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One-pot synthesis of biodiesel from high FFA shea butter in an optimisation study using response surface methodology

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ABSTRACT

Optimisation of biodiesel production from high free fatty acids (FFA) of 6.86% shea butter (SB) using a base catalyst, potassium hydroxide, was studied. Response surface methodology (RSM) was employed as a model for the optimisation of the process by varying temperature, agitation speed, mole ratio and catalyst loading in the range of 40–60°C, 200–1400 rpm, 8:1–12:1 (w/w) and 1–2% (w/w) respectively. The significance of the variables in both linear and nonlinear forms of the model was analysed by analysis of variance (ANOVA). The ANOVA results showed that the temperature, agitation speed, mole ratio and catalyst loading had a significant effect on the shea biodiesel yield with a *p*-value of < 0.0001 for each of the variables, adjusted R^2 of 0.9517 and predicted R^2 of 0.8625. The maximum %yield of shea biodiesel obtained was 90.83% at optimal operating temperature of 50°C, agitation speed of 800 rpm, mole ratio of 12:1 (w/w) and catalyst loading of 1% (w/w). This showed that the RSM optimised the one-pot synthesis of biodiesel from high FFA SB, an indication that it can be adopted successfully in the industry to maximise the yield of biodiesel from high FFA SB.

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Transesterification; biodiesel; shea butter; free fatty acid; optimisation; response surface methodology

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Introduction

Biodiesel is a renewable, sustainable and alternative replacement to petroleum-derived diesel with several benefits and advantages. It is biodegradable, has negligible presence of sulphur (<0.005%, w/w), is renewable and non-toxic, and has excellent lubricity. In terms of physical properties and functionality, it is very similar to petroleum diesel fuel, and therefore can be used to substitute petroleum diesel in existing diesel compression-ignition engines with little or no engine modifications [1,2]. Several feedstocks that consist of triacylglycerides (TG) such as vegetable oils, animal fats, and waste grease, can be converted through transesterification reactions or esterification of free fatty acids (FFA) in the presence of low molecular weight alcohols, to biodiesel; an alternative fuel which consists of methyl or ethyl esters. However, the conversion of feedstock with high FFA (>1%) to biodiesel requires a more complicated process, especially when a basic homogeneous catalyst such as alkaline metal alkoxides (CH_3ONa and CH_3OK) and alkaline hydroxides (NaOH and KOH) are used for the transesterification, which can lead to soap formation as by-products through the unwanted saponification reaction. The production of soap in the reaction leads to low quality in terms of physicochemical properties and also to a high cost of production through the separation of soap from the biodiesel [3,4].

Several authors employed esterification and transesterification in a two-stage process to overcome these challenges by using an acid-base catalyst. Goyal et al. [5] produced biodiesel from high FFA jatropha oil in a two-stage process using acid-base catalyst and obtained 98.3% at a reaction time of 240 min. Mazo and Rios [6] also studied the conversion of palm oil into biodiesel by the same process and got a 86.31% yield at a reaction time of 240 min. Lebnebiso et al. [7] investigated cottonseed oil for biodiesel production in a two-stage process of acid-base catalyst and yielded 66.4% biodiesel at a reaction time of 30 min for transesterification; the time of esterification was not mentioned, but the yield was low. Math et al. [8] reported the same process for the mixture of restaurant waste and pig fat of high FFA and obtained a yield of 85.5% biodiesel at a high reaction time of 225 min. Ajala et al. [9] also used high FFA shea butter for biodiesel production in a two-stage process and yielded 92.16% biodiesel at a reaction time of 360 min. For all the aforementioned studies, a large amount of methanol above methanol: oil molar ratio of 12:1 was consumed for the two-stage process which led to an increase of the overall cost of the biodiesel production. The presence of an acid catalyst in the two-stage process causes equipment corrosion, long reaction times and difficulty in catalyst recovery [10]. Due to these challenges of a two-stage process, one-pot synthesis is

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investigated. One-pot synthesis is a promising alternative method that can mitigate some of the challenges with the two-stage process.

One-pot synthesis is the treatment of high FFA present in the TG using a base catalyst for neutralisation of FFA and transesterification simultaneously to produce biodiesel. This process is expected to reduce the reaction time, production loss and the cost of production, and meet product quality standards. In order to achieve high performance of this process, optimisation of the process variables is essential with the aid of appropriate experimental design procedure such as response surface methodology (RSM).

