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Optimization of Biolubricant from *Terminalia catappa* (Almond) Seed Oil Using CaO Derived from Limestone as Catalyst

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ABSTRACT

This research aimed at developing a process optimization of biolubricant production that will be environmentally eco-friendly. Greater concern has been focused on the use of solid-base catalysts, such as calcium oxide from limestone, for the transesterification of vegetable oils. Limestone was pretreated and calcined to 900 °C for 5 hours to obtain calcium oxide (CaO). The tropical almond seed oil was extracted from the tropical almond seeds using the soxhlet extraction method with a yield of 53.91%. The biolubricant was produced via double transesterification of tropical almond seed oil with methanol and trimethylolpropane (TMP) under reflux at different reaction conditions. Optimization of process parameters for the transesterification of tropical almond seed oil was determined using a response surface based on Box-Beinkhen design. The optimum yield of the biolubricant, 81.9%, was achieved at a temperature of 150°C, catalyst dose of 0.75 % wt and reaction time of 3 hours, respectively.

1.0 INTRODUCTION

Environmental problem, health issues and reduction of petroleum reserves are of major concern to both the scientific and industrial communities. The manufacture of biologically based products that are environmentally friendly and biodegradable using natural materials as substrates increasingly becoming an interesting field of study (Thanaphorn *et al.*, 2018). The demand for many ecologically friendly and sustainable alternatives to current practices continues to attract maximum focus and attention in light of growing global climate change issues and the depletion of oil sources (Sukirno *et al.*, 2019). For instance, studies have revealed that 20% of the 5.2 million metric tons of lubricant used each year in Europe are discharged into the ecosystem, and 1 kg of said mineral oil has the potency to contaminate huge and significant volumes of water (Godfred *et al.*, 2021). Therefore, pollution instigated by lubricants cannot be touted as unimportant. Additionally, lubricants sourced from petroleum can degrade the soil directly and pollute the air (Kim *et al.*, 2020). Health-wise, there have been numerous studies recording the harmful effects of lubricants. In circumstances where there

is long-term breathing or skin exposure to mineral oil-based lubricants, there is a probability of causing a high degree of inflammatory effects on the respiratory system and locality of the contact, aside from being carcinogenic (Appiah *et al.*, 2021).

Lubricants are compounds that are used to decrease the friction between contact surfaces, and as a result, they reduce the amount of heat generated by these surfaces. Furthermore, according to Rajendra *et al.* (2022), it could transmit forces, shift foreign particles, and heat or cool surfaces. The aim of lubrication is to reduced wear of one or two surfaces in close vicinity, and moving relative to each other, by interposing a substance between the surfaces to carry other load between the opposing surfaces (Panchal *et al.*, 2017).

In this way, biolubricants derived from vegetable oils as well as biodiesel, which is more consolidated, are gaining significance for many applications because they are biodegradable, have low ecotoxicity, and do not contribute volatile organic compounds emissions.

Mustapha *et al.* (2023) conducted research on the production of a trimethylolpropane biolubricant based on sweet almond oil and the assessment of its temperature-dependent viscosity qualities. After extraction, refinement, and acid-alkaline transesterification, the oil was transformed into biodiesel by the transesterification method. After that, biolubricant was produced by further transesterifying trimethylolpropane with biodiesel at 105 °C in a 3.9:1 ratio for 60 minutes using a 1 wt% potassium hydroxide catalyst concentration. The biolubricant's pour point and index of viscosity were found to be -4 °C and 267.50, respectively, according to the American Standard Test Methods (ASTM). At 30, 40, 60, 80, and 100 °C, the measured viscosities were 42.80, 30.18, 21.39, 12.25, and 8.90 cSt, showing an inverse relationship between temperature and lubricant viscosity. The ester group is confirmed by the difference between the FTIR spectra of the biodiesel and the biolubricant at 1755.74 versus 1743.96 cm⁻¹. Sweet almond oil has 53.478% more unsaturated fatty acids than saturated fatty acids and 71.725% more unsaturated fatty acids for biolubricant than unsaturated glycerides (9.52 g of iodine in 100 g of oil sample). Palmitic, linoleic, and oleic acids were present in the biolubricant and oil from sweet almonds. The biolubricant's characterization results correlate highly with the ISO VG-32 standards, suggesting that it has the potential to serve as light gear oil for automobiles.

