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Optimization of Bio Lubricant Produced from Non-edible *Jatropha curcas* Seed Oil

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ABSTRACT. This paper presents the production and optimization of a bio-lubricant derived from non-edible Jatropha plant seed oil. Plant oil-based lubricants offer significant environmental benefits and are derived from renewable sources, making them an attractive alternative to conventional lubricants. They provide a potential solution for sustainable and low-cost feedstock for fuel oil and its derivatives without competing with food uses, as Jatropha oil contains toxic esters unsuitable for human consumption. Jatropha seed oil was extracted using a Soxhlet apparatus fitted with a heating mantle. A total of 500 g of the extracted oil was esterified in a round-bottom flask glass reactor with 25 wt% methanol and 1.0 wt% H_2SO_4 as a catalyst, reducing free fatty acids (FFA) to approximately 1%. The esterified oil was then subjected to transesterification in a reactor preheated to 60°C. A mixture of 1.0 wt% NaOH and methanol at a 6:1 ratio was added while stirring at 300 rpm for a reaction time of 90 minutes. The transesterification process was optimized using a 23 response surface methodology (RSM) experimental design. This design evaluated the effects of temperature (112.16°C to 160.00°C), reaction time (1.81 to 5.00 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 conditions. The results revealed that the optimal conditions for bio-lubricant synthesis were a temperature of 130°C, a reaction time of 3 hours, and a catalyst concentration of 0.8% w/w. A second-order quadratic model was developed to predict the yield of Jatropha curcas bio lubricant based on the process variables. The model predicted a yield of 88.74% with a desirability of 0.9739 and demonstrated a high coefficient of determination (R^2) of 0.9739, indicating strong predictive accuracy.

1. INTRODUCTION

There has recently been greater attention in the usage of renewable resources due to factors which include the decreasing quantity of the world's crude oil availability, an increasing rate of utilization, rising petroleum costs and scarcity, and conservation-related difficulties. There is consensus among scholars concerning the prospect for by-product sources to yield lubricants with higher efficiency. Vegetable oils are replacing petroleum-based lubricants in several industries, particularly the automobile industry, because of their exceptional lubricating efficiency, biodegradable properties and renewability compared to conventional fossil lubricants. Bio lubricants include feedstocks, physicochemical qualities, feedstock-to-raw-oil chemical transformation procedures, lubrication properties, and property-enhancement additives. Researchers are creating novel lubricants that are less hazardous and more biodegradable to meet environmental laws [3]. Accordingly, mineral oils made from petroleum have been replaced by lubricants derived from bio-based sources, or "bio-lubricants" [2].

Bio lubricants derived from renewable resources, including vegetable oils and animal fats, serve as a viable substitute to petroleum-based lubricants [7]. A major driving force behind the ongoing movement to replace petroleum-based industrial lubricants with more environmentally friendly vegetable oils as lubrication base stock is the depletion of fossil fuel reserves and increasing knowledge of the adverse effects which petroleum-based oils causes to the environment [5]. The presence of a closed carbon cycle further contributes to the reduction of greenhouse gas emissions. There is great concern about utilizing vegetable oils in bio lubricant manufacture; therefore, investigations are being explored into the possibility of utilizing inedible oils and waste by-products from vegetable oil processing as an environmentally friendly basis for bio lubricant production. It is possible to transform fatty acid polyol esters of vegetable oils (triglycerides), which are raw natural feedstock with significant lubricity potential, into a quick, biodegradable, renewable, and environmentally friendly lubricant base stock by the process of trans-esterification [5].

