IOP Conference Series:
Earth and
Environmental Science

PAPER • **OPEN ACCESS**

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To cite this article: J. Mohanraj *et al* 2022 *IOP Conf. Ser.: Earth Environ. Sci.* **1100** 012017

Intensification of Biodiesel Production by Optimizing Process Parameters from Waste Cooking Oil through Response Surface Methodology

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Abstract

This study focused catalytic transesterification process for the production of biodiesel from restaurant based waste cooking oil. Fifteen pre-designed experiments are used to explore the significance of three reaction parameters with three different levels such as methanol to oil ratio, catalyst concentration and agitation speed as well as their combined effect on biodiesel production. Box-Behnken experimental design based on Response Surface methodology (RSM) is utilized for optimizing the process parameters A quadratic model was developed to estimate biodiesel yield, and the R2 value was determined to be 0.99, indicating that the model is accurate. The result showed optimized process parameters for maximum biodiesel yield as methanol:oil molar ratio of 12.97:1, NaOH concentration of 0.1834 wt% and at the agitation speed of 1088 rpm. The obtained results reveal that the yield of 98.45% and the experimental yield of 97.80% respectively which shows the deviation of 0.7%.

Keywords: Waste cooking oil, transesterification, biodiesel, Box-Behnken, optimization, response surface methodology

1. Introduction

Biodiesel production has recently grown a lot of interest across the world as a result of increased global energy crisis. Many countries in the world have started to take a number of steps to address unexpected energy need particularly to replace conventional fuel fuels by alternative energy resources [1]. Vegetable oils were used as diesel fuels in al over the world since 1950. The need for energy has increased dramatically due to rapid development of modern industries and uncontrolled population growth [2]. Therefore, finding possible alternate energy sources are being investigated all over the world and the word "biodiesel" has cropped up in a lot of recent studies.

Biodiesel now are using up to 15% for worldwide transportation due to improved production and its lower emission. The developing countries are currently producing roughly 40% of the total global biofuel production. However, the increased biofuel production has brought serious difficulties on the existence of first-generation biofuels that represents the production of biodiesel from food sources [3]. The problems associated with first generation biodiesel are insisting the researchers to opt second generation biodiesel without affecting food chain. The second-generation biodiesels have the potential to save overall economy including use of waste oils and dumped land [4]. Biodiesel is biodegradable, renewable and safe for our environment. They are commonly derived from locally available sources which can contribute more to the rural economic growth [5]. The feedstock selection is critical since procuring 75% of the production cost. Availability, continuous supply, cost and its storage properties are the crucial parameters that should be considered for the selection of feedstock for biodiesel production. Uses of

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biodiesel in internal combustion engines are advisable one without any engine modification. The utilization of biodiesel for engine operation considerably reduces various poisonous gases due to oxygen content. But the emissions of nitrogen oxides (NOx) are more [6]. The issue with NOx emissions are also can be controlled by various modifications in engine including coating in engine components and exhaust gas recirculation etc [7, 8]. The use of raw vegetable oils for burning inside the engine has some drawbacks related to fuel spray and injection. Most of the vegetable oils have higher cetane numbers with reduced heating values. As compared to mineral diesel, their brake thermal efficiency is low, resulting in emission of harmful gases [9]. The higher viscosity of the fuel causes deposits in the combustion chamber, pump and injector [10]. Transesterification is the most cost-effective process for the production of biodiesel from high viscous oils with presence of suitable catalyst. It is suggested as the best technique that produces biodiesel with lower viscosity with improved quality. Due to higher conversion efficiency, an alkali catalysed transesterification is the commonly used technique. Several variables including process temperature, catalyst type, alcohol concentration, reaction time and stirring speed are the influencing parameters during biodiesel production [11]. The biodiesel production technology is considered as very complicated system engineering since it encompasses various processes including catalyst preparation, reaction and waste oil reuse.

