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OPTIMIZATION OF BIODIESEL PRODUCTION FROM MANGO KERNEL FAT USING CALCIUM OXIDE AS CATALYST

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ABSTRACT

The increase in the demand for energy and awareness on environmental concern necessitate consideration for renewable energy. The potential of mango kernel fat as an alternative source of biodiesel was investigated and the production process has been optimized in this study. Solvent extraction was employed to extract the fat from the mango kernel using n-hexane as solvent in a soxhlet extractor. Calcium oxide was used as the heterogeneous catalyst. Response surface methodology was employed to study the effect of methanol-to-oil molar ratio, reaction time and catalyst concentration on the yield of biodiesel. A statistically significant quadratic polynomial regression model was found to best fit the data with no significant lack of fit. The analysis of variance (ANOVA) further validated the strong correlation between the experimental and predicted data with a coefficient of determination (R^2) value of 0.985. Optimum biodiesel yield was found to be 87.37% at methanol-to-oil molar ratio of 8.9:1, catalyst concentration of 1% and reaction time of 89.3 minutes. In addition, the fuel properties of the produced biodiesel were in the acceptable ranges according to international standards for biodiesel specifications.

1. INTRODUCTION

The demand for energy is steadily increasing globally. This is due to the rapid growth in population, fossil fuel depletion as well as fluctuations in petroleum prices (Mcgowan et al., 2009; Narasimharao et al., 2007). This increase in demand for energy has necessitated the need to search for alternative sources to replace geological deposits of organic materials. In addition, pollution emission and environmental concerns have been a massive problem which has seen millions of dollars spent to discover new ways to mitigate them (Ferdous et al., 2013). This paved the ways for new studies on the

possibility of having alternative renewable energy sources as a substitute to fossil fuels (Bhuiya et al., 2014). Biodiesel, among other renewable energy sources, is gaining more attention as potential substitute for petro-diesel fuel (Sandip and Kok, 2017). The use of biodiesel derived from animal fat and vegetable oil to reduce dependence on fossil fuel and environmental pollution is already being practiced in some developed countries like United States of America and Germany.

Biodiesel, also known as fatty acid alkyl esters is a mixture of monoalkyl esters of long chain fatty acids. They are derivatives of lipid substances originating from vegetable oils, animal fats, waste oil etc (Palash et al., 2015). Biodiesel can be produced using methods such as transesterification, hydrodeoxygenation, dilution, pyrolysis and microemulsifiacation (Calero et al., 2015). Transesterification is the most frequently used method for biodiesel production in the industries. Transesterification offers the advantages of low production cost and high conversion efficiency. Viscosity problem often associated with other methods of biodiesel production is overcome with transesterification (Bhuiya et al., 2014). Transesterification is the catalytic reaction between triglycerides and alcohol to produce fatty acid alkyl ester and glycerol. Methanol is the preferred choice of alcohol for transesterification due to its low cost, polar and non-azeotropic nature (Fangrui and Milford, 1999).

Conventionally, homogeneous catalysts such as sodium hydroxide (NaOH), potassium hydroxide (KOH), sulphuric acid (H_2SO_4) and hydrochloric acid (HCl) have been employed for the transesterification of vegetable oil to produce biodiesel (Asakuma et., 2009).Transesterification process involving homogeneous catalysts generates large amount of wastewater from the catalyst washing process and the catalyst is not reusable. Supercritical transesterification of triglycerides could be a possible solution to these challenges. The use of heterogeneous catalysts can help to avoid the problems associated with the homogeneous catalytic process since the solid catalysts can be easily recovered and potentially reused. In addition, the wastewater producing neutralization step is eliminated. Heterogeneous catalytic process also can be operated under conditions similar to those used for the homogeneous system. Heterogeneous catalysts such as calcium oxide (CaO), magnesium oxide (MgO), aluminium oxide (Al_2O_3), zinc oxide (ZnO), Zeolite, and hydrotalcites have been employed in the transesterification process. The alkaline earth metal oxides (MgO,and CaO) among these other catalysts have demonstrated the highest activity in a typical transesterification process under moderate operating conditions (Kesic et al., 2016). Among the alkaline earth metal oxides, CaO is readily obtainable from environmental materials in the form of calcium nitrate ($Ca(NO_3)_2$),calcium carbonate ($CaCO_3$) or calcium hydroxide ($Ca(OH)_2$) which are the raw materials to produce CaO catalysts (Viriya-empikul et al., 2012).