Within the past three centuries, worldwide manufacturing of oil seeds has steadily increased. This is believed to be directly related to the rising need for oil seed derivatives and by-products. The bulk of seeds are grown primarily for human intake and lubricant. About eighty percent of the vegetable oil produced worldwide is used for human intake, with the other 20 percent heading toward livestock farming and the chemical production sector (which in turn produces biofuel). Edible seed oils encompass those from groundnuts, fluted seeds of pumpkin, and soybean seeds [6]. In view of the increasing demand for oil for both consumption and commercial uses, non-edible oil is essential as a bio-lubricant feedstock. The increasing adoption of vegetable oils has contributed to the identification of numerous kinds of seed oils. Still, the vast majority have not been fully explored; this is particularly the case with Jatropha curcas seed, which is one of these ignored plant species [7]. The high amount of free fatty acids (FFA) in non-edible oils has prevented their widespread application in the production of bio-lubricants. How successfully an oil seed fits into the developing industry will be determined by its application, rate of production, and access to processing technologies. Fatty acid methyl esters (FAMEs) and polyhydric alcohol are typically employed in a trans-esterification method for making bio lubricants. An acid catalyst, a base catalyst, or an enzyme could all be used in the course of trans-esterification [1]. The quest for renewable energy sources has since dominated most manufacturing industries with much emphasis on bio-products. Several researchers have agreed on the possibility of obtaining more efficient lubricants from such (bio-products) sources. Hence, there is need to investigate the possibility of obtaining an environmentally friendly and economically viable lubricant from one of such sources Nigerian Jatropha curcas seed oil.

2. MATERIALS AND METHODS

2.1 Materials and Reagents

The Jatropha oil and ethylene glycol used were purchased from local chemical store in Benin City, Edo state and all chemicals and reagents used in the study were obtained in a high analytical grade and they are:

a) Ethylene glycol; 99.5% pure, produced by JHD, Shatou, Guondghuo, China.
b) Sodium Hydroxide; 96% pure, produced by CDH, New Delhi, India.
c) Potassium Hydroxide; 96% pure, produced by CDH, New Delhi, India.
d) Sulphuric acid; 98% pure, produced by Fisons, Loughborough, England.
e) Benzene; 98% pure, produced by BDH Chemicals ltd, Poole, England.
f) Ethanol; 99.7% pure, produced by JHD, Shatou, Guondghuo, China.
g) Methanol; 99.7% pure, produced by JHD, Shatou, Guondghuo, China.
h) Phenolphthalein Indicator; produced by Kermel Chemicals Reagent Company Ltd, Tianjin, China.

Figure 1. Block Diagram of the Production Process

2.2 Feedstock Preparation

The Jatropha seed oil was extracted from Jatropha seeds using the Soxhlet Apparatus fitted with a heating mantle. After that, 1kg of seed was sun-dried for 24 hours to reduce moisture content. The dried seeds were then grinded to fine particle size using tabletop manual mechanical grinder. Furthermore, 100 g of the grinded seeds were charged into the Soxhlet apparatus assembled on a heating mantle. The extraction process was done with n-hexane as the solvent, and it was allowed to reflux several times for maximum extraction of the oil contained. The procedure was repeated 10 times until the whole grinded seeds was extracted hence, the oil was recovered from the hexane-oil mixture by distillation. The oil was thereafter analyzed for acid value to determine the percentage of free fatty acid.

2.3 Determination of Acid Value, ASTM D 664

Exactly 0.1N KOH solution was prepared by dissolving 5.61 g KOH (pellet) with 1000 ml distilled water. Furthermore, a mixture of 99.7% pure ethanol and 98% pure benzene in a ratio of 1:1 by volume was prepared by mixing 50 ml of benzene and 50 ml of ethanol. About 1 g of the oil was weighed and dissolved in the mixture of ethanol and benzene. The solution was titrated with 0.1N KOH solution in the presence of 2 drops of phenolphthalein as an indicator until the endpoint with the appearance of pale permanent pink. The titre volume of 0.1 N KOH (V) was noted. The total acidity (acid number) in mgKOH/g was calculated using the following equation:

$$AV = \frac{M_W\, xN\, x\, V}{W} \tag{1}$$

where:

M_W = Molecular weight of potassium hydroxide (56.1 g).

N = Normality of potassium hydroxide solution (0.1 N).

V = Volume of potassium hydroxide solution used in titration.

