



Optimization of Biodiesel Production from Castor Oil Using a Microwave Via Response Surface Methodology (RSM)

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The purpose of this research work was to investigate the optimum operating conditions for biodiesel production from castor oil using a microwave. The Box–Behnken design of experiment was carried out using the Design Expert 7. A Response Surface Methodology (RSM) was used to analyze the influence of the process variables (molar ratio of methanol to castor oil, catalyst concentration, reaction time, and microwave power) on the biodiesel yield. Optimizing biodiesel production reaction based on the yield of reaction by means of response surface method showed that 1.44 wt.% of catalyst concentration, 7.12:1 molar ratio of alcohol to oil, microwave power of 500 W and 120 seconds of reaction time produces the best results for maximizing the conversion percent. Finally, Biodiesel samples were analyzed using Gas Chromatography (GC) method for determination of fatty acid methyl ester yield. The optimum obtained yield of reaction was 92.15% with above operating conditions and the reaction temperature was 58 °C.

1. INTRODUCTION

Biodiesel is an alternative biodegradable and nontoxic diesel type fuel which is essentially Carbon neutral and free of sulfur. It is usually produced by a transesterification reaction of vegetable oils or animal fats with a low-molecular weight alcohol such as methanol or ethanol using sodium or potassium hydroxide as a catalyst. Industrially, the most common reaction type of biodiesel production is a basic homogeneous reaction [1]. In this reaction, triglycerides, as the main components of vegetable oils, react with an alcohol to produce fatty acid mono-alkyl esters and a glycerol as a by-product. Methanol is the most commonly used alcohol because of its low price compared to other alcohols. In this case, the reaction is referred to as methanolysis. Generally, this reaction is catalyzed by a basic or an acid catalyst. The alkali catalysts are the most commonly used, because they make the process faster and the reaction conditions are more moderated. The stoichiometry of the methanolysis reaction requires three moles of methanol and one mole of triglyceride to give three moles of Fatty Acid Methyl Esters (FAMEs) and one mole of glycerol. This is the general transesterification reaction that consists of a

number of consecutive reversible reactions. The first step is the conversion of triglycerides to diglycerides, which is followed by conversion of diglycerides to monoglycerides and of monoglycerides to glycerol, yielding one methyl ester molecule from each glyceride at each step [2, 3]. Several techniques have been used in the literature for the biodiesel synthesis which includes conventional heating [4], supercritical methanol [5], ultrasonic method [6], and microwave irradiation [7]. Efficiency of the microwave heating is significantly higher when compared to the conventional method of transesterification reaction. Microwave assisted biodiesel production requires less time for reaction as well as for separation of products [8]. The application of microwave irradiation in conjunction with the use of catalysts or mineral supported reagents enables organic reactions to occur expeditiously at ambient pressure, thus providing unique chemical processes with special attributes such as enhanced reaction rates, higher yields, and the associated ease of manipulation [9]. Castor oil, extracted from the seeds of *Ricinus communis* plant, is viscous, pale yellow, non-volatile, and non-drying oil. Opposed to other vegetable oils, it is characterized by its indigestibility, partial solubility in alcohol, high hygroscopicity, and high viscosity [10]. Like other vegetable oils, castor oil is mainly constituted by triglycerides which consist of three fatty acids and one glycerol molecule. The fatty acids of this oil consist of

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ricinoleic (which is the main fatty acid), linoleic, oleic, and saturated fatty acids [11]. Response Surface Methodology (RSM) is an effective statistical technique used for investigation of complex processes whose mechanisms are not completely known. RSM, a combination of some mathematical and statistical procedures, is applied to study relationships between independent variables and response. It uses quantitative data obtained from the accurately designed experiments to solve multivariate equations. Response surfaces are the graphical representations of these equations. These surfaces actually explain individual, cumulative, and interactive effects of test variables on response. Furthermore, it estimates optimal factorial combination of variables that results in maximum response [12].

