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Optimization of Trans-esterification Processes from Three Indigenous Feedstock using Calcium Oxide-Based Catalyst

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Abstract

Using calcium oxide-based catalyst, optimization of two-stages trans-esterification processes at a fixed catalyst (1.0 wt. %) and molar ratio (1:6) was achieved. Processes were tested on Jatropha, Sweet Almond and Sesame seed oils to produce and compare biodiesel yields. An optimization solution of speed (629.630 rpm), temperature (26.661 ^{o}C), and time (60 min) resulted in an 83.304% for refined jatropha biodiesel (RJB) production, with an overall attractiveness of 0.853. The expected optimal yield rates of 86.915–90% obtained from various models were higher than the ASTM D 6751 and EN 14214 standards, which both required an experimental range of 46–55 % in conventional biodiesel production. The effects of speed and temperature on biodiesel yield from the refined jatropha oil (RJO), refined sweet almond oil (RSAO), and refined sesame oil (RSO) were major parameters that greatly influenced the yield, although time only changed the yield moderately.

Introduction:

Biodiesel is a monoalkyl ester of fatty acids produced by esterification or trans-esterification of fatty acids with alcohol in the presence of a catalyst from vegetable oil or animal fat. It is a reversible process in which triglycerides react with methanol (most typically) or ethanol (less frequently) in the presence of a catalyst such as NaOH, KOH, or H_2SO_4 [1].

The transesterification process of edible oil is well-known for common alkaline catalysts (such as NaOH, KOH, and $NaOCH_3$). When compared to acid-catalyzed transesterification, the pace of alkaline-catalyzed transesterification is quick, and it has been reported that the rate might be as high as 4000 times faster. The free fatty acids (FFA) in waste cooking oil reacts with the alkaline catalyst (KOH, NaOH) and generates soap, the use of an alkaline catalyst in the transesterification of waste cooking oil is limited. The soap generated during the process

prevents glycerol separation, lowering the ester yield significantly. The methyl ester production is also influenced by the water in the waste cooking oil, which favors a saponification reaction [2].

Calcium oxide (CaO) has garnered a lot of interest among alkaline earth metals because of its low solubility in methanol, low toxicity, and large availability from natural resources. CaO is easier to make than many other solid catalysts, and its catalytic activity can be improved by using thermal calcination. CaO catalysts are made from a variety of precursor salts, including carbonate, hydroxide, oxalate, and acetate monohydrate. It's worth noting that even a few seconds of exposure to air will deactivate the catalyst. It can, however, be reactivated by calcination at high temperatures in a furnace. The economics of biodiesel production are improved by transesterification (the primary process for biodiesel production) using heterogeneous catalysis, using low-cost waste feedstocks for catalyst synthesis. Heterogeneous catalysts are chosen for industrial biodiesel production because of their durability and inexpensive prices, as well as their ease of separation and reusability. Calcium oxides, which are abundant in nature and can be found in seashells and eggshells, are potential candidates for heterogeneous catalyst production. However, technological improvements are required to create commercially viable calcium oxide-based catalysts. Biodiesel has been classified as a sustainable fuel because of its unique characteristics, such as being non-toxic, biodegradable, renewable, and environmentally friendly. Heterogeneous catalysts have recently gotten a lot of interest since they can avoid the difficulties that homogeneous catalysts might create, such as lengthened separation and purification steps, and so prevent the longer reaction time and wastewater creation. Figure 1 depicts the mechanism of CaO-catalyzed transesterification. To begin with, methoxide anion is formed when basic sites remove protons from methanol. Second, due to the impact of the methoxide anion on the carbonyl carbon of triglyceride, an alkoxy carbonyl intermediate form is formed. A methoxide cation draws the anion of a diglyceride, resulting in the production of a diglyceride. Fatty acid methyl ester (FAME) and an anion of diglyceride are converted into a stiffer structure. For the carbon chain of fatty acids, the pattern is repeated twice (R_2 and R_3) [3-7].

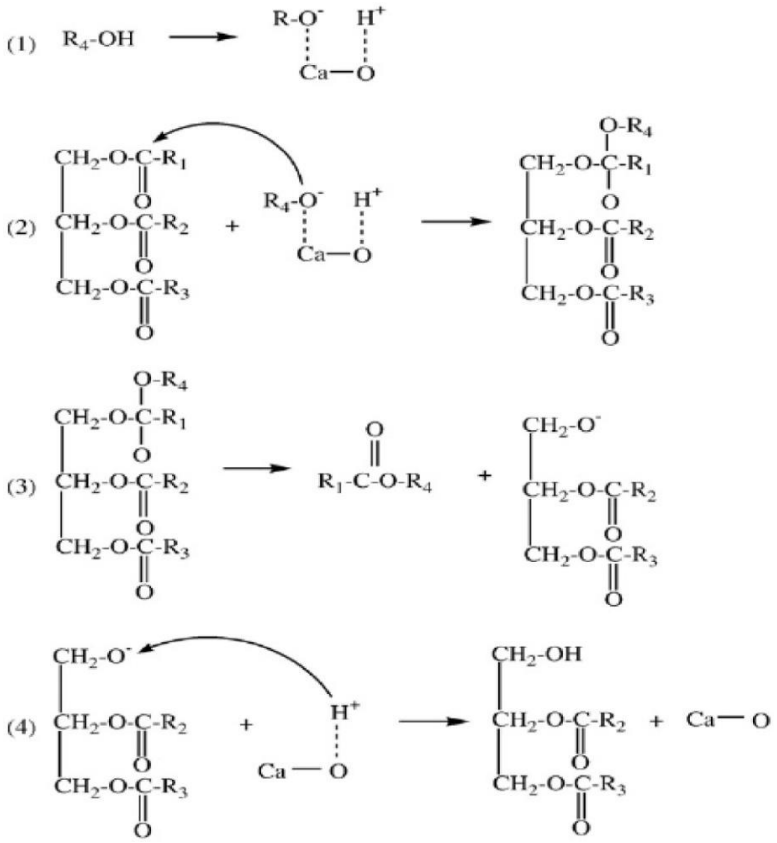

Figure 1: CaO-catalyzed transesterification mechanism [4]. R_1, R_2, R_3 are the carbon chains of fatty acids, while R_4 is the alcohol's alkyl group.

