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Transesterification of Algae Oil and Little Amount of Waste Cooking Oil Blend at Low Temperature in the Presence of NaOH

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Abstract: The present study describes the single-step transesterification method of biodiesel production from high free fatty acid (FFA) waste cooking oil blended with algae oil using a homogeneous base catalyst. Due to high FFA contents, two step transesterification is needed to convert oil into biodiesel and therefore the high FFA content of waste cooking oil is decreased by blending it with low FFA content algae oil, which would further lead only to single step transesterification of low FFA oil. The design and optimization studies were conducted using Response Surface Methodology (RSM). The box-Behnken design technique is applied to optimize the three process parameters, i.e., catalyst concentration (0–2 wt%), methanol concentration (*v/v*) (20–60%) and reaction time (60–180 min) at a uniform reaction temperature of 50 °C. The result of the current study indicates that an effective biodiesel yield of 92% can be obtained at the optimized condition of catalyst concentration of 1.5% (*w/w*), methanol/oil ratio of 21:1 and reaction time of 110 min at a constant reaction temperature of 50 °C. This analysis clearly shows that this study can resolve the storage problem of high FFA oils from different feedstock and RSM can be successfully used to model the reaction to maximize the biodiesel yield.

Keywords: transesterification; biodiesel production; optimization; free fatty acid (FFA); Response Surface Methodology (RSM); algae oil

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1. Introduction

The planet is dealing with the issue of the depletion of conventional energy sources and their hazardous environmental pollution. Due to this, researchers have decided to develop new sources of energy that can play a promising role as alternative fuels [1,2]. Among the multiple renewable sources of energy, biomass is the most easily accessible resource, having a low cost and being abundant worldwide [3]. Biofuels like biodiesel, biooil, syngas and methane obtained from biomass have huge potential for commercial usage [4,5]. Since there are multiple challenges for their large scale production, it is essential to develop novel technologies to fulfil the energy needs of tomorrow [6,7].

Biodiesel, in particular, has been identified as a beneficial substitute, as biodiesel and its blends used with a Compression Ignition (CI) engine create a significant reduction in the number of emissions such as CO and unburnt hydrocarbons, with no sulfur compounds, as it is sulfur-free [8,9]. Biodiesel can be made through the transesterification of vegetable oil, animal fats, dry biomass and other non-edible oils with alcohol and a catalyst [10]. Producing biodiesel from edible oils disturbs food security worldwide and also raises the price of biodiesel [11] Thus, more focus is being given to the non-edible sources for biodiesel synthesis so that the production cost of biodiesel can be lowered [12,13].

The application of waste cooking oil (WCO) has increased over the years and gained the attention of various countries for use as a feedstock for biodiesel production [14]. Using WCO, the cost of biodiesel can be lowered and waste disposal reduced. WCO is the oil being used for food preparation and is no longer usable. The main concern in using WCO for high-quality biodiesel production is the high free-fatty acid (FFA) concentration. Various pretreatment processes are required to reduce the FFA of WCO to make it suitable for transesterification [15]. Various researchers have studied WCO for producing biodiesel, using different heterogeneous catalysts to prevent the need for pretreatment of high FFA oils [16,17].

Mohadesi et al. [18] used KOH or clinoptilolite as a heterogeneous catalyst for biodiesel synthesis from WCO. The calcination time of the catalyst was 300 min. at 400 °C. A considerable yield of 97.45% was recorded for optimum production at an 8.1 wt% catalyst concentration and a 2.25:01 methanol and oil ratio for a reaction time (Rt) of 13.4 min at a reaction temperature (RT) of 65 °C. Hsiao et al. [19] studied $Ca(OH)_2$ as a heterogeneous catalyst for microwave-assisted transesterification of WCO. The calcination time for the catalyst was 120 min at 65 °C, with a considerable yield of 98.2% at 65 °C obtained for the conditions of 75-min reaction time, a 4.0 wt% catalyst concentration and 8:01 methanol/oil ratio. Aghel et al. [20] evaluated kettle limescale as a catalyst for biodiesel synthesis from WCO in a pilot-scale microreactor. Limescale was calcined at 900 °C for 120 min for catalyst preparation. The maximum production of 93.41% was achieved for the reaction conditions of 8.87 wt% catalyst concentration, 1.7:03 methanol/oil ratio with Rt of 15 min at a T of 61.7 °C. Degfie et al. [21] used the CaO heterogeneous catalyst to produce biodiesel from WCO. The catalyst was calcined at 500 °C. The optimum combination of 1% catalyst and an 8:1 methanol to oil ratio resulted in a 96% yield in 90 min at 50 °C. Komintarachat and Chuepeng [22] developed a CaO-KCl catalyst from green mussel shells by calcination at 500 °C for 300 min to produce biodiesel from WCO. An optimum biodiesel production rate of 97% was achieved, but at the cost of high energy consumption in catalyst preparation.