Owuna *et al.*, (2019) reported the application of palm oil and jatropha oil for the production of environment friendly biolubricants through two steps of transesterification processes. Methanol and potassium hydroxide were used to produce biodiesel in the first step; the second step was the synthesis of biolubricants using TMP and biodiesel at the same conditions of reaction time of 4 hours at a temperature of 130°C, to yield 97.80% and 98.20% of palm oil based trimethylolpropane ester and jatropha oil based trimethylolpropane ester respectively. FTIR and ASTM analyses were used for confirmation and analysis of the products.

Furthermore, using soybean oil as the feedstock, Parente *et al.* (2021) performed an esterification and transesterification biolubricant procedure. At reaction conditions of 6:1 methanol to oil molar

ratio, 1 g of sodium methyolate catalyst, 60 °C, and a reaction period of 1 hour, the esterification reaction resulted in free fatty acid methyl esters. The second-stage transesterification procedure took place under optimum free fatty acid methyl esters conditions, which included a molar ratio of 1:3 for 2-ethylhexanol, 10% weight for para-toluene sulfonic acid (PTSA), and 80°C for 4 hours. It has lubrication properties that are comparable to those of commercial hydraulic and transmission oils with a Viscosity Index of 78 and a Pour Point of 12.

Ferreira *et al.* (2019) studied the synthesis of TMP ester from pequi oil using methyl esters as the starting material. The free fatty methyl esters were synthesized using a homogeneous basic catalyst, KOH, which reduced the problem in the separation between the biodiesel glycerine phase, and also the biodiesel showed high thermal stability. The sodium methoxide heterogeneous catalyst was used to produce the pequi oil-based triester under a vacuum of 400 to 450 mmHg for 6 hours. The resultant TMP ester had a total acid value that was approximately eight times reduced and a viscosity that was significantly lower, similar to mineral-based lubricants.

Encinar *et al.* (2020) carried out research on methyl esters of rapeseed and castor oils, which were then transesterified with a combination of a number of alcohols and titanium isopropoxide as a catalyst. Temperature, the molar ratio of alcohol to methyl ester, the catalyst concentration, and other reaction parameters were modified. The yield for rape seed and castor oils in 2 hours was roughly 93%, according to the results. It was found that 165 °C and a catalyst concentration of 2% were the optimal conditions for the production of biolubricant.

2.0 MATERIALS AND METHOD

2.1 Sample Collection and Treatment

A matured tropical almond fruits was collected at Chemistry Department, Usmanu Danfodiyo University, Sokoto State, Nigeria. The samples were taken to Herbarium unit of Biological Science Department of Usmanu Danfodiyo University, Sokoto for identification and authentication. The sample was identified as *Terminalia catappa* of the *Combretaceae* family with voucher number

UDUH/ANS/0118. The tropical almond outer fleshes were manually removed by eating. The hard-shell nut was sun dried and the seeds were removed from the dried shell by manual breaking

with a hammer. The oil-bearing seeds were cleaned, sun dried and further grinded into powdered form using cleaned mortar and pestle to ease extraction of the oil (Muhammad *et al.*, 2018).



a. Tropical Almond fruits



b. Almond fruit shell breaking



c. Tropical almond seeds

Plate 1: Tropical almond seed Treatment.

2.2 Extraction of Oil

The oil from tropical almond seeds was extracted using soxhlet extractor with n-hexane. The pulverized seeds sample (50 g) was placed in thimble wrought with a piece of cotton wool and placed into a Soxhlet extractor. A reflux condenser was fitted onto Soxhlet extraction column and the set up was placed on heating mantle, at temperature

of 60°C to vaporize the solvent. The extraction lasted for 6 hours after which the flask was allowed to cool and the thimble was removed. The set up was reassembled to recover the solvent. The flask containing the extracted oil was dried in air oven at 65°C for 30 minute to expel the remnant solvent (Muhammad *et al.*, 2021). The flask was then weighed and percentage oil yield calculated using equation 1.1.