RSM is an optimisation technique using an experimental design to achieve optimal variables that would give an optimum response (biodiesel yield). The RSM is a set of mathematical and statistical techniques that are useful for modelling and analysing problems which are influenced by several variables (independent variables) with the aim of optimising the response of interest. The first step of the RSM is to obtain a mathematical model that describes the response as a function of the independent variables. The second step is to determine the validation of the model which is assessed by the analysis of variance (ANOVA) using an F-test [11]. RSM is an effective tool to finding the optimal variables for different processes using any of the different packages such as Box-Behnken, three-level factorial, hybrid, D-optimal, pentagonal, hexagonal and central composite design (CCD) [12].

The CCD has been applied in the optimisation of biodiesel production as it has several advantages: it reduces the number of experimental runs that would provide sufficient data to generate enough information for a statistically acceptable result; it reduces the cost of expensive analysis methods like finite element method or computational flow diagram analysis and their associated numerical noise; and it helps in finding improved or optimal process settings in troubleshooting process problems and in making a product or process more robust. Goyal et al. [5] had successfully used CCD for the optimisation of esterification and transesterification of jatropha caucus for biodiesel production and achieved 98.3% yield with methanol:oil molar ratio (11:1) using NaOH as catalyst (1% w/w) in 110 min at 55°C. Jahirul et al. [13] were also successful by using RSM to optimise biodiesel production using non-edible beauty leaf (*Calophyllum inophyllum*) and obtained 93% yield when methanol:oil molar ratio, catalyst concentration and reaction temperature were 7.5:1, 1% and 55°C respectively.

Therefore, this study was aimed at investigating the production of biodiesel using high FFA shea butter in a one-pot synthesis. One-pot synthesis was optimised using RSM based on a CCD to determine the effect of operating variables such as temperature, methanol:oil molar ratio, catalyst loading and agitation speed on the percentage yield of biodiesel from shea butter.

Materials and methods

Materials

Shea butter (SB) with FFA of 6.86% was obtained from *Fufu*, Ilorin South Local Government, Kwara State, Nigeria. A 4.5 L capacity reactor developed as part of this research was used to carry out the reaction. The reactor was made of stainless steel and had a heater band with temperature and electric motor speed controllers installed. The methanol and KOH used were of analytical grades.

Experimental design

The CCD with three-level-four-factor variables of RSM in Design Expert (version 8.06 Stat-Ease Inc., Minneapolis, MN, USA) was applied for the optimisation of the one-pot synthesis of biodiesel from high FFA shea butter using a base catalyst (KOH) only. A total of 30 experiments were designed for the process at different levels of reaction temperature (A) (°C), agitation speed (B) (rpm), methanol:oil molar ratio (mole ratio) (w/w) and catalyst loading of KOH (w/w) as the independent variables (Table 1) to determine the %yield of shea biodiesel. Multiple regression analysis was used to evaluate the best-fitting of the quadratic polynomial model used in the study.

Experimental procedure

The SB was melted and filtered to remove all insoluble impurities. The filtered SB was then heated at 100°C for 10 min to remove all the moisture and to obtain pure SB. The %FFA of the pure SB was determined using the standard analytical methods of American Oil Chemists Society (AOCS) for fats and oils as reported by Viele et al. [2] The standard method, that is, a titration procedure, was used to evaluate the quantity of KOH to neutralise the FFA in the butter for biodiesel production in a one-pot synthesis [2,14]. The required quantity of KOH was weighed and poured into a flask. The mole ratio of 8:1 was selected as the minimum for conversion of high FFA shea butter to biodiesel in a base-catalysed reaction and was added to the measured KOH to form potassium methoxide.

Table 1. Levels of variables for optimisation of one-pot synthesis of shea biodiesel.

Independent variables			Range and level		
Factor	Name	Units	−1	0	+1
A	Temperature	°C	40	50	60
B	Agitation speed	rpm	200	800	1400
C	Methanol:oil ratio	w/w	8	10	12
D	Catalyst loading (KOH)	(w/w, %)	1.0	1.5	2.0

Table 2. Physicochemical properties of shea biodiesel using one-pot synthesis.