The literature review showed that heterogeneous catalysts are difficult to prepare and require a high amount of energy for the calcination process. Some base heterogeneous catalysts are sensitive to FFA content, thus single-step transesterification is not possible when using these catalysts [23]. Homogeneous catalysts are easily available and have become more important with the commercialization of biodiesel production worldwide [24]. Mohadesi et al. [25] manufactured biodiesel from WCO in a pilot prototype reactor. The esterification process was carried out at 1% H_2SO_4 to keep the FFA of WCO below 1% at 06:01 methanol and oil concentration at a RT of 65 °C. The oil obtained was further trans-esterified with a 1.16 wt% KOH catalyst concentration. Biodiesel production of 98.26% was achieved using a 9.4:01 methanol and oil ratio at a RT of 62.4 °C.

Algae is supported by many researchers as a renewable source of energy and at the same time it has a smaller amount of FFA, which is good for the trans-esterification process [26].

From the literature review, it was found that homogeneous catalysts are more popular and easier to use. These catalysts require the pretreatment of high-FFA oils. As a result, they require a two-step transesterification process: an acid catalyzed transesterification process to lower the FFA content of the oil, followed by a base catalyzed transesterification process to produce biodiesel. This whole process, consisting of two steps of transesterification, is both a time and energy consuming process. Algae is receiving attention due to its high productivity and oil yield which is of low FFA, and due to this reason it can be converted to biodiesel using single step transesterification. On the other hand, WCO requires a two-step transesterification process to convert it into biodiesel due to high FFA. For this reason, to use WCO for biodiesel production with single step transesterification, the FFA of the oil can be reduced by blending the oil with low-FFA algae oil; therefore, in the present study, the production of biodiesel from high-FFA WCO blended with low-FFA algal oil using a homogeneous catalyst in a single-step transesterification process is proposed.

2. Materials and Methods

2.1. Materials

For this experiment, WCO was collected from city restaurants and algae oil was brought in from the Soley Biotechnology Institute. Analytical reactants included NaOH and Methanol, which were obtained from Naveen Traders, Roorkee. The properties of the fuels, WCO and algae oil were calculated using the usual techniques described in IS:15607 and then obtained after refinement. The FFA content of WCO was found to be 20%, which was very high for the base-catalyzed transesterification process. Only 0.50% of algal oil was discovered to contain FFA. Using a base catalyst causes the saponification reaction, which results in the creation of soap when FFA levels are more than 1.0%. Thus, there is a need to reduce the FFA of WCO below or equal to 1%. The two oils (algae oil: WCO) mentioned above were blended in a volumetric proportion to reduce the FFA content of WCO to 1% to make it suitable for the homogeneous-base catalyzed transesterification-process. The titration process was performed to obtain the optimum blend ratio of algae oil: WCO. The final blend ratio of 1:40 was found to be appropriate (algal oil is 40 times the volume of WCO) and the FFA was reduced to 1.0%. Table 1 shows the various fuel characteristics of WCO, algae oil and their blend used in this experimental study.

Table 1. Properties of Waste Cooking oil & algae-oil.

S.NO.	Properties	Waste Cooking Oil	Algae Oil	Blend of Waste Cooking Oil and Algae-Oil
1.	Flash point (°C)	256.00	102.00	105.85
2.	FFA (%)	20.00	0.50	1.00
3.	Density (kg/m^3)	0.919	0.86	0.8614
4.	Viscosity (cst) @40 °C	39.4	21.00	21.46

2.2. Method

This research intends to increase the transesterification of a mixture or blend of WCO and algae oil with respect to three process variables. Based on the literature review, the ranges of these parameters were decided [27]. These factors are: (a) reaction duration (60.00 to 180.00 min); (b) catalyst concentration (NaOH) (0.0% to 2.0% *w/w*); and (c) volumetric ratio of the methanol-oil blend (20.0% to 60.0% *v/v*). Due to the high reactivity of homogeneous catalysts, NaOH was used in the study [28]. Jain et al. [29] looked into the kinematics of transesterification of WCO and found 50 °C to be the optimum reaction temperature for acquiring a higher FAME yield. Thus, the transesterification reaction was performed at a steady RT of 50 °C. The experiments were created with RSM and the Box-Behnken Design (BBD) technique [30]. The statistical analysis and optimizations were carried out using Design of experiments

2.3. Experimental Procedure

Design of Experiments

There were a total of 17 trials designed with RSM based on BBD to obtain the optimal combinations of three process variables. The three varying parameters stated earlier were evaluated to determine their ideal blend using this experimental design. Table 2 displays the three parameters, A (reaction time (Rt) in minutes), B (methanol to oil ratio in volume percent) and C (catalyst amount in *w/w*%), at three different stages of the reaction mechanism within the predetermined range. A list of 17 experiments designed to gain experimental results for biodiesel yield is shown in Table 3.