$$\% \text{ oil yield} = \frac{\text{Weight of Extracted Oil}}{\text{Weight of Sample}} \times 100 \dots\dots\dots (1.1)$$



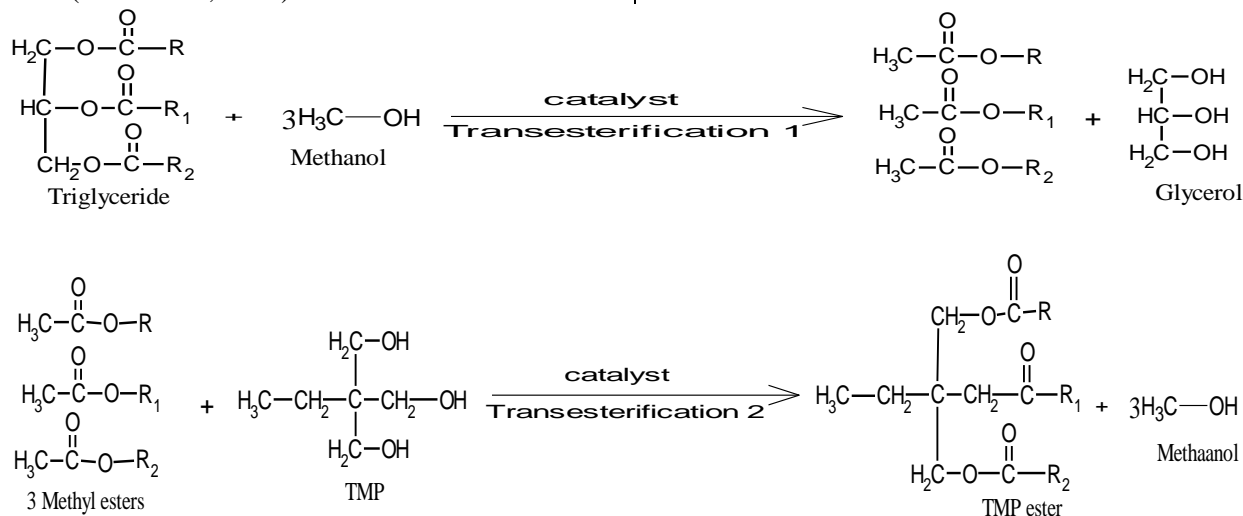
Plate 2: Pulverized seeds



Plate 3: Soxhlet extraction set-up

2.3 Limestone Collection and Catalyst Preparation

The limestone was collected at Kalambaina Area Sokoto. The catalyst was washed with deionized water to remove dust and impurities, and then dried overnight in an oven at 105°C. The acquired limestone was then crushed to powdered using a mortar and pestle and calcined at 900°C for 5 hours in a furnace to produce calcium oxide (CaO) and then was sieved through a mesh size of (35µm) and placed in airtight container before characterization and further studies, to avoid contact with CO₂ and H₂O (Sisca *et al.*, 2020).



2.4 Biolubricant Synthesis by Double Transesterification of Tropical Almond Seed Oil

This involves two steps as shown in scheme 3.1. The first was the production of methyl esters of fatty acid (Biodiesel) and the second was the conversion of the methyl esters to biolubricants by reacting with Trimethylolpropane (TMP).

2.2.1 Methyl Ester (Biodiesel) Synthesis

Tropical almond oil (40.0g) was transesterified with methanol using calcium oxide as catalyst. The weight ratio of oil to methanol was 3:1; the amount of catalyst was 0.5g of the oil. The reaction was conducted at a temperature of 60°C for 1 hour to produce biodiesel and glycerol. The contents (containing biodiesel at the top, glycerol at the middle and catalyst at the bottom) The biodiesel was allowed to be separated by gravity from the glycerol using separating funnel after leaving for about 20 hours (Muhammad *et al.*, 2019).

