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Modeling and Optimization of CN and HHV of *Parinari polyandra* Biodiesel Using Response Surface Methodology

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ABSTRACT

Transesterification process parameters can influence the quality of biodiesel produced from seed oils. The effects of selected process parameters were studied on the Cetane Number (CN) and Higher Heating Value (HHV) of *Parinari polyandra* biodiesel were studied using Response Surface Methodology (RSM). A two-level Box-Behnken Design (BBD) of RSM was applied to model and optimize the transesterification reaction for best CN and HHV. The study parameters, temperature (60 – 75 ^{o}C), time (60 – 180 minutes), and catalyst concentration (1 – 3 wt%) were studied in a seventeen experimental run using ethanol as the alcohol. The fuel properties, CN and HHV were found to be within the ASTM limits and very similar to that of diesel, making the biodiesel suitable for engine applications. The best CN and HHV values, which correspond to the highest values of 65.5 and 46.2 MJ/kg, were obtained experimentally from reaction with applied parameters of 67.5 ^{o}C temperature, 60 minutes reaction time, and 1 wt% of catalyst concentration. Optimized reaction conditions were validated to obtain optimal quality values of 65.3 and 46.5 MJ/kg for CN and HHV under experimental conditions set of 62.25 ^{o}C temperature, 61.15 minutes reaction time, and 1.58 wt%. The study presents the capability of RMS in predicting important fuel qualities of *Parinari polyandra* biodiesel, which are mostly needed for better performance and combustion in diesel engine applications,
.

Keywords: Optimization, higher heating value, transesterification, response surface methodology. cetane number

Aims Research Journal Reference Format:
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1. INTRODUCTION

Optimization and predictive studies using intelligent and robust algorithms can be employed to achieve maximum biodiesel production from oil extracted from seed bearing plants. Optimization is a powerful tool which is being used in process and control integration for the development of simulations and predictions that best satisfy a solution for maximum response function [1]. Optimization of biodiesel production process has been carried out in many ways for the purpose of achieving high and quality biodiesel yields at moderate reaction conditions [2]–[11]. In order to meet up with high demands for biodiesel fuel, both local and industrial production of biodiesel must be improved for high and quality biodiesel yields. Different researchers have shown that the properties of non-edible oil biodiesel may

vary significantly depending on their chemical compositions and fatty acid compositions, which give obvious effect on engine performance and emissions [12]–[15]. Therefore, when considering a specific non-edible biodiesel, a measurement of its properties is required. Even though biodiesel has similar physical and chemical properties with fossil diesel fuel, biodiesel properties can sometimes be superior because the former has higher flash point, low sulphur concentration, better lubricating efficiency, and better cetane number [16]–[18]. The ASTM standards for biodiesel comparison with petroleum-based diesel is shown in Table 1.

The physicochemical (fuel) properties of biodiesel are similar to those of fossil diesel fuels. Biodiesel fuels are characterized by their cetane number, higher heating value (HHV), viscosity, cloud and pour points, flash point, copper corrosion, ash content, distillation range, sulfur content, carbon residue, acid value, free glycerine content, total glycerine content and density etc. [19]. A thorough study of literatures has shown scientifically that parametric studies of process parameters can influence the quality and quantity of responses. Though the fuel quality seems to be the focus of this study, not all the chemical properties of the biodiesel were taken into consideration. The Cetane Number (CN) and Higher Heating Value (HHV), owing to their measure of the fuel's relative combustion index, were considered as measurable responses to assess the suitability of the biodiesel as recommended biofuel for engine applications. Obtaining the best properties from the process optimization would help in determining the process conditions under which these fuel qualities can be obtained best.

The CN is a measure of ignition quality of diesel fuels and is equivalent to the percentage by volume of cetane in the blend with heptamethylnonane, which matches the ignition quality of the test fuel (ASTM D-613) [20]. The CN is depicted as a dimensionless numerical value which indicates the ignition quality of a fuel and majorly considered for selection of biodiesel as an engine fuel [21] [22] [23]. The ignition quality of diesel fuels refers to their comparative ease to get ignited when they come into contact with compressed air in diesel cylinders [21]. The standard methods for measuring CN are ISO 5165:2017 (Petroleum products – Determination of the ignition quality of diesel fuels – Cetane engine method), ASTM D 613 (Standard Test Method for Cetane Number of Diesel Fuel Oil) and European standard DIN 16906:2017, which was formerly known by 51773:2010.

Table 1: Comparison of ASTM standards for diesel and biodiesel

Property	Diesel	Biodiesel
Standard number	ASTM D975	ASTM D6751
Composition	Hydrocarbon (C10–C21)	Fatty acid methyl ester (C12-C22)
Specific gravity (g/mL)	0.85	0.88
Flash point (K)	333–353	373–443
Cloud point (K)	258–278	270–285
Pour point (K)	243–258	258–289
Carbon (wt. %)	87	77
Water (Vol. %)	0.05	0.05
Cetane number	40–55	48–60
Sulphur (wt. %)	0.05	0.05
Hydrogen (wt. %)	13	12
Oxygen (wt. %)	0	11

Source: Helwani et al. [24]

Ignition quality of a fuel can either be measured in term of octane or cetane number. The ignition quality is a measure of the relative ease by which the fuel will ignite. Fuels which have high ignition quality are identified as fuels with short ignition delay, whereas fuels with low ignition quality have long ignition delay. The ignition delay time refers to the time between a start of fuel injection and fuel combustion. The ignition delay time is strongly dependent on engine conditions like pressure and temperature of the chamber into which the fuel is injected. An increase in the CN causes a shorter ignition delay, which has the effect of less fuel being injected during the premix burn and more during the diffusion burn portion, thus reducing the cylinder pressure rise, which may result in lower cylinder temperature. Conversely, the ignition timing of a lower cetane fuel is advanced because of the longer ignition delay, thereby increasing the combustion pressures and temperatures [25].

