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**Production And Process Optimization of Biolubricant from Neem Seed Oil****Zayyanu Iyya<sup>1</sup>, Saidu Rabi Saidu<sup>1</sup> Mohammed Misbahu Ladan<sup>2</sup>, Almustapha Nuruddeen Muhammad<sup>2</sup>, Sadiq Mas'ud Eneji<sup>3</sup>**<sup>1</sup>Department of Science Laboratory Technology, Federal Polytechnic Kaura-Namoda<sup>2</sup>Department of Energy and Applied Chemistry, Usmanu Danfodiyo University, Sokoto<sup>3</sup>Department of Biochemistry, Usmanu Danfodiyo University, Sokoto**Corresponding Author's Email:** [zayyanuiyya@gmail.com](mailto:zayyanuiyya@gmail.com)**Received on:** January, 2024**Revised and Accepted on:** March 2024**Published on:** March**ABSTRACT**

The current work investigates the conversion of neem seed oil to biolubricant. Furthermore, calcium oxide was prepared from egg shell by calcination and activated with phosphoric acid solution. The activated calcium oxide produced was characterized using FT-IR and TGA. The neem seed oil was transesterified using methanol over activated calcium oxide derived from egg shell as catalyst to produce methyl esters (biodiesel), which was again transesterified with trimethylolpropane (TMP) under reflux at different reaction conditions to produce the target biolubricant and characterized using FT-IR and GC-MS. using the same activated calcium oxide prepared from egg shell as catalyst. Optimization of process parameters for transesterification of neem seed oil was determined by Response surface based on Box-Beinkhen design. The optimum yield of the biolubricant (79.60%) was achieved at the temperature, catalyst dose and reaction time of 150 °C, 0.75% wt and 3 hours respectively.

**Keyword: Neem seed oil, egg shell, calcium oxide, methanol, TMP, RSM.****1.0 INTRODUCTION**

Energy plays an essential role in our lives in order to carry out our daily activities as well as to boost the nation's economic growth. Fossil fuels play a major role in fulfilling global energy demands for centuries. However, fossil fuels are depleting over the years due to the increasing energy demands resulting from industrialization and population growth. The ever-increasing consumption of these energy sources is alarming since the depletion of fossil fuels will have a serious impact on people's lives. Fossil-based lubricant is commonly available in Nigeria. Nigeria depends heavily on fossil fuels. Is a country with a large oil reserve. (Owuna, 2020). According to Owen *et al.*, (2010) the demand for mineral oils will likely surpass its supply by 2015 – however, this is constrained by strong recession pressures caused by the decrease in demand and thus, the demand for mineral oils is parallel with its supply. The depletion of fossil fuels and the growing concern on the detrimental impact of fossil fuels on the environment has led to the critical need to explore alternative sources of energy. The use of vegetable oils for lubrication purposes has been in practice for many years. However, this idea was scrapped due to the discovery of petroleum and the availability of low-cost oils. To date, fossil fuel-based crude oils are used as the raw materials to produce fuels and lubricants

Bio-based lubricants are promising alternatives for mineral oils since they retain the technical specifications of conventional lubricants. The use of edible vegetable oil to produce lubricants and cutting fluids is not feasible in view of the big gap in demand and supply of such oil. (Sahana VN and Chandra Kumars., 2019).

Lubricants obtained from petroleum based stocks have generally gained wider application; however, their non-biodegradability and non-renewability constitute a major environmental problem and sustainability challenges. The need to sort for renewable, eco-friendly and biodegradable feedstock for the production of lubricants has become urgent and inevitable. Renewable, non-toxic and biodegradable vegetable oil have become the first choice for ecofriendly lubricating oil ranging from hydraulic oils to greases. Therefore, modification and blending of bio-based oils, such as Neem Seed Oil to give them important qualities for various lubricating applications could provide suitable alternatives to petroleum based feedstock.

The objectives of the research is to produced biolubricant from neem seed oil using CaO source from waste egg shell as catalyst and optimize the process variable for biolubricant production i.e temperature, catalyst, time.

## 2.0 MATERIALS AND METHODS

### 2.1 Materials

Methanol (99.5%), Potassium hydroxide (85.0%), trimethylolpropane (98.0), Phosphoric acid (46.0%), and Sodium bicarbonate (95.0%) were all of analytical grade and were purchased from sigma-Aldrich (Merck).