Table 2. Blend oil transesterification via BBD with independent parameters.

Parameters	**Symbol**	**Unit**	**Levels**		
			−1	**0**	**1**
Reaction time (Rt)	A	Minutes	60	120	180
Methanol/oil ratio	B	%	20	40	60
Catalyst-concentration (CC)	C	%	00	01	02

Table 3. List of trials designed with BBD.

Run No.	**Time (in Minutes)**	**Methanol/Oil Ratio (%)**	**Catalyst-Concentration (CC) (%)**
	A	**B**	**C**
1	180	40	0
2	60	60	1
3	120	60	2
4	60	20	1
5	120	40	1
6	120	40	1
7	180	60	1
8	120	20	2
9	120	40	1
10	180	40	2
11	60	40	2
12	60	40	0
13	180	20	1
14	120	20	0
15	120	40	1
16	120	60	0
17	120	40	1

2.4. Transesterification

The blend of WCO and algae oil was trans-esterified by adding methanol (CH_3OH), a reactant with a base catalyst, i.e., sodium hydroxide (NaOH), for the production of methyl ester. With the aid of a heating plate stirrer and a batching reactor, all of the planned tests were carried out at a maintained temperature of 50 °C as illustrated in Figure 1. To produce pure biodiesel, pure FAME and glycerol layers were segregated by applying a separator funnel.

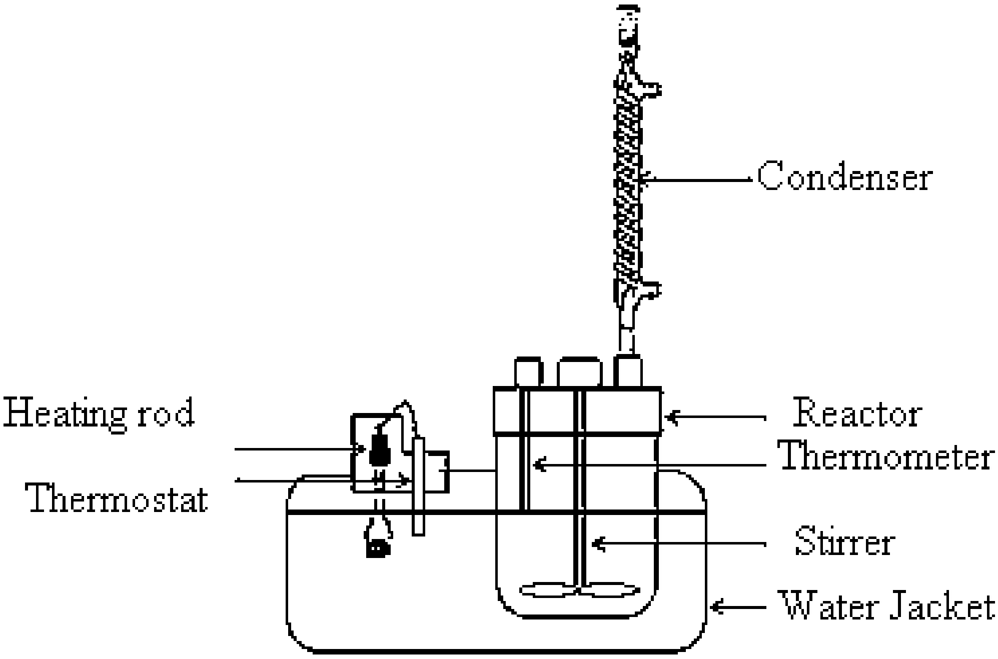

Figure 1. Representation of the reactor's schematics.

2.5. Statistical Analysis

An analysis of variance (ANOVA), a regression analysis and the development of the response-surface graphs were the three analytical stages that were used to analyze the relationship among the varying parameters and the response yield. With a confidence level of 95.0% ($p = 0.05$), Design Expert-11 has been used to assess the statistically significant results of the entire quadratic polynomial model. As the response values for the tests indicated, the highest experimental yield percentage rates were used. Then the developed model was employed for analyzing the interactive impact of individual parameters. Surface regression analysis was also conducted using the polynomial equation (Equation (1)):

$$R = \beta_0 + \sum_{j=1}^{k} \beta_j P_j + \sum_{j=1}^{k} \beta_{jj} P_j^2 + \sum_{i=1}^{j-1} \sum_{i=2}^{k} \beta_{ij} P_i P_j + \varepsilon \tag{1}$$

where P_i and P_j are the uncoded independent parameters, ε is the random disturbance term, k is the number of independent parameters maximized for testing (k = 3 in this work) and β_0 is the regression coefficient. R is the result (% Fatty-Acid Methyl-Ester Yield).