The Higher Heating Values (HHVs) is an important parameter used to quantify the energy content and combustion efficiency of fuels [26]. The standard measure of the energy content of a fuel is its HHV. The Higher Heating Values (HHVs) of biodiesels are relatively high as a result of high oxygen content but slightly lower than that of diesel. The oxygen content of biodiesel improves the combustion process and decreases its oxidation potential. The oxygen content of a fuel improves its combustion efficiency due to a rise in the uniformity of oxygen with the fuel during combustion [26]. The oxygen content of biodiesel generally varies between 10 wt% to 12 %wt depending on the degree of oxygenation of the feedstock [27]. Highly saturated oil, like *Parinari polyandra*, is more oxygenated and burns cleaner and stable when used as feedstock for biodiesel production.

Several studies have been conducted on biodiesel production to predict the yield response using RSM. Most of these studies are limited to transesterification method where the reaction variables affecting biodiesel yields are studied in different randomized experimental designs. The literature is scarce of robust information on prediction and optimization studies involving the quality of biodiesel produced from seed oil. There is a need to determine the effect of process parameters on the quality of the biodiesel obtained from transesterification of *Parinaric polyandra* oil to provide necessary information towards establishing the biofuel as a reliable quality alternative fuel. In this work, the optimization of transesterification process of *Parinari polyandra* oil for quality yield is reported following an earlier which focused on the optimization of biodiesel production from the oil. The effect of transesterification parameters on two selected combustion characteristics of *Parinari polyandra* biodiesel is reported and the process parameters are optimized for better quality biodiesel production from the oil.

2. METHODOLOGY

The fruits of fresh *Parinari polyandra* were harvested and decorticated to remove the fresh kernels. The seeds were dried under atmospheric temperature and milled to particle sizes. Oil was extracted from the milled seeds using solvent extraction method as reported by Ogunkunle and Ahmed [9]. Biodiesel was produced using standard alkaline transesterification process based on preliminary experimental studies [9], [28], [29]. The CN and HHV of *Parinari polyandra* biodiesel were studied, as measured from a seventeen box-behnken design (BBD) experimental runs, in order to study the effect of transesterification reaction parameters on these fuel combustion properties and optimize them under moderate reaction parameters. The experimental design was based on BBD of response surface methodology of the commercial package, Design-Expert 12 (Stat-Ease Inc, Minneapolis, USA).

Three process variables, temperature, time, and catalyst concentration were varied between low- and high-level experimental values of 60 °C, 60 minutes, 1 wt% and 75 °C, 180 minutes, 3 wt%, respectively. These were designated as coded variables A, B, and C. The summary of the experimental design is presented in Table 1.

2.1 Oil and extraction and biodiesel production from *Parinari polyandra* seeds

The oil extraction and biodiesel production were done exactly as reported by Ogunkunle and Ahmed [9]. A soxhlet system was used for extracting oil from the milled *Parinari polyandra* seeds. N-hexane was used as the extracting solvent in the soxhlet extraction system. The extraction conditions employed were 0.06 g/ml seed/solvent (g/ml), 70 °C extraction temperature and extraction time of 5 hrs. The properties of the extracted *Parinari polyandra* oil were determined using standard test methods of the Association of Official Analytical Chemists (AOAC). Biodiesel production from *Parinari polyandra* oil was carried out using the alcoholysis procedures reported by Ogunkunle and Ahmed [9]. The HHV and CN were determined using the ASTM D2015 and D613 methods respectively.

2.2 Statistical analysis and optimization

Seventeen (17) experimental runs, generated from a three-factor two-level Box-Behnken design of RSM, were used to produce biodiesel samples from varied experimental runs. Temperature, time and catalyst amount, were represented as independent variables A, B, and C, respectively. The two responses are CN and HHV.

The summary of the coded design factors and their levels are shown in Table 2. A second order polynomial equation was developed to model the CN and HHV properties of the biodiesel. The responses, CN and HHV, were predicted to obtain the best model for obtaining quality CN and HHV properties. The multiple regression analysis was used to investigate the significance effect of the three reaction variables on CN and HHV. The goodness of the fit of the model was evaluated using Analysis of Variance (ANOVA). Numerical optimization was done to find the best magnitude of variables that fulfil the criteria placed on the numeric factors and the response. The factor space was set in range between desirable limits in order to achieve optimum combination of conditions that satisfy the desired qualities.

Table 2: Design summary for factors and their levels for the biodiesel production

Factor	Name	Units	Type	Coded Low	Coded High	Mean	Std. Dev.
A	Temperature	Deg. C	Numeric	-1 ↔ 60.00	+1 ↔ 75.00	67.50	5.30
B	Time	minutes	Numeric	-1 ↔ 60.00	+1 ↔ 180.00	120.00	42.43
C	Catalyst amount	wt%	Numeric	-1 ↔ 1.00	+1 ↔ 3.00	2.00	0.7071

3. RESULTS AND DISCUSSION

The results and statistical analyses of CN and HHV determined from each experimental run are presented under this section. The interactivity plots among reaction parameters on CN and HHV are plotted and discussed. Optimized reaction variables and results from validation experiments for quality CN and HHV properties are reported as well.

3.1 Oil and biodiesel yields from *Parinari polyandra*

An average value of 57.75% of oil was obtained from the oil extraction process. This is quite similar to a yield of 64% obtained by Afolabi et al. [30] from the optimization of oil extraction from *Parinari polyandra* seeds. The biodiesel yields obtained ranged from 72.49 to 95.62%. The results of the biodiesel production from each experimental run can be found in the reports of Ogunkunle and Ahmed [9].