### 2.2. Methods

#### 2.2.1 Sample collection and preparation

The egg shell sample was collected from restaurant at Usmanu, Danfodiyo University Sokoto mini market. The CaO catalyst was prepared from waste egg shell by calcinations method according to the modified procedure reported by (Babatope and Racheal, 2020). The egg shells were washed using distilled water and sun dried. Then the cleaned egg shell was crushed and calcined in the furnace at 800°C for 1 hour. After cooling, the resultant solid product was grounded, sieved and kept in air tight sample bottles. The sample bottles were kept in the desiccator to prevent air contact.

#### 2.2.2. Activation of CaO catalyst

The catalyst was activated by impregnation of a known quantity of the powdered sample using phosphoric acid as the Activated agent. Impregnation ratio of 15 g of activation agent to 5 g of the calcined egg shells was mixed. This mixture was stirred for 30 minutes; until a paste is formed and was allowed to stand for 24hrs. The activated substrate was filtered using a filter paper. The mixture was washed by pouring distilled water gradually over the filter paper containing the sample while the filter paper was placed over a conical flask. The pH of the filtrate was checked regularly using a pH meter until neutrality. The activated substrate was then dried in an oven at 105°C for 10 minutes. (Babatope and Racheal, 2020).

#### 2.2.3 Characterization of the Raw and Activated Calcium oxide derived from Egg shell

The Fourier transform infrared (FT-IR) spectroscopic analysis of activated and raw calcium oxide was carried out at Central Advanced Science Laboratory, Usmanu, Danfodiyo University Sokoto, using Carry630 Model Spectrophotometer. The analysis was performed by mixing the dry calcium oxide samples with potassium bromide (KBr) in a 1:20 weight ratio and ground into fine powder. The mixture was dried at 100°C for 3 hours, and thin pellets were made in manual equipment. The spectra were acquired at this temperature by accumulating 100 scans at 4-

4000 cm<sup>-1</sup>.

#### 2.2.4 Thermogravimetric Analysis (TGA) of the activated Calcium oxide

Thermal gravimetric (TG) was carried out at Central Advanced Laboratory, Usmanu Danfodiyo University Sokoto using TA-60WS thermal analyzer. The 14.32 g of the sample was placed on pan and inserted in to the machine. The machine started with heating rate of 5 °C per min, starting from room temperature up to 800°C in inert condition.

#### 2.2.5 Biolubricant Synthesis by Double Transesterification of Neem Seed Oil

Methyl esters of fatty acid (biodiesel) were produced in the first step, and then were converted to biolubricant by reacting with trimethylolpropane (TMP).

The two processes were carried out as described below:

##### i. Methyl Ester Synthesis using CaO derived from waste egg shell

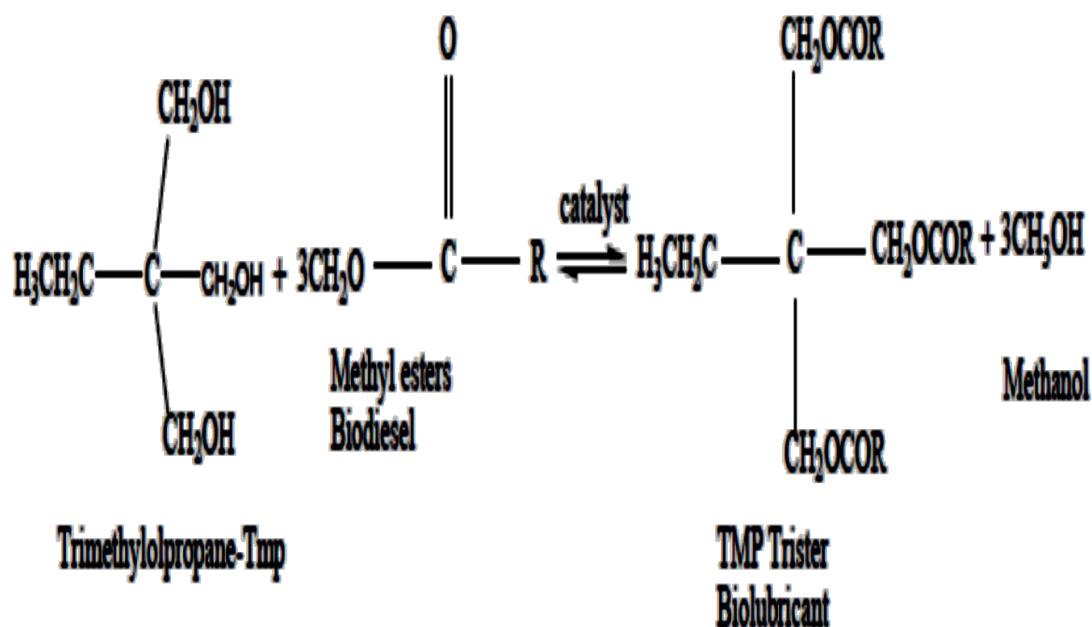
The neem seed oil (400g) was transesterified with methanol using calcium oxide as catalyst. The weight ratio of oil-to-methanol was 3:1; (i.e. 48g of methanol and 400g of oil) the amount of catalyst was 0.5% w/w of the oil (i.e. 2g of the catalysts). The reaction was conducted at a temperature of 60 °C for one hour to produce biodiesel and glycerol. The biodiesel was separated by gravity from the glycerin using separating funnel after leaving it to settle for 20 h. (Muhammad, *et al.*, 2019).