3. Results and Discussions

3.1. Transesterification Process

During the transesterification of the blend, two distinct response yields were obtained: the actual and expected value. The 17 trials yielded the empirical yield and RSM's analysis of the experimental outputs led to the prediction of the yield. The three independent process parameters, namely A: reaction time (Rt), B: methanol/oil proportion and C: catalyst concentration (CC), are listed in Table 4. These two response rates are expressed as percentages. The link between both the outcome and the input parameters was characterized using second-order polynomial equations, including interaction effects, by using multiple regression analysis on the experimental response. Equation (2) displays the finalised techniques developed by coded factors.

$$\begin{aligned} \text{Yield\%} = \; & +28.6475 + 0.1810 * A - 0.6176 * B + 98.3164 * C + 0.01186 * A \\ & * B - 0.0512 * A * C - 0.3175 * B * C - 2.4063 * 10^{-3} * A^2 \\ & -0.0107 * B^2 - 27.8689 * C^2 \end{aligned} \tag{2}$$

where A is Rt, B is Methanol/Oil proportion and C is CC.

Table 4. Observations to the combination of waste cooking oil and algae oil being trans-esterified.

Run	Time (Minutes)	Methanol/Oil Ratio (%)	Catalyst Concentration (CC) (%)	Blend Biodiesel Yield (%)		Error (%)
				Experimental	Predicted	
	A	B	C			
1	180	40	0	20.8	26.78	22.33
2	60	60	1	31.01266	46.11	32.742
3	120	60	2	69.23077	60.14	−15.116
4	60	20	1	91.90556	89.46	−2.7336
5	120	40	1	82.8125	82.36	−0.5494
6	120	40	1	80.71429	82.36	1.998
7	180	60	1	75.33333	77.81	3.1829
8	120	20	2	79.27928	87.71	9.6120
9	120	40	1	81.95489	82.36	0.4918
10	180	40	2	61.42857	68.10	9.7965
11	60	40	2	76.97842	71.02	−8.3897
12	60	40	0	24.06015	17.41	−38.197
13	180	20	1	79.27928	64.20	−23.487
14	120	20	0	18.42105	27.54	33.1116
15	120	40	1	83.33333	82.36	−1.1817
16	120	60	0	33.77483	25.37	−33.129
17	120	40	1	82.92683	82.36	−0.6882

The graph was plotted for the anticipated value and actual value of response yield (%) as presented in Figure 2. The graph shows that the anticipated values around the zero-error line are scattered at lower values of yield, with some of the points of higher yield values being near the experimental values. Therefore, it confirms the validity of the model created to set up a significant relationship between the response yield of the oil blend and the three independent process variables.

3.2. ANOVA

The most appropriate method for determining how accurate the tests and modelling are is analysis of variance (ANOVA). Variance analysis for performed experiments is shown in Table 5. The quadratic model was formed and analysed using RSM. The model was found to be significant and the lack of fit was insignificant. The study of the coefficient of determination (R^2) provided more insight into the model's suitability. A model can be deemed appropriate specifically if the R^2 value is higher than 0.95, showing that the model can account for up to 95% of the variability of the response data. The observed R^2 value of 0.9904 demonstrates that the parameter estimates and experimentally obtained results correspond well. Additionally, there was a 0.2 discrepancy between the anticipated R^2 and modified R^2 values, which is a respectable level of agreement.

