OPTIMIZATION OF BIODIESEL PRODUCTION FROM LOOFAH OIL USING RESPONSE SURFACE METHODOLOGY

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ABSTRACT

Biodiesel derived from the trans-esterification of inedible oils with simple alcohol in the presence of catalyst is now attracting great attention as a renewable fuel for diesel engines. Response Surface Methodology (RSM) based on D-Optimal was used to optimize biodiesel production process from loofah oil. Oil was extracted from loofah seeds using mechanical extraction method. Biodiesel (ethyl ester) was produced from raw loofah oil using potassium hydroxide (KOH) and ethanol as alcohol. The effect of variables including reaction time, reaction temperature, amount of ethanol and amount of catalyst on the biodiesel yield was examined and optimized by RSM. The optimum reaction time, temperature, amount of alcohol and amount of catalyst were found to be 75 minutes, 70 ^{o}C, 20ml, and 0.81g respectively, resulting in optimum loofah biodiesel yield of 88%. The RSM indicates an optimum ridge in yield by increasing amount of catalyst but with decreasing amount of alcohol.

Keywords: *Biodiesel, Trans-esterification, Response Surface Methodology, Optimization, Inedible oil.*

1.0 INTRODUCTION

Biodiesel is a substitute for diesel fuel made wholly or partly from an organic product, especially processed vegetable oils such as soybean oil and peanut oil (Encarta Dictionaries, 2009). Consumption of fossil fuels has increased to a greater extent and the use of these energy resources is seen as having major environmental impact. Energy diversification is seen as an insurance policy against geopolitical risks and government insecurity about fossil fuel costs and fuel scarcity (Refaat *et al.,* 2007). Biodiesel derived from vegetable oil or animal fats by trans-esterification with alcohol like methanol and ethanol is recommended for use as a substitute for petrol-diesel mainly because biodiesel is an oxygenated, renewable, biodegradable and environmentally friendly biofuel with similar flow and combustion properties and also low emission profile (Siti, 2009).

Researches into production and optimization of biodiesel is encouraged due to rising demand for energy globally and major crude oil importers expressing concerns in the prices and sustainability of supplies couple with the fact that biodiesel has excellent lubricating properties (Alamu *et al*., 2007). The future of biodiesel lies in the world's ability to produce renewable feedstocks such as vegetable oils and fats to keep the cost of biodiesel competitive with petroleum, without supplanting land necessary for food production, or destroying natural ecosystems in the process (Meher *et al*., 2006). The use of under-utilized inedible oil seeds such as loofah seeds is suitable for the production of biodiesel thereby eliminating food-fuel competition.

Since the efficiency of ester synthesis depends on the applied reaction conditions, it is of great importance to investigate their influence on the esters yield in order to determine the optimal. The classic way of investigation by varying one process variable while others are held constant is time-consuming and expensive. Instead, the statistical methods, known as design of experiments, are frequently used as a power tool for the process optimization in the past decade such as the Response Surface Methodology (RSM) combined with the central composite rotatable design (Da Silva *et al*., 2006), factorial design (Da Silva *et al*., 2009) or Box–Behnken factorial design (Liao and Chung, 2009). Although biodiesel has been successfully produced from loofah oil (Ajiwe *et al*., 2005), there is scarcity of literature on the optimization of biodiesel production from loofah oil. Hence, this work focused on the use of RSM for optimizing biodiesel production from loofah oil which would describe the relationships between variables and obtain minimum and maximum yields.

2.0 MATERIALS AND METHODS

2.1 Collection of Seeds

The loofah fruits were harvested from bushes and uncompleted buildings within Ogbomoso, Oyo State, Nigeria. The exterior brownish part was peeled, and the interior spongy fruit was dissected into four parts using a knife, for easy removal of the seeds. The seeds were parboiled in a cooler filled with hot water (heated to 100 ^oC) to soften the shell for easy deshelling of the seeds. The seeds were eventually sundried so as to remove the moisture absorbed during parboiling in cooler.

2.2 Mechanical Extraction of Loofah Oil

The oilseeds were ground to fine particle sizes and roasted in the oven preset to a temperature of 100 ^oC for about 30 minutes. After roasting, the samples were wrapped in cheese cloth and introduced into the pressing cylinder of a laboratory press. The oil expressed was collected into a funnel beneath the cylinder and then drained into a container for storage.

