



Response surface optimization of biodiesel production from snail shell catalysed *Jatropha* oil

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Abstract: Snail shell was obtained in Ungwan Sunday area in Kaduna South of Kaduna Metropolis. It was washed, cleaned, dried, ground and sieved to a particle size of 250 microns, 180 microns and 63 microns. Calcined at a temperature of 900°C for 3 hours to remove the remaining moisture content. The calcined catalyst was employed in transesterification process between *Jatropha* Oil and methanol. The synthesis parameters such as the reaction time (60-180min), reaction temperature (30-60°C), molar ratio (6:1) and the amount of catalyst (1-4 wt%) were optimized using Response Surface Methodology (RSM) based on Box Behnken Design (BBD). The results of the optimization showed an optimum yield of 86.3 % biodiesel at 60 °C, 120 minutes using 4 wt% catalyst. Biodiesel characteristics from the experiment were qualified according to the ASTM D975 standard. The results showed that the characteristics of the produced biodiesel fall within the standard.

Keywords: *biodiesel, jathropa oil, Response Surface Methodology; Box Behnken Design.*

1. INTRODUCTION

The renewable energy source has become the main source of energy for many nations, including Nigeria, although it has been identified that its manufacturing and use negatively affects the environment in the form of an unusual rise in the level of atmospheric carbon dioxide, environmental degradation, environmental pollution (greenhouse gas emissions), and anthropogenic global warming. As a result, there has been improved awareness of the environment and concern about the determination to reduce the progress of climatic change. Thus, in both studies, growth, and business, the transition from fossil fuels to pure and sustainable energy sources is growing rapidly.

In Nigeria, diesel usage has been confirmed to be on the rise (Isa *et al.*, 2013) and with its determination to industrialize as stated in Vision 2020, this pattern is most probable to continue; because of the need for local growth of renewable, sustainable and environmentally friendly alternative power sources. Several energy sources are currently being researched and applied in practice, with varying degrees of achievement, and in various stages of research and execution.

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Examples include, among others, solar energy, thermal or photovoltaic, hydroelectric, geothermal, wind, biofuels, and carbon sequestration. Each has its own benefits and issues and distinct choices will be best suited depending on the region of implementation.

Boroet *al.*, (2012) documented that biodiesel has attracted significant worldwide interest in reducing global warming and dependency on conventional fossil fuels considerably. Chakraborty (2011) also indicated that it can be generated using fundamental, acidic, enzymatic or other catalysts with heating features by transesterification of oil or esterification of fats. Buasri (2015) reported that biodiesel manufacturing uses an alkaline catalyst most frequently, but this method is hard to adapt for use with some disposal oils and fats. The standard homogeneous technique has technical difficulties in removing catalysts after response and generates big quantities of wastewater which makes it hard to distinguish the catalyst and clean the products. Therefore, for biodiesel production, the use of heterogeneous catalysts with many benefits over homogeneous catalysts is very significant (Sanjay, 2013).

Calcium oxide (CaO) is among the most exciting heterogeneous alkali catalysts used for biodiesel production because it is inexpensive and abundant in nature and some of its sources are renewable (Suryaputra, 2013). Buasri (2015) said it strongly resembled a material that is eco-friendly. Akande *et al.*, (2016) indicated that calcium carbonate (CaCO₃) usually produces CaO. Akande *et al.*, (2016) revealed that a number of naturally occurring of calcium from waste such as snail shell, eggshell, crab shell, and periwinkle are used to generate elevated output and quality calcium oxide. Buasri (2015) indicated that using biomass as raw material for catalyst synthesis could reduce waste while producing high-cost efficiency catalysts concurrently (Viriya-empikul, 2010). The waste shell synthetic catalyst provides the gate to a sustainable catalyst while recycling the waste produced (Buasri, 2015). These shells may also find use in other base catalyzed significant organic reactions that add value to the produced waste (Boroet *al.*, 2012). In several areas of Nigeria, snail shells are discovered. The preparation also generates important quantities of shell waste in the manufacturing of Snail shell meat on a very big scale. The objective of this research is to explore the optimization of Biodiesel Production from Jatropha oil using CaO generated as a catalyst from a snail shell.