Cavalcante et al. [13] studied trans-esterification of castor oil with ethanol using a central composite rotatable design. They determined the optimum reaction conditions as ethanol to oil molar ratio of 11, catalyst amount of 1.75 wt.% KOH, and reaction time of 90 min and obtained $\approx 86.0\%$ of biodiesel yield. Ramezani et al. [14] used Taguchi method for optimization of castor oil transesterification. Optimum reaction conditions were determined as methanol to oil molar ratio of 8, catalyst amount of 0.5 wt.% NaOH, reaction time of 120 min, reaction temperature of 65 °C, and mixing intensity of 400 rpm. According to their results obtained using Taguchi method, the optimum reaction conditions produces biodiesel yield of 87.0%. Yeong and Park [15] optimized the biodiesel production from castor oil using response surface methodology. They determined that the optimum process parameters for 92.0% yield of biodiesel are the reaction time of 40 min, reaction temperature of 35.5 °C, methanol to oil molar ratio of 8.24, and catalyst concentration of 1.45 wt.% KOH. Production of biodiesel from castor oil using iron II doped zinc oxide nanocatalyst with maximum yield of 91% has been performed by Baskar and Soumiya [16]. The optimum conditions were 50 min of reaction time, 55 °C temperature, 14 wt.% of catalyst loading and 12:1 methanol/oil ratio. Their results also revealed that the iron II doped ZnO nanocatalyst is a one of the suitable heterogeneous catalysts for the production of biodiesel under mild reaction conditions.

Recently, methanolysis of purified castor oil using a microwave energy and parametric optimization have been studied by Hailegiorgis et al. [17]. The yield of treated castor oil was 95.6% in comparison with the yield of 84% for the untreated oil. The reaction time has also been reduced from 60 min to 30 min using the microwave.

With the above considerations, this research work investigates the optimization of biodiesel production from castor oil using a microwave by application of Box–Behnken design. Box–Behnken design is a spherical, revolving response surface methodology (RSM) design that is consisted of a central point and the

middle points of the edges of the cube circumscribed on the sphere [15]. RSM was used to optimize the reaction parameters and determine the relationship between the reaction parameters. Using the experimental design method, optimized effects of process parameters such as molar ratio of methanol to castor oil, catalyst concentration, reaction time, and microwave power on the biodiesel yield have been studied.

2. MATERIALS AND METHODS

2.1. Materials and equipment

Purified castor oil was purchased from a local departmental store (Tehran, Iran) with negligible FFA less than 1 wt.% and MW of 927 g/mol with typical compositional analysis measured by a gas chromatography (GC) model PerkinElmer-Clarus 580 model. The inert temperature of GC was 250 °C and helium gas (purity 99.999%) with 0.001 L/min flow used as mobile phase. The oil compositions determined by the GC are given in Table 1 as shown in Fig. 1. Methanol (99.9% purity, Merck, Germany) was used as a reactant for the esterification reaction. Potassium hydroxide (KOH) pellets (99.99% purity, Merck, Germany) were applied as the reaction catalyst. 2-Propanol alcohol (99.9% purity, Merck) has been used for the titration of castor oil to find the amount of free fatty acids (FFAs) in the oil before esterification reaction.

TABLE 1. Compositional analysis of castor oil

| Fatty acid composition | wt% |
|------------------------|-------|
| C15:0 | 7.38 |
| C16:0 | 3.51 |
| C18:0 | 3.67 |
| C18:1 | 1.38 |
| C18:2 | 12.15 |
| C18:3 | 1.03 |
| C20:1 | 0.61 |
| Ricinoleic | 60.81 |
| C24:1 | 9.46 |

Experimental runs have been conducted in a modified domestic microwave oven model Samsung M 2030 W, made in Malaysia with exiting variable power. A Teflon tube with an inner diameter of 6 mm and an overall length of 4.8 meter was placed spirally inside the microwave chamber as a reactor. A Peristaltic pump model P566 made by Green Bio-Kimia Co., (Tehran, Iran) was used for material transfer to the microwave reaction chamber. The oil flow rate could be controlled by motor rate of 5-110 RPM between 0.5 to 230 ml/min which corresponds to 0.06-7.2 lit/hr. An external

agitator was included to ensure uniform mixing of the reaction mixture.