2.3 The Transesterification Reactor

The main design consideration for the trans-esterification process is that the transesterification reactor should be closed to the atmosphere with no fumes escaping. Esters are very volatile and most of it may escape to the atmosphere as vapour if open reactors are used. Therefore, close reactors were used for this work. Also, the reactor must be made of materials that can resist corrosion of the chemicals used for trans-esterification. For example, the potassium ethoxide formed by adding potassium hydroxide to ethanol. Therefore, glass and metal batch, stirred reactors were used for this work.

2.4 Production of Biodiesel Fuel

Loofah ethyl ester was produced by base catalyzed trans-esterification. The biodiesel production process was carried out using the transesterification reaction mechanism reported by Ogunkunle *et al*. (2017). Potassium hydroxide was used as the alkali and ethanol as alcohol. The corresponding yield for each run was recorded and the experiment was carried out for 25 runs as analyzed by the computer program.

Three operations were performed in the production of the biodiesel fuel. These are:

i. Transesterification: The process of transesterification to produce ethyl esters was a two-step process carried out by Abowei *et al.* (2013). The advantage of the two-step process is the ease with which each of the trans-esterification process can be monitored. Different yields of biodiesel were obtained under different reaction variables. The variables are reaction time, temperature, amount of alcohol and amount of KOH.
ii. Phase separation: When the transesterification reaction was completed, the reaction products were separated into two layers by allowing the product mixture to settle overnight under ambient condition. After separation, the ester product formed the upper layer and by-product glycerol formed the lower layer. The residual catalyst and the un-reacted excess alcohol were distributed between the two phases.
iii. Washing and heating: After separation of the phases, the dissolved catalyst and alcohol were washed from the ester with distilled water. The ethyl ester obtained was then heated at 120 ^{o}C for 30 minutes to remove all moisture residues present in it. The ester product was then stored in sample bottles.

2.5 Determination of the Yield of Esters

The yield of the ethyl esters, Y, produced was calculated using Equation 1:

$$Y = \frac{V_e}{V_r} \times 100\% \qquad \ldots \qquad \ldots\ldots (1)$$

Where:

Y= Yield of the ethyl esters, %

V_e= Volume ethyl esters produced, milliliters.

V_r = Volume of raw oil used, milliliters.

2.6 Optimization of the Transesterification Reaction

Optimization of the production process of biodiesel from the loofah oil was carried out experimentally as randomized by Response Surface Method. Design Expert software 6.0.8 Version was used for regression and graphical analysis of the data obtained. Analysing the experimental yield data supplied into the Design Expert, the predicted optimum values from the 25 experimental runs were found to be 25 The optimal yield conditions for biodiesel production from the transesterification of loofah oil were obtained from the optimization of the yield responses for the experimental reaction data. The experimental reaction conditions were set in range and the yield response was set on maximum level in order to obtain an ideal optimized reaction conditions for biodiesel production. The optimal solution predicted for biodiesel yield are favourable optimum levels based on economic considerations (reduced temperature and reaction time and moderate alcohol amount and catalyst amount) according to Oniya *et al*. (2016). Table 1 shows the 2^k factorial values for the experimental design.

Table 1: 2^k factorial values for the experimental design

Variables	Units	Low	High
Reaction time	Minutes	60	90
Temperature	Degree Centigrade	40	70
Amount of Alcohol	Milliliters	15	30
Amount of KOH	Grams	0.81	1.11

3.0 RESULTS AND DISCUSSION

The results of the 25 randomized experimental runs and the percentage biodiesel yield are presented in Table 2. From Table 2, the optimum yield was obtained at run 13 under the following conditions of variables: reaction time of 75 minutes, temperature of 70^{o}C, amount of alcohol of 20 ml and amount of KOH of 0.81g. While the lowest yield was obtained at run 7 and 8 under the following conditions: reaction time of 60 minutes, temperature of 55^{o}C, amount of alcohol of 20 ml and amount of KOH of 0.81g for run 7 and 0.91g for run 8. This result was also similar to the values obtained by Yusuf and Sirajo (2009).