##### ii. Synthesis of Biolubricant

The previously prepared Neem oil methyl esters (Biodiesel) was weighted (100 ml) in a 500 ml reaction vessel and heated in a water bath to 70°C. Then 0.9 g of CaO based catalyst solution was added. After 10 minutes, 20 g of TMP crystals was added to the reaction vessel and the reaction was allowed to proceed for 4 hours at 100°C under reflux. The experimental set up is shown in plate 3.1. The reaction mixture was allowed to cool to room temperature. The mixture was transferred into a separatory funnel and the TMP triester (biolubricant) was collected as the bottom viscous layer as shown in plate 3.2. (Ribeiro, *et al.*, 2014). The biolubricant yield was calculated using Equation (3.1)

$$\% Yield = \frac{V_p}{V_s} \times 100 \quad 3.1$$

Where;  $V_p$  is the volume of product and  $V_s$  the volume of sample taken.



Scheme 2.1: Synthesis of biolubricant from Neem oil methyl esters (NOME) via transesterification with trimethylolpropane (TMP)

### 2.2.6 Design of Experiment

The experiment was designed using Response surface based on Box-Behnken design on MINITAB17

### 3. RESULTS AND DISCUSSION

#### Characterization of Calcium oxide, Biodiesel and Biolubricant produced

The calcium oxide, biodiesel and biolubricant produced were analyzed using FTIR, TGA, and GC-MS and the results were discussed as follows;

statistical software. The effects of three (3) factors i.e. reaction temperature (120150 °C), catalyst loading (0.5-1 wt%), and reaction time (1-3 hours) on the yield of biolubricant were determined