3.2 CN and HHV properties of *Parinari polyandra* biodiesel

The results of the CN and HHV obtained from each experimental run are presented in Table 3. Comparing the CN and HHV of fossil diesel, which is usually between 40 – 52 and 44.80 – 45.6 MJ/kg, with those obtained from the experimental showed that HHVs of the biodiesel fuels are very good owing to the closeness to that of diesel.

Table 3: CN and HHV measured from transesterification of *Parinari polyandra* oil

	Factor 1	Factor 2	Factor 3	Response 1		Response 2	
Run	A: Temperature	B: Time	C: Catalyst amount	CN	Predicted value	HHV	Predicted value
	Deg C	minutes	wt%			MJ/kg	
1	60	120	3	65.3	65.30	45.2	45.07
2	75	120	3	65.2	65.22	44.8	45.00
3	60	60	2	65.4	65.44	45.8	46.09
4	67.5	180	1	65.3	65.34	45.8	45.96
5	67.5	120	2	65.5	65.48	46.1	45.94
6	75	180	2	65.4	65.36	46.1	45.81
7	75	60	2	65.2	65.21	44.9	44.86
8	75	120	1	65.2	65.20	44.8	44.92
9	67.5	180	3	65.5	65.51	45.9	45.99
10	60	180	2	65.4	65.39	45.2	45.24
11	67.5	60	1	65.5	65.49	46.2	46.11
12	67.5	120	2	65.5	65.48	45.7	45.94
13	60	120	1	65.4	65.37	45.7	45.50
14	67.5	120	2	65.5	65.48	46.1	45.94
15	67.5	60	3	65.3	65.26	45.9	45.74
16	67.5	120	2	65.4	65.48	45.8	45.94
17	67.5	120	2	65.5	65.48	46	45.94

3.2.1 Response surface regression models for CN and HHV

The statistical summary for the two measured responses is presented in Table 4. The ANOVA model values for the measured CN are shown in Table 5. Model F-value of 9.49 and low probability value of 0.0036 show that the model is significant.

Table 4: Statistical summary for the responses

Response	Name	Units	Runs	Analysis	Min	Max	Mean	Std. Dev.	Ratio	Model
R1	HHV	MJ/kg	17	Polynomial	44.8	46.2	45.65	0.4771	1.03	Quadratic
R2	Cetane number		17	Polynomial	65.2	65.5	65.38	0.1131	1.00	Quadratic

Table 5: ANOVA for Quadratic model for CN

Source	Sum of Squares	df	Mean Square	F-value	p-value	Comment
Model	0.1892	9	0.0210	9.49	0.0036	significant
A-Temperature	0.0312	1	0.0312	14.11	0.0071	
B-Time	0.0050	1	0.0050	2.26	0.1766	
C-Catalyst amount	0.0013	1	0.0013	0.5645	0.4769	
AB	0.0100	1	0.0100	4.52	0.0712	
AC	0.0025	1	0.0025	1.13	0.3233	
BC	0.0400	1	0.0400	18.06	0.0038	
A^2	0.0684	1	0.0684	30.91	0.0009	
B^2	0.0000	1	0.0000	0.0119	0.9162	
C^2	0.0253	1	0.0253	11.42	0.0118	
Residual	0.0155	7	0.0022			
Lack of Fit	0.0075	3	0.0025	1.25	0.4028	not significant
Pure Error	0.0080	4	0.0020			
Cor Total	0.2047	16				

There is only a 0.36% chance that an F-value this large could occur due to noise. P-values less than 0.0500 indicate model terms are significant. In this case A, BC, A^2, C^2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. The Lack of Fit F-value of 1.25 implies the Lack of Fit is not significant relative to the pure error. There is a 40.28% chance that a Lack of Fit F-value this large could occur due to noise. Non-significant lack of fit is good for the fitness of the model. The R-square and Adjusted R-square values were given as 0.9243 and 0.8269 respectively, which shows the acceptability of the regression model. A ratio of 8.659 Adequate Precision indicates an adequate signal. Statistically, a ratio greater than 4 is desirable. This model can be used to navigate the design space. The final quadratic model for CN in terms of coded factors is given in Equation 1.

$$CN = +65.48 - 0.0625A + 0.025B - 0.0125C + 0.05AB + 0.025AC + 0.1BC - 0.1275A^2 - 0.0025B^2 - 0.0775C^2 \quad (1)$$

The significance and adequacy test of the model showed that the main effects were due to the coded terms, A, AB, BC, A^2, C^2, with significance values less than 0.05. The regression model equation indicated that temperature was the most significant model term with more significant probability values of individual, interactive and quadratic effect on CN model. The normal probability plots of residuals obtained from *Parinari polyandra* biodiesel CN analysis is shown in Figure 1. The diagnostics studies showed CN values had insignificant residual values of 0.0000, -0.0250, -0.0375, -0.0375, 0.0200, 0.0375, -0.0125, 0.0000, -0.0125, 0.0125, 0.0125, 0.0200, 0.0250, 0.0200, 0.0375, -0.0800, and 0.0200 from the first to the seventeenth experimental run in the standard order. This confirmed the validity of the predicted model in covering the design space.