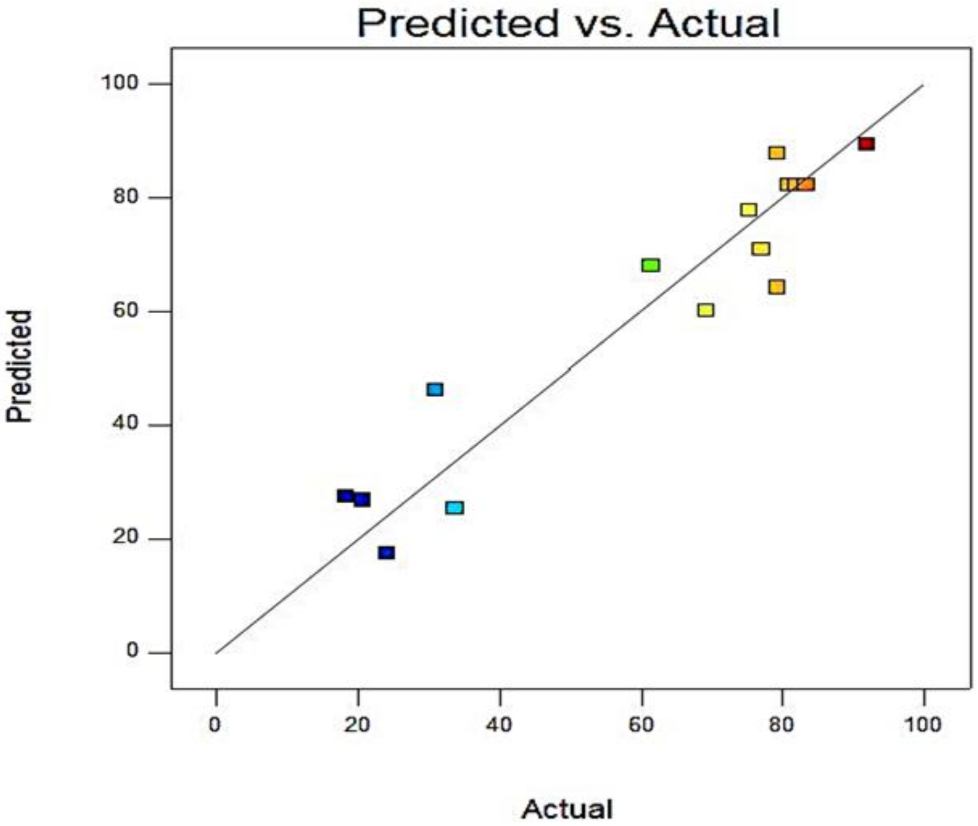

Figure 2. Comparison of theoretical and measured yield percent values.

Table 5. Evaluating the RSM modeling.

S.No.	Parameters	Value
1.	Model	Significant
2.	Lack of fit	Non-Significant
3.	R^2	0.9904
4.	Adjusted R^2	0.9745
5.	Predicted R^2	0.9044

3.3. Effects of Process Variables on Yield

Figure 3 depicts how changes in reaction time (Rt), methanol/oil ratio and catalyst concentration (CC) affect the blend's responsive yields. As Rt of the method increases (curve A), the yield shows an increasing trend, but after reaching the point of optimum yield, a small decrease in yield % was observed. Due to excess Rt, the biodiesel yield decreases because of the occurrence of the reverse reaction. At the initial level, the methanol/oil ratio (curve B) results in a high biodiesel yield. The decreasing trend of biodiesel yield was observed with any further increase in the methanol/oil ratio from the initial level. The figure shows that increasing the catalyst concentration (curve C) in the reaction raises the initial response yield of the blend. When the catalyst percentage was raised after the optimal yield point, a pattern toward lower yield was seen. This reaction yield behaviour may be the result of adding too much catalyst. Excessive catalysts speed up triglyceride conversions and prevent the creation of water, which could increase the probability of a secondary reaction producing soap.

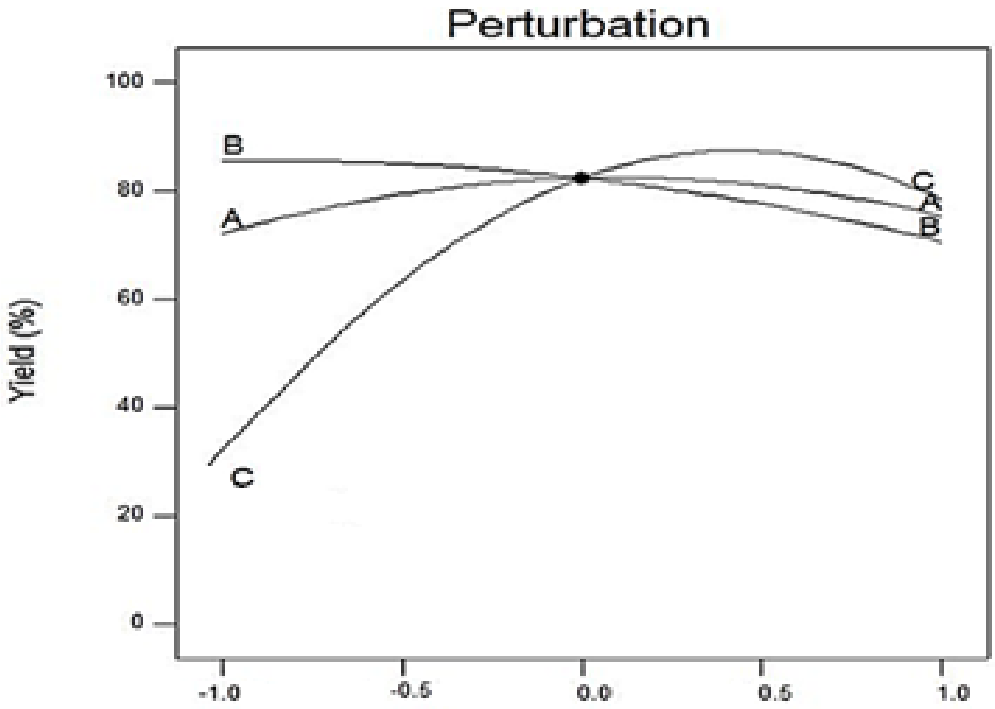

Figure 3. Effect of CC, Rt and methanol/oil ratio on yield (%).