2 MATERIALS AND METHODS

For this research, a snail shell was chosen. It was bought from the Kaduna South metropolis market of Ungwan Sunday in the state of Kaduna, Nigeria. To prevent differences in features, the sample was thoroughly chosen. The snail shell sample gathered before the experiment was clearly washed, cleaned and dried to remove the impurities associated with it, i.e. silica. Using jaw crusher, the snail shell sample was crushed. Using the Ball Mill grinding machine, the sample was gathered and ground. Mechanical agitator with distinct Tyler mesh arranged to achieve a particle size of 250 microns, 180 microns and 63 microns was used to collect and sieve the sample

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T. This methodology determines how the snail shell produces a catalyst. In this research, the optimum calcination parameters acquired from the research done by Birmah (2016) were used for peak snail shell transformation to the catalyst.

2.1 Calcination of the snail shell sample

The mass of 100 g of the snail shell sample of 63 microns was positioned on an empty crucible and weighed on an electronic weighing balance. It noted the weight of the empty crucible and recorded the weight of the crucible with the sample (A). The crucible containing the snail shell sample was loaded into a carbonite oven shown in Plate II and the temperature was set at 900 °C for 150 minutes and the sample was removed and put in a glass desiccator containing silica gel as a desiccant to cool down. The crucible containing the sample of the cold calcinated snail shell (B) was then reweighed and registered to determine the current weight.

2.2 Biodiesel manufacturing optimization from Jatropha Oil using Box-Behnken Design

The methodology provided in this chapter for producing Biodiesel from Jatropha Oil using Box Behnken design is Design Expert Model 12.

Experimental Design Matrix

The experimental matrix was constructed based on assigning the +/-Alpha value to the extreme points (operating window), see Table 1.0. The software allocated the high and low settings for the factorial points based on the specified extreme point values. This was to ensure that the modelling covering the widest possible range of operating window for the respective parameters could be carried out. Therefore, factorial point values were not adequately rounded.

Table 1.0: Operating conditions for trans esterification process parameters

Independent variables	Coded Levels		
	-1	0	+1
x_1 : Reaction Temperature (°C)	30	45	60
x_2 : Reaction Time (min)	60	120	180
x_3 : Calcined Snail shell Quantity (wt% of oil)	1	2.5	4

As shown in Table 1, the experimental matrix developed based on the design of the RSM Box-Behnken and the +/- values in Table 1. Each experimental run consisted of three samples as

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represented in Table 2 and the sequence of experiments following the randomly designated run number.

2.3 Production of biodiesel

The snail shell catalyst's catalytic activity was examined in a batch process by trans esterifying Jatropha oil with methanol. Conical flask, electric magnetic stirrer, and beaker were used to perform the reaction process. The method was performed by mixing a required quantity of catalyst and methanol followed by adding oil. The required temperature, time, and quantity of catalyst loading acquired from the above-mentioned experimental design were maintained. Finally, for biodiesel separation, the mixture was transmitted to the separating funnel. The set-up was left untouched for the separation of the phase. The lower phase (glycerol) was removed and the biodiesel-containing upper phase was analyzed for biodiesel yield calculation. The catalyst was removed and regenerated at the bottom. The biodiesel yield values generated using the experimental design in Table 3.5 were entered into the yield column.

Table 2: Experimental design of the BBD RSM

SN	Temperature (°C)	Time (min)	Amt of Catalyst (wt% of oil)	Yield (%)
1	30	120	1	
2	60	60	2.5	
3	45	60	4	
4	45	180	4	
5	60	180	2.5	
6	45	120	2.5	
7	45	180	1	
8	60	120	4	
9	45	120	2.5	
10	45	60	1	
11	45	120	2.5	
12	60	120	1	
13	30	120	4	
14	30	60	2.5	
15	30	180	2.5	

3 RESULTS AND DISCUSSION OF RESULTS

This section presents the results of the model relating to reaction time and temperature to biodiesel yield. The results of the choice of models and the significance of models are also shown.

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The developed mean and variance models show the optimization basis to determine the reaction temperature, catalyst loading and time that will maximize biodiesel output.

3.1 Biodiesel yield model

The experimental analysis of Table 2 was used to obtain the second order statistical model coefficients of Equation 1. The empirical model equations for the biodiesel yield were developed using experimental data in Table 4.4 and are described as Equation 1 below, as function of the reaction temperature, time and catalyst loading.