Waste cooking oil is a possible source to replace food based oils for the production of biodiesel. They are the huge restaurant output obtained from cooking food. Due to higher free fatty acid components, repeated usage of vegetable oil for frying food is avoided to certain limit [12]. The disposal of this waste oil creates many environmental issues including water and soil pollution, human health concerns and disruption of the aquatic ecology [13]. As it is readily available, waste cooking oils are the cost-effective feedstock rather of being discarded into the environment. The waste oil production is depends on the population growth and the current outcome is huge all over the world. The properties of the waste cooking oil is completely different from raw vegetable oil since it undergone many chemical reactions during frying. Its characteristics can fluctuate based on frying conditions including temperature and time. The cooking and frying process breaks down the raw oil, causing the formation of diglycerides, monoglycerides and free fatty acids [14]. Furthermore, the viscosity and saponification number of the oil is increased owing to oxidation and polymerization. However, it is necessary to explore the conversion process in order to make better use of waste cooking oil into biodiesel. This study deals with the optimization of biodiesel preparation process using Response Surface Methodology. Design of experiments is a new way to conducting tests that is focused on process parameters in order to analyze and observe the results (response). They assist the researchers to get good quality finished products that is biodiesel in this case [15] by identifying most influencing parameters. RSM investigates the interaction between several explanatory variables with one or more response variables.

This study utilized Box-Behnken design matrix in order to determine the best experimental parameters for producing maximum biodiesel from waste cooking oil via alkaline transesterification process. To anticipate the optimum process condition, methanol:oil ratio, NaOH catalyst concentration, and agitation speed are modified within three-level in the design matrix.

2. Materials and Methods

2.1. Materials

The waste cooking oil was obtained from a KFC restaurant used especially for frying applications. The main reason for collecting waste oil from KFC is due to unique cooking technique which can make production of biodiesel with identical properties. The experimental facilities available with RVS Technical campus Coimbatore was used for the process. The tests were conducted using small scale laboratory reactors. Methanol, sodium hydroxide and the filter paper (Whatman) used for this process were supplied by the department of chemistry, RVS Technical Campus.

2.2. Biodiesel production

The set up is well equipped with reactor, temperature measuring unit, condenser and magnetic stirrer. The oil has an acid value of around 0.97, indicating the transesterification process is more enough to convert it into biodiesel.

The experiment initially was conducted with 50 g of waste cooking oil with measured quantity of alcohol and NaOH catalysts. The optimization experiments was conducted by taking three different combinations of methanol:oil molar ratio (5:1, 10:1 and 15:1), NaOH catalysts (0.1325, 0.1767 and 0.2209 wt%) and agitation speed (1000, 1100 and 1200 rpm). Fig. 1 shows general flow chart about the current experimental study.

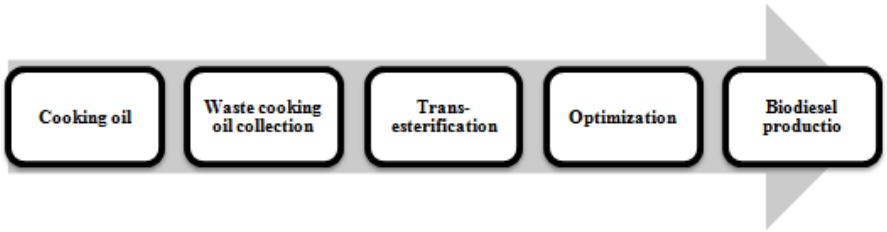


Fig. 1 Process flow chart

The process was performed at room temperature. The raw waste cooking oil was processed with 1:10 molar ratio of oil to methanol utilizing NaOH (0.1325 wt%). For the process the oil was heated initially up to 60 °C. The powdered NaOH is mixed thoroughly with methanol using magnetic stirrer. After that the mixture is slowly poured into oil and again the mixing process is performed using magnetic stirrer at 1000 rpm. The temperate of the mixture is maintained at 65 °C for 8 hour. In a separating funnel, the mixture is then allowed to settle down by gravitational force. Glycerin is identified at the bottom, whereas methyl ester is separated at the top. The treated mixture was again transferred to another glass vessel for further phase separation. For that 1 litre of water is mixed with 0.3 milliliters of sulphuric acid. The solution is then slowly put into the biodiesel and thoroughly mixed and finally allowed to settle. The washed biodiesel is filtered by a Whatman fiter paper in order to remove further traces. The yield of biodiesel is calculated by the following equation.

$$Biodiesel\ yield\ (\%) = \frac{Weight\ of\ biodiesel\ produced}{Weight\ of\ untreated\ oil\ used}\ x\ 100 \qquad (1)$$

3. Results and Discussion

The untreated waste cooking oil before the experiment were analyzed for its various basic properties including density, viscosity, flash point, acid value and calorific value. Table 1 and 2 illustrates the properties of waste cooking oil and its fatty acid contents respectively. The waste cooking oil has majority of unsaturated fatty acids, including linoleic acid and oleic acid.