3.4. *Effect of Reaction Parameters on Yield %*

Utilizing the Box-Behnken Modeling approach in the Design of experiments technique, three distinct 3D graphs were created in order to explore the effects of individual variables, or processing parameters, on related parameters, or the response yield of the mix. The area contained within the smallest oval in the contour graphic provided the maximum expected values for the response yield of the mix as determined by the program. The interaction of various independent components in this procedure resulted in these elliptical outlines. As seen in Figures 4–6, the two sequential variables were selected at their minimal level to create the three 3D representations. The curve surface inside the shortest ellipse in the contours graphic illustrates the interaction between each pair of processing parameters, AB, BC and AC.

3.4.1. Effect of Reaction Time on Yield %

To study the effect of reaction time (Rt) on biodiesel yielding (%), Rt was varied from 60–180 min. Initially, the yield was very high when the reaction was performed for 60 min. This may be because reactants required a certain amount of minimal time to react i.e., the activation energy. Identical outcomes were recorded by Zhao et al. [31]. On further increasing the reaction time from 60 to 120 min and then to 180 min, a decreasing trend was observed for the yield % for all the ranges of catalyst concentration and methanol/ratio. This decrease can be attributed to the fact that the transesterification process is reversible [32]. Thus, beyond the optimum point, the decrease in yield % can be observed.

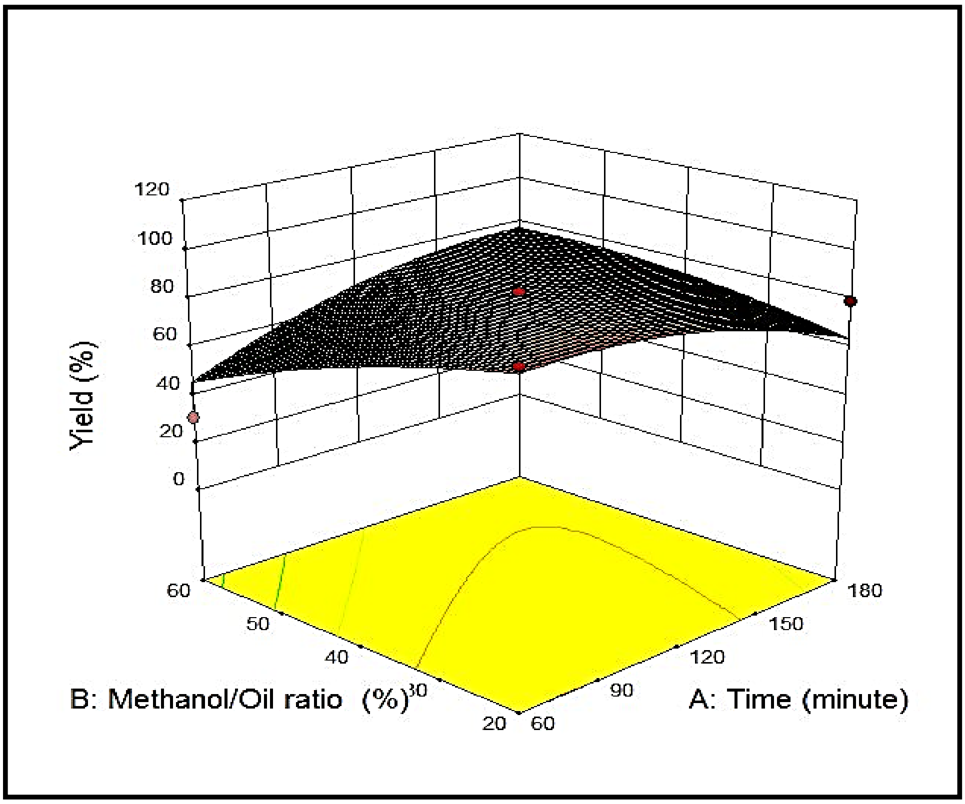

Figure 4. Biodiesel yield (%) vs. methanol/oil ratio (%) and Rt (min).