Mango kernel fat is obtained from the kernel of mango fruit, a member of the *genus mangifera* of the family of *anacardiaceae*. The plant is a tropical fruit bearing tree which thrives well in Africa and Asia (Nzikou et al., 2010). Mango seed kernels have oil content of 9-13% depending on variety. The oil contains high content of unsaturated fatty acid with 46.22% oleic acid which compares favourably with that of *Jatropha curcas* oil, containing 44.7% oleic acid (Nzikou et al., 2010; Musa et al., 2014).

Following the industial processing or consumption of the fruits, mango seeds are often disposed of indiscriminately causing environmental concern (Puravankara et al., 2000; Kittiphoom, 2012). This paper aimed at addressing this problem by utilizing mango seed kernel fat for the production of biodiesel using heterogeneous (CaO) catalyst. Optimization of biodiesel yield, effect of operating variables (catalyst concentration, methanol-to-oil molar ratio and reaction time) and their interactions have been studied using response surface methodology.

2. MATERIALS AND METHODS

2.1. Materials

All chemicals used in this study were reagent grade and were used without further purification. Methanol, n-hexane, calcium chloride ($CaCl_2$), sodium carbonate (Na_2CO_3) potassium iodide and phenolphthalein were purchased from Whosco Chemicals Ltd, Nigeria. Mango seeds (*mangifera indica)* were collected from different locations in Benin City, Nigeria. The collected seeds were cracked to remove the kernels. The kernels were then ground in a grinder to reduce the particle sizes to a maximum of 600µm as described by Kittiphoom and Sutasinee (2013). The ground seeds were placed in a metal tray after being cleaned and dried in an oven at a constant temperature of 60 ^oC for 24 hours in order to reduce its moisture content to the barest minimum.

2.2. Mango Seed Kernel Fat Extraction

Solvent extraction method was employed in the extraction of the mango seed kernel fat using n-hexane as the solvent in a soxhlet extractor. In a cellulose paper, 50 g of the ground mango kernel was placed and extracted with 200ml n-hexane for 8 hours (Nzikou et al., 2010). A rotary evaporator (TT107R; Techmel and Techmel, USA) was employed to recover the oil while the n-hexane was collected and reused. The extracted oil was then dried in an oven at 60 ^oC to remove the residual solvent.

2.3. Catalyst Preparation

The calcium oxide catalyst was prepared using the method described by Aminu et al. (2015). To a 100 cm^3 of 1M solution of Na_2CO_3 in a beaker,100 cm^3 of 1M solution of $CaCl_2$ was slowly added while the mixture was stirred vigorously for 30 minutes. The residue of the reaction, $CaCO_3$ was filtered off using filter paper. The $CaCO_3$ was then washed with distilled water and subsequently dried at a temperature of 110 ^oC for 3 hours. The dried $CaCO_3$ was subjected to calcination in an oven at a temperature of 800^oC for 7 hours. This resulted in the formation of CaO which was stored in a glass jar until when required.

2.4. Design of Experiment

Response Surface Methodology (RSM) was used to design the experiments for the determination of the optimum conditions for the production of biodiesel from mango seed kernel fat. Box-Behnken statistical experimental design was employed to study the effects of operating parameters (independent variable) on the transesterification of mango seed kernel fat. Methanol to oil molar ratio (X_1), reaction time (X_2) and catalyst concentration (X_3) were the operating variables investigated and optimized. The actual variables were coded to two levels; low (-1) and high (+1) as shown in Table 1. The response model consisting of 17 runs had 5 central points to provide information regarding the interior of the experimental region, making it possible to evaluate the curvature effect. The experimental runs were randomly carried out so as to maximize unexplained variability effect (Yi et al., 2009). The experimental design, analysis and optimization were done using Design Expert software version 7.0.0 (Stat-ease Inc., Minneapolis, USA).