Table 1. Properties of the waste cooking oil

Property	Values
Density (kg/m^3)	935
Kinematic viscosity (cSt)	10.6 at 40 °C
Flash Point (°C)	252
Acid number (mg of KOH/g)	0.97
Calorific Value (MJ/kg)	31.2

Table 2. Fatty acid composition of waste cooking oil

Fatty acid	Structure	Composition (wt%)
Pamitic Acid	16:0	8.81
Stearic Acid	18:0	6.96
Oleic Acid	18:1	15.4

Linoleic Acid	18:2	61.8
Linolenic Acid	18:3	1.35
Arachidic Acid	20:0	3.62
Lognoceric Acid	24:0	1.01

3.1. Process optimization

For the statistical analysis was carried out based on Box-Behnken response surface method. For this purpose Minitab 17 and analysis of variance (ANOVA) was used to achieve process optimization. The optimization was done by considering three parameters at three stages resulting in a total of fifteen runs. Table 3 list out the components and its levels considered for the analysis.

Table 3 Parameters and its levels

Component	Symbol	Range		
		-1	0	1
Methanol to oil ratio (mol/mol)	M	5:1	10:1	15:1
NaOH concentration (wt%)	C	0.1325	0.1767	0.2209
Agitation speed (rpm)	S	1000	1100	1200

The experimental design matrix is shown in Table 4. To avoid systemic mistakes, the experiments were conducted in a random order. The biodiesel output ranged from 30.14% to 94.45% during various runs. To forecast biodiesel yield as a function of selected input parameters an empirical equation response to the independent variables is defined by equation (2), the obtained regression coefficients, estimated T-values, and P-values using ANOVA are summarized in Table 5.

$$Yield\ of\ biodiesel\ (\%) = -1966 + 19.96M + 5184C + 2.681S - 0.3555M^2 - 10820C^2 - 0.001161S^2 - 40.84MC - 0.00300MS - 0.622CS \quad (2)$$

The ANOVA table shows that the confidence level is 95% hence the model is extremely significant. The findings show that the model is very significant due to higher F value and lower P value. During this study, M, C and MC have significant effects on transesterification process. The value of coefficient of determination and adjusted coefficient of determination is 99.62% and 99.13% respectively which shows the model is accurate. Table 6 shows ANOVA results with respect to reaction variables.

Table 4. Experimental design matrix

Experiment Number	Run	M	C	S	Methanol	NaOH	Agitation Speed	Experimental Yield	Predicted Yield
1	2	-1	-1	0	5	0.1325	1100	30.14	31.91
2	11	1	-1	0	15	0.1325	1100	76.32	73.29
3	5	-1	1	0	5	0.2209	1100	74.09	73.61
4	13	1	1	0	15	0.2209	1100	76.11	78.90
5	3	-1	0	-1	5	0.1767	1000	59.01	62.10
6	9	0	0	0	10	0.1767	1100	93.11	94.45
7	12	1	0	-1	15	0.1767	1000	88.00	88.43
8	7	-1	0	1	5	0.1767	1200	62.00	62.48
9	15	1	0	1	15	0.1767	1200	84.42	82.81
10	6	0	0	0	10	0.1767	1100	94.27	94.45

11	10	0	-1	-1	10	0.1325	1000	47.72	48.44
12	4	0	1	-1	10	0.2209	1000	76.00	77.59
13	1	0	-1	1	10	0.1325	1200	52.00	51.32
14	14	0	1	1	10	0.2209	1200	70.09	69.47
15	8	0	0	0	10	0.1767	1100	94.14	94.45

Table 5. Regression coefficient

Term	Effect	Coefficients	Standard errors	T-Value	P-Value
Constant		-1966	0.783	120.11	0.000
M	23.375	-19.96	0.619	18.89	0.000
C	23.625	5184	0.619	19.09	0.000
S	-2.750	2.681	0.619	-2.22	0.062
M*M	-17.775	-0.3555	0.853	-10.42	0.000
C*C	-42.275	-10820	0.853	-24.78	0.000
S*S	-23.225	-0.001161	0.853	-13.62	0.000
M*C	-18.050	-40.84	0.875	-10.31	0.000
M*S	-3.000	-0.003	0.875	-1.71	0.130
C*S	-5.500	-0.622	0.875	-3.14	0.016