The uncoded biodiesel yield empirical model is shown in Equation 1

$$\begin{aligned} \text{Yield} = & -106.67611 + 2.20039T + 0.89542t + 42.79389C_a + \\ & - 0.006939Tt - 0.166889TC_a - 0.028667tC_a \\ & - 0.005456T^2 - 0.001767t^2 - 4.408778C_a^2 \end{aligned} \quad (1)$$

Where T is Temperature, t is time and C_a is Catalytic Amount

3.2 Analysis of variance for biodiesel yield model

The findings of Table 3 from ANOVA indicates that p-value of less than 0.05 are significant for the response factor of the biodiesel. The reverse elimination technique was used to test the model for the insignificant parameters. The ANOVA results from Table 3 indicates that reaction temperature (A), reaction time (B) and Catalyst amount (C) are significant and affect biodiesel production, as well as interaction terms (AB, AC and BC) and quadratic terms (A^2 , B^2 , C^2).

Table 3: Analysis of Variance (ANOVA) Table for Biodiesel Yield Response Surface Reduced Quadratic Model

Source	Sum of Squares	Df	Mean Squares	F-value	p-value	Remark
Model	3595.706	9	399.5229	39.11402	0.000417	Significant
A-Temperature	379.0881	1	379.0881	37.11342	0.001724	
B-Time	206.1465	1	206.1465	20.18212	0.006444	
C-Catalyst Amount	2341.332	1	2341.332	229.2207	2.28E-05	
AB	155.127	1	155.127	15.18722	0.011443	
AC	56.4001	1	56.4001	5.521672	0.065574	
BC	26.6256	1	26.6256	2.606695	0.167334	
A²	5.643006	1	5.643006	0.552461	0.490741	
B²	149.8812	1	149.8812	14.67364	0.01224	
C²	311.7531	1	311.7531	30.52119	0.002662	
Residual	51.07158	5	10.21432			

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Lack of Fit	51.07138	3	17.02379	170237.9	0.58745	not significant
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The statistical findings in Table 3 also shows that the model of biodiesel production established is significant because the value of the biodiesel lack of fit in relative to the pure error is 17.02. Thus, it is 5.8 % likely that noise will lead to an inadequate fitness value of 0.5874558 and template F-value of 170237.9. The insignificant lack of fit obtained for the empirical formula thus reveals that the model actually represents the experiment.

Table 4. Model Summary Fit Statistics for biodiesel Yield Response Surface Reduced Quadratic Model

Std. Dev.	R-Squared	Adj R-Squared	Pred R-Squared
3.20	98.60%	96.08%	77.59%

The *Predicted R²* of 0.7759 is in reasonable agreement with the *Adjusted R²* of 0.9608; i.e. the difference is less than 0.2. The 77.59% of the "predicted R²" of Table 4 corresponds with the 96.08% "Adjusted R²" of the noise signal ratio. In a low standard deviation value of 3.20 the model shows that design spacing can be predicted. Furthermore, as its "Prob> F" is of significance, the terms (A, B,C, AB, AC, BC, A², B²,C²) are of less than 0.0500 as described in Table 3. The primary residual analysis diagnostic tool was also studied. Figure 4.6 illustrates the normal probability plot of the residuals. Normally the data points are distributed as a straight line. The predictions of the model are right as the real values are based on the expected values in