Property	Shea biodiesel	Diesel	ASTM method	Limits
Density kg m^{-3}@15°C	885.5	860.4	4052-11	860–900
Specific gravity kg m^{-3}@15°C	885.9	860.8	4052-11	
Kinematic viscosity mm^2/s@40°C	5.71	2.6	445-12	1.9–6.0
Flash point (°C)	170.0	73.0	93-02a	130 min
Cloud point (°C)	14	2.4	2500-11	−3–12
Pour point (°C)	10	−9	97-12	−15–10
Cetane	49	49	976-11	47–60
Total sulphur (% mass)	0.001	0.300	4294-10	0.005 max
Water content (% vol.)	<0.05	<0.05	95-13	0.05 max
Colour	L1.0	L2.0	1500-12	L2.0
Total acid value (mg KOH/g)	0.37		974-12	0.80 max
Residue	1.0	1.0	–	
Loss	0.5	0.5	–	
Diesel index	24	28	*IP21	
Aniline point (°F)	86	86	611-12	

*CALC (*IP21), ASTM = American Society Testing and Materials, BC = base catalysed and ABC = acid-base catalysed samples
Source: Ajala et al. (2015b))

The SB (2000 g) was measured into the 4.5 L reactor and 70% of the potassium methoxide required was added to the reactor. This was done to facilitate the neutralisation of FFA in the butter. The mixture was heated at stipulated temperatures of 40, 50 or 60°C, and agitation speeds of 200, 800 or 1400 rpm for 1 h. The agitation was stopped after 1 h while the reaction temperature was kept constant for another 1 h. Thereafter, the remaining 30% of the potassium methoxide was added to complete the transesterification reaction.

The reaction continued for another 1 h at the stipulated temperature and agitation speed. The product was discharged and poured into a separating funnel for 24 h. The biodiesel was thereafter separated from glycerol to obtain crude biodiesel, which was further purified by washing and a further purification process.

Statistical and optimisation analysis

A mathematical model that described the response variables (Y) as a function of the independent variables (X_1, X_2...X_3) was chosen (Equation 1), which considered the significance of the coefficients. The coefficient Y is the response at a central point, and the other coefficients measured the main effects and interactions of coded variables (X_i) on the response variable (Y). The model obtained from the regression analysis was used to generate the response surface and contour plots for the study. For selecting the significant terms from the model, the significance level was set at a confidence interval of 95% and p-value < 0.05. The validity of the model was determined by the analysis of variance (ANOVA) using an F-test.[11]

$$Y = \beta_0 + \sum_{i=1}^{n} \beta_i x_i + \sum_{i=1}^{n} \beta_{ii} x_i^2 + \sum_{i=1}^{n-1} \sum_{j=i+1}^{n} \beta_{ij} x_i x_j \quad (1)$$

The optimal values of selected variables were obtained by solving the regression Equation (1) using the design expert. The model was used to determine the value of the process variables that produced optimum %yield of shea biodiesel.

Purification process and percentage yield of shea biodiesel

The modified methods of biodiesel purification by Ajala et al. [9] and Mishra et al. [15] were used to obtain pure biodiesel. The quantity of the pure biodiesel obtained was determined using a weighing balance and the percentage yield of the biodiesel was calculated using Equation (2):

$$\text{Yield of SBD } (\%) = \frac{\text{Total weight of pure biodiesel}}{\text{Total weight of oil used}} \times 100\% \quad (2)$$

Analysis of the sample

The shea biodiesel produced was characterised for physicochemical properties and fatty acid methyl esters (FAME) profile. The results have since been published in the literature [10] and shown in Tables 2 and 3, respectively.

Results and discussion

Experimental results and model fitting

The matrix of the operating variables and the %yield of shea biodiesel in 30 experimental runs are shown in Table 4. The predicted results of the model and the

Table 3. GC-MS quantitative analysis of FAME of shea biodiesel from one-pot synthesis.

Serial number	Retention time (min)	FAME compound	Molecular formula	% content for shea biodiesel
1	24.5	Methyl palmitate	C16:0	6.72
2	28.0	Methyl oleate	C18:1	50.26*
3	28.5	Methyl stearate	C18:0	38.82
4	31.7	Methyl arachidate	C20:0	4.21

*Monounsaturated fatty acid methyl esters and the remaining are saturated.
Source: Ajala et al. (2015b))

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Table 4. The matrix of variables and %yield of shea biodiesel using CCD of RSM.