Table 1: Range and level of independent variables

Variable	Unit	Level		
		-1	0	1
Methanol-to-oil molar ratio (X_1)	mol/mol	4:1	7:1	9:1
Reaction time (X_2)	min	30	60	90
Catalyst concentration (X_3)	(% w/w)	0.5	0.7	1

2.5. Transesterification Procedure

The reactor (Flat bottom flask) was initially filled with 150 g of the oil and preheated to 60 ^oC in order to remove excess moisture from the oil. The dehydration of the oil was followed by esterification to minimize the free fatty acid content of the oil. This was done by reacting the oil with 100 ml of methanol and 1 v/w% H_2SO_4 as catalyst under constant agitation at a temperature of 60 ^oC for 1 hour. The product mixture was allowed to stand for 30minutes in a separating funnel. The lower layer which was the esterified oil was separated from the upper layer containing methanol and water (Musa et al., 2014). The methanol was later recovered from the methanol-water mixture through distillation.
Transesterification reaction was carried out by constantly agitating 100 g of the esterified oil with a mixture containing the catalyst and methanol in a known proportion as designed in Table 1. Reaction started when the methanol-catalyst mixture was added to the warm agitated oil at a temperature of 35 ^oC in the reactor. The reactor was immediately sealed using polythene bag to prevent the escape of methanol. Reaction occurred for a given time as shown in the design matrix. Upon completion of the reaction, the product was allowed to stand under gravity for a period of 24 hours and the biodiesel (upper layer) was recovered using a separating funnel. Through centrifugation at a speed of 4000 rpm for 20 minutes, the spent catalyst was recovered from the glycerol (bottom) layer of the product. The residual methanol in the biodiesel was removed by heating the biodiesel in an evaporating dish at a temperature of 85^oC for 20 minutes (Aminu et. al., 2015). Biodiesel yield was then calculated using Equation (1). The experiment was performed under different condition ranges: amount of catalyst (0.5-1.0%wt (by weight of the oil used)), reaction time (30-90min) and methanol-to-oil molar ratio (4:1–9:1). The experiment was done for various ranges of independent variables as shown in Table 1.

$$Biodiesel\ yield\ (\%) = \frac{Weight\ of\ biodiesel\ produced}{Weight\ of\ fat\ used} X100 \quad (1)$$

2.6. Fuel Characterization

The fuel and the physico-chemical properties of the produced biodiesel were determined using ASTM standard (Density, specific gravity, flash point -ASTM D 93; kinematic viscosity at 40^oC-ASTM D445; Sulfated Ash -ASTM D847; Cloud point -ASTM D2500; Iodine value -ASTM D1959; Carbon residue -ASTM D4530; Acid value-ASTM D664).

3. RESULTS AND DISCUSSION

The results of the biodiesel yield from the different experimental runs are shown in Table 2. Under the conditions investigated, the biodiesel yield obtained varied from a minimum of 57.8% to a maximum of 81.7% with an average yield of 71.9%. From experimental result obtained, a quadratic model was suggested by the Design Expert software to represent the relationship between biodiesel yield and the methanol-to-oil molar ratio, reaction time and catalyst concentration. The quadratic model for the biodiesel yield was given by Equation 2. The predicted values of the biodiesel yield are also shown in Table 2.

$$Y = 75.94 + 8.94X_1 + 4.79X_2 + 1.98X_3 + 3.74X_1X_2 + 1.01X_1X_3 - 0.43X_2X_3 - 6.87X_1^2 - 2.13X_2^2 + 0.41X_3^2 \quad (2)$$

Where:
Y= Biodiesel yield (%)
X_1=methanol-to-oil ratio (mol/mol)
X_2=reaction time (min)
X_3=catalyst concentration (%w/w)