W = Weight of oil sample.

$$\%FFA \cong \frac{AV}{2} \tag{2}$$

2.4 Moisture Content

The moisture content of oil was quantitatively determined by oven drying method at 1100 °C for 1 hour. 5 g of oil was weighed in a crucible using the electronic mass balance. The weight of the crucible and oil obtained together was placed in an oven at 110 degrees. At a time interval of 10 minutes, the crucible with oil was taken out and weighed with a new mass for both oil and crucible obtained. The process was continued till the constant weight of the oil was obtained respectively.

The moisture content was calculated using the following equation:

$$\%\text{ moisture} = \frac{Wm - Wd}{wm} x100 \tag{3}$$

where, W_m is the weight of a moist sample, and W_d is weight of a dry sample

2.5 Determination of Flash Point, ASTM D 93

The flash point of biodiesel was determined using Pensky Martens Closed Cup method (Figure 2.). The cup was filled with the biodiesel up to the mark (about 75 ml) and placed in the tester. The machine was then set to heat at about 5^{o}C/min until a rise in temperature with simultaneous stirring was observed. Small open flame was maintained from an external supply of petroleum gas. Periodically, the flame was passed over the surface of the oil. When the flash temperature is reached the surface of the oil catch flame, the temperature at the moment was noted and thus reported as flash point temperature.

Figure 2. SYD-261 Pensky Martens Closed Cup Flash Point Tester

2.6 Esterification Reaction

Exactly 500 g of Jatropha oil in a round bottom flask glass reactor was esterified with 25 wt% of methanol using 1.0 wt% H_2SO_4 as the catalyst to reduce the free fatty acids to about 1% FFA. The mixtures were placed on a constant temperature magnetic stirrer set to heat at a constant temperature of 60 ºC for 1.5 hour transesterification reaction. The procedure was repeated until %FFA was about 1 %.

Figure 3. Experimental set-up for biodiesel synthesis

2.7 Transesterification Reaction

The production of biodiesel from Jatropha seed oil and methanol was carried out in a 1000ml round bottom flask reactor equipped with a condenser and placed on a constant temperature magnetic stirrer at atmospheric pressure. 500 g of esterified oil was weighed into the reactor and preheated to 60 ºC and then added a mixture 1 wt% of NaOH and methanol at a ratio of 6:1, estimated to about 23 wt% of the oil while stirring at 300rpm for reaction time of 90minutes. The product from the reactions was poured into a 1000ml separating funnel and allowed to settle into two very distinct layers of biodiesel and glycerol.

2.8 Crude biodiesel purification

After obtaining the maximum separation, the crude biodiesel was purified by warm water washing with distilled water using a separation funnel. Since both glycerol and methanol are highly soluble in water, crude biodiesel is mixed with distilled water and agitated gently [8] to avoid emulsion formation, then slowly percolating water droplets through the ester. The process was repeated until colorless wash water was obtained, indicating the complete removal of impurities.

2.9 Bio lubricant Production

Bio lubricant was synthesized from fatty acid methyl ester as described by [9] with some modification. A batch experimental set up which comprise of a two neck 1000ml flat bottom flask, a reflux condenser and constant temperature magnetic stirrer was used. The synthesis was optimized using central composite experimental design and response surface methodology, with reaction temperature (T), Ethylene Glycol to Jatropha oil mole ratio (EG:JO) and time (t) as factors. A required amount of catalyst was added in the reaction mixture and at the end of the reaction, the product mixture was brought to room temperature and washed with warm water to remove the catalyst from the liquid mixture. The washed Jatropha bio lubricant was analyzed to determine the product composition, Pour point, viscosities, flash point and total base number (TBN).

2.10 Bio lubricant Production Experimental Design

A three-factor Central Composite Design (CCD) was employed for the experimental design on bio lubricant synthesis. The responses obtained from the CCD were optimized using response surface methodology. Factors optimized were coded at 3 levels which gave range for the reaction temperature (T) (1.0 - 5.0ºC), Ethylene Glycol to Jatropha biodiesel mole ratio (EG:BD) (2:1 – 5:1), and Time (t) (1.0 – 5.0hours). The bio lubricant yield was taken as a response for process optimization using RSM. The experimental design carried out made up to 20runs.