2.2.1 Polyolester (Biolubricant) Synthesis

The biolubricant synthesis was performed by adopting the method of Alang *et al.* (2018) and

Mustapha *et al.* (2023). In a typical run, 20 cm³ of previously produced methyl esters was placed into 250cm³ three neck round bottom flask and heated in a water bath to 70°C for 10 minutes. 0.9 g of calcium oxide catalyst was measured out and dissolved in 5 cm³ of methanol. The catalyst mixture was heated for 10 mins before being added to the reaction vessels. Then 1 g of TMP crystals was added to the reaction vessels and the reaction was allowed to proceed under reflux at 100°C for 4 hours. The reaction mixture was then allowed to cool to room temperature. The mixture was further transferred into a separatory funnel and the TMP triester (biolubricant) was collected as the bottom viscous layer after leaving it to stand for 20 hours. The same procedure was repeated at different reaction condition. The experiment setup is shown in plate 4 below.

The biolubricant yield was calculated using equation 1.2.

$$\% \text{ yield} = \frac{V_p}{V_s} \times 100 \dots\dots\dots (1.2)$$

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



Plate 1: Experimental Set Up For Biolubricant Synthesis



Plate 2: Biolubricant

2.5 Design of Experiment for Biolubricant Production

Response surface (Box-Bechnken) statistical experimental design was used to design the optimization. Three independent variables namely reaction time (1-3 hours) and temperature(120-150°C) and catalyst (0.5-1g) was selected for optimization of the yield as response. The biolubricant yied was determined. The experiment was designed using MINITAB 17 statistical software.

Table 1: Process Variables and their levels used in the Box-Bechnken Design

Independent Variables	Unit	Level of Variables	
		Low(-)	High(+)
Temperature	(°C)	120	150
Time	Hour(s)	1.00	3.00
Catalyst	Wt(g)	0.50	1.00

2.6 Data Analysis and Presentation

The Biolubricant yields obtained from the experiments conducted was analyzed on MINITAB 17 statistical software base on Box Beikhen design to estimate the main interaction effects on the reaction variables (i.e. temperature, time and catalyst), on the biolubricant products yield. The percentage products yield was fitted with a full quadratic polynomial model using regression analysis. The fitness of the model was evaluated by the coefficient of determination (R^2) and the effects of terms evaluated using analysis of variance (ANOVA) at 95% confidence level. Also, contour plots were developed using the fitted quadratic polynomial equation. Optimizer on the MINITAB

17 was used to optimize the factors and the optimal level obtained was experimentally validated (Muhammad *et al.*, 2018).

3.0 RESULTS AND DISCUSSION

3.1 Effect of the process variables on the yield of transesterification reaction

The experiments performed on biolubricant production from biodiesel transesterification with trimethylolpropane (TMP) presented in Table 1, gives the experimental conditions and percentage of biolubricant yield from the ruined experiments. The percentage biolubricant yield obtained ranges from 36.0% to 81.9% with the highest yield of 81.9% at 150°C, 3 hours' reaction time and 0.75 wt% catalyst dos.

Table 2: 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	37.9
2	5	2	1	150	1	0.75	55.8
3	14	2	1	120	3	0.75	64.3
4	3	2	1	150	3	0.75	81.7
5	10	2	1	120	2	0.50	39.4
6	2	2	1	150	2	0.50	58.4
7	8	2	1	120	2	1.00	50.0
8	13	2	1	150	2	1.00	68.1
9	12	2	1	135	1	0.50	36.0
10	4	2	1	135	3	0.50	47.5
11	7	2	1	135	1	1.00	50.2
12	1	2	1	135	3	1.00	72.1
13	15	0	1	135	2	0.75	56.3
14	6	0	1	135	2	0.75	56.2
15	9	0	2	135	2	0.75	56.1
16	29	2	2	120	1	0.75	38.0
17	16	2	2	150	1	0.75	55.6
18	19	2	2	120	3	0.75	64.5
19	26	2	2	150	3	0.75	81.9
20	18	2	2	120	2	0.50	39.2
21	22	2	2	150	2	0.50	58.3
22	24	2	2	120	2	1.00	49.9
23	21	2	2	150	2	1.00	68.4
24	30	2	2	135	1	0.50	36.2
25	28	2	2	135	3	0.50	47.7
26	27	2	2	135	1	1.00	50.0
27	23	2	2	135	3	1.00	72.3
28	25	0	2	135	2	0.75	56.3
29	17	0	2	135	2	0.75	56.1
30	20	0	2	135	2	0.75	56.0

The analysis of variance (ANOVA), (Table 2) shows that, the model F-value for biolubricant yield is significant. This was confirmed by relatively higher Fischer's F-statistics' value (48.50) and lower value of probability (P value =

0.00) (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* which has p-value of (0.555). The results further deduce 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 $\alpha = 0.05$). The lack of fit (F -value = 690.63) 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 ($R^2 = 96.23\%$) suggests that, the model adequately accounts for the empirical relationship between the biolubricant yield and the process variables.