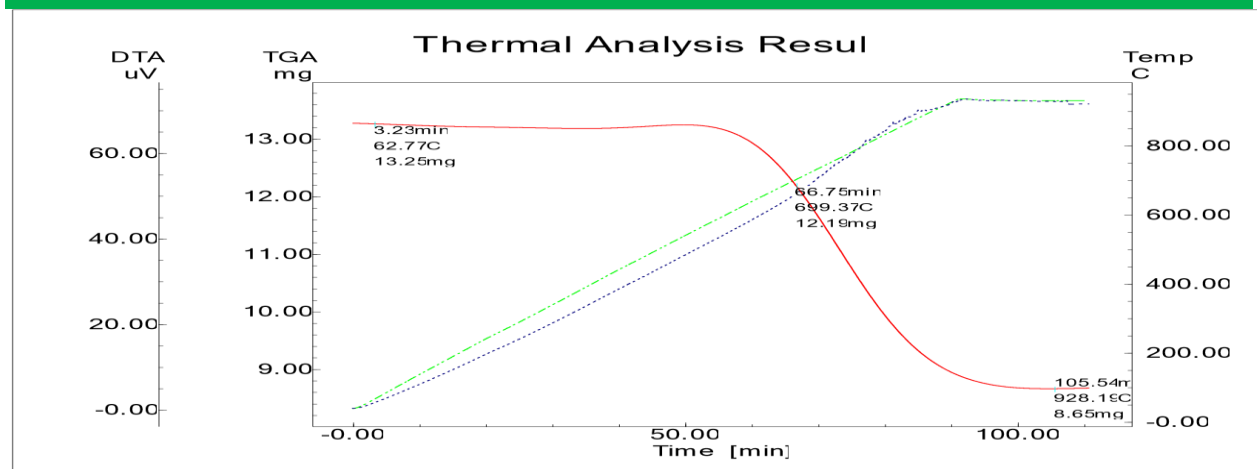


Figure 4.1: TGA of calcium oxide produced

### 3.1.2 FT-IR characterization of the biodiesel produced

The esterification of neem seed oil with methanol was carried out in a 250cm<sup>3</sup> round-bottomed flask heated and a hot plate equipped with magnetic stirrer using activated calcium oxide prepared from egg shell as catalyst. The FT-IR analysis of the biodiesel obtained shows the frequency of the main absorption band at 2929 cm<sup>-1</sup> which illustrates the C-H bond vibration of alkanes. The stretching absorption C=O seen at around 1748 cm<sup>-1</sup> indicates a characteristic absorption of the ester group, and a sharp stretching absorbance band at 1377 cm<sup>-1</sup> probably indicates C-O-H bending vibration of alcohol. Absorption was also observed at 1456 cm<sup>-1</sup> which is

characteristics absorption of CH<sub>2</sub> alkanes. C-O stretching absorption for alcohol was also observed at 1162 cm<sup>-1</sup> and sharp absorption at 721 cm<sup>-1</sup> was also observed indicating CH aromatic bending vibration. Similar absorptions to the ones observed here were also reported by Banik *et al.* (2018). However, from the spectrum obtained in the analysis of the biodiesel produced, it shows the presence of major peaks corresponding to C-H for alkyl, C=O absorption for carbonyls, and a C-O absorption band for esters. Thus, it is safe to say that, the spectrum obtained was for an aromatic ester group. Again the absorption band and possible functional groups observed were presented in Table 3.2.

Table 3.2: Absorption bands and functional groups

Absorption band (cm <sup>-1</sup> )	Bond Type
1377	O-H
1748	C=O
1162	C-O
2929	C-H
1456	CH <sub>2</sub>
721	C-H

### 3.1.3. FT-IR characterization of the biolubricant produced

The double esterification of methylesters produced from transesterification of neem seed oil with trimethylolpropane (TMP) was carried out in a 250cm<sup>3</sup> round-bottomed flask heated and a hot plate equipped with magnetic stirrer using activated calcium oxide prepared from egg shell as catalyst. From the FT-IR analysis, the esters obtained shows the frequency of the main absorption band at about 3326cm<sup>-1</sup> which illustrates the O-H bond for alcohols. There is also significant absorption at 2847 cm<sup>-1</sup> which indicates the presence of C-H bonds. The stretching absorption C=O seen at

around 1742 cm<sup>-1</sup> indicates a characteristic absorption of the carbonyl group, and a sharp stretching absorbance band at 1168 cm<sup>-1</sup> probably indicates C-O stretching for C-O-C linkage in ester. However, from the spectrum obtained in the analysis of the bio lubricant produced, it shows the presence of major peaks corresponding to O-H for alcohols, C=O absorption for carbonyls, and C-O absorption band for esters. Thus, it is safe to say that, the spectrum obtained was for an alcohol compound attached with the ester group. Again the absorption band and possible functional groups observed are presented in Table 3.3.

**Table 3.3: Absorption bands and functional groups**

Absorption band (cm <sup>-1</sup> )	Bond Type
3326	O-H
1742	C=O
1459	C-O
2847	C-H

The mixture of neem seed oil and methanol was subjected to esterification reaction using activated calcium oxide derived from egg shell as a catalyst. The reaction was carried out in a 250 cm<sup>3</sup> round bottom flask and heated under reflux on a hot plate equipped with magnetic stirrer. The product was analyzed using GC-MS. The results in Table 4.4 show the distribution of the esters obtained from esterification of neem seed oil with methanol using activated calcium oxide catalyst derived from egg shell. The esters obtained are: Hexadecanoic

### 3.1.4 GC-MS profile of the esterified (biodiesel) Product

acid methyl ester, 8,8-dimethyl-7,9-dioxabicyclo (4.3.0) nonane-3-carboxylic acid methyl ester, (Z,Z)-9,15octadecadienoic acid methylester, (Z,Z)-9,12-octadecadienoic acid methyl ester, Octadecanoic acid methylester and Methyl stearate. Similar esters were also observed by (Babatope and Racheal, 2020). Furthermore, the analysis of GC-MS of the products showed the presence of other organic products most likely by-products of the reaction.