Experimental observations from the reaction process were analyzed and fitted according to a quadratic design model as a second order polynomial equation. Analysis of variance (ANOVA) and response surface plots were generated using Design Expert software. The optimized value of the independent variables for optimum response was determined using numerical optimization.

Table 1. Central Composite Design for Extraction

Factor	Name	Units	Minimum	Maximum	Mean	Std. Dev.
A	EG:BD	mol : mol	2.0000	5.000	3.50	1.13
B	Time	hours	1.0000	5.000	3.00	1.38
C	Temperature	ºC	130.00	170.00	150.00	13.80

2.11 Determination of the physical and chemical properties of bio-lubricant

The physical and chemical properties of bio lubricant analyzed in this study were density, dynamic viscosity at 30ºC to 100ºC, viscosity index and acid number. Density was determined by pycnometer 5 mL (IWAKI) according to the ASTM D1217. Kinematic viscosity at 30ºC to 100ºC was determined using a rotary viscometer according to the ASTM D445 standard method. The viscosity index was calculated according to the ASTM D2270 standard method. Acid number was determined by titration method according to the D974 standard method.

3. RESULTS AND DISCUSSION

3.1 Characterization of Jatropha Seed Oil

The Jatropha seed oil was analyzed and the results are shown in Table 2 below. The amount of FFA in the oil was found to be 5.266 %. The oil was treated with sodium hydroxide (a mild alkaline solution) to neutralize the Free Fatty Acid. Studies have shown that high FFA reduces catalyst effectiveness and decreases the production yield; therefore, the recommended amount of FFA in Jatropha seed oil should not exceed 1 wt% [10]. The saponification value (238mg KOH/**g**) was slightly higher due to the fatty acid composition (9.0%), which affected the iodine value [11].

Table 2. Results of characterized jatropha seed oil

Properties	Unit	Value
Acid value	mgKOH/g	10.532
FFA content	%	5.266
Water content	wt%	0.21
Flash point	ºC	186

3.2 Properties of biodiesel comparing with ASTM standard

In order to determine the quality of biodiesel after purification by distilled water, some properties were determined using the American Society for Testing and Materials standard (ASTM). ASTM identifies the parameters that pure biodiesel (B100) should fulfil before being used as a pure fuel or blended with diesel fuel [12]. The summarized results (Table 3.) show that all of the measured values were in the range of the test limit.

Table 3. Physicochemical Properties of Jatropha Seed Oil Biodiesel

	Experiment	ASTM
Density @ 30ºC (g/ml)	0.87	0.88
Kinematic viscosity @ 30ºC (mm^2/s)	4.97	1.9 – 6.0
Free Fatty Acid (%)	0.17	NA
Flash Point (ºC)	133	100 – 170

Note: NA = Not Available

3.3 Fourier Transform Infra-Red Spectrometry (FTIR) of Bio lubricant

The FTIR spectrum shows absorption peaks in the range of 3377.0 cm-1 to 723.1 cm-1, indicating the presence of various functional groups. The peaks are attributed to hydroxyl group (-OH) absorption in the range of 4000-

3700 cm-1. The IR spectrum also exhibits a broad peak in the range of 2643 cm-1 - 1707 cm-1, indicating the presence of acidic groups, specifically carboxylic acids, and a sharp peak at 3140.2 cm-1, which verifies the presence of alkynes. A sharp peak at 1645.8 cm-1 indicates the presence of (C=C) group in the extracted compound, and its geometric configuration is inferred to be cis, as indicated by a sharp peak at 770.0 cm-1. The presence of –C=O (esters) and >C-O (ethers) groups in the compound is indicated by sharp peaks in the range of 1300 cm-1 - 1000 cm-1, specifically at 1220.2 cm-1 and 1112.0 cm-1. Furthermore, the presence of another ester group (C=O-CH_3) in the isolated compound is evident from a peak at 1017.4 cm-1. Aliphatic C-H stretching is observed at 2963 cm-1, and the presence of O-CH_3 group is indicated by peaks around 1220-1017 cm-1. The analysis suggests the presence of various functional groups, including hydroxyl, acidic, alkyne, ester, and ether groups, which are indicative of the molecular structure of the extracted compound. The presence of these functional groups enables the production of bio lubricants with desirable properties such as high lubricity, low viscosity, good biodegradability, low toxicity and not limited to this list, chemical stability. t

