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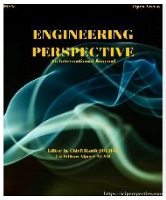

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Research Paper

Response Surface Method Based Optimization of the Viscosity of Waste Cooking Oil Biodiesel

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Abstract

In this study, biodiesel fuel production from waste sunflower oil and viscosity optimization was carried out. During the production process, catalyst ratio, alcohol ratio and reaction temperature were determined as variable parameters. Transesferication method was used as the production method. During the production process, the use of NaOH catalyst and methyl alcohol was provided. Biodiesel production steps with the transesterification method were discussed in detail. A total of 27 different biodiesel fuels were obtained with a catalyst ratio varying between 0.03% and 0.07%, alcohol content between 15% and 25%, and reaction temperature between 50 ° C and 70 ° C. All biodiesel fuels were analyzed and their characteristics were determined. In the optimization process, catalyst ratio, temperature and alcohol ratio were considered as input parameters, and viscosity as output parameters.Both 3D surface plots and 2D contour plots were developed using MINITAB 19 to predict optimum biodiesel viscosity. To predict biodiesel viscosity a quadratic model was created and it showed an R2 of 0.95 indicating satisfactory of the model. Minimum biodiesel viscosity of 4.37 was obtained at a temperature of 60, NaOH catalyst concentration of 0.07% and an alcohol ratio of 25%. At these reaction conditions, the predicted biodiesel viscosity was 4.247. These results demonstrate reliable prediction of the viscosity by Response surface methodology(RSM).

Keywords: Response surface methodology, biodiesel production, viscosity, waste cooking oil

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1. Introduction

Due to the rapid increase in the world population and technological developments, the number of vehicles and energy need are increasing. Diesel engines are widely used in passenger and freight transportation, agricultural and industrial activities due to their high efficiency and durability [1]. These vehicles are dependent on fossil fuels to meet the energy needs [2,3]. But fossil energy sources are non-renewable energy sources. For this reason, it becomes difficult to provide it in a sustainable way and to ensure energy security [4]. Emissions from diesel engines bring negative effects along with it. This means that the exhaust emissions from vehicles will cause problems for the environment and human health [5,6]. In order to close the gap between consumed energy and produced energy and to obtain sustainable clean energy, there has been a tendency towards alternative, renewable energy sources [7,8].

An alternative fuel should be economically competitive with diesel fuel, be environmentally safe and readily available. The most important alternative fuels for diesel engines are biodiesel fuels under biomass energy [9,10]. Biodiesel fuel can be produced from vegetable, animal or marine products. In addition, biodiesel can be produced from waste oils [11,12]. Different methods are used for biodiesel production. Biodiesel production can be realized by dilution, micro-emission, pyrolysis and transesterification methods [13,14]. The method of producing biodiesel as a result of the reaction of vegetable and animal oils with alcohol is called transesterification. Transesterification method is widely used due to its low cost, mild reaction conditions, ease of production and properties close to standard diesel fuel. This method also has disadvantages such as the difficulty of separation processes, the risk of side reactions and the large amount of water waste [15,16]. There are almost no aromatic compounds, carcinogenic substances and sulfur in its structure. It can be used in standard diesel engine without any change.

Biodiesel fuel has many advantages. Biodiesel fuel has basic advantages such as no risk of extinction, supporting agricultural development, reducing foreign dependency, and being environmentally friendly. The high cetane number decreases the knock value. Its high flash point increases transport and storage safety. It is a more lubricating fuel than standard diesel fuel and reduces wear values. Besides these advantages, there are also disadvantages. Its calorific value is lower than petrol based diesel fuel. This feature causes a slight decrease in power as a result of combustion in the engine, decrease in engine performance and increase in specific fuel consumption. Its viscosity is higher than diesel fuel, it is affected more quickly by cold weather conditions. This situation is a factor limiting the use of biodiesel in engines in cold climates and in pure form [17,18,19,20].