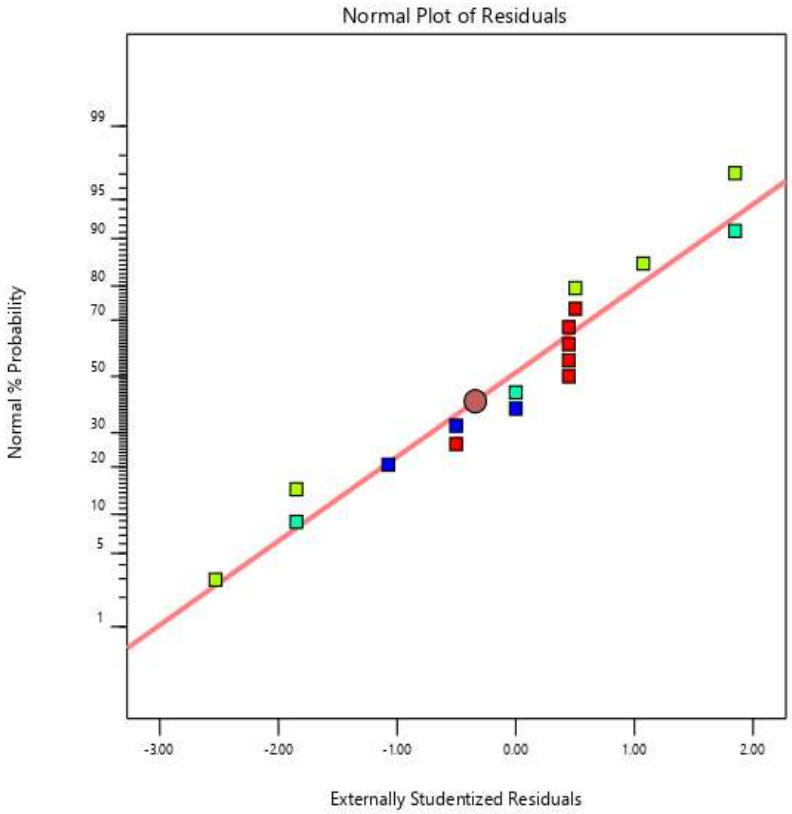

Figure 1: Normal probability plot of residuals for CN obtained from response data analyses

The ANOVA model values for the measured HHV are shown in Table 6. The Model F-value of 5.13 and a probability value of 0.0212 imply that the model is significant. There is only a 2.12% chance that an F-value this large could occur due to noise. P-values less than 0.0500 indicate model terms are significant. In this case AB and A^2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. The Lack of Fit F-value of 3.51 implies the Lack of Fit is not significant relative to the pure error. There is a 12.83% chance that a Lack of Fit F-value this large could occur due to noise. Non-significant lack of fit is good for the fitness of the model. R-square and Adjusted R-square values were calculated as 0.8684 and 0.6991. A ratio of 6.227 Adequate Precision indicates an adequate signal. Statistically, a ratio greater than 4 is desirable.

This model can be used to navigate the design space. The model equation for HHV in terms of coded factors is given in Equation 2.

Table 6: ANOVA for Quadratic model for HHV

Source	Sum of Squares	df	Mean Square	F-value	p-value	Comments
Model	3.16	9	0.3514	5.13	0.0212	significant
A-Temperature	0.2113	1	0.2113	3.08	0.1225	
B-Time	0.0050	1	0.0050	0.0730	0.7948	
C-Catalyst amount	0.0613	1	0.0613	0.8942	0.3758	
AB	0.8100	1	0.8100	11.82	0.0109	
AC	0.0625	1	0.0625	0.9124	0.3713	
BC	0.0400	1	0.0400	0.5839	0.4697	
A^2	1.68	1	1.68	24.59	0.0016	
B^2	0.1560	1	0.1560	2.28	0.1750	
C^2	0.1402	1	0.1402	2.05	0.1956	
Residual	0.4795	7	0.0685			
Lack of Fit	0.3475	3	0.1158	3.51	0.1283	not significant
Pure Error	0.1320	4	0.0330			
Cor Total	3.64	16				

$$HHV = +45.94 - 0.1625A + 0.025B - 0.0875C + 0.45AB + 0.125AC + 0.1BC - 0.6325A^2 + 0.1925B^2 - 0.1825C^2 \quad (2)$$

The significance and adequacy test of the model showed that the main effects were due to the coded terms, AB and A^2, with significance values less than 0.05. The regression model equation indicated also that temperature had more significant effect on HHV model as measured by the low probability values of interactive and quadratic model terms. The normal probability plots of residuals obtained from *Parinari polyandra* biodiesel HHV analysis is shown in Figure 2. The diagnostics studies showed HHV values had insignificant residual values of 0.1250, -0.2000, -0.2875, -0.1625, 0.1600, 0.2875, 0.0375, -0.1250, -0.0875, -0.0375, 0.0875, -0.2400, 0.2000, 0.1600, 0.1625, -0.1400, and 0.0600 from the first to the seventeenth experimental run in the standard order. This confirmed the predicted model is valid in covering the design space.

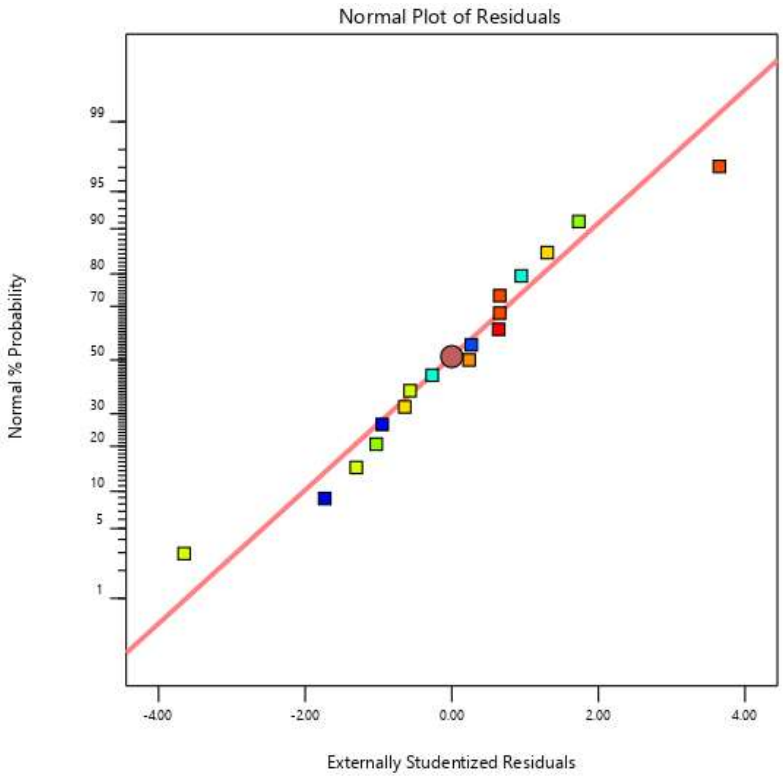

Figure 2: Normal probability plot of residuals for HHV obtained from response data analyses

3.2.2 Influence of reaction parameters on CN and HHV

Three dimensional surface plots, as shown in Figure 3 and 4, were plotted from the model equations to show the interactivity among the transesterification parameters on CN and HHV. The interactivity between temperature and time, temperature and catalyst concentration, and time and catalyst concentration on CN is shown in Figure 3.