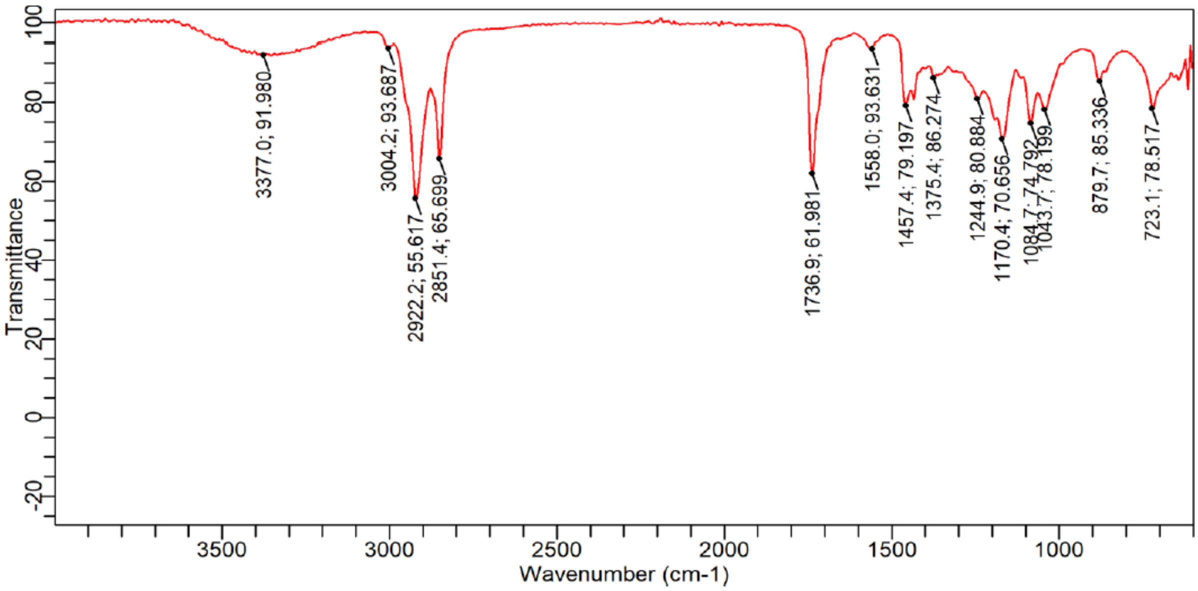

Figure 4. FTIR spectrum of synthesized bio-lubricant

Table 4. Fourier Transform Infra-Red Spectrometry (FTIR) Spectrum Analysis showing functional groups and compounds.

Wavenumber (cm^{-1})	Intensity	Functional Group	Compound
1558.0	Strong	C=C Stretching	Cyclic alkene
3004.2	Strong broad	O=H Stretching	Carboxylic acid
3376.9	Strong sharp	O=H Stretching	Alcohol
879.6	Strong	C=H Bending	1,2,4– trisubstituted
1244.9	Strong	C–O Stretching	Alkyl aryl ether
1457.3	Medium	C=H Bending	Alkane
723.1	Strong	C=C Stretching	Akene cis disubstituted
1084.6	Strong	C–O Stretching	Primary alcohol
1170.3	Strong	C–O Stretching	Ester
1736.9	Strong	C=O Stretching	Aldehyde