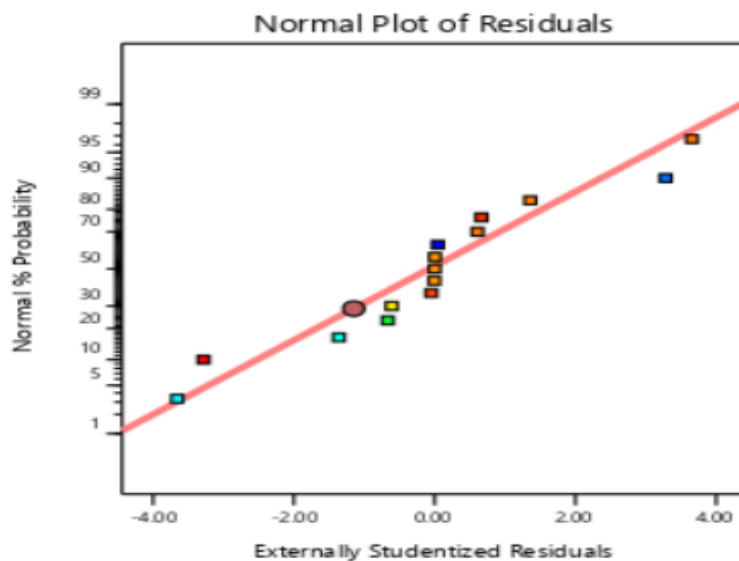


Figure 1: Normal Plot of Residual for Biodiesel Yield

Table 5: Model Report showing Actual and predicted value

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Run	Actual Yield	Predicted Yield	Residual
	44.42	41.36875	3.05125
2	81.01	79.9675	1.0425
3	80.6	78.59125	2.00875
4	83.5	83.58375	-0.08375
5	80.8	77.665	3.135
6	79.54	79.54	0
7	52.52	54.52875	-2.00875
8	86.3	89.35125	-3.05125
9	79.53	79.54	-0.01
10	39.3	39.21625	0.08375
11	79.55	79.54	0.01
12	61.52	62.64625	-1.12625
13	84.22	83.09375	1.12625
14	50.61	53.745	-3.135
15	75.31	76.3525	-1.0425

3.3 Response Optimization of Biodiesel Yield

The goal is to find optimum reaction-process variables that optimize the efficiency of biodiesel in the production process of biodiesel. For this necessity, the results of biodiesel production optimizing the reaction process variables are shown below.

3.4 Effect of process variables on biodiesel yield using the optimized Yield model

The 2-D contour plot and the 3-D yield model surface are shown in Figures 2 and 3. The contour and response surface plots are used to identify the relationship of each process-factor experimental levels with the biodiesel yield. The plot shows the effect of different parameters of the system while keeping the catalytic load at zero point constant.

Figures 2 and 3 display the quadratic reaction temperature variable and major reaction time influence on biodiesel yield, thus ignoring the catalyst loading and keeping it constant at zero. The plots show that increased biodiesel yield with increased response time and temperature values were observed. Increased biodiesel yield with increased reaction temperature and time values has been recorded in Figures 2 and 3. Further increase in the reaction temperature, however, was above the optimum value of 60 °C and below the reaction time of 104.85 min. The yield of biodiesel started to decrease when the reaction temperature was above 60 °C as it decreased below 104

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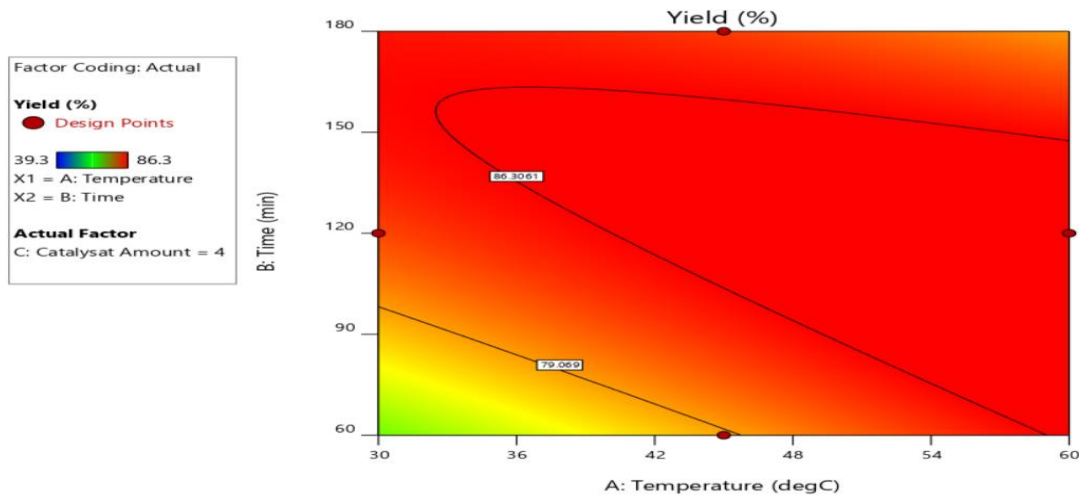


Figure 2: Model Contour Plot Showing the Effect of Reaction Temperature and Time on Biodiesel Yield for Simultaneous Optimization of Biodiesel Yield Model.