In experimental studies, it is necessary to know the experiment design, parameters and what to expect from the test result in order to reach a correct result. Even if all these conditions are met, it may be necessary to make a large number of the same analysis or experiment. Reducing the number of experiments provides labor, time and cost savings. For this reason, a design determined with the correct parameters and levels is a reason for preference [21]. Response Surface Method (RSM) is taking its place in current statistical methods [22,23]. RSM was developed by Box and Wilson in 1951 and was first applied to the chemical industry [24]. RSM is widely used in the formulation of a new product, improvement of existing product design, process optimization, process development and improvement [25]. Response Surface Methodology can be used in experiments with at least two or more parameters. It is used for mathematical modeling and optimization of the relationship between outputs and inputs based on the results obtained from experiment combinations consisting of different levels of parameters. This method is also known as the experimental design method [21]. RSM uses experimental modeling techniques used to determine the relationship between the system's response and the independent variables affecting it. It includes experimental strategies and optimization techniques to investigate the experimental space of process variables [26].

Anwar et al., have produced biodiesel from Australian native stone fruit by transesterification method. They took methanol oil molar ratio, catalyst ratio and temperature as variable parameters to use in the production process. Both 3D surface plots and 2D contour plots were developed using MINITAB 18 to estimate the optimum biodiesel yield. After optimization for maximum biodiesel yield, they found a methanol: oil molar ratio of 6: 1, KOH catalyst ratio 0.5% and a reaction temperature of 55 C. They determined the biodiesel yield produced under these reaction conditions as 95.9% [27]. Nayak et al., examined the optimization of methyl ester yield of biodiesel fuel produced from papaya oil using response surface method. They took temperature, catalyst amount, methanol / oil molar ratio and reaction time as variable parameters. Based on the optimum condition, the predicted biodiesel yield was 99.9% and the actual experimental value was 99.3% [28]. Latchubugata et al., optimized parameters such as temperature value, reaction time and methanol / oil molar ratio used in the process of biodiesel production from palm oil with the Response surface method. They produced biodiesel fuel at the parameter values reached as a result of optimization. As a result of the analysis they applied to the produced biodiesel fuel, they found that the optimization reached the result with high accuracy [29]. The response surface method has been applied in different working areas and successful results have been achieved. Güvercin et al., using the Response Surface Method, optimized the cutting parameters that affect the surface roughness. They successfully performed the determination of the parameter and the optimum value that most affected the results obtained in the experiments [26]. Ozmetin et al., conducted an experimental study on paint chemistry and optimized the results with the Response Surface Method. With the result they achieved by optimization, they reached the targeted values [21].

In this paper, biodiesel fuel production from waste sunflower oil and viscosity optimization were carried out depending on the alcohol ratio, catalyst ratio and temperature values. In the optimization performed with the response surface method, it has been concluded that biodiesel should be produced at 66.96 ° C temperature, 0.07% NaOH catalyst ratio and 25% alcohol ratio to obtain a minimum biodiesel viscosity of 4.227.

2. Material and Method

In this study, biodiesel fuel was produced from waste sunflower oil, depending on the alcohol ratio, catalyst ratio and temperature values. Transesterification method is used in the production of biodiesel fuel. The biodiesel production process and the response surface method stages applied to the analysis results obtained were examined in detail.

2.1 Biodiesel production

Transesterification method was used for the production of biodiesel from waste sunflower oil. This method consists of 6 steps: mixing alcohol and catalyst, reaction, separation, alcohol removal, glycerin neutralization and methyl ester washing process. The stages of biodiesel production by the transesterification method can be seen schematically in figure 1 [30-34].

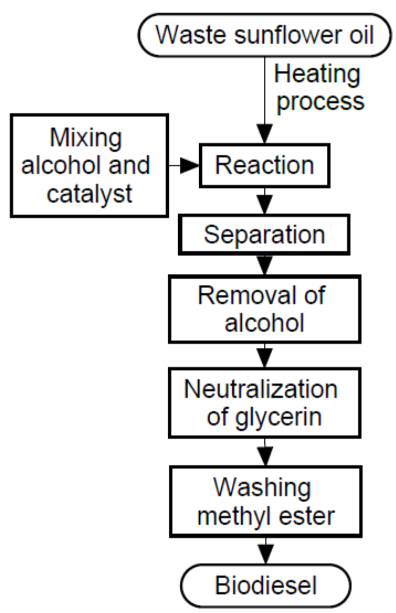


Figure 1. Biodiesel production process with transesterification method[30]

Beaker, glass bubble, cooler, density measuring device, separation funnel, heated magnetic stirrer with thermocouple, magnetic fish and precision scales were used for biodiesel production. The technical characteristics of the materials used in biodiesel production are given in table 1.