Calcium oxide has received a lot of interest among heterogeneous catalysts because of its poor solubility in methanol, low toxicity, and abundant availability from natural resources. Among the aforementioned catalysts (such as pure, supported, and mixed oxides), calcium oxide catalysts made from seashells are environmentally beneficial and have no negative impact on the ecosystem. CaO is readily derived from several types of seashells and eggshells, and it can be used as a heterogeneous catalyst in transesterification in three different forms: neat, supported, and mixed [4].

The goal of this study was to compare the optimization of biodiesels from jatropha (*Jatropha curcas*), sweet almond (*Prunusamygdalus dulcis*), and sesame (*Sesamum indicum*) seed oils using a fixed dosage of calcium oxide-based catalyst and molar ratio at varying mixing speeds, temperatures, and times.

Materials and Method

The three seeds of Jatropha (Jatropha curcas), sweet almond (Prunusamygdalus dulcis), and sesame (Sesamum indicum) were collected in Ilorin markets in Kwara State, Nigeria. Sigma Aldrich provided the chemicals and equipment (Gillingham, Dorset, UK).

Sample preparation

Cold oil extraction was used to extract refined Jatropha oil (RJO), Sweet Almond oil (RSAO), and sesame oil (CSO) from crude Jatropha oil (CJO), Sweet Almond oil (CSAO), and sesame oil (CSO) (RSO). Following refinement, the oils were transesterified to obtain refined jatropha biodiesel (RJB), Sweet Almond biodiesel (RSAB), and sesame biodiesel (RSB) using the two-step method recommended by the American Standard for Testing Materials [8, 9], the Association of Official Analytical Chemists [10, 11].

Product analysis and a two-step biodiesel synthesis process

Cold extraction of crude vegetable oils was carried out at room temperature using n-hexane. The removal of contaminants before economic transesterification and the lowering of free fatty acid (FFA) levels to below 0.5 % were the first steps in the refining and pretreatment (degumming, alkaline treatment and bleaching) of 100 mL of crude oil. The percentage of free fatty acid (FFA) present and moisture are the most important parameters in the transesterification reaction since two elements favor side reactions like saponification and lower the ester yield (biodiesel). As a result, before starting the transesterification process, the crude vegetable oils were characterized in order to make a decision on whether to proceed in one or two phases. If the presence of free fatty acids exceeds the limit, a two-step method should be used to obtain a larger yield of ester, first neutralizing the free fatty acid with an acid catalyst (esterification) and then transesterification with a base catalyst. With a mixture of oils and methanol at a pre-determined molar ratio, reaction temperature, reaction duration, reaction speed, and CaO as a dosage catalyst, trans-esterification procedures were carried out according to Mustapha *et al*. [12].

Biodiesel Optimization Using the Response Surface Method (RSM)

To establish correlations between independent and response variables, the RSM approach is utilised. Box and Wilson [13] were the first to develop a model or optimal response for experimental data, but other techniques to process optimization have broadened its applicability. The p-value for each of the models may be calculated using ANOVA. When the values were less than 0.0500, the *p*-value of 0.05 for most process variables was helpful, suggesting that model terms were significant. Design Expert II was chosen as the statistical tool because it has the three minimal categories of input and response variables, as well as anticipated and experimental values, which are necessary for the adequacy evaluation.

Experimental Design

In order to produce valid ANOVA models, the RSM must create a design of experiments (DoE) that uses the least amount of data. Because Box–Behnken Designs (BBD) does not contain axial points, all design points must lie between operational constraints, a design matrix (inputs) must be developed using a BBD. It demands a decrease in the number of treatment options available. The input components (temperature, speed, and duration) in a given catalyst molar ratio were chosen in various combinations to create yield as an output.

Design levels with independent variables

Production independent factors	
Temperature (^{o}C)	20, 40, 60
Speed (rpm)	500, 750, 1000
CaO (%)	1
Molar ratio	1:6
Time (min)	20, 40, 60

Results and Discussions

Using a fixed calcium oxide dose, test matrices for biodiesel optimization were created.

A fixed calcium oxide dose of 1.0 %, molar ratio of 1:6, and temperature of 60 ^{o}C were randomly optimised in experimental settings with varying speed (500, 750, 1000 rpm), and time (20, 40, 60 min) based on the recommended range of biodiesel production in the literatures [14]. All the parameters in Table 1 were employed in the biodiesels (RJB, RSAB, RSB) made from refined oils (RJO, RSAO, RSO).

Optimized biodiesel made from refined jatropha (RJB)

Test matrices for biodiesel optimization was established with a fixed calcium oxide dosage. The Design Expert software generated the most runs based on the three levels of input. The relationship between the actual yield values obtained experimentally (Table 1) and the yield values predicted by different models are depicted in Figure 1. The results from Tables 1 - 3 demonstrate desirability functions from three separate criteria employing various input components (speed, temperature and time) at fixed CaO, molar ratio and the combination of processes that were evaluate.

Table 1: The experimental matrix at various speeds, temperatures, and times.