**Table 3.4: Composition of Biodiesel produced**

Name of esters	Molecular Formula	Molecular Weight(g)
Hexadecanoic acid methyl ester	C17H34O2	270
8,8-dimethyl-7,9-dioxabicyclo (4.3.0) nonane-3-carboxylic acid methyl ester	C11H18O4	294
(Z,Z)-9,15octadecadienoic acid methylester	C19H34O2	294
(Z,Z)-9,12- octadecadienoic acid methyl ester	C19H34O2	294
Octadecanoic acid methyl ester		
Methyl stearate	C19H36O2	296
	C19H34O2	298

The mixture of previously produced methylesters (biodiesel) from neem seed oil and TMP was subjected to esterification reaction using activated calcium oxide derived from egg shell as a catalyst. The reaction was carried out in a 250 cm<sup>3</sup> round bottom flask and heated under reflux on a hot plate equipped with magnetic stirrer. The product was also analyzed using GC-MS.

### 3.1.5 GC-MS profile of the biolubricant produced

The results in Table 4.5 show the distribution of the esters obtained from esterification of methylesters with TMP using activated calcium oxide catalyst derived from egg shell. Furthermore, the analysis of GC-MS of the products showed that, other organic products detected by (Attia, et al., (2020). GC-MS are most likely by-products of the reaction.

**Table 3.5: Composition Of Biolubricant Produced**

Name of Esters	Molecular formula	Molecular Weight
Tridecanoic acid-12-methyl esters	C15H24O2	242
Methyl-8-methyldecanoate	C12H24O2	200
Hexadecanoic acid methyl esters	C17H34O2	270
Methyl state	C19H38O2	298
Hexadecanoic acid-2-hydroxy-1(hydroxyl methyl) ethyl ester	C10H38O4	330
Hexadecanoic acid-2,3-dihydroxypropyl ester	C19H38O4	330
Glycerol-1-palmitate	C10H38O4	

9-octadecanoic acid (Z)-2-hydroxy-1hydroxymethyl ester	C21H40O4	356
Octadecanoic acid-2,3-dihydroxypropyl esters	C21H42O4	358

### 3.2 Effect of the Process Variables on the Yield of Esterification Reaction

The experiments performed on biolubricant production from biodiesel esterification with TMP presented in Table 3.6 gives the experimental conditions and

percentage of biolubricant yield from the runned experiments. The percentage biolubricant yield obtained ranges from 35.0% to 79.6% with the highest yield of 79.6% at 150 °C, 3 hours reaction time and 0.75wt% catalyst dose

**Table 3.6: Optimization process for Biolubricant production and the yield obtained**

StdOrder	PtType	RunOrder	Blocks	Temp. (°C)	Time(hrs)	Cat.load(%wt)	Yield (%)
1	11	2	1	120	1	0.75	38.0
2	5	2	1	150	1	0.75	56.1
3	14	2	1	120	3	0.75	63.3
4	3	2	1	150	3	0.75	79.4
5	10	2	1	120	2	0.50	39.0
6	2	2	1	150	2	0.50	58.0
7	8	2	1	120	2	1.00	49.3
8	13	2	1	150	2	1.00	68.5
9	12	2	1	135	1	0.50	35.2
10	4	2	1	135	3	0.50	47.1
11	7	2	1	135	1	1.00	50.2
12	1	2	1	135	3	1.00	71.6
13	15	0	1	135	2	0.75	56.0
14	6	0	1	135	2	0.75	55.9
15	9	0	2	135	2	0.75	56.2
16	29	2	2	120	1	0.75	38.2
17	16	2	2	150	1	0.75	56.4
18	19	2	2	120	3	0.75	63.5
19	26	2	2	150	3	0.75	79.6
20	18	2	2	120	2	0.50	39.2
21	22	2	2	150	2	0.50	58.0
22	24	2	2	120	2	1.00	49.0
23	21	2	2	150	2	1.00	68.9
24	30	2	2	135	1	0.50	35.0
25	28	2	2	135	3	0.50	47.0
26	27	2	2	135	1	1.00	50.0
27	23	2	2	135	3	1.00	71.3
28	25	0	2	135	2	0.75	56.3
29	17	0	2	135	2	0.75	56.1
30	20	0		135	2	0.75	56.0