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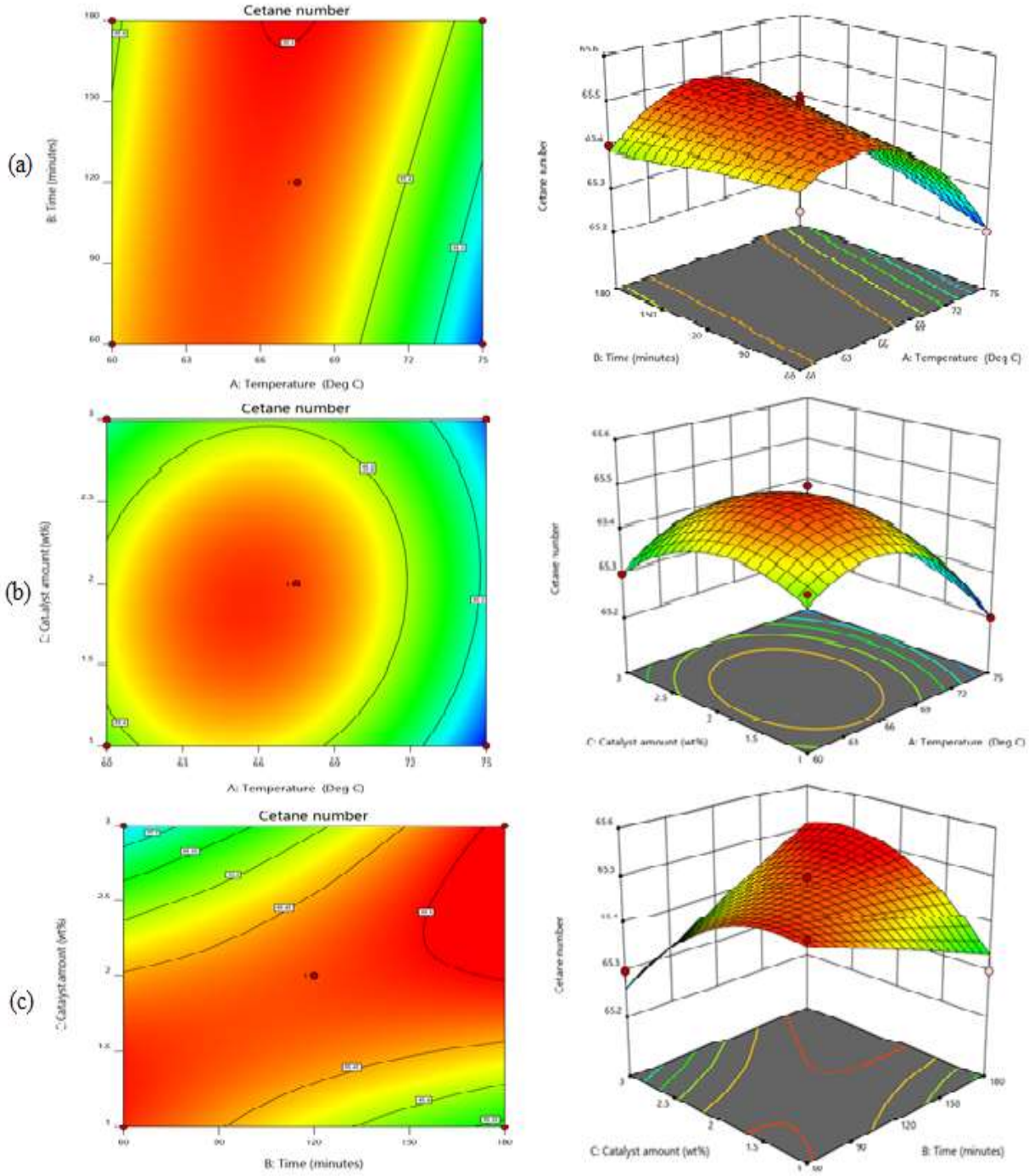

Figure 3
(a): Contour and 3D plots of interactive effect of temperature and time on CN
(b): Contour and 3D plots of interactive effect of temperature and catalyst amount on CN
(c): Contour and 3D plots of interactive effect of time and catalyst amount on CN

Considering the interactive effect of temperature and time, the CN was found to increase with increasing transesterification reaction temperature and time. Higher CN values were relatively obtained at moderate temperature of 65.5 ^{o}C and higher reaction time of 180 minutes. Reaction temperatures beyond 65.5 ^{o}C showed a decline in CN values, which was more visible at low reaction time. These results indicated that moderate reaction temperatures, which is quite lower than the boiling point of ethanol, and higher reaction time are desirable to obtain higher CN values from alcoholysis of *Parinari polyandra* oil.

A similar trend was observed for temperature and catalyst amount in their interactive effect on CN. An appreciable increase was observed from low level until medium reaction values while further increases in temperature and catalyst amount led to decrease in CN values. Reaction variables which consist of temperature and catalyst amount above 67.5 ^{o}C and 2 wt% respectively appeared to be the critical points which combinedly led to reduction of CN.