Experimental run	Temperature (°C)	Agitation (rpm)	Methanol ratio (w/w)	Catalyst (KOH, w/w)	%Yield of shea biodiesel
1	40	200	8	1.00	79.14
2	60	200	8	1.00	53.9
3	40	1400	8	1.00	65.31
4	60	1400	8	1.00	59.9
5	40	200	12	1.00	90.03
6	60	200	12	1.00	86.54
7	60	1400	12	1.00	66.78
8	40	1400	12	1.00	61.72
9	40	200	8	2.00	59.28
10	60	200	8	2.00	37.72
11	40	1400	8	2.00	56.14
12	60	1400	8	2.00	46.64
13	40	200	12	2.00	70.4
14	60	200	12	2.00	51.33
15	40	1400	12	2.00	50.99
16	60	1400	12	2.00	43.42
17	60	800	10	1.50	39.62
18	40	800	10	1.50	52.48
19	50	200	10	1.50	38.97
20	50	1400	10	1.50	29.45
21	50	800	12	1.50	69.03
22	50	800	8	1.50	61.9
23	50	800	10	1.00	72.3
24	50	800	10	2.00	45.75
25	50	800	10	2.00	45.75
26	50	800	10	1.50	43.03
27	50	800	10	1.50	42.56
28	50	800	10	1.50	42.67
29	50	800	10	1.50	42.77
30	50	800	10	1.50	43.01

actual results of the experimental runs are shown in Figure 1. The figure shows that the predicted values are quite close to the experimental values, which validates the reliability of the model and showed a good fit of the model. This established a correlation between the operating variables and the %yield of shea biodiesel.

Table 5 shows the coefficients of the model and *p*-values of the variables as they affect the transesterification of high FFA shea butter for biodiesel production in a one-pot synthesis. The analysis of variance (ANOVA) and a *p*-value of the model are shown in Table 6. The coded terms A, B and D in the linear coefficients, BC in the interactive coefficients and B^2, C^2 and D^2 in the

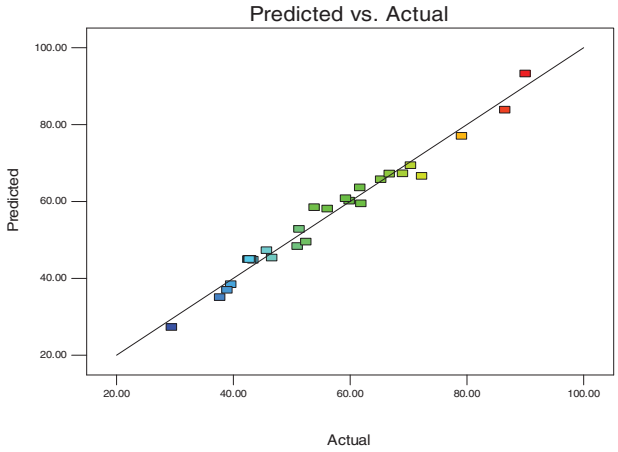


Figure 1. Predicted against actual results of %yield of shea biodiesel in one-pot synthesis.

Table 5. Estimated coefficient of the model and variables for %yield of shea biodiesel.

	%Yield of shea biodiesel		
Factors	Coefficient	F-value	*p*-value
Intercept	44.94	41.84	<0.0001
A	−5.54	50.36	<0.0001
B	−4.83	38.36	<0.0001
C	3.91	25.08	0.0002
D	−9.66	153.48	<0.0001
AB	3.25	15.39	0.0014
AC	2.29	7.66	0.0144
AD	−1.79	4.67	0.0472
BC	−4.58	30.69	<0.0001
BD	2.15	6.74	0.0203
CD	−1.90	5.29	0.0362
A^2	−1.01	0.24	0.6318
B^2	−12.85	39.03	<0.0001
C^2	18.41	80.17	<0.0001
D^2	11.97	33.89	<0.0001

Table 6. ANOVA for %yield of shea biodiesel from high FFA shea butter.