The analysis of variance (ANOVA), (Table 3.7) shows that, the model F-value for biolubricant yield is significant. This was confirmed by relatively higher Fischer's 'F-statistics' value and lower value of probability (P value) (Chaudhary and Balomajumder,

2014). The ANOVA results revealed that all the linear interactions terms of the process variables are statistically significant ( $p < 0.05$ , at  $\alpha = 0.05$ ). It also shows that all the interactions of square terms are statistically significant with the exception of time<sup>2</sup> which

has p-value of (0.842). The results also indicates that with the exception of time and catalyst dosage interaction, all other 2way interaction terms of the process variables are statistically insignificant ( $p > 0.05$ , at  $\alpha = 0.05$ ). The —lack of fit ( $F$ -value = 428.13) and ( $p$ -value = 0.000) implies that, the lack of fit of the data is

statistically significant ( $p < 0.05$ , at  $\alpha = 0.05$ ). Again, the high coefficient variation ( $R^2 = 96.59\%$ ) suggests that, the model adequately accounts for the empirical relationship between the biolubricant yield and the process variables.

**Table 3.7: Analysis of Variance (ANOVA) for Biolubricant yield**

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	10	4248.52	424.85	53.84	0.000
Blocks	1	0.02	0.02	0.000	0.964
Linear	3	3900.68	1300.23	164.76	0.000
Temp ( $^{\circ}$ C)	1	1321.32	1300.23	167.44	0.000
Time (hrs)	1	1674.86	1674.86	212.24	0.000
Catalyst (% wt)	1	904.51	904.51	114.62	0.000
Square	3	301.32	100.44	12.73	0.000
Temp( $^{\circ}$ C)* Temp( $^{\circ}$ C)	1	67.39	67.39	8.54	0.009
Time(hrs)* Time(hrs))	1	0.32	0.32	0.04	0.842
Catalyst(% wt)* Catalyst (% wt)	1	212.69	212.69	26.95	0.000
2 way Interaction	3	46.49	15.50	1.96	0.154
Temp( $^{\circ}$ C)*Time(hrs)	1	2.10	2.10	2.10	0.612
Temp( $^{\circ}$ C)*Cat.(% wt)	1	0.21	0.21	0.03	0.872
Time(hrs)*Cat.(% wt)	1	44.18	44.18	5.60	0.029
Error	19	149.94	7.89		
Lack-of-Fit	15	149.84	9.99	428.13	0.00
Pure Error	4	0.09	0.02		
Total	29	4398.45			

Regression analyses of the results (Table 3.8) shows that all the linear interactions of the process variables are statistically significant, also all square terms interaction of the process variables with the exception of time<sup>2</sup> are statistically significant ( $p < 0.05$  at  $\alpha = 0.05$ ). Furthermore, regression analyses also indicates that with the exception of time and catalyst dosage

interaction, all other 2-way interaction terms of the process variables are statistically insignificant ( $p > 0.05$ , at  $\alpha = 0.05$ ). With the elimination of insignificant terms from the model, the new regression model (Equation 4.2) with nine significant terms is better than the previous model (adjusted  $R^2 = 95.63\%$  compared to 94.80%), with six terms.

**Table 3.8: Results of Regression Analysis showing the Estimated Coefficients of the Model and their Significance**

Term	Effect	Coef	SE Coef	T-value	P-value	VIF	Significance
Constant		56.08	1.15	48.90	0.000		S
Blocks 1		-0.023	0.513	0.513	0.964	1.00	NS
Temp( $^{\circ}$ C)	18.175	9.088	0.702	12.94	0.000	1.00	S
Time (hrs)	20.462	10.231	0.702	14.57	0.000	1.00	S
Cat.(% wt)	15.037	7.519	0.702	10.71	0.000	1.00	S
Temp.*Temp. ( $^{\circ}$ C)	6.04	3.02	1.03	2.92	0.009	1.01	S
Time(hrs)*Time(hrs)	0.42	0.21	1.03	0.20	0.842	1.01	NS
Cat.*Cat.(% wt)	-10.73	-5.37	1.03	-5.19	0.000	1.01	S
Temp.( $^{\circ}$ C)*Time(hrs)	-1.025	-0.512	0.993	-0.52	0.612	1.00	NS
Temp.( $^{\circ}$ C)*Cat.(% wt)	0.320	0.163	0.993	0.16	0.872	1.01	NS
Time (hrs)*Cat.(% wt)	4.700	2.350	0.993	2.37	0.029	1.01	S