Table 1. Technical properties of materials used in biodiesel production

Material name	Waste sunflower oil	Sodium hydroxide (NaOH)	Methyl alcohol (CH_3OH)
Density (g/cm^3)	0.922	2.13	0.790-0.793
Molecular weight (g/mol)	-	40	32.04
Boiling point	-	-	64-65
Melting point	-	319-322°C	-
Resolution	1090 g/l	-	-
Refractive index	-	-	1.328-1.331

Table 2. Technical properties of viscosity device

Device Name	Brand / Model	Measuring Range	Temperature Range	Sample Volume
Viscosity	Omnitek D445	0.15-25,000 mm^2/s @ 40 °C	15-150 °C	8-16 ml

Waste sunflower oil was heated up to 80 ° C and then filtered. In order to evaporate the water molecules in the oil, it was kept at 120 ° C for about 1 hour. A homogeneous solution was prepared with NaOH catalyst and methyl alcohol and mixed with waste sunflower oil. Twenty-seven different biodiesel production was carried out, with catalyst ratio varying between 0.03% and 0.07%, alcohol content between 15% and 25%, and reaction temperature between 50 ° C and 70 ° C. The reaction time was kept constant at 1.5 hours in all production processes. The setup where the reaction taking place during the production process takes place is shown in Figure 2.

Figure 2. Reaction setup

After the reaction, the separation of glycerin from biodiesel took approximately 10 hours. The glycerin separation process in the biodiesel production process is shown in Figure 3.The fatty acids, catalysts and unreacted alcohols contained in the biodiesel obtained as a result of the reaction were removed by washing. The washing process was repeated until the fuel cleared. For this study, 5 repetitive washing processes were carried out. The washing process is shown in figure 4. After the washing process, drying and filtering process was applied to biodiesel fuel again.

Figure 3. Separating glycerin in biodiesel

Figure 4. Biodiesel fuel washing process

2.2 Design of Experiments

Response surface methodology was used for statistica analysis of the experimental data using the MINITAB 19 software. The Box-Behnken is one of the most commonly used response surface methodology designs. This design was used for statistical analaysis and designing of this experiment. The Box-Behnken design matrix was utilised to find the optimum conditions for minimum biodiesel viscosity. The experimental optimization was achieved via ANOVA (analaysis of variance) using MINITAB 19 software. The effect of process factors such as alcohol, NaOH catalyst concentration, and temperature were tested. Using these three factors at three levels required a total of 15 runs for identifying the optimum conditions for biodiesel viscosity. The ranges, coded symbols and levels of the factors are shown in Table 3. The design matrix fort he three factors was varied at three levels, namely -1, 0 and +1.

Table 3. Range and levels coded for independent factors

Factors	Unit	Symbol Coded	Range and Levels		
			-1	0	+1
Alcohol	(%)	A	15	20	25
NaOH catalyst concentration	(%)	C	0.03	0.05	0.07
Temperature	°C	T	50	60	70

Alcohol ratio ranged from 15% to 25%, NaOH catalyst concentrations were 0.03 – 0.07% and the reaction temperature was varied from 50 °C to 70 °C. The response factor (biodiesel viscosity) was correlated to the parameters using a full quadratic model. The general form of full quadratic model is expressed as follows,

$$Y = B_0 + B_1X_1 + B_2X_2 + B_3X_3 + B_{1,2}X_1X_2 + B_{1,3}X_1X_3 + B_{2,3}X_2X_3 + B_{1,1}{X_1}^2 + B_{2,2}{X_2}^2 + B_{3,3}{X_3}^2 \quad (1)$$

where Y is the predicted biodiesel viscosity; B_0 is a constant; B_1, B_2, and B_3 are regression coefficients; $B_{1,1}$, $B_{1,2}$, $B_{1,3}$, and $B_{2,3}$ are quadratic coefficient; and X_1, X_2, and X_3 are independet variables.

3. Simulation Results

The results of the Box-Behnken design model to optimize biodiesel viscosity parameters are shown in Table 4. In the experimental results, the viscosity of biodiesel ranged from 4.376 to 5.478 mm2/s. This design matrix also show the run order, experimental viscosity values and predicted viscosity values. To avoid systematic errors, all run orders were randomised.