3.4 Optimization of the Process Parameters Using Response Surface Methodology.

Table 5. Central composite design (CCD) of bio lubricant production experiment

Run	Ethylene Glycol: Fatty Acid Esters, mol/mol	Time, h	Temperature, ºC	Bio Lubricant Yield, %
1	3.50	3.00	130.00	88.63
2	3.50	5.00	130.00	75.34
3	2.61	1.81	112.16	42.19
4	3.50	3.00	130.00	88.02
5	3.50	3.00	130.00	88.74
6	3.50	3.00	160.00	76.25
7	4.39	1.81	112.16	53.06
8	2.61	1.81	147.84	57.38
9	3.50	3.00	130.00	88.74
10	5.00	3.00	130.00	72.92
11	3.50	3.00	100.00	51.42
12	3.50	1.00	130.00	48.74
13	2.61	4.19	147.84	75.45
14	4.39	1.81	147.84	65.05
15	4.39	4.19	147.84	79.63
16	3.50	3.00	130.00	86.74
17	4.39	4.19	112.16	61.12
18	2.61	4.19	112.16	54.41
19	3.50	3.00	130.00	88.47
20	2.00	3.00	130.00	57.72

3.5 Model Fitting and Analysis of Variance (ANOVA)

The Central Composite Design (CCD) was used to optimize bio-lubricant synthesis, examining the combined effects of temperature, time, and mole ratio on the percentage yield of bio-lubricant. A total of 20 experiments were conducted, including six centre points, to determine the optimal conditions for maximum yield. The results showed that the highest yield of Jatropha tri-ester (JTE) was 88.74%, achieved at a temperature of 130°C, a time of 3.0 hours (180 minutes), and a mole ratio of 3.5:1 for Ethylene Glycol: Biodiesel. This optimization process helped identify the ideal conditions for maximizing bio-lubricant yield.

Table 6. Fit Summary of bio lubricant yield

Source	Sequential p-value	Lack of fit p-value	Adjusted r^2	Predicted r^2
Linear	0.0498	< 0.0001	0.2615	0.1626
2FI	0.9865	< 0.0001	0.1006	-0.6570
Quadratic	**< 0.0001**	**0.0501**	**0.9924**	**0.9739**
Cubic	0.1962	0.0407	0.9948	0.7808

The summary of P-values statistics is shown in Table 6. A quadratic model was suggested from the CCD module. The cubic model is always aliased because the CCD does not contain enough runs to support a full cubic model. A significance level of 95% was used hence all terms whose P-value are less than 0.05 were considered significant.

Table 7. ANOVA for Biolubricant Yield

Source	Sum of squares	Df	Mean square	F-value	P-value
Model	4693.48	9	521.50	278.24	< 0.0001
A-EG:BD	221.45	1	221.45	118.15	< 0.0001
B-Time	698.45	1	698.45	372.65	< 0.0001
C-Temperature	861.83	1	861.83	459.82	< 0.0001
AB	7.32	1	7.32	3.90	0.0764
AC	4.10	1	4.10	2.19	0.1697
BC	19.13	1	19.13	10.21	0.0096
A^2	1006.98	1	1006.98	537.27	< 0.0001
B^2	1305.76	1	1305.76	696.68	< 0.0001
C^2	1137.45	1	1137.45	606.88	< 0.0001
Residual	18.74	10	1.87		
Lack of Fit	15.64	5	3.13	5.04	0.0501
Pure Error	3.10	5	0.6203		
Cor Total	4712.23	19			

3.6 The ANOVA Results

The ANOVA results in Table 7. shows that the model variables A, B, C, BC, A^2, B^2, and C^2 are significant, with a highly significant regression F-value of 278.24 ($P < 0.0001$). This indicates that the model is significant and that the variables significantly impact the response. The P-values were used to evaluate the significance of each coefficient, with smaller P-values indicating higher significance. The high F-test value and small P-values confirm the significance of the corresponding coefficients. However, the Lack of Fit F-value of 5.04, with a probability of 5.01%, suggests that the model may not fit well, which is a concern. The results indicate that the model is significant but may require further refinement to improve its fit.

Table 8. Fit Statistics

Std. Dev.	1.37	**R^2**	0.9960
Mean	70.01	**Adjusted R^2**	0.9924
C.V. %	1.96	**Predicted R^2**	0.9739
		Adeq Precision	47.5285

The Predicted R^2 of 0.9739 is in reasonable agreement with the Adjusted R^2 of 0.9924; i.e. the difference is less than 0.2. Adequate Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 47.529 indicates an adequate signal. This model can be used to navigate the design space.