		Factor 1	Factor 2	Factor 3	Response 1
Std	Run	A:Speed	B:Temp	C:Time	Yield
		rpm	0C	min	%
8	1	1000	20	62.5	77.77
7	2	500	40	65	11.11
3	3	750	40	62.5	100
13	4	1000	40	65	83.33
14	5	1000	40	60	5.55
12	6	750	40	62.5	100
17	7	750	40	62.5	100
1	8	500	20	62.5	61.11
4	9	750	40	62.5	100
6	10	750	60	65	77.77
5	11	500	40	60	77.77
16	12	750	20	60	72.22
15	13	750	20	65	38.88
2	14	1000	60	62.5	72.22
9	15	750	60	60	27.77
10	16	500	60	62.5	27.77
11	17	750	40	62.5	100

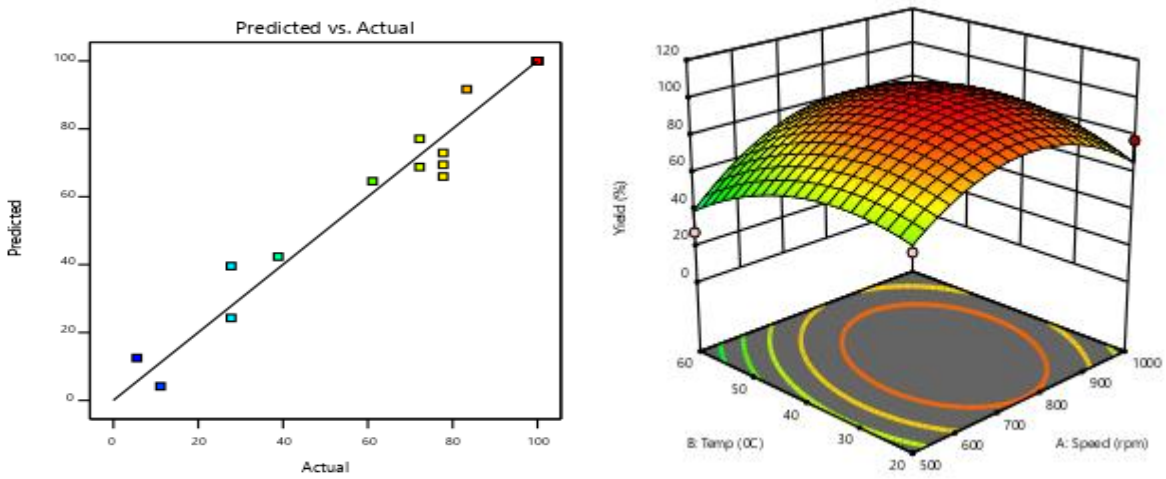

Figure 1: RJB provides scatter diagram with associated 3D surfaces

Variance Analysis (ANOVA)

The equation depicts the second polynomial functions in terms of real-world parameters used to model yield

Final Equation in Terms of Actual Factors:

$$Yield = -2828.02500 + 0.35400Speed - 2.79125Temp + 93.21000Time - 0.000900Speed * Temp + 0.002560Speed * Time + 0.115000Temp * Time - 0.000099\ Speed^2 - 0.043313Temp^2 - 0.788000Time^2 \quad (1)$$

Table 2: "N" Table for the Quadratic Model "RJB Yield"

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	16305.92	9	1811.77	20.81	0.0003	significant
A-Speed	466.80	1	466.80	5.36	0.0538	
B-Temp	246.98	1	246.98	2.84	0.1360	
C-Time	96.47	1	96.47	1.11	0.3275	
AB	193.07	1	193.07	2.22	0.1801	
AC	5215.73	1	5215.73	59.90	0.0001	
BC	1736.39	1	1736.39	19.94	0.0029	
A^2	2631.84	1	2631.84	30.23	0.0009	
B^2	983.23	1	983.23	11.29	0.0121	
C^2	3931.95	1	3931.95	45.16	0.0003	
Residual	609.50	7	87.07			
Lack of Fit	609.50	3	203.17			
Pure Error	0.0000	4	0.0000			
Cor Total	16915.42	16				

Note: df = degree of freedom

Table 4 displays the optimization methods discovered based on the biodiesel optimization scenario. The optimization solution with the speed (629.630 rpm), temperature (26.661 ^{o}C), and time (60 min) gave biodiesel (RJB) yield of 83.304 %, with the specified overall desirability of 0.853, was obtained using fixed catalyst of 1.0 wt %, and at molar ratio of 1:6.

The findings of the analysis of variance (ANOVA) revealed that, speed, temperature and time were all significant determinants in biodiesel synthesis.

Table 4: Constraints

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
A:Speed	minimize	500	1000	1	1	3
B:Temp	minimize	20	60	1	1	3
C:Time	minimize	60	65	1	1	3
Yield	maximize	5.55	100	1	1	3

Table 5: A solution was found according to the biodiesel (RJB) optimization scenario.

Number	Speed	Temp	Time	Yield	Desirability	
1	**629.630**	**26.661**	**60.000**	**86.916**	**0.853**	**Selected**
2	628.418	26.755	60.000	86.915	0.853	
3	631.519	26.514	60.000	86.915	0.853	
4	624.292	27.074	60.000	86.895	0.853	
5	636.490	26.122	60.000	86.886	0.853	

Optimized biodiesel made from refined sweet almond (RSAB)

Based on the three levels of input, the Design Expert program created the most number of runs. Figure 2 depicts the link between the actual yield values acquired experimentally (Table 5) and the yield values projected by several models (Figure2).