This relatively moderate temperature requirement favours a more economical transesterification process when considering heating costs amongst other costs. The CN values appeared to increase continuously as the reaction time increases.

This demonstrated that there was room for complete conversion of *Parinari polyandra* triglycerides to quality biodiesel, as long as the reaction variables (temperature and catalyst amount) proceed at moderated levels. Although, from the experimental results and analyses, high CN hovers around 65.5 from reaction variables of 67.5 ^{o}C temperature, 180 minutes reaction time and 3 wt% catalyst concentration. Considering a cost-effective transesterification reaction, this same high CN value was obtained at 67.5 ^{o}C temperature, 120 minutes reaction time and 2 wt% catalyst concentration, as evident from the results obtained (Table 3).

The interactivity between temperature and time, temperature and catalyst concentration, and time and catalyst concentration on HHV shown in Figure 4. The HHV was found to increase as temperature increase and then decrease at higher temperature values beyond 67.5 ^{o}C. The reaction time was found to a positive increasing effect on the HHV, with a slight decline midway as depicted by the elliptical curve. The HHVs were quite high as the reaction progressed but showed a declining curve until 120 minutes reaction time.

Further increases in HHVs were observed as the reaction progressed till 180 minutes, where the highest HHV was obtained. The elliptical nature of the curve shows that the reaction variables had appreciable significant effects on HHV [9]. A noticeable increase in HHV was obtained at temperature of 67.5 ^{o}C, which was more profound as the reaction time approached 180 minutes.

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A Multidisciplinary & Interdisciplinary Journal
Volume 7, No 1, March 2021 Series

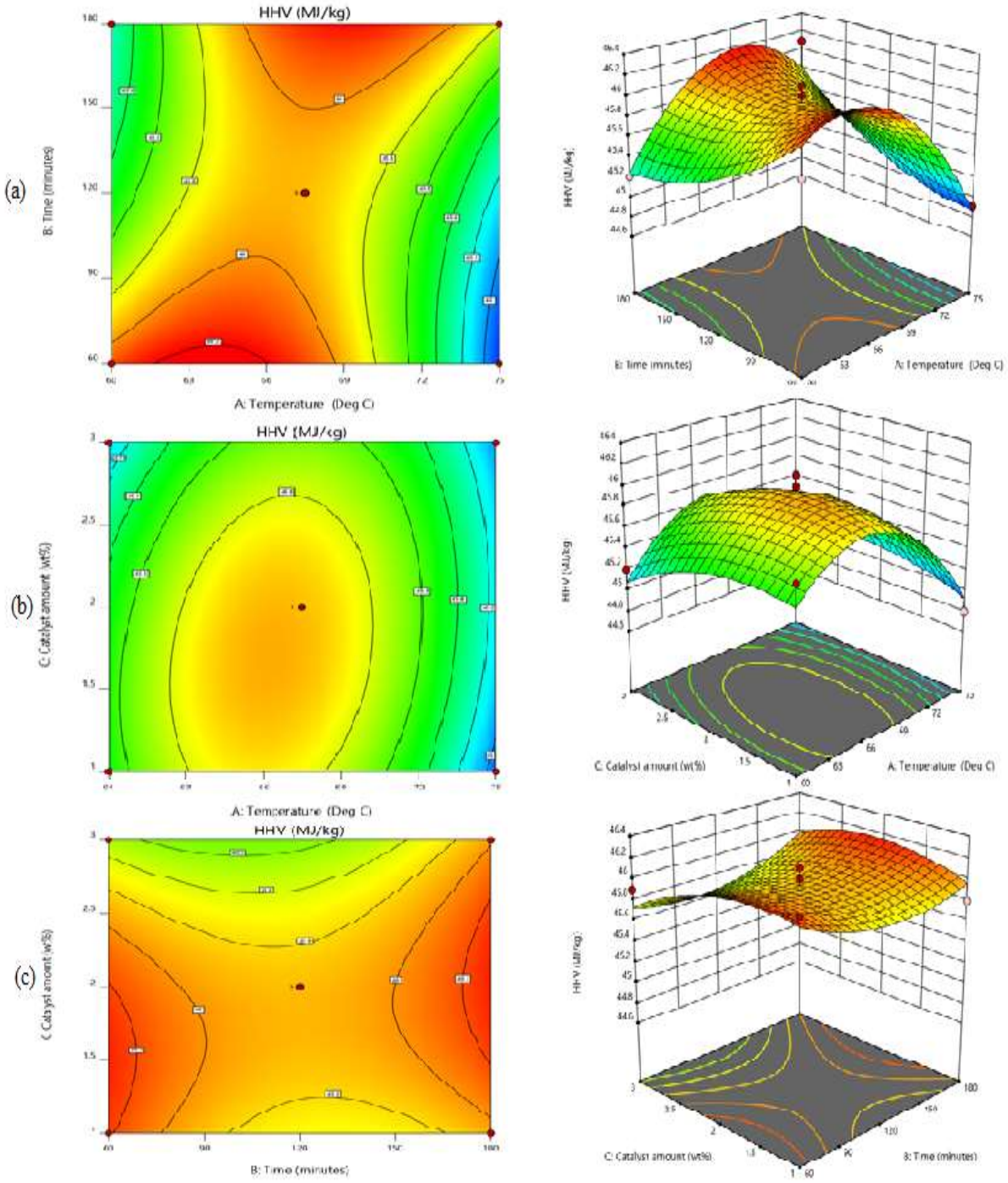

Figure 4
(a): Contour and 3D plots of interactive effect of temperature and time on HHV
(b): Contour and 3D plots of interactive effect of temperature and catalyst amount on HHV
(c): Contour and 3D plots of interactive effect of time and catalyst amount on HHV

The HHV was found to increase gradually as the catalyst concentration increased and suddenly decreased as the concentration increased beyond 2 wt%. This can be explained by the undulating curve of the catalyst concentration on HHV. At optimal value of 2 wt% of catalyst amount, high values of HHV was obtained at temperature of 67.5 ^{o}C and reaction time of 120 minutes. Any increase beyond these values, with exception of higher reaction time, led to reduction in HHV.