3.7 Final Equation in Terms of Coded Factors

$$\text{Bio lubricant Yield} = +\,88.28 + 4.03A + 7.15B + 7.94C - 0.9563AB - 0.7163AC + 1.55BC - 8.36A^2 - 9.52B^2 - 8.88C^2 \qquad (4)$$

The equation in terms of coded factors can be used to make predictions about the response for given levels of each factor. By default, the high levels of the factors are coded as +1, and the low levels are coded as -1. The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients.

3.7 Response Surface Analysis

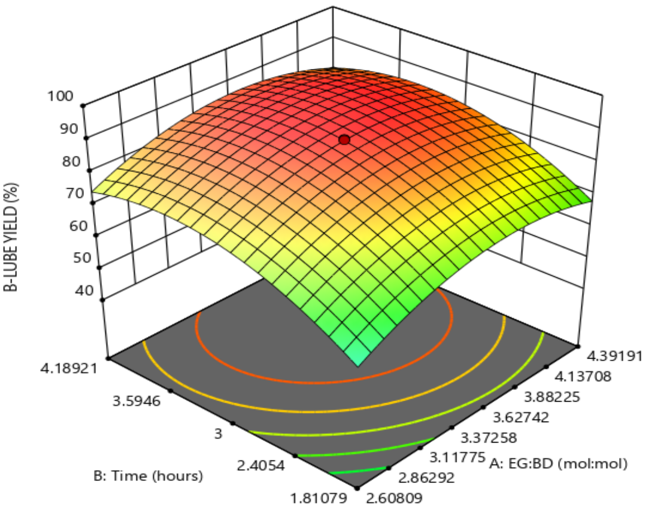

Figure 5. Interaction of time and Ethylene glycol: Fatty acid methyl Ester on biodiesel yield

The yield of bio lubricant in relation to the interactions between ethylene glycol: FAME ratio, temperature and time is shown in Figures 5 to 7. Figure 5. shows the surface plot of the yield of bio-lubricant synthesis as a function of the ratio of ethylene glycol and time [13]. Figure 6. shows that time and EG: FAME directly affect the yield of tri-ester poly-ol (bio-lubricant). The yield starts to decrease with increasing time and the mass ratio of ethylene glycol: Fatty acid methyl ester.

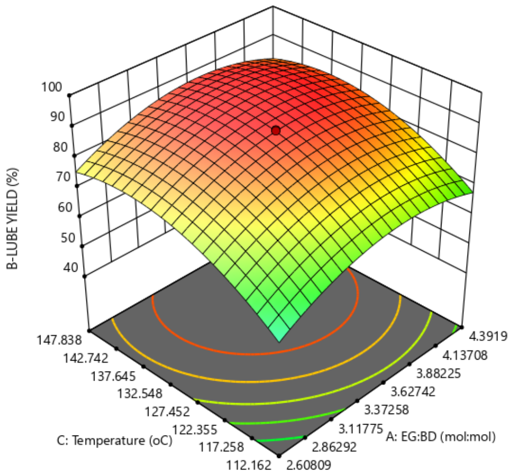

Figure 6. Interaction of temperature and Ethylene glycol: Fatty acid methyl Ester on biodiesel yield

The temperature influences reaction speed, leading to a higher conversion of FAME to tri-ester poly-ol. With an increase in the reaction temperature, bio-lubricant yield increased quickly [2]. The yield of bio lubricant reduced when the concentration of the ratio EG:BD increased beyond 3.5:1 wt%, as shown in Figure 4.3, because the increasing concentration of ethylene glycol exponentially leads to fewer contacts between FAME and methanol and thus, a lower amount of less amount of bio lubricant formation. The excess ethylene glycol lowers the conversion of tri-ester poly-ol and causes other problems associated with phase separation [10].