Source	Sum of square	df	Mean square	F	*p*-value
Regression	6414.82	14	458.27	41.84	<0.0001
Linear	2927.35	4	731.84	5.01	0.0042
Square	771.66	6	128.61	0.85	0.5491
Interaction	2716.82	4	679.20	62.02	<0.0001
Residue error	164.29	15	10.95		
Pure error	0.18	5	0.036		
Total	6580.11	29			
R^2 0.9750	**Adj. R^2** 0.9517		**Pred. R^2** 0.8625	**C.V. %** 6.03	

square coefficients were highly significant ($p < 0.0001$), and only A^2 terms in all the coefficients for the chosen model were insignificant ($p > 0.5$). The F-value of 41.84 and *p*-value less than 0.0001 showed that the model and its terms were significant. A low value of the coefficient of variation (CV) of 6.03% indicated a high degree of precision and that the experimental values were highly reliable. From the table, regression coefficient (R^2) was 0.9750, which is the fraction of the variation of the response from the model, an indication that 97% of the experimental data is compatible with the predicted data of the model. The adjusted (Adj.) R^2 of 0.9517 and predicted (Pred.) R^2 of 0.8625 also supported the significance and indicated a reasonable precision of the model. Regression analysis was performed on the designed experimental data and a model equation for the production of shea biodiesel was obtained and is shown in Equation (3).

$$\begin{aligned} \%\ Biodiesel\ Yield\ \left(\frac{w}{w}\right) &= +44.94 - 5.54A - 4.83B + 3.91C - 9.66D \\ &\quad + 3.25AB + 2.29AC - 1.79AD - 4.58BC \\ &\quad + 2.15BD - 1.90CD - 1.01A^2 - 12.85B^2 \\ &\quad + 18.41C^2 + 11.97D^2 \end{aligned} \tag{3}$$

where A–D are the studied independent variables in terms of temperature (°C), agitation speed (rpm), mole ratio (w/w) and catalyst loading (w/w) respectively.

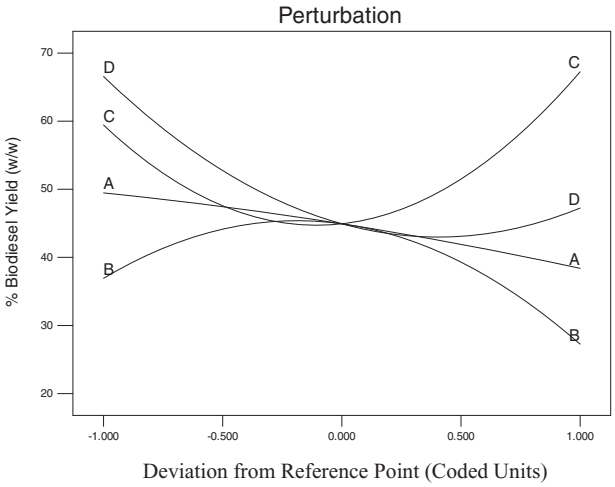


Figure 2. Effect of temperature (A), agitation speed (B), methanol/oil molar ratio (C) and catalyst loading (D) on %yield of shea biodiesel (coded).

Figure 2 shows the effect of temperature (A), agitation speed (B), mole ratio (C) and catalyst loading (D) on %yield of shea biodiesel using coded level of −1 and +1 on the horizontal axis. The figure shows that %yield of shea biodiesel decreases significantly with increase in temperature. This is because transesterification occurs at different temperatures depending on the oil used and it affects the yield of the biodiesel. For this study, the reason for the low yield of shea biodiesel at higher temperature was due to the acceleration of the saponification reaction of triglycerides; and the loss of methanol at a temperature beyond its boiling point of 65°C [16].

In terms of the agitation speed, the %yield decreases along both directions, an indication that both excess and insufficient agitation speed reduces the %yield of shea biodiesel. The deviation of mole ratio from the centre to +1 shows that the yield increases as the mole ratio increases. Biodiesel yield, however, can be improved from the triglycerides by introducing excess alcohol into the reaction, since the reaction is a transesterification which is a reversible process that can shift the reaction to the product when excess alcohol is used [17].

The %yield of shea biodiesel decreases as the catalyst loading increases from −1 to +1. This is because the addition of excessive catalyst causes more triglyceride to react with the alkali catalyst leading to the formation of soap, thereby causing an increase in biodiesel yield [17]. In this context, the yield of shea biodiesel increases with moderate temperature, agitation speed, and high mole ratio, but decreases with increase in catalyst concentration. These results showed that the CCD model of RSM is suitable to predict %yield of shea biodiesel in this study.