It can also be seen from Table 2 that the ester yield increases with decreasing amount of catalyst and increasing temperature but progressively decreases at lower level of temperature. This can be explained by the formation of by-products, possibly due to triglycerides saponification process, a side reaction which is favoured at high temperature. This side reaction produces potassium soaps and thus, decreases the yield of biodiesel. The varying biodiesel yield values presented in Table 2 are indications that loofah oil transesterification reaction parameters considerably affected the biodiesel yield. The Model F-value of 7.06 obtained for biodiesel yield implies the model is significant. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case reaction time, temperature, amount of alcohol, amount of KOH and the interactive effects are significant model terms.

Standard deviation of 4.37, mean of 72.40, C.V. of 6.04, R^2 of 0.9082, adj R^2 of 0.7796, pred R^2 of -1.0171 and adeq precision of 9.910 were obtained for biodiesel yield. A negative "Pred R-Squared" implies that the overall mean is a better predictor of your response than the current model. "Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. In this case, a ratio of 9.910 indicates an adequate signal. This model can be used to navigate the design space. Guan and Yao (2008) reported that an R^2 should be at least 0.80 for the good fit of a model. In this case, the R^2 value of 0.9082 indicated that the sample variation of 90.82% for the biodiesel production is attributed to the independent factors (temperature, time, and alcohol to oil molar ratio) and only 9.18% of the total variations are not explained by the model.

The interaction term for reaction time and temperature indicates these two factors did not affect percentage yield independently. Thus, the effect of one factor on percentage yield depends on the specific level of the other factor. This interaction can be observed in the fitted response surface plot shown in Figure 1. There is significant interaction (95% CI) between the response surface of percentage yield vs. time and amount of alcohol as observed in Figure 2. There is reduction in percentage yield at initial time and amount of alcohol. According to Oniya *et al.* (2016), biodiesel yield is always low for low reaction time because there is not yet a phase formation between the reactants at low reaction time. Also, Ogunkunle *et al.* (2017) reported that higher molar ratio resulted into higher production of biodiesel. Higher volume of would allow complete transesterification of the seed oil and favour the forward reaction. Lower volume of alcohol can only allow partial transesterification of the oil, hence the lower yield of biodiesel obtained.

Also, the significant interaction term for amount of KOH and amount of alcohol indicates these two factors did not affect percentage yield independently. Thus, the effect of one factor on percentage yield depends on the specific level of the other factor. The fitted response surface plot for the interaction can be observed in Figure 3.

The effect of the amount of catalyst was also very significant upon biodiesel production the transesterification process. The relationship between percentage yield and amount of KOH with time in Figure 4 was curvilinear with a positive linear coefficient. This suggests that percentage yield was inhibited at high catalyst concentration. And this result is consistent with previous research carried out by Hem (2008).

Table 2: Result of yield of biodiesel produced from optimization of transesterification reaction of loofah oil

Run	Amount of oil (ml)	Reaction time (mins)	Reaction temperature (^oC)	Amount of Alcohol (ml)	Amount of KOH (g)	Yield (%)
1	70.0	70.0	70.0	30.0	0.96	80
2	70.0	70.0	70.0	22.5	1.11	74
3	70.0	75.0	40.0	20.0	0.91	68
4	70.0	70.0	70.0	15.0	0.81	82
5	70.0	90.0	60.0	20.0	0.91	69
6	70.0	70.0	40.0	20.0	0.96	68
7	70.0	60.0	55.0	20.0	0.81	54
8	70.0	60.0	55.0	20.0	0.91	54
9	70.0	75.0	70.0	15.0	0.81	83
10	70.0	60.0	70.0	20.0	0.81	76
11	70.0	70.0	40.0	20.0	0.81	68
12	70.0	70.0	70.0	30.0	0.91	78
13	70.0	75.0	70.0	20.0	0.81	88
14	70.0	75.0	70.0	15.0	0.91	82
15	70.0	75.0	70.0	22.5	0.81	74
16	70.0	70.0	40.0	22.5	0.81	68
17	70.0	80.0	70.0	15.0	0.81	82
18	70.0	75.0	40.0	22.5	0.96	68
19	70.0	70.0	70.0	30.0	0.81	83
20	70.0	70.0	55.0	22.5	0.96	74
21	70.0	60.0	55.0	20.0	1.11	55
22	70.00	70.0	40.0	15.0	1.11	68
23	70.00	70.0	70.0	30.0	0.91	80
24	70.00	60.0	60.0	15.0	1.11	66
25	70.00	75.0	40.0	20.0	0.81	68