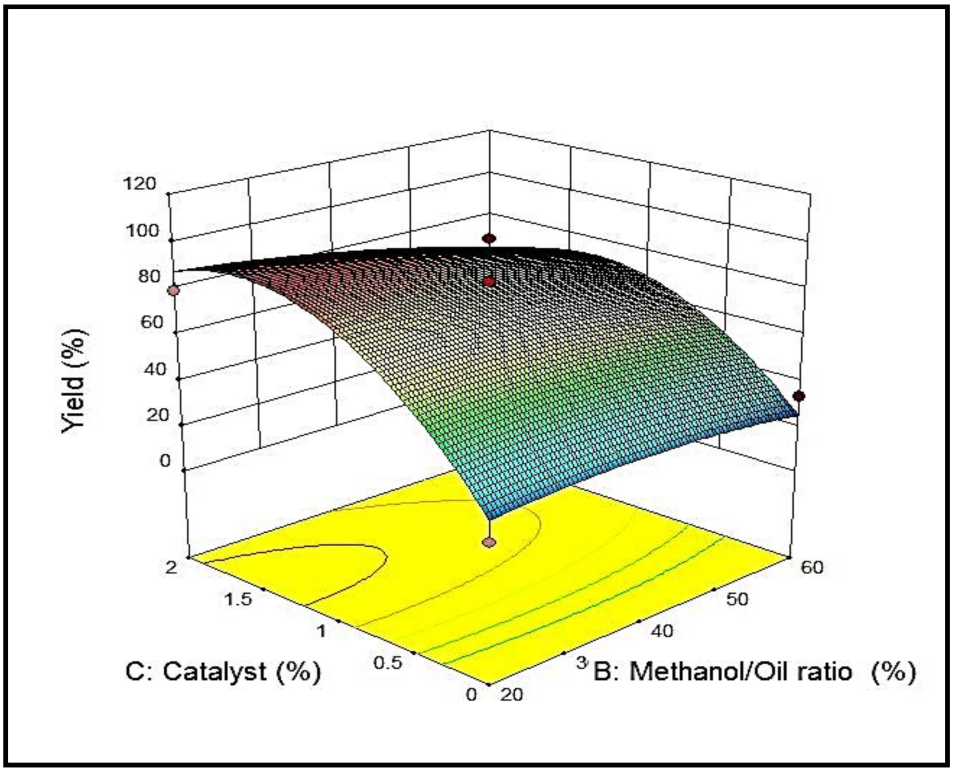

Figure 5. Biodiesel yield (%) vs. CC (%) and methanol/oil ratio (%).

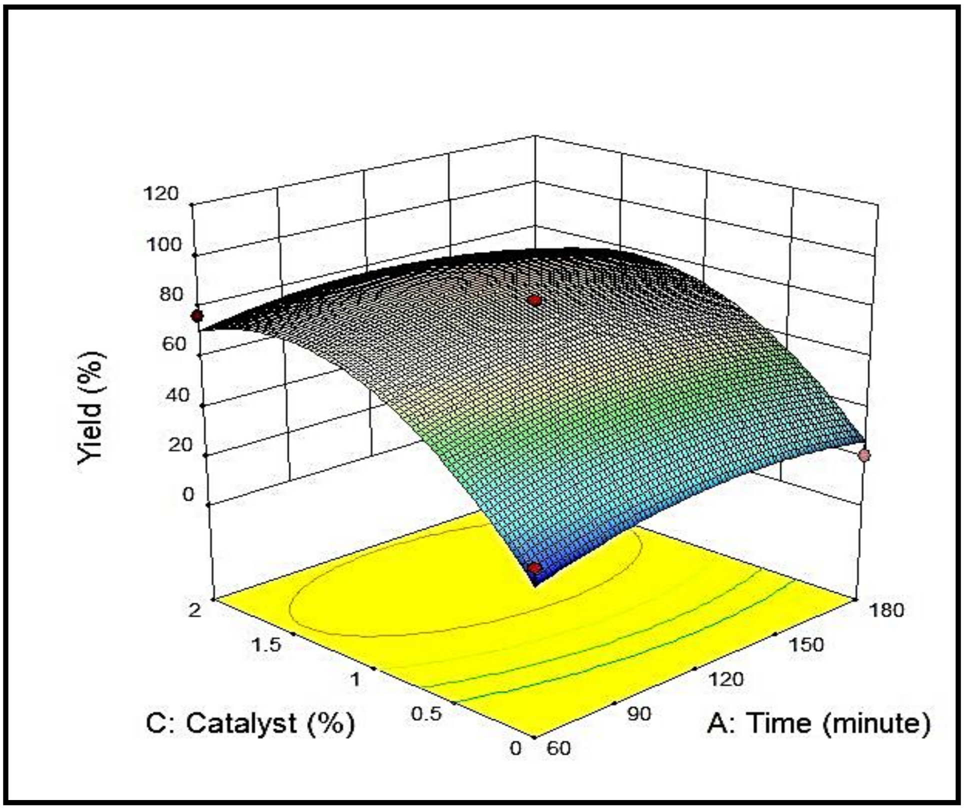

Figure 6. Biodiesel yield (%) vs. CC (%) and reaction time (minutes).

3.4.2. Effect of Methanol/Oil Ratio on Yield %

To study the effect of methanol concentration on yield %, the methanol/oil ratio was varied from 20–60%. The highest yield of 91.90% was obtained using a methanol concentration of 20%. It was found that, on further raising the methanol content, there was a decrease in yield % recorded. The reversible nature of transesterification necessitates a greater alcohol concentration in order to move the reaction along [33], but in the current study, this decrease may be because adding excess methanol dissolves the glycerol into methanol. The side product glycerol formed during the reaction decreases the reactivity of methanol with the fatty acids. The same result was observed by Behera et al. [34]. The biodiesel yield decreased beyond the methanol concentration of 20:1. Recovery of excess methanol is also an energy-consuming process [35]. Thus, the optimum amount of methanol/oil ratio is required for high yield.