**Regression equation with insignificant terms**

$$\text{Yield (\%)} = 137.8 - 2.98 \text{ Temp. (}^{\circ}\text{C)} + 7.0 \text{ Time (hrs)} + 134.2 \text{ Cat. load (\% wt)} \\ + 0.01343 \text{ Temp. (}^{\circ}\text{C)} * \text{Temp. (}^{\circ}\text{C)} + 0.21 \text{ Time (hrs)} * \text{Time (hrs)}$$

$$- 85.9 \text{ Cat. load (\%wt)} * \text{Cat. load (\%wt)} - 0.0342 \text{ Temp. (}^{\circ}\text{C)} * \text{Time (hrs)}$$

$$+ 0.043 \text{ Temp. (}^{\circ}\text{C)} * \text{Cat. load (\%wt)}$$

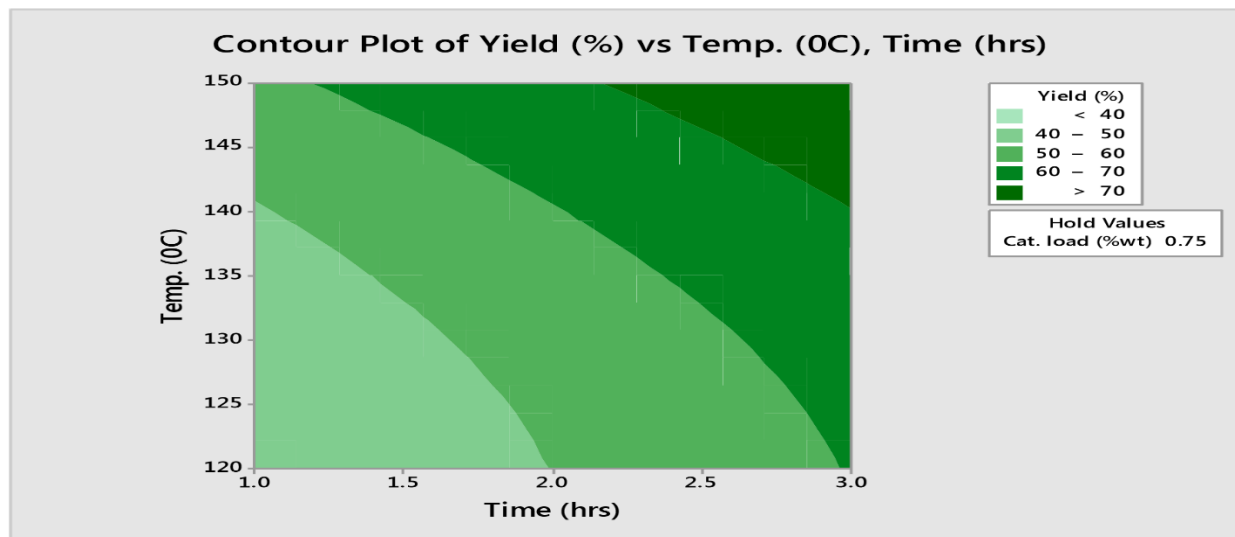
$$+ 9.40 \text{ Time (hrs)} * \text{Cat. load (\%wt)} \dots\dots\dots 4.1$$

**Regression equation with six terms after elimination of insignificant terms**

$$\text{Yield (\%)} = 140.5 - 3.00 \text{ Temp. (}^{\circ}\text{C)} + 3.18 \text{ Time (hrs)} + 140.5 \text{ Cat. load (\%wt)}$$

$$+ 0.01335 \text{ Temp. (}^{\circ}\text{C)} * \text{Temp. (}^{\circ}\text{C)} - 86.1 \text{ Cat. load (\%wt)} * \text{Cat. load (\%wt)}$$