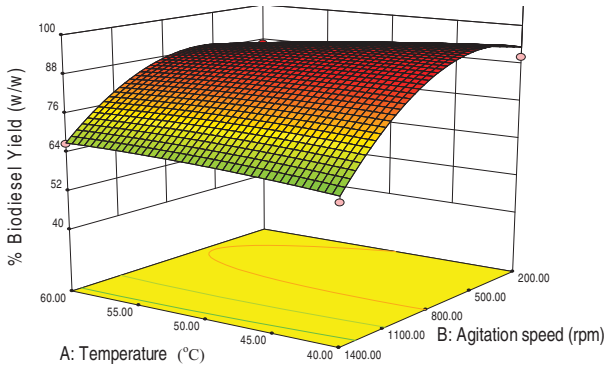


Figure 3. Effect of temperature and agitation speed on %yield of shea biodiesel in a 3D surface plot.

Effects of the process variables on %yield of shea biodiesel using one-pot synthesis

Figure 3 presents the effects of temperature, agitation speed and their interaction on the %yield of shea biodiesel. The figure shows that as the temperature increases from 40 to 60°C, the %yield of shea biodiesel decreased but the yield increased significantly when the agitation speed was increased from 200 to 800 rpm. However, a further increase in the agitation speed to 1400 rpm resulted in a decrease in the %yield of shea biodiesel. This was because the mixing effect was most significant at the slow rate region when the reaction was at the initial stage, and once the single phase was established, increase in the mixing intensity becomes insignificant. Besides, when mixing intensity is high, the mass transfer controlled region is shortened and the mixing effect is not significant [18,19]. This shows that the agitation speed affects the %yield of shea biodiesel. The interaction of the two variables gave an optimum yield of 93.21% shea biodiesel at a temperature of 40°C, agitation speed of 800 rpm when mole ratio was 12:1 and catalyst loading (KOH) was 1% (w/w).

Figure 4 shows the effects of temperature, mole ratio and their interaction on the %yield of shea biodiesel. The effect of temperature on this reaction seems

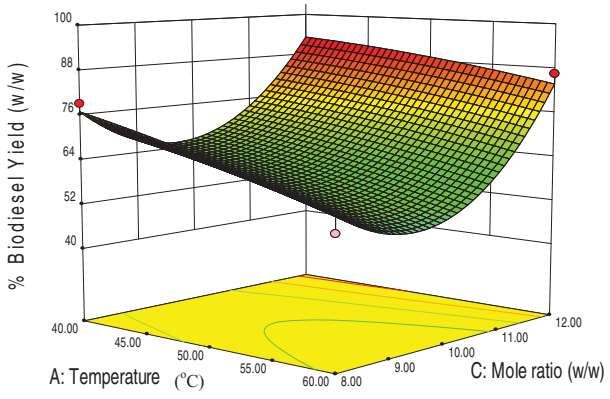


Figure 4. Effect of temperature and methanol/oil molar ratio on %yield of shea biodiesel in a 3D surface plot.

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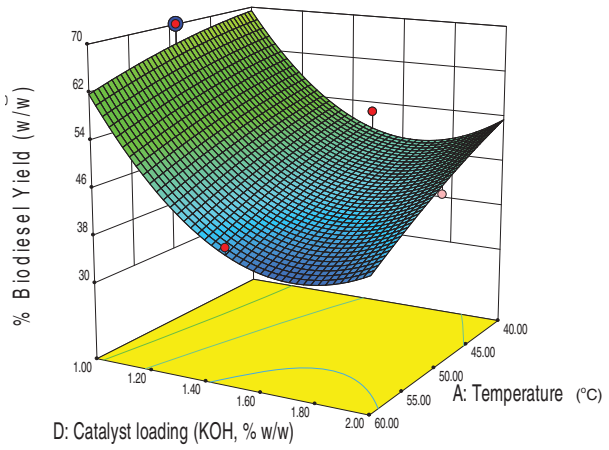


Figure 5. Effect of temperature and catalyst loading on %yield of shea biodiesel in a 3D surface plot.