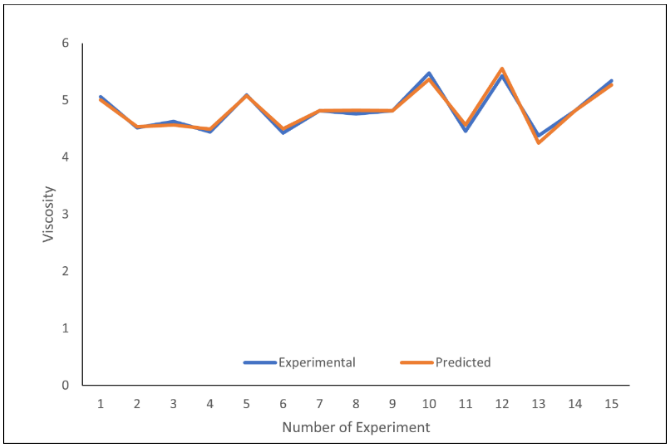
Table 4. Experimental matrix and Box-Behnken results

Exp. Number	Run Order	T	A	C	Temp. (°C)	Alcohol (%)	Catalyst (%)	Biodiesel Viscosity	
								Experimental	Predicted
1	5	-1	0	-1	50	15	0,05	5,058	5,00575
2	7	-1	0	1	50	25	0,05	4,518	4,53250
3	10	0	1	-1	60	15	0,07	4,627	4,56562
4	8	1	0	1	70	25	0,05	4,442	4,49425
5	6	1	0	-1	70	15	0,05	5,090	5,07550
6	4	1	1	0	70	20	0,07	4,423	4,49887
7	14	0	0	0	60	20	0,05	4,816	4,81600
8	11	0	-1	1	60	25	0,03	4,759	4,82037
9	15	0	0	0	60	20	0,05	4,816	4,81600
10	2	1	-1	0	70	20	0,03	5,478	5,36437
11	3	-1	1	0	50	20	0,07	4,453	4,56662
12	9	0	-1	-1	60	15	0,03	5,429	5,55713
13	12	0	1	1	60	25	0,07	4,376	4,24787
14	13	0	0	0	60	20	0,05	4,816	4,81600
15	1	-1	-1	0	50	20	0,03	5,341	5,26512

The predicted biodiesel viscosity values were obtained from Minitab software version 19.0 using a quadratic regression model by means of response surface methodology (RSM) analysis of experimental data. Minitab 19 program was used to calculate each parameter and the effects of their interactions with other parameters. Biodiesel viscosity was correlated with other parameters using the quadratic regression model shown in Equation (2).

$$V = 7.22 - 0.0302T - 44C + 0.059A + 0.000435T^2 + 161C^2 - 0.0033A^2 - 0.209TC - 0.00054TA + 1.047KA \quad (2)$$

Here, V is response, C is catalyst concentration, T is a reaction temperature, and A represents alcohol ratio.



5. Experimental and RSM fitting

Comparison of experimental and predicted biodiesel are shown in Fig. 5. It is seen that there is a sufficient correlation between RSM predictive values and experimental values confirming the acceptability of the model.

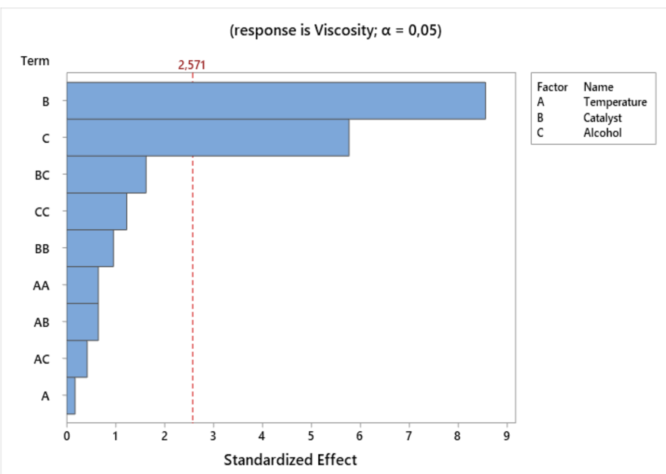


Figure 6. Effects of factors

Table 5. Regression coefficients

Term.	Coefficients	Standard Errors	T-Value	P-Value
Constant	4,8160	0,0746	64,52	0,000
T	0,0079	0,0457	0,17	0,870
C	-0,3910	0,0457	-8,55	0,000
A	-0,2636	0,0457	-5,77	0,002
$T \times T$	0,0435	0,0673	0,65	0,546
$C \times C$	0,0643	0,0673	0,95	0,383
$A \times A$	-0,0825	0,0673	-1,23	0,275
$T \times C$	-0,0417	0,0646	-0,65	0,547
$T \times A$	-0,0270	0,0646	-0,42	0,694
$K \times A$	0,1048	0,0646	1,62	0,166

In order to investigate the effects on biodiesel viscosity, linear, quadratic and interaction effects of parameters were taken into account. Table 5 and Figure 6 show the importance of these parameters in terms of the probability value (p-value). It also shows the obtained regression coefficients and calculated T-values. In the model, positive coefficients T, T2, K and KA had a positive effect on biodiesel viscosity, while A, A2, TC, TA and C had negative effects on biodiesel viscosity. Analysis of variance (ANOVA) was used to determine the importance and appropriateness of the quadratic model.