$$+ 9.40 \text{ Time (hrs)} * \text{Cat. load (\%wt)} \dots\dots\dots 4.2$$

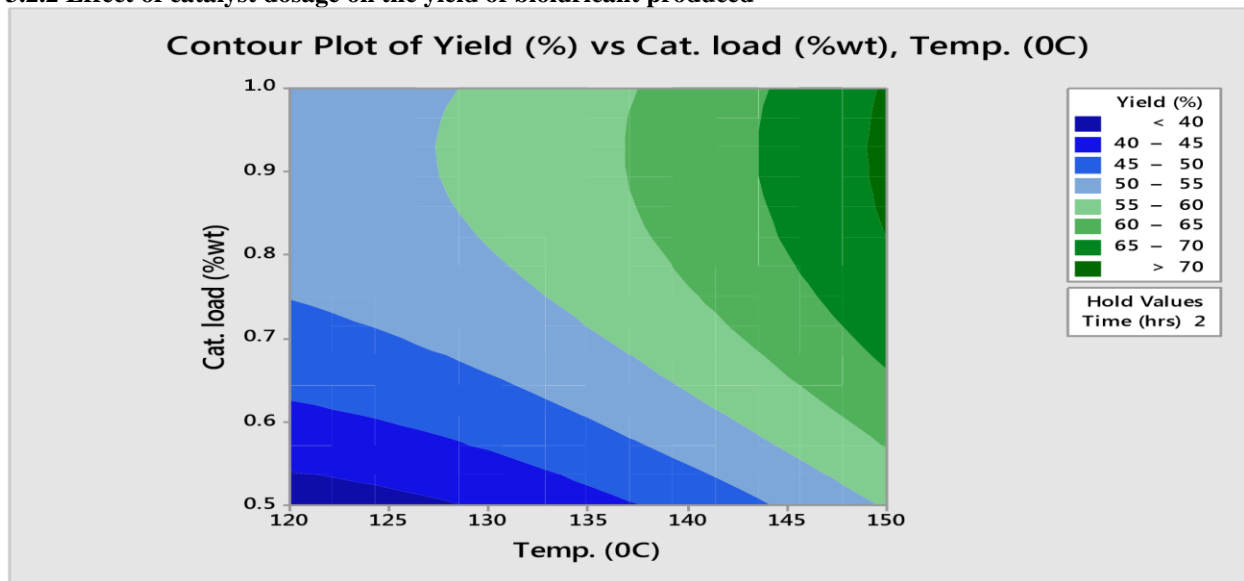


**Figure 3.2: Effects of temperature and reaction time on the yield of Biolubricant**

The contour plot presented in Figure 4.2, indicates that temperature has significant impact on the yield of biolubricant produced. A yield 79.60% was obtained at a temperature of 150 °C and reaction time of 3 hours, while the catalyst dosage was maintained at 0.5%wt and 3 hours. The yield slightly decreased to 71.6% as the temperature decreases from 150 to 130 °C However, the

lowest yield 35.0% was achieved as temperature and time were decreased to 135 °C and 1 hour respectively. Esterification reaction of methyl esters (biodiesel) with TMP to produce biolubricant is an equilibrium reaction. Therefore, reaction variables such as temperature, time and catalyst dosage can shift the equilibrium towards the formation of the products.

### 3.2.2 Effect of catalyst dosage on the yield of biolubricant produced



**Figure 3.3: Effects of catalyst dosage on the yield of biolubricant**

Figure 3.3 shows the biolubricant from esterification of methyl ester with TMP in different catalyst dosage. The effect of catalyst loading on methyl ester esterification was determined by ranging the catalyst load from 0.5-1wt%. From Figure 4.3, it was observed that the biolubricant yield increases with increased in catalyst dose with respect to temperature. The optimal yield was determined to be 0.75%wt with biolubricant yield of 79.6%. However, the minimum biolubricant yield of 35.0% was observed at catalyst dose of 1wt. The methyl ester was found to increase as the catalyst increases from 0.5 to 0.75%wt but decreases as the catalyst dosage increases to 1%wt. Therefore, the optimization catalyst dose was about 0.75%wt since no changes in methyl ester was observed above 1%wt. In this study the presence of catalyst enhanced biolubricant conversion (79.6%)

#### 4. Conclusion

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This study demonstrated the production of biolubricant via esterification of methyl esters with TMP over activated calcium oxide prepared from egg shell. Egg shell can be successfully utilized as calcium oxide source to synthesize solid acid catalyst (activated calcium oxide) through calcination and activation with phosphoric acid solution. The produced catalyst was successfully and effectively utilized to catalyze the esterification reaction with TMP and produced higher yield of biolubricant (79.6%). The optimum yield of yield of 79.6% was achieved at 150 °C, 3 hours reaction time and 0.75wt% catalyst dose.

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