3.2.3 Optimization and validation of *Parinari polyandra* CN and HHV

The plots for desirability and point prediction of reaction conditions on HHV and CN are shown in Figures 5, 6, and 7. Numerical optimization of the experimental data produced different solutions, out of which the most fitting reaction conditions with the best desirability and highest CN and HHV was selected. Values of 65.51 and 46.29 MJ/kg were statistically optimized for CN and HHV from a combination of 65.25 ^{o}C reaction temperature, 61.15 minutes reaction time, and 1.58 wt% catalyst amount. A desirability level of 1.00 shows the effectiveness of the chosen solution. Measurements of CN and HHV from the validation experiments gave average values of 65.3 and 46.5 MJ/kg for CN and HHV respectively. The values obtained from the validation experiments have proved to be close to the statistically optimized quality parameters, which confirms the validity of the models for predicting the biodiesel quality. These two fuel properties of *Parinari polyandra*, have also shown that the raw oil is a very stable feedstock for biodiesel production, whose properties remain stable under moderate reaction conditions.

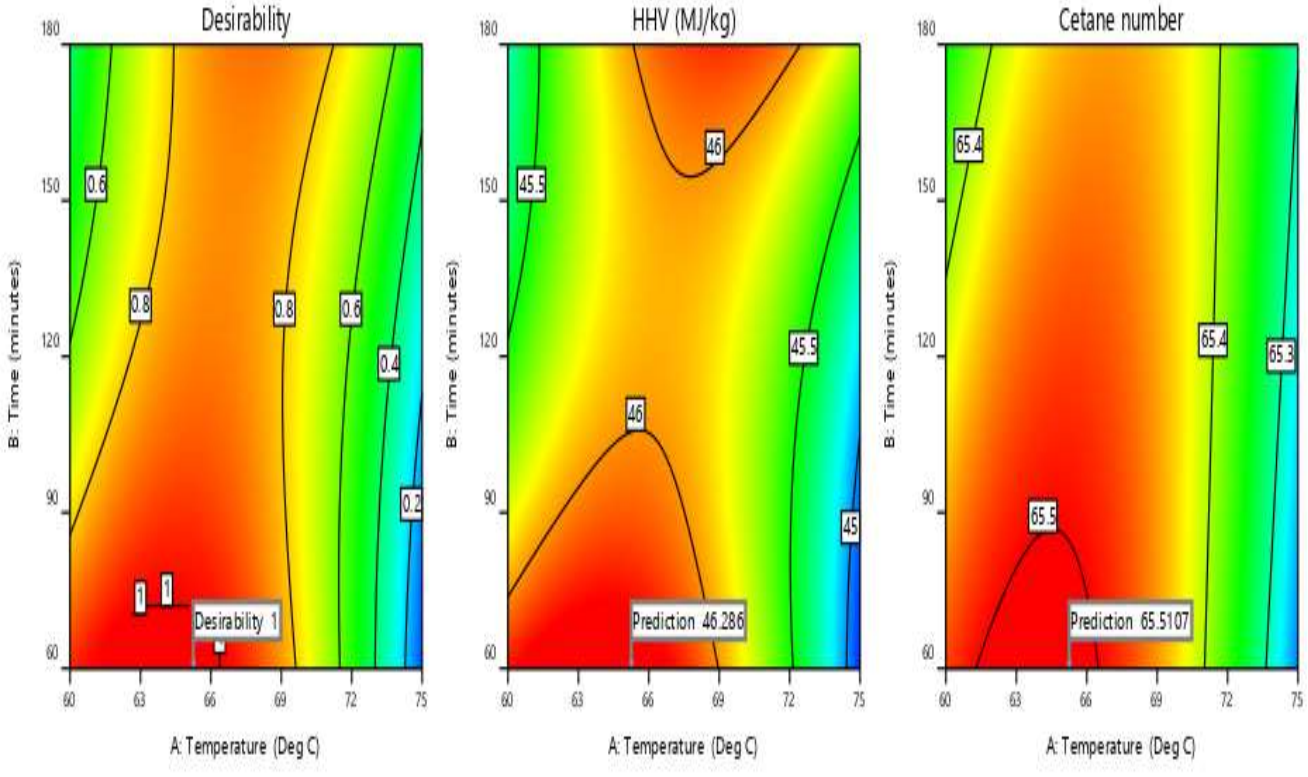

Figure 5: Desirability and point prediction of temperature and time interactivity on HHV and CN

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A Multidisciplinary & Interdisciplinary Journal
Volume 7, No 1, March 2021 Series