Table 5: The experimental matrix at various speeds, temperatures, and times

		Factor 1	Factor 2	Factor 3	Response 1
Std	Run	A:Speed rpm	B:Temp 0C	C:Time min	Yield %
8	1	1000	40	65	68.89
7	2	500	40	65	90
3	3	500	60	62.5	86.44
13	4	750	40	62.5	42.22
14	5	750	40	62.5	35.56
12	6	750	60	65	61.11
17	7	750	40	62.5	42.22
1	8	500	20	62.5	84.44
4	9	1000	60	62.5	85.56
6	10	1000	40	60	75.56
5	11	500	40	60	66.66
16	12	750	40	62.5	42.22
15	13	750	40	62.5	42.22
2	14	1000	20	62.5	82.22

9	15	750	20	60	66.67
10	16	750	60	60	57.78
11	17	750	20	65	63.42

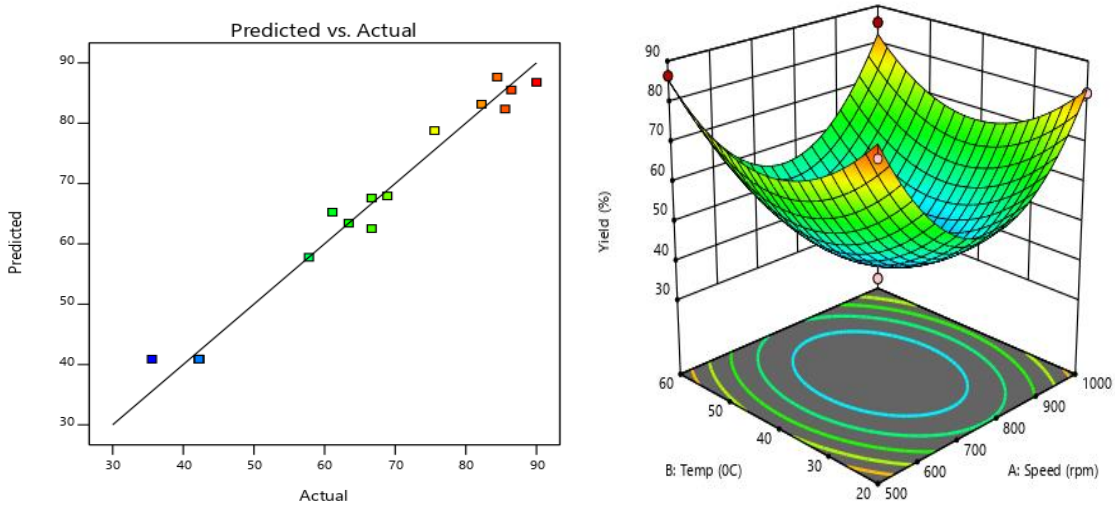

Figure 2: RSAB provides scatter diagram with associated 3D surfaces

Variance Analysis (ANOVA)

The equation depicts the second polynomial functions in terms of real-world parameters used to model yield

Final Equation in Terms of Actual Factors:

$$Yield = +3574.92350 + 0.58201Speed - 5.21757Temp - 111.17050Time + 0.00067Speed * Temp - 0.012004Speed * Time + 0.032900Temp * Time + 0.000454Speed^2 + 0.038431Temp^2 + 0.957560Time^2 \quad (2)$$

Table 6: "N" Table for the Quadratic Model "RSAB Yield"

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	5203.54	9	578.17	35.36	< 0.0001	significant
A-Speed	29.30	1	29.30	1.79	0.2225	
B-Temp	4.29	1	4.29	0.2625	0.6242	
C-Time	35.07	1	35.07	2.14	0.1865	
AB	0.4489	1	0.4489	0.0275	0.8731	
AC	225.15	1	225.15	13.77	0.0075	
BC	10.82	1	10.82	0.6620	0.4427	
A^2	3397.18	1	3397.18	207.76	< 0.0001	
B^2	994.97	1	994.97	60.85	0.0001	
C^2	150.81	1	150.81	9.22	0.0189	
Residual	114.46	7	16.35			
Lack of Fit	78.97	3	26.32	2.97	0.1603	not significant
Pure Error	35.48	4	8.87			
Cor Total	5318.00	16				

Table 7: Constraints

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
A:Speed	minimize	500	1000	1	1	3
B:Temp	minimize	20	60	1	1	3
C:Time	minimize	60	65	1	1	3
Yield	maximize	35.56	90	1	1	3
Specific Gravity	minimize	0.903	0.989	1	1	3
Density	minimize	4.118	9.116	1	1	4

Table 8: A solution was found according to the biodiesel (RSAB) optimization scenario.

Number	Speed	Temp	Time	Yield	Desirability	
1	**545.480**	**20.000**	**60.000**	**77.241**	**0.875**	**Selected**
2	534.766	20.000	60.000	79.059	0.874	
3	550.557	20.000	60.012	76.390	0.873	
4	546.552	20.142	60.000	76.828	0.873	
5	557.057	20.000	60.070	75.237	0.865	

Tables 6–8 show desirability functions for three different criteria using various input components (speed, temperature, and time) for fixed CaO, molar ratio, and the combination of processes that were evaluated. The optimization strategies identified based on the biodiesel optimization scenario is shown in Table 8. Using a constant catalyst of 1% and a molar ratio of 1:6, the optimization solution with the speed (545.480 rpm), temperature (20.0 ^{o}C), and time (60 min) gave biodiesel (RSAB) yield of 77.241 %, with the specified overall desirability of 0.875. Speed, temperature, and time were all important variables in biodiesel synthesis, according to the results of the analysis of variance (ANOVA).

Optimized biodiesel made from refined sesame (RSB)

Based on the three levels of input, the Design Expert program created the most number of runs.

Figure 3 depicts the link between the actual yield values acquired experimentally (Table 9) and the yield values projected by several models (Figure 3).

Table 9: The experimental matrix at various speeds, temperatures, and times.