3.4.3. Effect of Catalyst Concentration on Yield %

The catalyst concentration (CC) having g significant effect on yield %, in this study the catalyst amount had a range from 0–2%. The results indicated that the yield % improves rapidly when raising the catalyst amount from 0–1%. At lower concentrations, it was observed that the increase in yield % was very low, but with increasing catalyst concentration there was a high-rate reaction process which increased the yield %. Beyond the optimum point, the yield % increased very slowly until, after a point, a slowdown in yield % was found. The same trend was observed by Bhatia et al. [36]. The reason behind this decrease may be the cause of soap formation due to excess catalyst, which negatively affects the yield % [37].

4. Optimization of Response Yield

In addition, the optimizing method was used on every response to get the most out of the blending in terms of responses. The optimization process was based on the quadratic model (Equation (2)) made by the RSM. The conclusion of the optimal value of processing parameters for the maximized biodiesel yield, which is 95.15%, is shown in Table 6. The experimental study was conducted relying on the optimal values of the process variables as indicated in Table 6 to verify the findings at constant temperature of 50 °C. Table 7 also showed that the projected optimized response was closer to the experimentally optimized solution with a 3.314% error. Finally, a 92% response yield for the biodiesel blends was reached employing a 21:1 methanol/oil ratio and a 1.5% w/w NaOH catalyst in 110 min at 50 °C. According to ASTM D-6751 and IS-15607, physicochemical testing of biodiesel samples was performed, results are shown in Table 8 and according to this table it is found that the biodiesel prepared on optimized parameters has physicochemical properties within the range mentioned by IS-15607.

Table 6. Optimizing for optimum possible biodiesel production.

Parameters	Objective	Optimized Value RSM
Reaction time (Rt)	In-range	110
Methanol volumetric ratio	Minimized	21
Catalyst concentration (CC)	In-range	1.5
Biodiesel-yield	Maximized	95.15

Table 7. Results of model evaluation under optimal conditions.

S.No.	Methanol/Oil Volumetric Ratio	Reaction-Temperature (°C)	Catalyst-Loading (wt%)	Reaction-Time (min)	Predicted-Biodiesel Yield (%)	Experimental-Biodiesel Yield (%)	Error (%)
1	21	50	1.5	110	95.15	92	−3.423

Table 8. Physicochemical properties of biodiesel.

S.NO.	Property (Unit)	IS-15607	IS-15607 Limits	Biodiesel
1	Flashpoint (°C)	IS 1448	-	105.85
2	Viscosity at 40 °C (cSt)	IS 1448	6.18	4.20
3	Water and sediment (vol%)	D: 2709	Max 0.05	0.04

5. Conclusions

This study focused on the single-step transesterification method of biodiesel production from a blend of high-FFA WCO and low-FFA algal oil using a homogeneous base catalyst. Because WCO contains high levels of FFA and a two=-step transesterification has to be used for converting this high FFA oil to biodiesel which consumes time and energy, therefore algae oil was chosen to blend with WCO in a proportion such that the resulting oil contains 1% or less FFA. By using this process, WCO can be used in combination with algal oil to produce biodiesel using single step transesterification. Three process variables, catalyst concentration (0–2 wt%), methanol concentration (v/v) (20–60%) and reaction time (60–180 min) at a constant reaction temperature of 50 °C, were selected. The design and optimization studies were conducted using the Box-Behnken Design Technique in Response Surface Methodology (RSM). The maximum biodiesel yield of 92% is obtained under optimized conditions of catalyst concentration of 1.5% (w/w), methanol/oil ratio of 21:1 and reaction time of 110 min. RSM can be successfully used to model the reaction in order to maximize the biodiesel yield. By using this process, one can eliminate the time and energy consuming acid catalyzed transesterification process used for FFA reduction.

Author Contributions: The main contributions of the authors are as follows: conceptualization: S.J., N.K., M.B., V.P.S. and S.M.; methodology: N.K.S., N.K., M.B. and S.M.; structuring of paper: T.M.Y.K., S.J., M.B. and V.P.S.; investigation and data collection: M.B., N.K. and S.M.; resources: V.P.S., S.J., M.B., T.M.Y.K. and S.M.; data curation: M.B., N.K. and S.M.; writing—original draft: N.K., S.J., M.B. and V.P.S.; writing—review and editing: S.J., M.B. and N.K.S.; visualization: M.B., V.P.S. and S.J.; supervision: M.B., S.J. and S.M.; project administration: M.B., S.J. and T.M.Y.K. All authors have read and agreed to the published version of the manuscript.

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