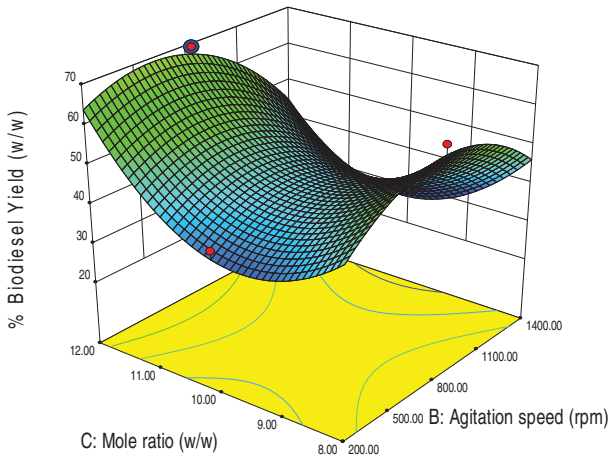


Figure 6. Effect of agitation speed and methanol/oil molar ratio on %yield of shea biodiesel in a 3D surface plot.

insignificant as shown in the figure, but when mole ratio was increased from 8:1 to 10:1, the %yield of shea biodiesel decreased drastically. However, an increase of the ratio to 12:1 brought about a significant increase in the %yield. The interaction of the two parameters shows a %yield of 93.21% biodiesel at a temperature of 50°C, mole ratio of 12:1 and at an agitation speed of 800 rpm with a catalyst loading of 1.5% (w/w).

The effect of temperature, catalyst loading, and their interactions is shown in Figure 5. The figure shows that the interaction at 50°C and catalyst loading of 1% (w/w) significantly affected the %yield of shea biodiesel, resulting in a maximum %yield of 69.3%. The %yield of shea biodiesel increases with the increase in temperature until 50°C at low catalyst loading (1%, w/w). It was evident that when the temperature was constant, there was a decrease in %yield of shea biodiesel with an increase in catalyst loading. This may be due to the formation of an emulsion from the excess catalyst in the process [20]. Also, when the catalyst loading was kept constant, at a high temperature of 60°C, a decrease in the %yield of biodiesel was noticed. This was as a result of the mechanism of the process which involves the interaction of the catalyst with the protons generated from the alcohol solvent and formed catalyst oxide. The triglycerides react with the catalyst oxide to form a nucleophile and proton. These compounds block catalyst active sites and lead to the decrease in the %yield of biodiesel [20].

The effect of agitation speed and mole ratio on the %yield of shea biodiesel illustrated by the response surface plot at a temperature of 50°C and catalyst loading of 1.5% (w/w) is shown in Figure 6. This figure shows that mole ratio increased from 8:1 to 12:1 to obtain a maximum %yield of shea biodiesel of 70% at an agitation speed of 800 rpm. It was also noticed that an increase in agitation speed from 200 to 800 rpm led to an increase in the %yield but a further increase in agitation speed to 1400 rpm led to a decrease in the %yield. This showed that the two variables of agitation speed at 800 rpm and mole ratio at 12:1 had a significant effect on the %yield, resulting in a maximum 69.03%.

Figure 7 shows the effect of agitation speed and catalyst loading, and their combined interaction on the %yield of shea biodiesel at 50°C and mole ratio of 10:1. It was observed that the interaction of the two variables showed a significant effect on the %yield of shea biodiesel. At the lowest speed (200 rpm) and 1% (w/w) catalyst loading, and the highest speed (1400 rpm) and 2% (w/w) catalyst loading, 60% and 30% of %yield of shea biodiesel were obtained. However, at 800 rpm and catalyst loading of 1% (w/w), 72.3% of yield was obtained. This is because the transesterification reaction is a diffusion-control and poor diffusion between the phases resulted in a slow reaction rate. At the initial stage of the reaction, the two-phase liquid system is formed and as the agitation speed increased, the diffusion of the two-phase changed into one-single-phase which led to the formation of methyl esters. The mixing effect is most significant within the slow rate region

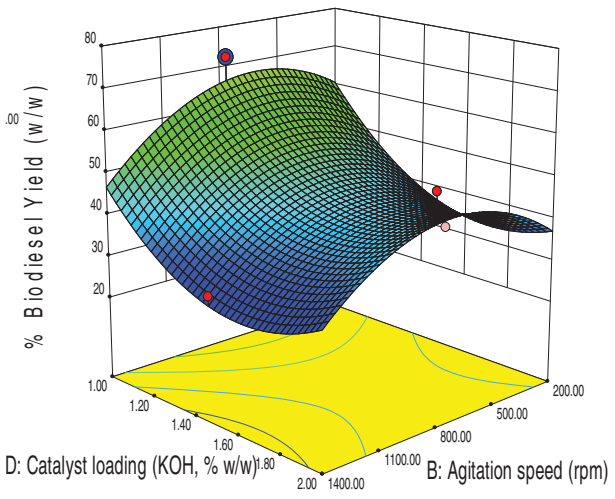


Figure 7. Effect of agitation speed and catalyst loading on %yield of shea biodiesel in a 3D surface plot.