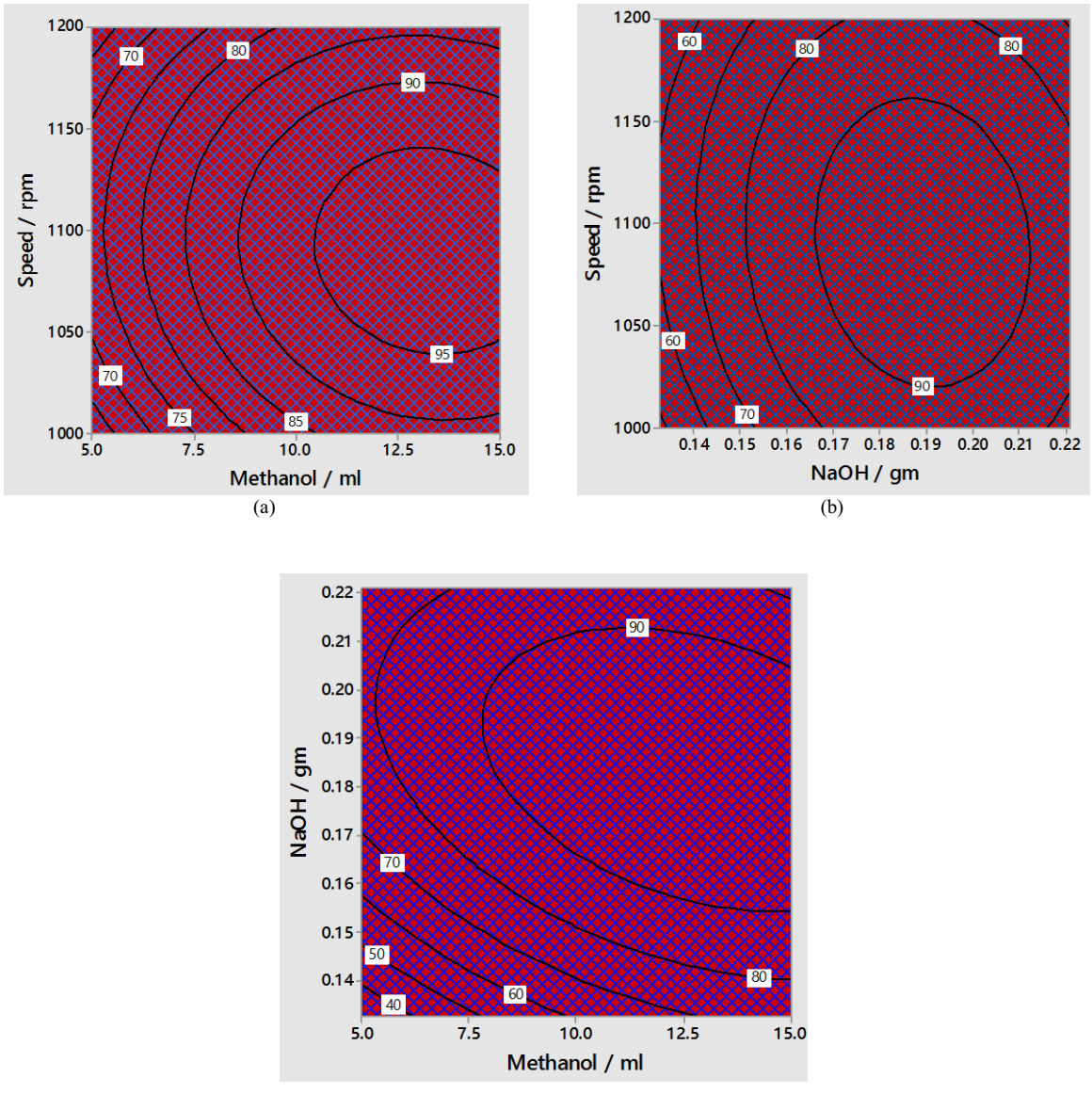
Table 6. ANOVA results for biodiesel yield with respect to reaction variables