Table 2: Experimental design matrix and the biodiesel yield

Run	Factors			Response	
	Actual values			Biodiesel yield (%)	
	X_1	X_2	X_3	Experimental	Predicted
1	6.5	90	1	81.7	80.55
2	6.5	60	0.75	75.7	75.94
3	9	60	0.5	76.98	75.43
4	6.5	30	1	72.54	71.84
5	4	30	0.75	57.8	56.95
6	6.5	30	0.5	65.88	67.03
7	6.5	60	0.75	77.68	75.94
8	6.5	60	0.75	74.45	75.94
9	6.5	60	0.75	76.68	75.94
10	4	90	0.75	59.45	59.05
11	6.5	60	0.75	75.2	75.94
12	4	60	1	59.96	61.51
13	4	60	0.5	59.87	59.57
14	9	30	0.75	66.96	67.36
15	9	90	0.75	83.56	84.41
16	9	60	1	81.11	81.41
17	6.5	90	0.5	76.75	77.45

3.1. Analysis of Variance (ANOVA)

ANOVA was applied in order to evaluate the suitability of the model, adequacy of the fit of the model and the significance of the model. The results of the ANOVA are presented in Tables 3 and 3. P-values (prob > F) less than 0.05 indicate significant model terms while values greater than 0.10 indicate that the model terms are insignificant. The results in Table 3 show that the model has a probability value (prob > F) of less than 0.0001 and an F-value of 52.96, an indication that the response model was statistically significant. All the model terms were significant except X_1X_3, X_2X_3 and X_3^2. These terms were however maintained in the model in order to maximize the accuracy of the model and minimize the deviation of experimental data from the predicted data. The adequacy of the model was further checked using the lack of fit's F-value. According to Montgomery (2001), the lack of fit is an indication of the failure of the model to represent the experimental data at which some points not included in the regression or variations in the model cannot be accounted for by random errors. This means that if there was a significant lack of fit, the model should be discarded. The lack of fit value of 2.15 indicates that it was not significant.

Table 3: ANOVA for response surface quadratic model for biodiesel yield

Source	Model coefficient	df	Mean square	F value	p-value
Model	75.94	9	126.62	52.96	< 0.0001
X_1	8.94	1	639.56	267.51	< 0.0001
X_2	4.79	1	183.16	76.61	< 0.0001
X_3	1.98	1	31.32	13.10	0.0085
X_1X_2	3.74	1	55.87	23.37	0.0019
X_1X_3	1.01	1	4.08	1.70	0.2327
X_2X_3	-0.43	1	0.73	0.30	0.5975
X_1^2	-6.87	1	198.63	83.08	< 0.0001
X_2^2	-2.13	1	19.12	7.99	0.0255
X_3^2	0.41	1	0.69	0.29	0.6063
Residual		7	2.39		
Lack of Fit		3	3.44	2.15	0.2363
Pure Error		4	1.60		
Cor Total		16			

Table 4: Statistical information for ANOVA for biodiesel yield

Source	Response Value
R-Squared	0.986
Adj R-Squared	0.967
Std. Dev.	1.55
Mean	71.9
C.V%	2.15

The coefficient of determination (R^2) was another important parameter used in analysing the accuaracy of the predicted model. The R^2 value for the predicted model was 0.9855. This means that 98.55% of the variability in the data was accounted for by the model. Thus, only 1.45% of the total variance could not be explained by the model. This is an indication of a very good model. For a good fit, R^2 values should not be lower than 0.8 (Haaland, 2009). R^2 value close to unity shows that the model fits the experimental data with very little or no variations in data.