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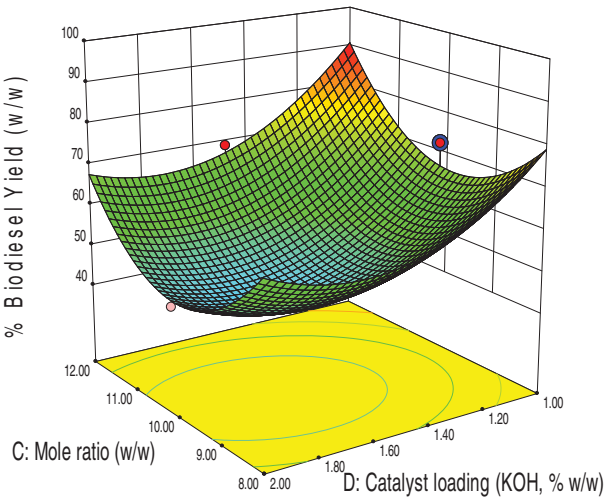


Figure 8. Effect of methanol/oil molar ratio and catalyst loading on %yield of shea biodiesel in a 3D surface plot.

Table 7. Optimal variables for %yield of shea biodiesel from high FFA shea butter.

Optimum condition				%Yield (w/w)		
Temp. (°C)	Agitation speed (rpm)	MeOH/ oil ratio (w/w)	Catalyst loading (% w/w)	Actual result	Predicted result	% Error
50	800	12	1	90.83	90.50	0.36

of the reaction. As the single phase is established, mixing becomes insignificant [21].

The effects of mole ratio and catalyst loading at a temperature of 50°C and agitation speed of 800 rpm are shown in Figure 8. The %yield of shea biodiesel increases with a decrease in a mole ratio from 12:1 to 8:1 at 2% (w/w) catalyst loading. It was observed that at a mole ratio of 10:1 and catalyst loading (KOH) of 1% (w/w), the %yield was 72.3%.

Optimisation of process variables for shea biodiesel production in one-pot synthesis

The optimal variables predicted by the model were the temperature of 50°C, agitation speed of 800 rpm, mole ratio of 12:1, catalyst loading (KOH) of 1% (w/w) and with the theoretical yield of 90.50% shea biodiesel as shown in Table 7. The prediction of the model was tested by validating the variables in a repeat experimental run. The %yield of shea biodiesel obtained was 90.83%, which is very close to the model prediction with %error of 0.36 (Table 5). This result demonstrates that RSM with good and accurate experimental design can be effectively applied to optimise the relevant parameters for shea biodiesel production in one pot synthesis.

Conclusion

Shea butter of 6.85% FFA was converted to biodiesel in a one-pot synthesis using a response surface methodology based on a CCD. The CCD was employed to plan feasible experimental runs that would optimise the operating variables to give an optimum yield of shea biodiesel. The effects of the operating variables such as temperature, agitation speed, mole ratio and catalyst loading were statistically investigated for the synthesis. The predicted optimal conditions according to the statistical modelling for the one pot synthesis were temperature 50°C; agitation speed 800 rpm; mole ratio 12:1 (w/w) and catalyst loading 1% (w/w) with the highest achievable %yield of shea biodiesel as 90.83%. The variables were found to be highly significant with p-values < 0.0001, so it can be concluded that they all had a significant effect on the %yield of shea biodiesel. Based on the obtained experimental results, the RSM is an effective optimisation tool for a simultaneous neutralisation and transesterification reactions for biodiesel production using high FFA shea butter and potassium hydroxide as the only catalyst. The information obtained from the model and the experiment is very significant and can be applied in the industrial production of biodiesel from high FFA vegetable oil such as shea butter.

Disclosure statement

No potential conflict of interest was reported by the authors.

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