Table 6. ANOVA results for biodiesel viscosity

Source	Degree of Freedom	Sum of Squares	Mean square	F-Value	P-Value	Remarks
Model	9	1,88910	0,20990	11,22	0,008	Significant
Linear	3	1,77953	0,59318	31,71	0,001	Significant
T-Temperature	1	0,00050	0,00050	0,03	0,877	Not significant
C-Catalyst	1	1,22305	1,22305	65,37	0,000	Highly significant
A-Alcohol	1	0,55599	0,55599	29,72	0,003	Significant
Square	3	0,05580	0,01860	0,99	0,467	Not significant
T^2	1	0,01271	0,01271	0,68	0,447	Not significant
C^2	1	0,02329	0,02329	1,24	0,315	Not significant
A^2	1	0,01674	0,01674	0,89	0,388	Not significant
TC	3	0,05378	0,01793	0,96	0,480	Not significant
TA	1	0,00697	0,00697	0,37	0,568	Not significant
CA	1	0,00292	0,00292	0,16	0,709	Not significant
Lack-of-Fit	1	0,04389	0,04389	2,35	0,186	Not significant
Pure Error	5	0,09354	0,01871			
Total	3	0,08358	0,02786	5,59	0,155	
R^2=0.9575	2	0,00996	0,00498			

Table 6 shows the significance of the individual terms and their interaction on the selected response. The P value is used to check the significance of each regression coefficient by representing the error probability. The interaction effect of each cross product can be revealed through the p value[27]. It is found, C (Catalyst concentration), A (Alcohol) have significant effects on biodiesel viscosity. It is seen that C value has the lowest p value (0.000) and the highest F value (73.16) according to all other parameters. These results show that the C value is the most important parameter in biodiesel viscosity. According to the regression model in Equation (1), A has a positive effect and both C and reaction temperature (T) have negative effects on biodiesel viscosity. This implies that increasing A will increase the viscosity of the biodiesel. However, increase in C and T will decrease the viscosity of biodiesel. The ANOVA results showed that the linear term of T with p value was not significant (more than 0.05) and its quadratic term T2 with p value also was significant (more than 0.05). R2 also shows good correlation between independent parameters. In this study, R2 was found to be 95.75% and the corrected coefficient of determination (Adj. R2) was found to be 88.11%. This means that the model explains 95.75% of the variation in experimental data. As a result, the regression model developed for biodiesel viscosity was valid and showed a satisfactory experimental relationship between response and parameters.

3.1 Interaction effect of Alcohol and Temperature

The interaction effect of alcohol A and catalyst concentration, C on biodiesel viscosity in both the 3D surface plot and the contour plot are shown in Figure 3. With an increase of catalyst concentration 0.07(highest) and alcohol 25% (highest) biodiesel viscosity decrease. The minimum viscosity of biodiesel value of 4.227 mm2/s was found for NaOH 0.07 % (Run 12). Table 3 design matrix indicated that lowest NaOH concentration at 0.03% and mid-value of alcohol ratio at 20% resulted in highest biodiesel viscosity. When the Alcohol ratio remains unchanged at 25% and catalyst concentration is at lowest value of 0.03, the biodiesel viscosity decreases 4.759 mm2/s (Run 11). When the alcohol ratio was reduced to 15 % (lowest level), and with the highest value of catalyst concentration of 0.07%, the biodiesel viscosity was found to be 4.627 (Run10). Again, at alcohol ratio of 25%, and with the mid-value of catalyst concentration of 0.05%, the viscosity was found to be 4.442% mm2/s (Run 7). On the other hand, when the alcohol ratio was reduced to 15, and with the mid-value of catalyst concentration of 0.05% the yield rose up to 5.09 mm2/s (Run 6). Alcohol ratio affected total biodiesel viscosity. ANOVA from Table 6 confirmed that both A and C interaction were significant. The 2D contour plot with A and C interaction along with biodiesel viscosity is shown in Figure 7. It is easy to identify the optimum operating conditions and the related response values (viscosity) through the 2D contour plot. Therefore, both A and C are significant for lower biodiesel viscosity.