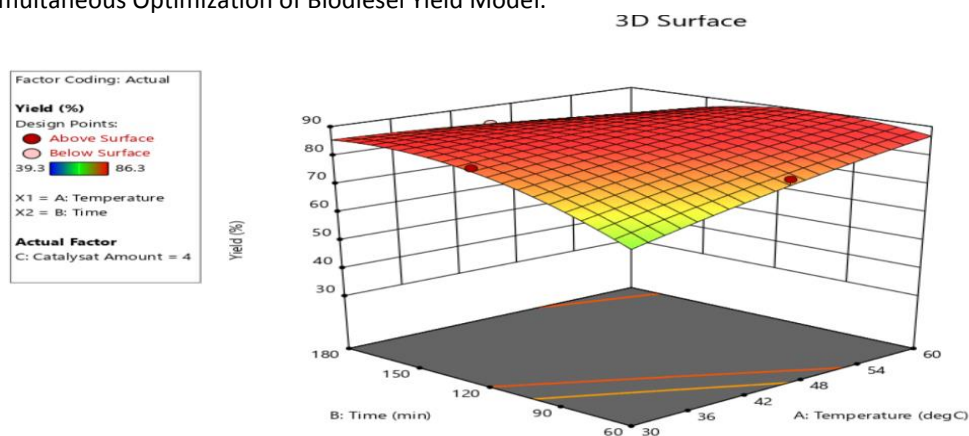


Figure 3: Mean Model Response Surface Plot Showing the Effect of Reaction Temperature and Time on Biodiesel Yield for Simultaneous Optimization of Biodiesel Yield Model.

3.5 Response Optimization of Biodiesel Yield Model

The Response Surface Optimization aims to find the appropriate operating conditions for process values that reach the goal. Figure 4 shows that an optimum biodiesel yield of 86.3 percent is achieved at 60 °C, a reaction time of 120 minutes, and a 4 catalysts amount. Suich and Derringer Approach criterion for optimization is desirable to be between 0 and 1.0. The value of desirability depends on how close the lower and upper limits are to the real maximum (Aggarwal et al., 2009). Optimizing the response to the development spacing constraints for biodiesel yield models using Design Expert has shown that the optimum solutions have been achieved at 0.76934 desirability as shown in figure 4

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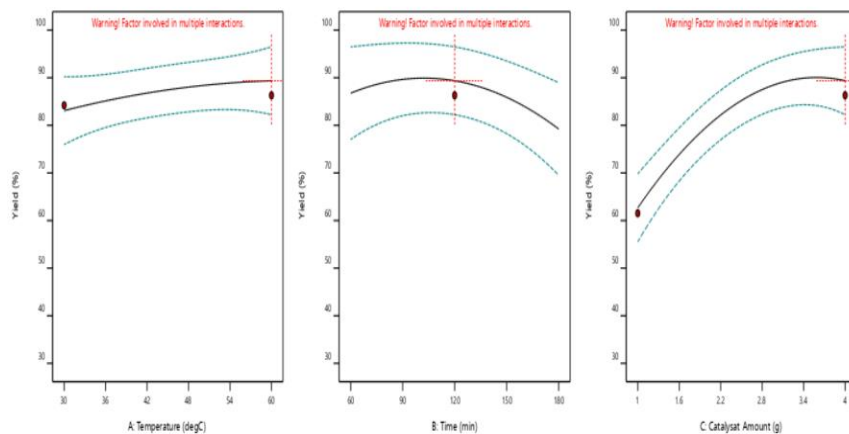


Figure 4: Response Surface Plot Showing the Effect of Reaction Temperature, Time and Catalyst Loading on Biodiesel Desirability for Response Optimization of Biodiesel Yield Model.