Table 3: Analysis of Variance (ANOVA) for Biolubricant yield

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	10	4408.91	440.89	48.50	0.000
Blocks	1	0.01	0.01	0.00	0.981
Linear	3	4044.20	1348.07	148.29	0.000
Temp (°C)	1	1314.06	1314.06	144.55	0.000
Time (hrs)	1	1855.46	1855.46	204.10	0.000
Catalyst (% wt)	1	874.68	874.68	96.22	0.000
Square	3	308.18	102.73	11.30	0.000
Temp(°C)* Temp(°C)	1	72.31	72.31	7.95	0.011
Time(hrs)* Time(hrs))	1	3.28	3.28	0.36	0.555
Catalyst(% wt)* Catalyst (% wt)	1	210.05	210.05	23.11	0.000
2 way Interaction	3	56.52	18.84	2.07	0.138
Temp(°C)*Time(hrs)	1	0.06	0.06	0.01	0.935
Temp(°C)*Cat.(% wt)	1	0.28	0.28	0.03	0.862
Time(hrs)*Cat.(% wt)	1	56.18	56.18	6.18	0.022
Error	19	172.72	9.09		
Lack-of-Fit	15	172.66	11.51	690.63	0.000
Pure Error	4	0.07	0.02		
Total	29	4581.63			

Regression analyses of the results (Table 3) shows that all the linear interactions of the process variables are statistically significant, it also confirmed that all square terms interaction of the process variables with the exception of time* are statistically significant ($p < 0.05$ at $\alpha = 0.05$). Furthermore, regression analyses also indicate 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 six significant terms is better than the previous model (adjusted $R^2 = 95.15\%$ compared to 94.25%) with six terms.

Table 4: 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		56.17	1.23	45.63	0.000		S
Blocks 1		-0.013	0.550	-0.02	0.981	1.00	NS
Temp(°C)	18.125	9.063	0.754	12.02	0.000	1.00	S
Time (hrs)	21.538	10.769	0.754	14.29	0.000	1.00	S
Cat.(% wt)	14.787	7.394	0.754	9.81	0.000	1.00	S
Temp.*Temp. (°C)	6.26	3.13	1.11	2.82	0.011	1.01	S
Time(hrs)*Time(hrs)	1.33	0.67	1.11	0.60	0.555	1.01	NS
Cat.*Cat.(% wt)	-10.67	-5.33	1.11	-4.81	0.000	1.01	S
Temp.(°C)*Time(hrs)	-0.17	-0.09	1.07	-0.08	0.935	1.00	NS
Temp.(°C)*Cat.(% wt)	-0.38	-0.19	1.07	-0.18	0.862	1.01	NS
Time (hrs)*Cat.(% wt)	5.30	2.65	1.07	2.49	0.022	1.01	S

Regression equation with insignificant terms

$$\text{Yield (\%)} = 148.3 - 3.10 \text{ Temp. (}^\circ\text{C)} + 0.9 \text{ Time (hrs)} + 143.1 \text{ Cat. load (\%wt)} \\ + 0.01391 \text{ Temp. (}^\circ\text{C)} * \text{Temp. (}^\circ\text{C)} + 0.67 \text{ Time (hrs)} * \text{Time (hrs)}$$

$$\begin{aligned}
 & - 85.3 \text{ Cat. load (\%wt)} * \text{Cat. load (\%wt)} - 0.0058 \text{ Temp. (}^{\circ}\text{C)} * \text{Time (hrs)} \\
 & - 0.050 \text{ Temp. (}^{\circ}\text{C)} * \text{Cat. load (\%wt)} \\
 & + 10.60 \text{ Time (hrs)} * \text{Cat. load (\%wt)} \dots\dots\dots 1.3
 \end{aligned}$$