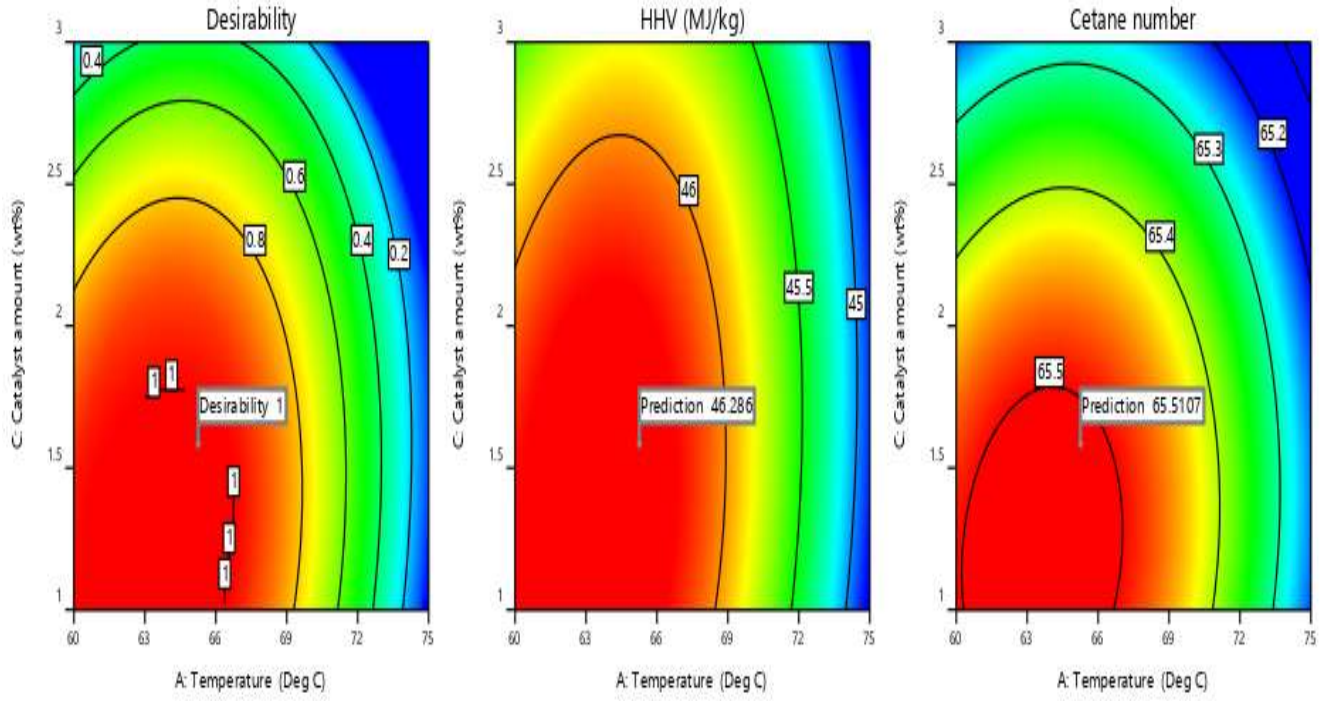

Figure 6: Desirability and point prediction of temperature and catalyst amount interactivity on HHV and CN

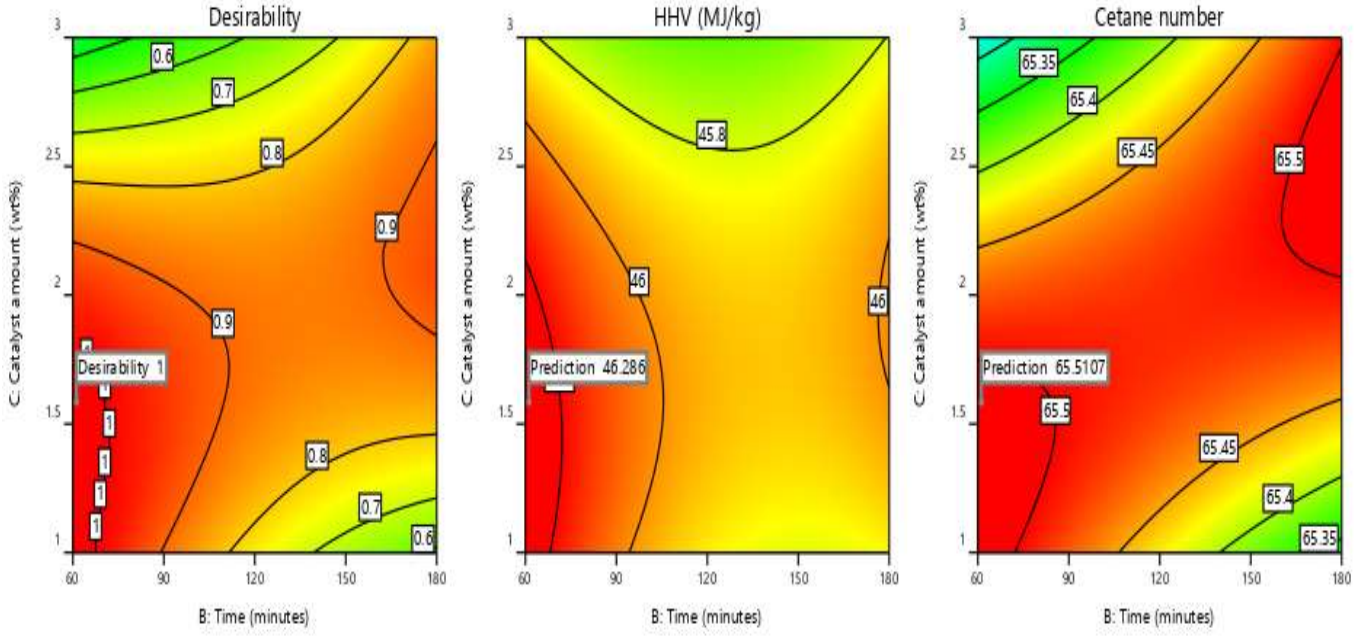

Figure 7: Desirability and point prediction of time and catalyst amount interactivity on HHV and CN

4. CONCLUSION

From the results obtained, the following conclusions can be drawn:

- Transesterification parameters were found to have varying significant, but very close, effects on the CN and HHV.
- At 95% confidence interval, the transesterification reaction temperature had the most significant effect on both the CN and HHV. The optimum conditions for achieving the desired CN and HHV were obtained at reaction temperature of 65.25 ^{o}C, reaction time of 61.15 minutes, and catalyst amount of 1.58 wt%.
- The CN and HHV of *Parinari polyandra* biodiesel were found to be within the ASTM D6751 standard for biodiesel usage as fuel. The biodiesel obtained will be viable as a vehicular fuel for sustainable development.

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