		Factor 1	Factor 2	Factor 3	Response 1
Std	Run	A:Speed	B:Temp	C:Time	Yield
		rpm	^{o}C	min	%
8	1	750	20	60	65
7	2	750	40	62.5	90
3	3	750	40	62.5	90
13	4	1000	60	62.5	74
14	5	750	60	60	61
12	6	750	60	65	82
17	7	750	40	62.5	90
1	8	500	40	60	79
4	9	1000	20	62.5	70
6	10	1000	40	65	82
5	11	500	20	62.5	50
16	12	1000	40	60	76
15	13	750	40	62.5	90
2	14	500	40	65	78.6
9	15	750	20	65	63
10	16	500	60	62.5	72
11	17	750	40	62.5	90

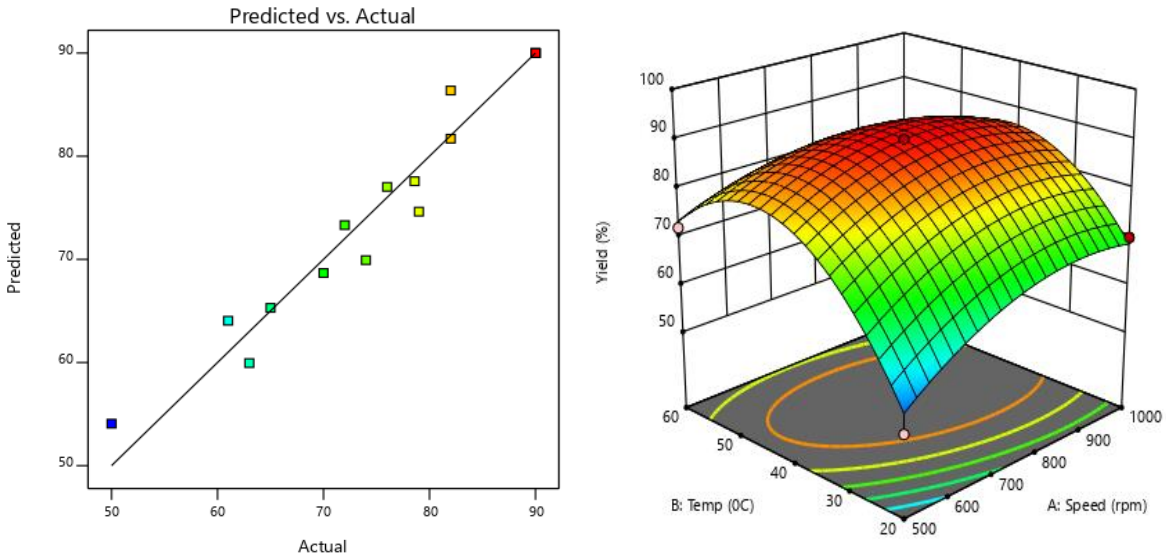

Figure 3: RSB provides scatter diagram with associated 3D surfaces

Variance Analysis (ANOVA)

The equation depicts the second polynomial functions in terms of real-world parameters used to model yield

Final Equation in Terms of Actual Factors:

$$Yield = 2828.02500 + 0.035400Speed - 2.7915Temp + 93.21000Time - 0.000900Speed * Time + 0.002560Speed * Time + 0.115000Temp * Time - 0.00099Speed^2 - 0.043313Temp^2 - 0.788000Time^2 \quad (3)$$

Table 10: "N" Table for the Quadratic Model "RSB Yield"

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	2212.26	9	245.81	17.94	0.0005	significant
A-Speed	62.72	1	62.72	4.58	0.0697	
B-Temp	210.13	1	210.13	15.34	0.0058	
C-Time	75.65	1	75.65	5.52	0.0511	
AB	81.00	1	81.00	5.91	0.0453	
AC	10.24	1	10.24	0.7475	0.4159	
BC	132.25	1	132.25	9.65	0.0171	
A^2	160.55	1	160.55	11.72	0.0111	
B^2	1263.81	1	1263.81	92.26	< 0.0001	
C^2	102.13	1	102.13	7.46	0.0293	
Residual	95.89	7	13.70			
Lack of Fit	95.89	3	31.96			
Pure Error	0.0000	4	0.0000			
Cor Total	2308.15	16				

Tables 9 - 11 show desirability functions for three different criteria using different input components (speed, temperature, and time) at a given CaO, molar ratio, and the combination of processes that were examined. Based on the biodiesel optimisation scenario, Table 11

shows the optimisation approaches developed. Using a constant catalyst of 1% and a molar ratio of 1:6, the optimisation solution with the speed (750 rpm), temperature (40.0 oC), and duration (62.50 min) yielded 90.0 % biodiesel (RSB) with the stated overall desirability of 0.895. Speed, temperature, and time were all important variables in biodiesel synthesis, according to the results of an analysis of variance (ANOVA).

Table 11: Constraints

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
A:Speed	is target = 750	500	1000	1	1	3
B:Temp	is target = 40	20	60	1	1	3
C:Time	is target = 62.5	60	65	1	1	3
Yield	maximize	50	90	1	1	3
Specific Gravity	minimize	0.854	1.0179	1	1	3
Density	minimize	3.025	5.483	1	1	4

Table 12 displays the optimization methods discovered based on the biodiesel optimization scenario. The optimization solution with the speed (750 rpm), temperature (40.0 ^{o}C), and time (62.50 min) gave biodiesel (RSB) yield of 90.0 %, with the specified overall desirability of 0.895, was obtained using fixed catalyst of 1.0 wt %, and at molar ratio of 1:6. The findings of the analysis of variance (ANOVA) revealed that, speed, temperature and time were all significant determinants in biodiesel synthesis.

Table 12: A solution was found according to the biodiesel (RSB) optimization scenario.