Regression equation with six terms after elimination of insignificant terms

$$\begin{aligned}
 \text{Yield (\%)} = & 148.0 - 3.09 \text{ Temp. (}^{\circ}\text{C)} + 2.82 \text{ Time (hrs)} + 137.6 \text{ Cat. load (\%wt)} \\
 & + 0.01368 \text{ Temp. (}^{\circ}\text{C)} * \text{Temp. (}^{\circ}\text{C)} - 86.2 \text{ Cat. load (\%wt)} * \text{Cat. load (\%wt)} \\
 & + 10.60 \text{ Time (hrs)} * \text{Cat. load (\%wt)} \dots\dots\dots 1.4
 \end{aligned}$$

3.2 Effects of Temperature and reaction Time on the yield of Biolubricant

Transesterification 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. The contour plot presented in Figure 1, indicates that temperature has significant impact on

the yield of biolubricant produced. A yield 81.9% was obtained at a temperature of 150 °C and reaction time of 3 hours, while the catalyst dosage was maintained at 0.75 %wt. The yield slightly decreased to 72.3% as the temperature decreases from 150 to 130 °C. However, the lowest yield 36.0% was achieved as temperature and time were decreased to 135 °C and 1 hour respectively.

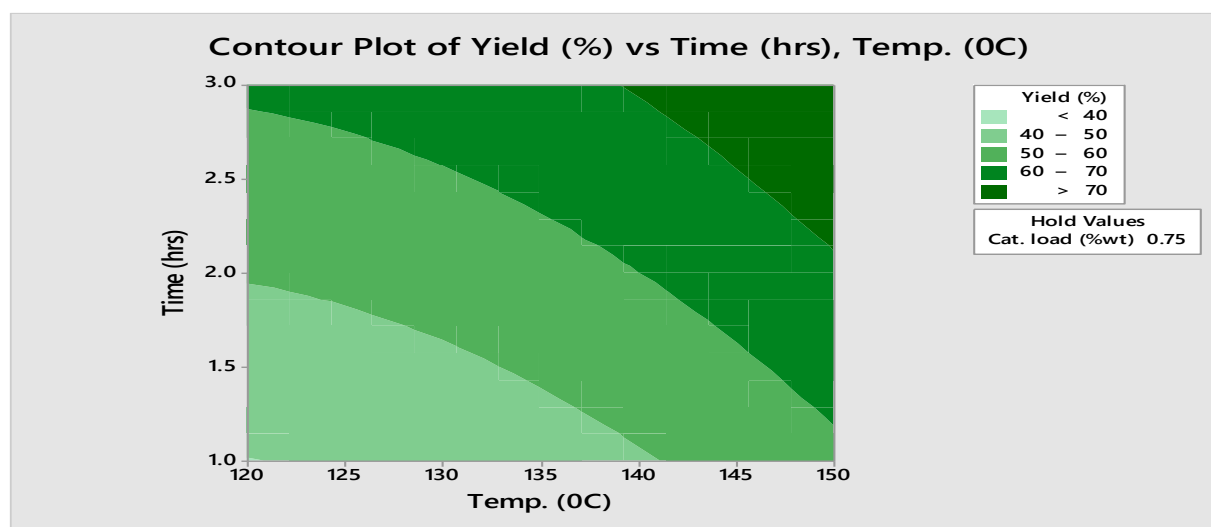


Figure 1: Contour Plot Effects of Temperature and Reaction Time on the Yield of Biolubricant

3.3 Effect of Catalyst Dosage and Reaction Time On the Yield of Biolubricant Produced

Figure 2, shows the biolubricant from transesterification 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 2, it was observed that the biolubricant yield increases with increased in catalyst dose with respect to time. The optimal yield was determined to be 0.75%wt with biolubricant yield of 81.9%. However, the

minimum biolubricant yield of 36.0% was observed at catalyst dose of 1%wt. 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 up to 81.9%.