Source	Sum of squares	Degree of freedom	Mean square	F-value	P-value	Remarks
Model	3604.44	9	400.49	435.15	0.000	Significant
M	1512.50	1	1512.50	1742.21	0.000	Significant
C	1104.50	1	1104.50	1310.20	0.000	Significant
S	2.00	1	2.00	2.09	0.062	
M*M	157.96	1	157.96	168.42	0.000	Significant
C*C	410.59	1	410.59	426.37	0.000	Significant
S*S	55.33	1	55.33	57.22	0.000	Significant
M*C	342.25	1	342.25	352.76	0.000	Significant
M*S	30.25	1	30.25	31.69	0.130	
C*S	20.25	1	20.25	21.06	0.016	
Lack of fit	3.244	4	1.302	16.21	0.041	
Pure error	0.212	3	0.031			
Total	3604.44	16				
R^2=99.62	Adj R^2=99.13					

3.2. Interaction effects between process components

Fig. 2 depicts the 2D contour plot between the methanol:oil molar ratio, concentration of NaOH catalyst and agitation speed. Addition of methanol with oil has a major role in regulating the transesterification rate. The yield of biodiesel is increased roughly in this case for methanol:oil molar ratio maintained up to 13:1. According to the study reported by Leung et al [16, 17] an addition of methanol shifts the reaction equilibrium to the biodiesel yield.

However, the quality of the oil is important one for selecting ratio of methanol to oil [18]. Further increase of methanol decrease the yield of biodiesel. From figure it can be observed that the agitation speed interact with methanol ratio has lower impact on the biodiesel yield. Fig. 3 shows the optimization plot for biodiesel yield based on the selection of input parameters. Waste cooking oil mixture at methanol:oil molar ratio of 12.97:1, NaOH concentration of 0.1834 wt% and at the agitation speed of 1088 rpm predicted the maximum biodiesel yield of 98.45% which is also confirmed with the experimental yield of 97.80%.



(a) (b)

(c)

Fig. 2. Biodiesel yield with respect to (a) methanol:oil molar ratio and agitation speed (b) catalyst and agitation speed (c) methanol:oil molar ratio and catalyst

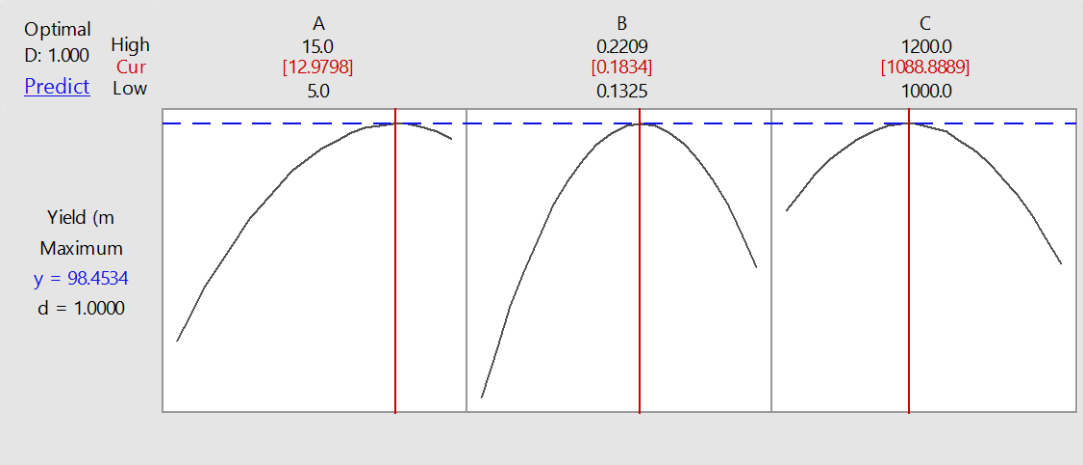


Fig. 3. Optimization plot for biodiesel yield

4. Conclusion

In this study, a homogeneous transesterification method was used for producing biodiesel from waste cooking oil. In order to enhance the production, the reaction parameters were optimized using Box-Behnken design based on response surface methodology (RSM). The plan optimized the parameters to produce maximum biodiesel of 98.45% at the optimized parameters of 12.97:1 methanol:oil molar ratio, 0.1834% NaOH concentration and 1088 rpm agitation speed. The obtained results revealed the experimental yield of 97.80% with low level error of 0.7% compared to experimental results. According to ANOVA results, the methanol:oil molar ratio and concentration of NaOH catalyst has significant effect rather than agitation speed on the yield of waste cooking oil biodiesel.

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