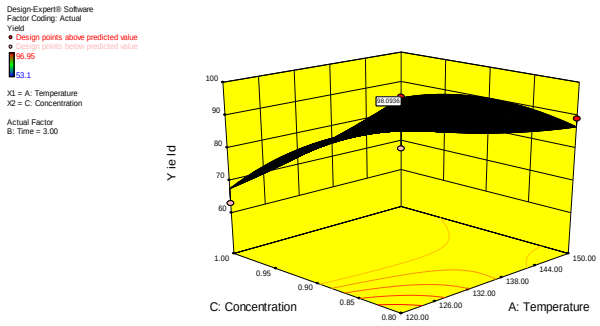

Figure 5: Response Surface Contour for Interaction on Optimum Yield of Biolubricant between Catalyst Concentration and Temperature.

IV. CONCLUSIONS

This research was carried out to study the effects of key process parameters on the yield of Jatropha curcas biolubricant. The optimization of transesterification process of JC biolubricant was made possible by three-factorial central composite design using response surface methodology in 20 experimental runs. A second-order quadratic model capable of predicting the Jatropha curcas biolubricant yield based on the process variables was developed. 98.09 % optimum yield was predicted with desirability of 0.905 at optimum conditions of temperature at 120 ^{o}C, reaction time at 3 hours and catalyst concentration of 0.8 % w/w KOH. Statistical analysis of variance (ANOVA) of results show that temperature has a positive effect on the biolubricant yield, reaction time effect is less significant and catalyst concentration has a negative influence on the biolubricant yield, however temperature has higher effect than the catalyst concentration.

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Table 1: Codes, Ranges and Levels of Independent Variables in RSM Design.

Symbols	Independent Variables	Coded Levels				
		-1.68	-1	0	1	1.68
X_1	T (^oC)	109.8	120	135	150	160.2
X_2	t (hours)	0.32	1	2	3	3.68
X_3	C (% w/w)	0.732	0.8	0.9	1	1.068

Table 2: Composite Experimental Design Biolubricant Yield.

Run	**CCRD component**	**X_1**	**X_2**	**X_3**	**Experimental**	**Predicted**
1.	Factorial	-1	-1	-1	88.15	88.15
2.	Factorial	1	-1	-1	96.95	96.96
3.	Factorial	-1	1	-1	93.9	92.96
4.	Factorial	1	1	-1	89.2	89.83
5.	Factorial	-1	-1	1	53.1	59
6.	Factorial	1	-1	1	93.05	88.75
7.	Factorial	-1	1	1	63.9	67.26
8.	Factorial	1	1	1	85.15	87.26
9.	Axial	-1.682	0	0	90	90
10.	Axial	1.0682	0	0	92.6	92.93
11.	Axial	0	-1.682	0	82.5	91.67
12.	Axial	0	1.0682	0	91.5	87.35
13.	Axial	0	0	-1.682	88.4	90.09
14.	Axial	0	0	1.0682	60.9	65.38
15.	Center	0	0	0	86.25	85.65
16.	Center	0	0	0	85.3	84.96
17.	Center	0	0	0	87.3	85.24
18.	Center	0	0	0	85.75	85.24
19.	Center	0	0	0	86	86
20.	Center	0	0	0	86.5	85.64

Mole ratio of methyl ester to TMP = 3.9:1

Table 3: Analysis of Variance (ANOVA) for the Quadratic Response Surface Model

Source	**Sum of squares**	**Degree of freedom**	**Mean square**	**F-value**	**Prob>F**
Model	2287.33	9	254.15	10.81	0.0005
Residual	235.17	10	23.52		
Lack of fit	232.33	5	46.47	82.00	<0.0001
Pure error	2.83	5	0.57		
Correction total	2522.5	19			

Adj R-Squared = 0.8229. R-Squared = 0.9068

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