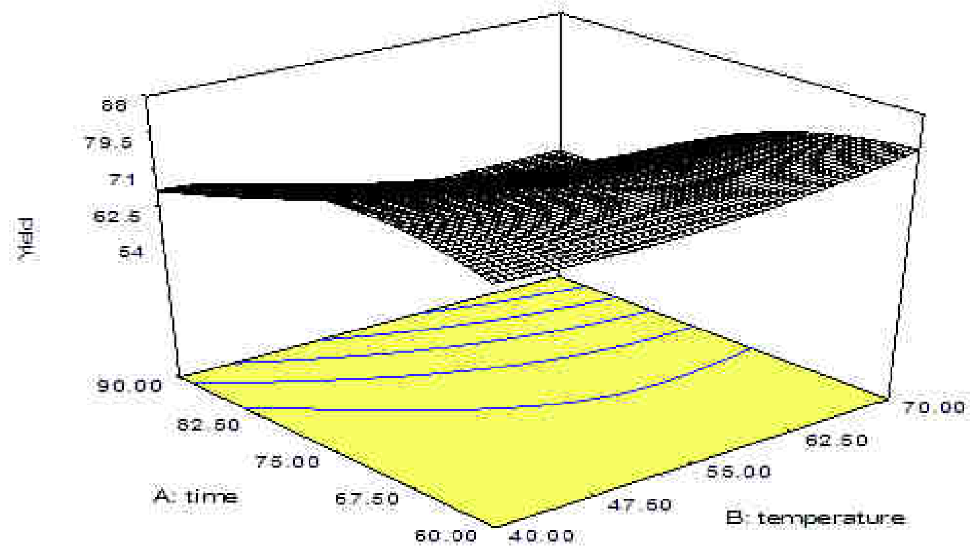

Figure 1: Response surface plot showing the effect of reaction time and temperature on percentage yield

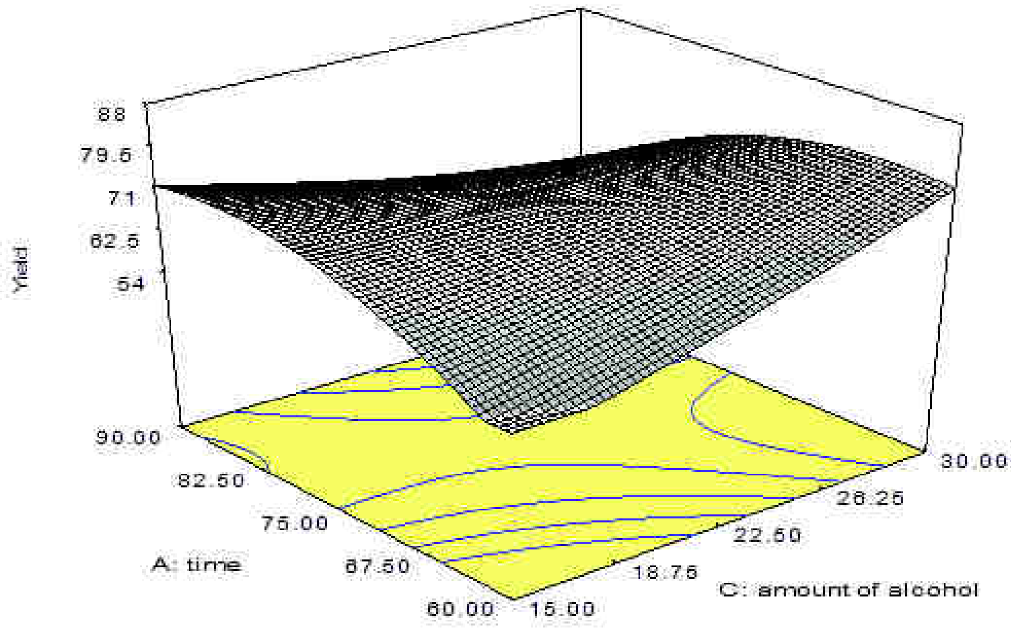

Figure 2: Response surface plot showing the effect of time and amount of alcohol on percentage yield