Number	Speed	Temp	Time	Yield	Desirability	
1	750.0	40.0	62.50	90.0	0.895	Selected

The biodiesel results obtained by the three sets of combination variable tests were used to test the accuracy of the predicted technique (Table 13). When compared to the criteria of ASTM D 6751 [8] and EN 14214 [9], which both mandated an experimental range of 46-55 % in normal biodiesel production, these predicted yield results of 86.915 – 90% from various models were higher.

Table 13: Optimization solutions for the three biodiesel optimizations (RJB, RSAB, and RSB)

Number	Speed	Temp	Time	Yield	Desirability	
RJB	629.630	26.661	60.0	86.916	0.853	Selected
RSAB	628.418	26.755	60.0	86.915	0.875	Selected
RSB	750.0	40.0	62.50	90.0	0.895	Selected

Sinha *et al.* [14] used sodium hydroxide (NaOH) as a catalyst to make rice bran biodiesel. Sodium or potassium methoxide (CH_3ONa or CH_3OK) has recently gained popularity as a catalyst, the goal is to reduce or eliminate the moisture content related to biodiesel manufacturing [15, 16]. Meanwhile, in the field of catalysis, researchers are concentrating their efforts on the utilization of heterogeneous solid catalysts (acid and base) as well as enzymes [17, 18]. Du *et al*. [19] produced palm biodiesel using a single-step and two-step trans-esterification process. They adjusted the process parameters and reported a 1.0 wt. % NaOH, a 1:6 molar ratio, a reaction temperature of 60°C, and a reaction time of 1 hour [20]. Singh *et al.* (4) made feasibility investigation of non-refined mustard oil for prospective biodiesel extraction. Zhang *et al*. [21] used leftover cooking oil to make biodiesel. They discovered that acid-catalyzed processes were superior to alkali-catalyzed processes. Olaoluwa *et al*. [22] made biodiesel from vegetable oil with a higher free fatty acid concentration; they employed an alkali-catalyzed trans-esterification procedure after pretreating the oil with H_2SO_4 due to the high free fatty acid (FFA) content. In Fourier Transform Infra-red (FT-IR) and Gas Chromatograph (GC) experiments, they discovered 97 % methyl ester conversion, and the biodiesel contained more esters than alkanes and alkenes in diesel fuel. Ramadhas *et al* [23, 24] used rubber seed oil to make biodiesel.

For trans-esterification of Karanja oil, Meher *et al* [25, 26] employed potassium hydroxide.The best parameters for maximal conversion were found to be 1.0 wt. % KOH, a molar ratio of 1:6, a reaction temperature of 65°C, and a reaction time of 2 hours. Rashid *et al* [27, 28] compared the effects of several catalysts on the trans-esterification of cotton seed oil and evaluated fatty acids effects on biodiesel. Rushang*et al* [29] reviewed production of biodiesel using various catalysts, including their emissions and general performances. They discovered that the viscosity of biodiesel increases as the temperature drops. They also devised a formula for calculating viscosity from temperature and ethyl ester volume fraction.CaO was utilized to make soybean biodiesel and CrO as a solid heterogeneous catalyst. Biodiesel generation utilizing calcium ethoxide as a catalyst was also investigated The best parameters for the synthesis of soybean biodiesel to achieve 95 percent conversion were a molar ratio of 1:12, 3.0 wt.% calcium ethoxide by, reaction temperature of 65 ^oC, and reaction time of 1.5 hours [29-32].

Supriya *et al.* [33] investigated the manufacture of jatropha methyl ester from eggshell calcium oxide and found that the conversion was good and the ester characteristics were within ASTM limits.According to the researchers, the catalyst can be reused up to six times without losing its catalytic activity. Brito *et al.* [34, 35] employed multiple Y-type zeolites to trans-esterify used vegetable oil varying the proportions of Al_2O_3. They discovered that reaction temperature and duration were critical factors in ester conversion. They discovered that putting 5% by weight of KNO_3 on flash resulted in a maximum conversion of 87.5 %. Babajide *et al* [36, 37] showed the best reaction conditions to be a 15:1 molar ratio of methanol to oil, an 8-hour reaction time, a 443K reaction temperature, and 15% catalyst loading. They discovered that reusing the catalyst reduced the catalytic activities. Taufiq-yap *et al* [38, 39] used a calcium-based mixed oxide catalyst to study the trans-esterification of jatrophacurcas oil.

Dalai *et al* [2] investigated the performance of different heterogeneous catalysts for biodiesel generation and discovered that NaOH and KOH performed better than diethylamine (DEA), dimethylethanol amine (DMAE), tetramethyl diaminoethane (TEMED), and tertramethylammonium hydroxide that are four amine-based catalysts (TMAH), though they discovered that a mixture of canola ester and methanol worked well as a lubricant additive.
The best conditions for achieving 98 percent conversion at a reaction temperature of 200 °C, a molar ratio of 18, a stirring speed of 600 rpm, and a catalyst quantity of 3.0 wt % was reported by Atadashi *et al* [40], using enzymes to catalyze the manufacturing of biofuel is extremely beneficial to the environment, but the cost is prohibitively high. Due to cost considerations, the use of lipase enzymatic catalysts is not practical and the cost of producing biodiesel on an industrial scale has long been a stumbling block [41, 42]

Conclusions

Using the surface response methodology of Box-Behnken Design, the best parameters for biodiesel were investigated in this study. It illustrated and compared the desirability package's capacity to integrate production parameters to yield three optimal biodiesel productions under different speed, temperature, and time optimization scenarios with a fixed catalyst and molar ratio. For each of these biodiesels, the optimal yield outputs were acquired and the impacts of speed and temperature on biodiesel yield from the RJO, RSABO, and RSO were key factors that influenced the yield significantly, whereas time changed just marginally.