3.2. Combined Effect of Variables on Biodiesel Yield

Three-dimensional response surfaces were plotted on the basis of the model equation (Equation 2) to investigate the interaction among the variables and to determine the optimum condition of each factor for maximum yield transesterification for biodiesel production. The variation of biodiesel yield with methanol to oil molar ratio and reaction time is shown in Figure 1. It was observed that both variables affected the biodiesel yield positively. Biodiesel yield increased with increase in time and methanol-to-oil molar ratio. This was due to the fact that higher mole ratio of reactant increases the contact between the methanol and triglyceride molecules. The biodiesel yield increased with increasing methanol to oil molar ratio, to an maximum value of 75.5% at methanol molar ratio of 8:1, after which it decreased. The drop in biodiesel yield at higher ratio of methanol to oil used could be attributed to lack of access of triglyceride molecules to contact the catalyst's active sites thereby reducing reactivity of the reactants (Seid and Omprakash, 2014).

Figure 2 shows the effect of catalyst concentration and reaction time on biodiesel yield. The yield of biodiesel increased with reaction time and catalyst concentration. It was observed that as reaction time increased from 30 minutes to 90 minutes, the yield of biodiesel increases from about 57% to 72% without any drop in the yield. Also as catalyst concentration increased from 0.5% to 1% by mass of catalyst the yield increased to its maximum at a catalyst concentration of 1% and began to drop thereafter. This observation is due to the fact that increasing the amount of catalyst leads to increased active sites of the catalyst and thereby speeding up the reaction and increased fatty acid methyl ester (biodiesel) yield. This observation is consistent with the findings of Fangrui and Milford (1999) and Wu et al. (2014).

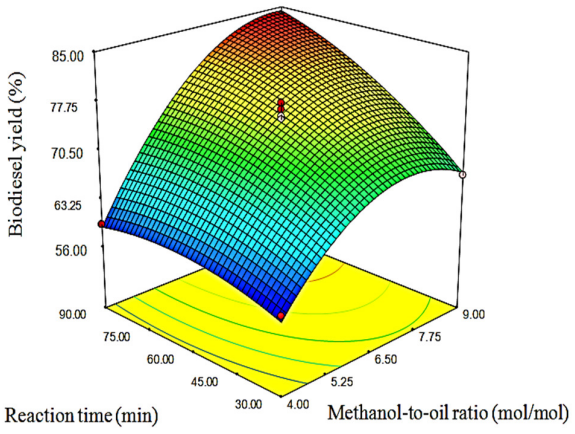

Figure 1: Effect of methanol to oil ratio and reaction time on the yield of biodiesel

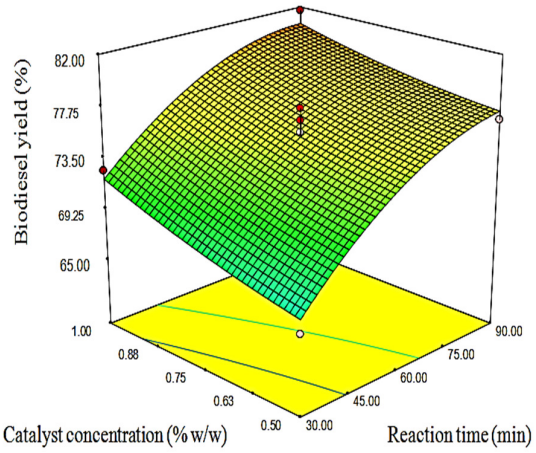

Figure 2: Effect of catalyst concentration and time on the yield of biodiesel

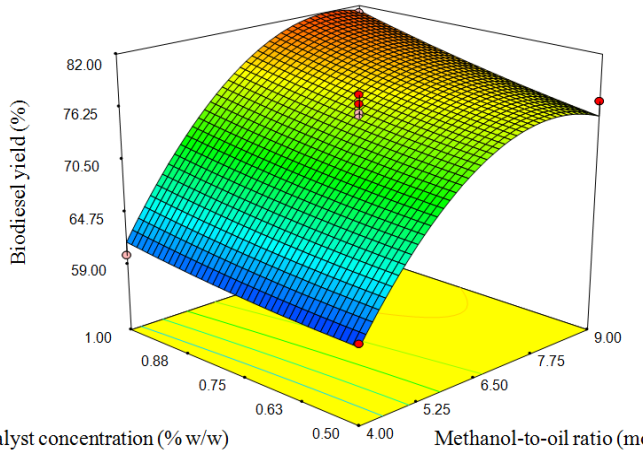

Figure 3: Effect of catalyst concentration and methanol-to-oil molar ratio on the yield of biodiesel