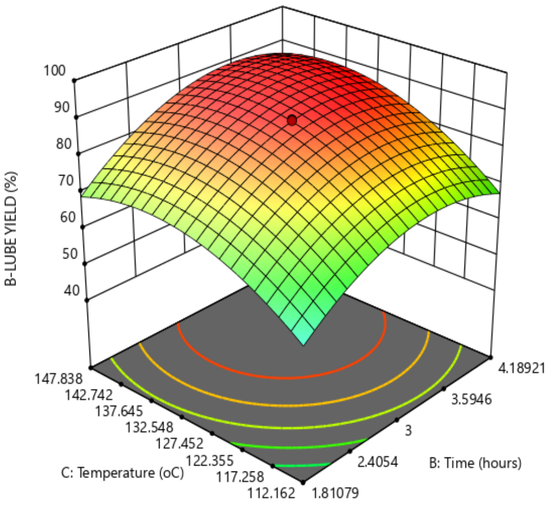

Figure 7. Interaction of time and temperature on biodiesel yield

The interaction effect of reaction time and temperature shown in Figure 7. revealed that the effect of temperature on the time on the yield and the value of interaction coefficient ($p>0.05$) demonstrate this fact.

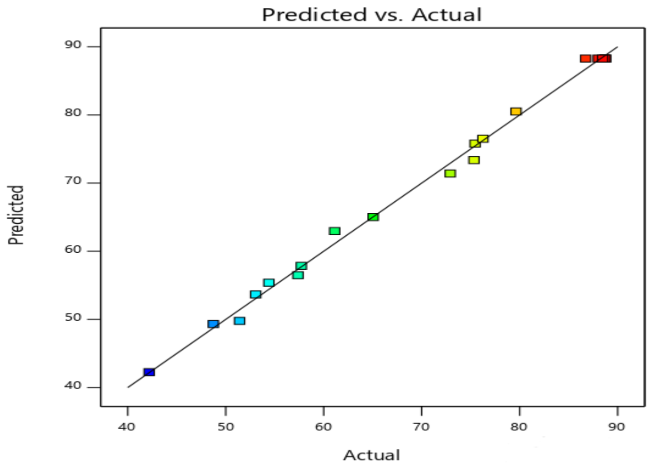

Figure 8. Plot of predicted versus actual Yield

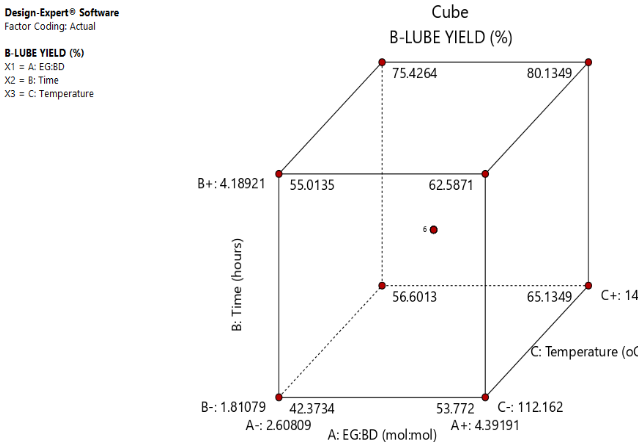

Figure 9. A cubic view of bio lubricant production factors on the yield

4. CONCLUSION

This research was carried out to study the effects of key process parameters on the yield of Jatropha curcas bio lubricant. The optimization of JC bio lubricant transesterification process was made possible by a three-factorial central composite design using response surface methodology in 20 experimental runs. Based on the process variables, a second-order quadratic model was developed to predict the Jatropha curcas bio-lubricant yield. 88.74% optimum yield was predicted with desirability of 0.9739 at optimum temperature conditions at $130^{O}C$, reaction time at 3 hours and catalyst concentration of 0.8 % w/w KOH. Statistical analysis of variance (ANOVA) of results shows that temperature has a positive effect on the bio lubricant yield, reaction time effect is less significant and catalyst concentration has a negative influence on the bio lubricant yield. However, temperature has a higher effect than the catalyst concentration.

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