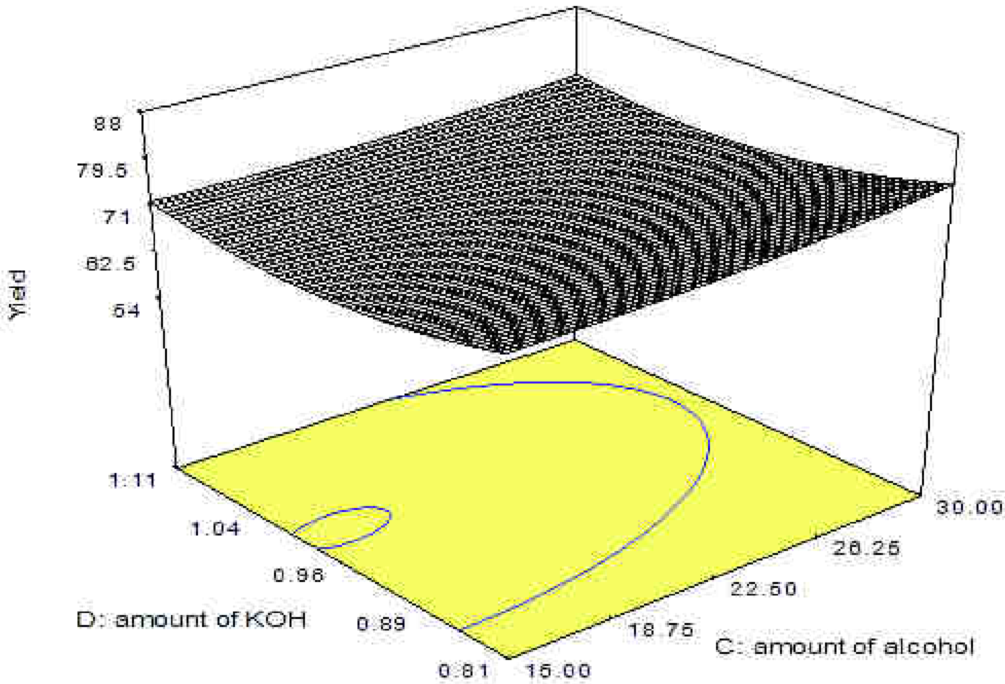

Figure 3: Response surface plot showing the effect of amount of KOH and amount of alcohol on percentage yield

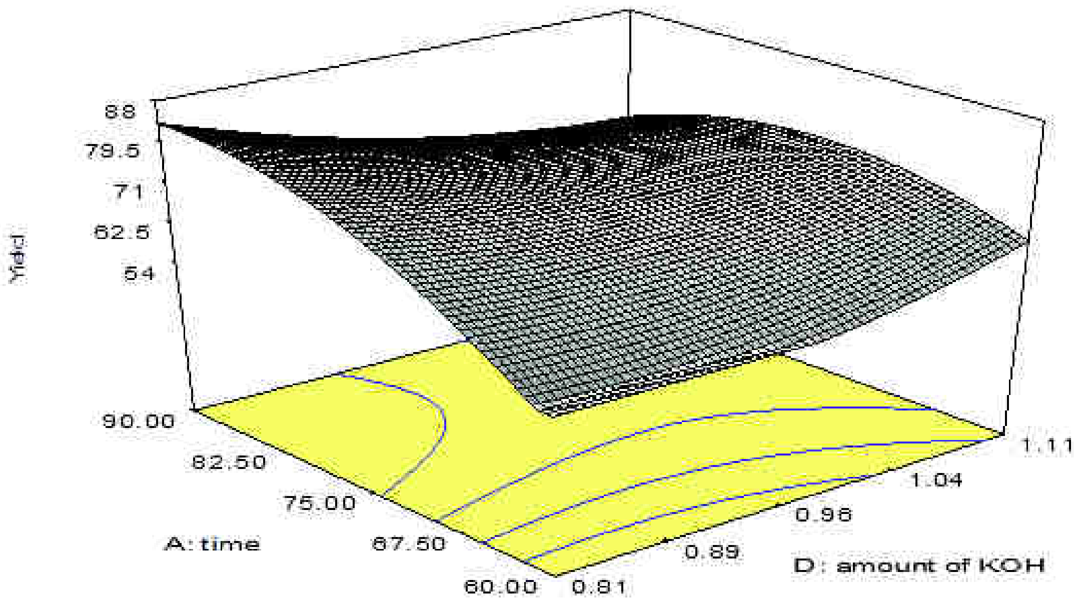

Figure 4: Response surface plot showing the effect of time and amount of KOH on percentage yield

The design summary for the optimization of biodiesel production from loofah oil is shown in Table 3. The final empirical model in terms of coded factors (reaction parameters) for the biodiesel yield is given in Equation 1. The model shows that reaction parameters are significant on biodiesel production from loofah oil. A, B, C and D represent time, temperature, amount of alcohol and amount of KOH, respectively. The expected yield can thus be predicted using the model with the inclusive of quadratic and interactive terms of the reaction parameters.