The effect of methanol-to-oil molar ratio and catalyst concentration on the yield of biodiesel is shown in Figure 3. At lower methanol-to-oil molar ratio, the conversion of triglyceride to biodiesel increased with increase in catalyst concentration. It seemed that increase in methanol-to-oil molar ratio had significant effect on the conversion to biodiesel at different catalyst amounts. This could be due to the fact that methanol-to-oil molar ratio has a higher coefficient value and a lower p-value than the catalyst. The nature of the contour plot between the methanol-to-oil molar ratio and catalyst

concentration indicated that interaction between these two variables had a positive significant effect on the conversion to biodiesel.

3.3. Optimization of Process Variables

The process variables (methanol-to-oil ratio, reaction time and catalyst concentration) were optimised on the basis of the statistical model given by Equation 2 and the first few results are given in Table 5. The first solution having the highest desirability was selected as the optimum conditions of the transesterification reaction of mango seed kernel fat.

Table 5: Optimum solution of transesterification of mango seed kernel fat

S/N	Methanol-to oil ratio (mol/mol)	Reaction time (min)	Catalyst concentration (w/w)	Yield (%)	Desirability
1	8.9	89.34	1.0	87.37	0.983
2	8.8	89.26	1.0	87.36	0.983
3	9.0	89.32	1.0	87.34	0.982
4	9.0	89.12	0.9	87.32	0.982
5	9.0	88.57	0.9	87.17	0.982

Two experimental runs were performed using the selected optimum transesterification conditions presented in Table 4 to confirm and validate the statistical model. The experimental biodiesel yield of 87.09% under the given optimum conditions compared favourably with the predicted optimum value of 87.37%. This result validates the statistical model and it shows a strong correlation between the actual and predicted values.

Table 6 shows the physico-chemical properties of the produced biodiesel from mango kernel fat. All the properties were within the standard of America Society for Testing and Materials (ASTM) except the carbon residue and sulphated ash which are slightly above the recommended maximum. The fact that the important properties of the biodiesel produced were well within the ranges of the recommended ASTM standard implies that the biodiesel produced from mango kernel fat could satisfactorily be used as fuel for internal combustion engine.

Table 6: Physico-chemical properties of biodiesel produced from mango kernel fat

Biodiesel properties	Measured values	ASTM Standard
Kinematic viscosity at 40^{o}C (cst)	5.45	4.0-6.0
Specific gravity	0.85	0.88max
Density (1b/gal)	7.11	7.3
Acid number (mg KOH/g oil)	0.81	0.8max
Iodine value	-	7.03
Flash point (^{o}C)	150	100-170
Cetane number	58	48-65
Pour point (^{O}C)	-2	-5-10
Sulphated ash	0.03	0.02max
Carbon residue	0.26	0.15max

4. CONCLUSION

This study suggested that optimum yield of biodiesel from mango seed kernel fat is obtained at methanol-to-oil molar ratio of 8.9, reaction time of 89.3 minutes and catalyst concentration of

1.0%w/w. The findings presented in this paper also indicated that reasonable yields of biodiesel can be produced from mango seed kernel fat using calcium oxide as heterogeneous catalysts under operating conditions similar to those used for homogeneous system. This will help mitigate challenges such as the generation of large amount of wastewater, non-reusability of the catalyst and the difficulty of separation of the catalyst from the products which are often associated with the use of homogenous catalyst in the production of biodiesel. The use of mango kernel fat in the production of biodiesel will also help to checkmate the problem of environmental concerning arising from indiscriminate disposal of mango seeds after consumption.

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6. CONFLICT OF INTEREST

The authors declare that there is no conflict of interests with respect to the publication of this paper..

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