References

[1] Aransiola,E.F., Ojumu, T.V., Oyekola,O.O., Madzimbamuto,T.F., Ikhu-Omoregbe,D.I.O. (2014). A review of current technology for biodiesel production: State of the art, *Biomass and Bioenergy,* (6)276-297

[2] Dalai, A. K., Kulkarni, M. G., &Meher, L. C. (2006). Biodiesel productions from vegetable oils using heterogeneous catalysts and their applications as lubricity additives. In 2006 IEEE EIC Climate Change Conference (pp. 1-8). IEEE. https://doi.org/10.1109/EICCCC.2006.277228

[3] Akubugwo, I.E., Chinyere., & Ugbogu., A.E. (2008). Comparative Studies on Oil from Some Common Plant Seeds in Nigeria. *Pakistan Journal. of Nutrition* 7(4): 570-573.

[4] Singh, T. S., Verma, T. N., Singh, L. D., Rajak, U., Nashine, P., Khan, A., & Asiri, A. M. (2021). Case study of non-refined mustard oil for possible biodiesel extraction: feasibility analysis. In Advanced Technology for the Conversion of Waste into Fuels and Chemicals (315-336). Woodhead Publishing

[5] Mazaheri, H., Ong, H.C., Amini, Z., Masjuki, H.H., Mofijur, M., Su, C.H., Anjum Badruddin, I., & Khan, T.M.Y. (2021). An Overview of Biodiesel Production via Calcium Oxide Based Catalysts: Current State and Perspective. Energies, (14), 3950. https://doi.org/10.3390/en14133950

[6] Mnam, Y., Nwm, Z., NL, S. (2020). Sustainability of Palm Biodiesel in Transportation: a Review on Biofuel Standard, Policy and International Collaboration between Malaysia and

Colombia. *Bioenergy* Res. 1-18.

[7] Tabatabaei, M., Aghbashlo, M., Dehhaghi, M., Panahi, H.K.S,Mollahosseini A., Hosseini M., &Soufiyan, M.M (2019). Reactor technologies for biodiesel production and processing. *A review Progress in Energy and Combustion Science,* 74 https://doi.org/10.1016/j.pecs.2019.06.001

[8] ASTM, C. (2003). Standard Test Method for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar-Bar Method)", Annual Book of ASTM Standards, Concrete and Mineral Aggregates. American Society for Testing and Materials, Philadelphia, USA, 227-03.

[9] EN14214. (2003). Automotive fuels-fatty acid methyl esters (FAME) for diesel engines requirements and test methods, Berling, Germany: Beuth-Verlag.

[10] AOAC, AOCS Official Method Cd 8-53 (2006). Peroxides in fats and oils. American Oil Chemists' Society. Champaign, Illinois, USA: American Oil Chemists' Society.

[11] AOAC. Official methods of analysis.(2012). Washington D.C., USA.: Association of official analytical chemist 19th edition," 2012.

[12] Mustapha, A.O., Adepoju, R.A., &Afolabi, Y.T. (2020). Optimization of vegetable oil-based biodiesels by multi-response surface methodology (MRS) using desirability functions,*Journal of the Chemical Society of Nigeria, JCSN*, (45)5, 917 – 924 https://doi.org/10.46602/jcsn.v45i5.517

[13] Box, G.E., &Behnken, D.W. (1960). Some new three level designs for the study of quantitative variables, *Technometrics,* (2), 455-475 https://doi.org/10.1080/00401706.1960.10489912

[14] Sinha, S., Agarwal, A.K., &Garg, S. (2008). Biodiesel development from rice bran oil: Trans-esterification process optimization and fuel characterization. *Energy Conversion and Management,* 49(5), 1248-57 https://doi.org/10.1016/j.enconman.2007.08.010

[15] Knothe, G. (2010). Biodiesel and renewable diesel: a comparison. *Program Energy Combust*, (36), 364-373.https://doi.org/10.1016/j.pecs.2009.11.004

[16] Knothe, G., &Razon, L.F. (2017). Biodiesel fuels. *Energy Combustion. Science*, (58), 36-59. https://doi.org/10.1016/j.pecs.2016.08.001

[17] Araújo, R.A.D., Neiva, J.N.M., Rogério, M.C.P., Pimentel, P.G., Furtado, R.N., Mariz, L.D.S., Cândido, M.J.D., &Pompeu, R.C.F.F. (2019). Ingestive behavior and physiological parameters of lactating goats fed diets containing detoxified castor cake. *Biol. Rhythm Res*. 1-11.

[18] Baskar, G., Selvakumari, I.A.E., &Aiswarya, R. (2018). Biodiesel production from castor oil

using heterogeneous Ni doped ZnOnanocatalyst. *Bioresour. Technol.* (250),793-798. https://doi.org/10.1016/j.biortech.2017.12.010

[19] Du, L., Ding, S., Li, Z., Lv, E., Lu, J., & Ding, J. (2018), Transesterification of castor oil to biodiesel using NaY zeolite-supported La2O3 catalysts. *Energy Convers. Manag.* (173), 728-734. https://doi.org/10.1016/j.enconman.2018.07.053

[20] Shahid, E.M., Jamal, Y. (2011.) Production of biodiesel: A technical review. Renewable and Sustainable Energy.*Reviews,* (15), 4732-4745 https://doi.org/10.1016/j.rser.2011.07.079

[21] Zhang, F., Fang, Z., & Wang, Y.T. (2015). Biodiesel production direct from high acid value oilwitha novel magnetic carbonaceous acid. *Application. Energy* (155), 637-647.https://doi.org/10.1016/j.apenergy.2015.06.044

[22] Olaoluwa, R.O., Abolanle, S. A., John, A. O. O., Efere, M. O., Olatunji, S. O., Adedayo, M. S., MuibA, A &Oyedare M. A (2017). Refining, Toxicology Study and Biodiesel Potentials ofUsed Vegetable Oils.*American Journal of Food Science and Technology*, (5), 3, 78-88.