3.6 Performance Characterization of the Biodiesel Produced

The biodiesel produced has been characterized at Kaduna's Spectral Laboratory Services and the results are shown in Table 4.8. The result corresponds to the acceptable biodiesel standard limits by (ASTMD975). The cloud point ($-4\text{ }^{\circ}\text{C}$) and pour point ($-12\text{ }^{\circ}\text{C}$) of the produced biodiesel are within the limit set for (ASTMD975) biodiesel standard (-15 to $-5\text{ }^{\circ}\text{C}$) and (-35 to $-15\text{ }^{\circ}\text{C}$) respectively. The pour point is the temperature below which the fuel will not flow. The flash point is one of the important characteristics of any fuel; it is defined as the lowest temperature at which an inflammable air mixture can be produced. According to ASTM D 975 biodiesel standards (60 - $80\text{ }^{\circ}\text{C}$), biodiesel has a higher flash point of $157\text{ }^{\circ}\text{C}$. The biodiesel product is therefore safe for use and handling. Fuels with lower flash points often appear to fire at lower temperatures, which makes them highly dangerous if not properly stored and used. The amount of cetane is another important characteristic of fuel and is an indicator of the combustion value of diesel during the ignition of compression. Due to the number of cetane, the ignition quality affects engine performance, cold starting, warm up and engine combustion roughness. The volatility of fuel in which more volatile carburant have a higher rating is associated with cetane Rating. A high cetane fuel can also lead to incomplete combustion and smoking if the fuel ignites too soon, as fuel cannot be mixed with air for total combustion. Normally, diesel motors operate at values of between 45 and 55 and the biodiesel generated is 44.69 , within the range of ASTM D975 biodiesel (40 - 55 , for use in diesel motors). The biodiesel output is therefore suitable for use with diesel motors. The viscosity of the fuel is a measure of resistance to fluid flow due to the internal frictions of the portion of a liquid flowing over the other. Knothe et al. (2005) reported another important value; this is a very important property in fuels. The viscosity of a fuel is important because the fuel is atomised in the combustion chamber of the engine.

Table 5: Performance Characteristic of Biodiesel produced

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SN	Properties	Produced Biodiesel	ASTMD 975
1	Biodiesel Yield (%)	86.3	
2	Specific Gravity	0.86	0.85
3	Density (g/cm ³)	0.873	0.82 – 0.845
4	Kinematic Viscosity (mPaS)	3.1	2 – 3
5	Iodine Value	106	18.5
6	Flash Point (°C)	157	60 – 80
	Cloud Point (°C)	-4	-15 – -5
8	Pour Point (°C)	-12	-35 – -15
9	Refractive Index	1.446	1.665
10	Cetane Number	50.7	40-55
11	Saponification Value	193	NS
12	Acid Value	0.4488	< 0.50
13	Free Fatty Acids (FFA)	0.2244	NS
14	Higher Heating Value (HHV)	39.927	39 – 43.33

A small drop of fuel is desired to allow for complete combustion. In an engine combustion chamber, high viscosity fuel will generate a larger drop of fuel which cannot be cleaner like fuel that produces smaller drops. The motor will build up unburned oxidized fuel around valves, injector tips, and on the sidewalls and rings of a piston. The fuel is likely to cause problems such as engine part obstruction due to higher viscosity. However, the viscosity of the produced biodiesel is slightly higher (3.09mPaS) than that provided for in ASTM biodiesel standards (2-3 mPaS). The key factor for good motor efficiency is the density of a fuel: the higher the density the harder it gets to pump the oil therefore the density obtained is 0.837 which is within the standard range (0.82 – 0.845). The specific gravity of the produced biodiesel is 0.86 which is within the standard range (0.85).

4 CONCLUSION AND RECOMMENDATION

This study showed that the calcined snails shell has been characterized, biodiesel production has been optimized with the Box-Behnken Model and biodiesel derived from optimum parameters obtained from optimized models has been characterized. The Response Surface Optimization results for Box-Behnken Design showed that the optimum biodiesel yield of 90.4274% was achieved at the reaction temperature of 60 °C, 105.256 minutes reaction time and 3.64074(wt% of Oil) mass of calcined snail shell. Biodiesel performance characterization is adequate, because their characterized properties are relatively close to the ASTM D975 biodiesel standards, using the best design parameters.

However, it is recommended that, the cetane number of the biodiesel obtained should be experimentally evaluated and additional research should be done regarding biodiesel blends with petro diesel.

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