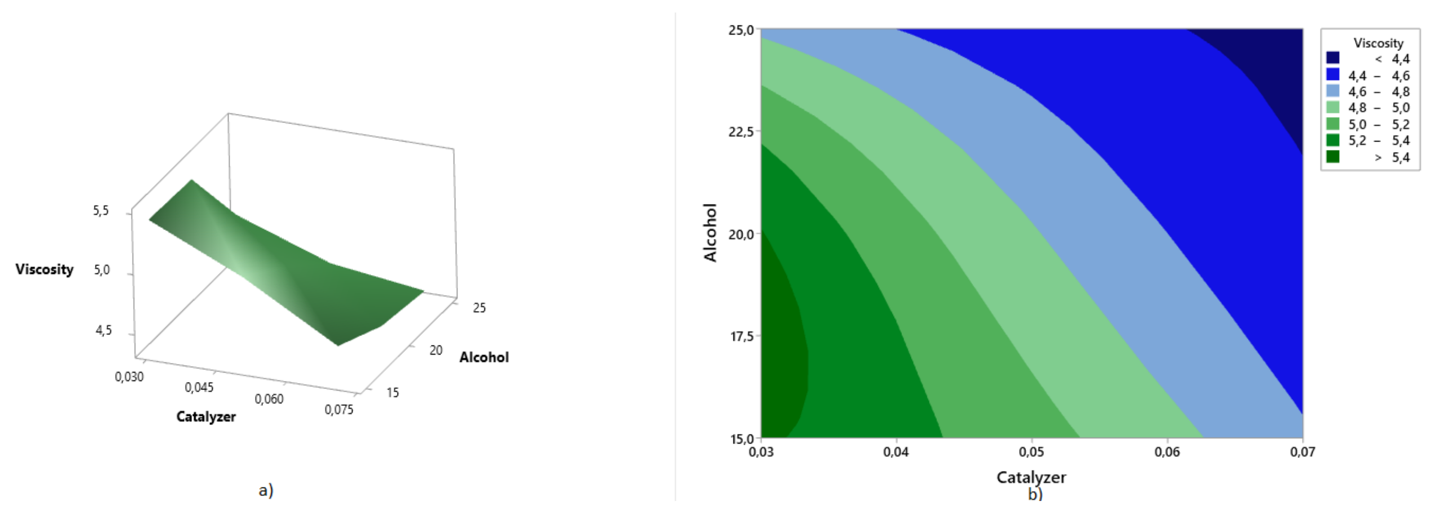


Figure 7. Interaction effect of Alcohol and catalyst concentration on bioediesel viscosity a) 3D surface plot, b) Contour plot

4. Conclusions

In biodiesel production process, catalyst ratio, alcohol ratio and reaction temperature were determined as variable parameters. Transesferication method was used as the production method. During the production process, the use of NaOH catalyst and methyl alcohol was provided. Biodiesel production steps with the transesterification method were discussed in detail. A total of 27 different biodiesel fuels were obtained with a catalyst ratio varying between 0.03% and 0.07%, alcohol content between 15% and 25%, and reaction temperature between 50 ° C and 70 ° C. All biodiesel fuels were analyzed and their characteristics were determined. A response surface methodology based on Box–Behnken design matrix was applied to achieve the optimum biodiesel viscosity. Three main parameters were changed separately at different intervals to estimate the biodiesel viscosity in this matrix. Based on the results, optimum biodiesel viscosity were found to be Alcohol ratio of 25%, catalyst concentration 0.07%, and a reaction temperature of 66.96 ◦C. The minimum biodiesel viscosity under such conditions was 4.376 mm2/s, which also confirmed the RSM model prediction of 4.227 mm2/s. ANOVA statistics of this study confirmed that catalyst concentration ratio has the most significant effect on the biodiesel viscosity, whereas reaction temperature does not seem to have any significant impact.

Nomenclature

ANOVA	Analysis of Variance
RSM	Response Surface Method

Conflict of Interest Statement

The authors declare that there is no conflict of interest.

CRediT Author Statement

Sedef Köse: Biodiesel production, methodology, article editing, **Mustafa Babagiray:** Supervision, using RSM, **Tolga Kocakulak:** Writing original draft and revision

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