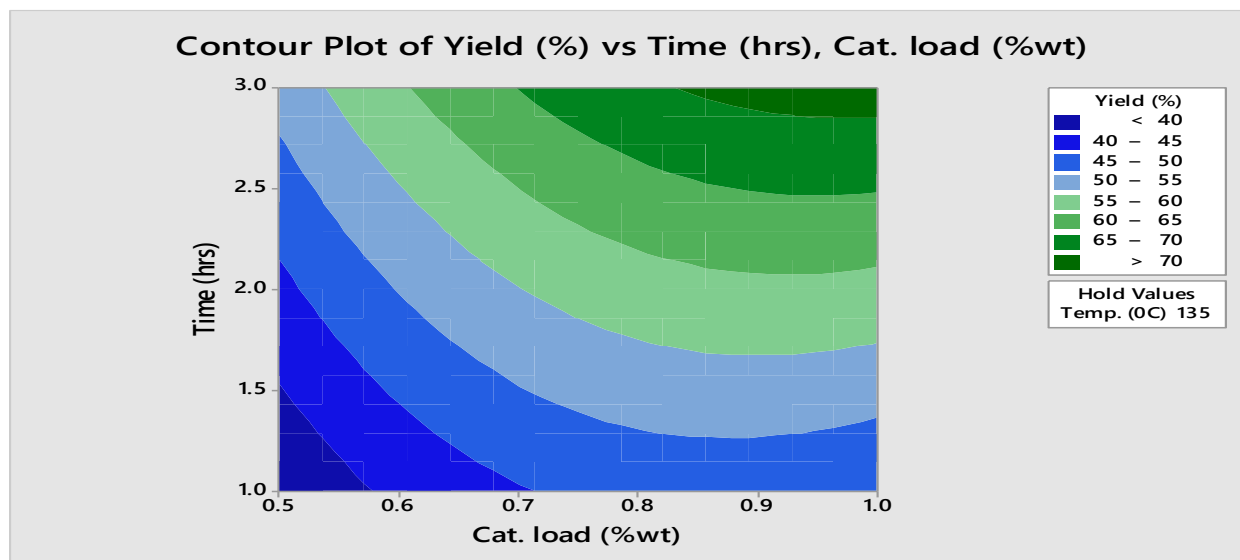


Figure 2: Contour Plot Effects of Catalyst Dosage and Reaction Time on the Yield of Biolubricant

3.4 Response optimization and validation

From Table 5 demonstrate the predicted results for optimal solutions obtained from the optimization. The solutions predicted maximum yield of 84.17 and 80.63% and desirability of one 1 and 0.99 for the two solutions with optimal process conditions of temperature 150°C and 147°C, time 3hours, catalyst dose (0.98 and 1wt%) for the two solutions respectively.

Biolubricant yield of 85.47 and 80.63% was obtained from validation of experiment carried out at the levels of the process variables predicted in solutions 1 and 2. It was observed that the optimized predicted and experimental validated results for the two solutions were 84.17%, 81.89% and 85.47%, 80.63% respectively. Thus, with predicted yields of 84.17% and 81.89% which is similarly closer to the experimental yields of 85.47% and 80.63%, it can be concluded that the optimization model is reliable and may therefore be more attractive for larger scale biolubricant production from tropical almond seed.

Table 5: Predicted Result of Optimization and Validation of Biolubricant produced

Solutions	Temperature (°C)	Cat. Loading (%wt)	Reaction Time(hr)	Predicted Yield(%)	Experimental validated Yield (%)	Desirability
1.	150.00	0.98	3.00	84.17	85.47	1.00
2.	147.00	1.00	3.00	81.89	80.63	0.99

4.0 CONCLUSION

This study showed that an eco-friendly biolubricant could be produced from *Terminalia catappa* (almond) seed oil by transesterifying methyl esters with TMP over calcium oxide (CaO) made from limestone. The process optimization of the process variables revealed that process parameters, i.e., catalyst dose, reaction temperature, and time, have a significant effect on the yield of biolubricant produced. The optimum yield of 81.9% was achieved at a temperature of 150 °C, a reaction

time of 3 hours, and a catalyst dosage of 0.75 % wt. The analysis of variance (ANOVA) and regression analysis of the process variable further proved that the interaction of the variables is statistically significant. Experimental validation gives an optimum yield of 85.47%. From the findings of this research, it can be concluded that a response surface based on the Box-Behnken experimental design can be successfully employed in the transesterification of tropical almond seed oil with methanol and TMP to produce biolubricant.

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