[23] Ramadhas, A.S., Jayraj, S., Muraleedharan, C. (2005). Biodiesel production from high FFA rubber seed oil. *Fuel,* (84), 335-340 https://doi.org/10.1016/j.fuel.2004.09.016

[24] Ramadhas, G. R., Ghadge, S.V., &Raheman, H. (2005). Biodiesel production from mahua (Madhucaindica) oil having high free fatty acids. *Biomass and Bio-energy,* (28), 601-605 https://doi.org/10.1016/j.biombioe.2004.11.009

[25] Meher, L.C., Vidya, Sagar, D., &Naik, S.N. (2006). Technical aspects of biofuel production by trans-esterification-a review. *Renewable and Sustainable Energy Reviews*, (10), 248-268 https://doi.org/10.1016/j.rser.2004.09.002

[26] Meher, Z.Y., Dube, M.A., McLean, D.D., &Kates, M. (2003). Biodiesel production from waste cooking oil: 1. Process design and Technological assessment. *Bio-resource Technology*, (89), 1-16 https://doi.org/10.1016/S0960-8524(03)00040-3

[27] Rashid, U., Anwa,r F., &Knothe, G. (2009). Evaluation of biodiesel from cotton seed oil. *Fuel Processing Technology,* (90), 1157-63 https://doi.org/10.1016/j.fuproc.2009.05.016

[28] Rashid, U., Rodriquez, L., & Perez, A. (2008). Influence of fatty acid composition of raw materials on biodiesel properties. *Bioresource Technology,* (100), 8175-8179.https://doi.org/10.1016/j.biortech.2008.03.066

[29] Rushang, Basha SA, Gopal KR, Jebraj S. (2009)a review on biodiesel production, combustion, emissions and performance. *Renewable and Sustainable Energy Reviews,* (13): 1628-1634 https://doi.org/10.1016/j.rser.2008.09.031

[30] Liu, H., Su, L., Shao, Y., &Zou, L. (2012). Biodiesel production catalyzed by cinder supported CaO/KF particle catalyst. *Fuel Review,* 97, 651-657. https://doi.org/10.1016/j.fuel.2012.02.002

[31] Liu, X., Huayang, H., Wang, Y., & Zhu, S. (2007), Trans-esterification of soybean oil to biodiesel using SrO as a solid base catalyst. *CatalCommun,* (8), 1107-11https://doi.org/10.1016/j.catcom.2006.10.026

[32] Liu, Y., Koh, C.M.J., &Ji, L. (2011).Bioconversion of crude glycerol to glycolipids in Ustilagomaydis. *Bioresour. Technology,* 102, 3927-3933. https://doi.org/10.1016/j.biortech.2010.11.115

[33] Supriya, C., Meena, Y., Reena, S., &Yogesh, C. S.(2017). Production of biodiesel from three indigenous feedstock: Optimization of process parameters and assessment of various fuel properties.*Environmental Progress & Sustainable Energy* 36(3) DOI: 10.1002/ep.12606https://doi.org/10.1002/ep.12606

[34] Brito, A., Borges, M., & Otero, N.Y. (2007) Zeolite as a heterogeneous catalyst in biodiesel fuel production from used vegetable oil. Energy Fuel, 21(6), 3280-3https://doi.org/10.1021/ef700455r

[35] Brito, F.B., Butterfield, R., &Pryde, E. (1986). Trans-esterification kinetics of soybean oil. *J American Chemical Society*, (63), 1375-80 https://doi.org/10.1007/BF02679606

[36] Babajide, O., Petrik, L., Musayoka, N., Amigun, B., &Ameer, F. (2010). Use of coal fly ash as a catalyst in the production of biodiesel. *Petrol coal,* 52(4), 261-272

[37] Babajide, V.T., Melissa, A., & Robert, O. (2005). Fuel production and nitrogen oxide emission levels of biodiesel produced from animal fats. *Journal of American Oil Chemists Society,* (82), 585-591 https://doi.org/10.1007/s11746-005-1113-2

[38] Taufiq-Yap, Y. H., Lee, H. V., & Lau, P. L. (2012). Transesterification of jatrophacurcas oil to biodiesel by using short necked clam (orbicullariaorbiculata) shell derived catalyst. *Energy Exploration & Exploitation,* 30(5), 853-866 https://doi.org/10.1260/0144-5987.30.5.853

[39] Tabatabaei, M., Aghbashlo, M., Dehhaghi, M., Panahi, H.K.S,Mollahosseini A., Hosseini M., &Soufiyan, M.M (2019). Reactor technologies for biodiesel production and processing. *A review Progress in Energy and Combustion Science,* 74 https://doi.org/10.1016/j.pecs.2019.06.001

[40] Atadashi, I.M., Aroua, M.K., Aziz, A.R.A., &Sulaiman, N.M.N. (2012). Production of biodiesel using high free fatty acid feedstocks. *Renewable and Sustainable Energy Reviews,*(16), 3275-3285 https://doi.org/10.1016/j.rser.2012.02.063

[41] Mnam, Y., Nwm, Z., NL, S. (2020). Sustainability of Palm Biodiesel in Transportation: a Review on Biofuel Standard, Policy and International Collaboration between Malaysia and Colombia. *Bioenergy* Res. 1-18.

[42] Bencheikh, K., Atabani,A.E., Shobana, S., Mohammed, M.N., Uğuz, G., Arpa, O., &Bokhari, A. (2019). Fuel properties, characterizations and engine and emission performance analyses of ternary waste cooking oil biodiesel-diesel-propanol blends, *Sustainable Energy Technologies and Assessments,* (35)321-334 https://doi.org/10.1016/j.seta.2019.08.007