Table 3: Design Summary for the Optimization of Biodiesel Production from Loofah Oil

Study Type	Response Surface			Experiments	25		
Initial Design	D-optimal			Blocks	No Blocks		
Design Model	Quadratic						
Response	Name	Units	Obs	Minimum	Maximum	Trans	Model
Y1	Yield	%	25	54.00	88.00	None	Quadratic
Factor	Name	Units		Low Actual	High Actual	Low Coded	High Coded
A	Time	minutes		60.00	90.00	-1.000	1.000
B	Temperature	^oC		40.00	70.00	-1.000	1.000
C	Amount of alcohol	ml		15.00	30.00	-1.000	1.000
D	Amount of KOH	Grams		0.81	1.11	-1.000	1.000

Final Equation in Terms of Coded Factors:

Biodiesel Yield = $70.23 + 0.074*A + 1.37*B + 1.92*C - 1.75*D - 8.33*A^2 + 2.80*B^2 + 1.11*C^2 + 1.11*C^2 - 4.00*D^2 - 6.69*A*B - 10.77*A*C - 7.85*A*D - 4.38*B*D - 0.40*C*D$ … (2)

Where A, B, C and D were the coded values of the independent variables i.e. time, temperature, amount of alcohol and amount of KOH.

3.1 Optimization of the Transesterification of Loofah Oil Reaction Variables

Optimum reaction conditions for maximizing biodiesel yield were determined by statistically analyzing the experimental data. The reaction variables (time, temperature, amount of alcohol and amount of catalyst) were set in range reaction time in order to provide an ideal case for optimized biodiesel yield from loofah oil transesterification using ethanol and KOH as alcohol and catalyst respectively. Based upon the requirements of the reaction parameters, biodiesel yield was set on maximum level. The predicted optimum values of reaction time, temperature, amount of alcohol, and amount of catalyst were found to be 62.54 minutes, 69.05 ^{o}C, 23.48 ml and 1.00 g respectively, to achieve 89.98% maximum loofah biodiesel yield; while desirability was 1.00 for the experiment. This optimal solution chosen for biodiesel yield are favourable predicted optimum levels based on economic considerations (reduced reaction time, temperature and moderate amount of alcohol and catalyst which corresponds to reduced operating costs of the transesterification process for biodiesel yields) and not necessarily the highest biodiesel yield value.

The biodiesel yield in the optimized conditions was 89.98% while the highest biodiesel yield from experimental work was 88%. The results clearly indicated the effectiveness of process variables optimization in biodiesel production using RSM. Ogunkunle *et al*. (2016) used a 2k factorial Box-Behnken design (BBD) to maximize biodiesel production from milk bush seed oil and obtained optimized reaction variables of 10.18:1 of alcohol to oil, 1.89 hours of time and a 63.67oC temperature. Under these conditions, a methyl ester (biodiesel) yield of 81.45% was predicted. Further validation experiments conducted at the predicted optimal conditions yielded an average of 90.72% of biodiesel. This is in reasonable agreement with the optimized experimental conditions.

4.0 Conclusion

The response surface analysis was performed to assess the effect of reaction time, reaction temperature, amount of alcohol and amount of catalyst on the percentage yield and for biodiesel produced from loofah oil. The response surface indicates an optimum ridge in yield by increasing amount of catalyst but with decreasing amount of alcohol was near the experimental maximum, a significant reduction in yield was noted possibly due to potential reaction reversal. Temperature of 70 ^{o}C, reaction time of 75mins, amount of alcohol of 20ml (5.0:1 molar ratio of alcohol to oil) and amount of KOH of 0.81g (1.2% weight of oil) for transesterification reaction gave the optimum yield. It is recommended that optimization of transesterification reaction for the production of biodiesel should be extended to other inedible oils seeds so that attention will be shifted from edible oils to inedible oil seeds for the large scale production of biodiesel.

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