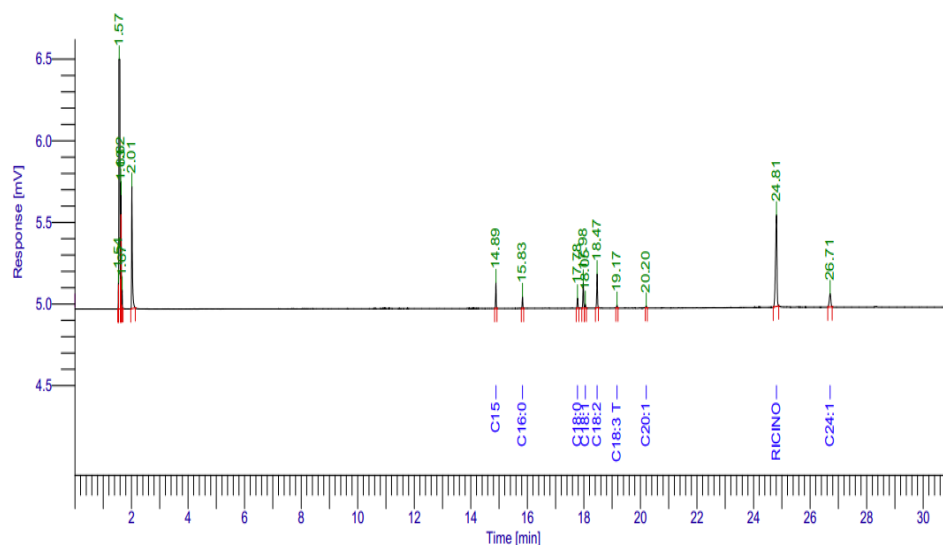


Figure 1. GC results for castor oil used for the esterification reaction

A temperature sensor was used to record the reaction temperature in the reactor outlet. In this research the gas chromatography apparatus of PerkinElmer-Clarus 580 with the BPX - BIOD5 column ($12\text{ m} \times 0.32\text{ mm} + 2\text{ m} \times 0.1$) with FID detector was used to determine the percentage of methyl esters according to the BSEN 14103 standard. Stabinger viscometer (SVM3000) was used for measuring the viscosity and density of biodiesel. Mini Flash FLP device that is manufactured by Grubbs was used to measure the flash point by closed cup method. Freezing Point Tester (PT-1220) device that is manufactured by an Iranian company (AbzarAzma) was used to measure the cloud point of biodiesel. Trial version of Design Expert software has been used for the experimental design analysis.

2.2. Experimental procedure

For each experiment, 200 grams of castor oil were added to the premixed homogeneous solution of methanol and KOH catalyst. The mixture was stirred for 10 seconds and then pumped into the microwave oven, under matrix conditions: reaction times of 60, 120 and 180 seconds; catalyst concentrations in the range of 0.5–1.5 wt.%; molar ratio of methanol to castor oil as 6–12 and microwave power of 200–600 watts. The schematic diagram of the biodiesel production system using a microwave is shown in Fig. 2 For analyzing the samples, 15 ml of the reaction products are taken by a falcon conical bottom tube after 60, 120, and 180 seconds, and immediately placed into an ice container and were kept for about 15 minutes to freeze the undesired reactions. The samples were put in the centrifuge for 2 minutes at 4000 rpm to separate the methyl ester phase. After centrifugation, two phases of methyl esters and glycerin were obtained. The upper phase consisted of methyl esters was weighed and

separated. Finally, 50 mg of biodiesel phase was transferred into micro-tubes and mixed with one ml of internal standard solution (C17) with a concentration of 7 mg/ml.

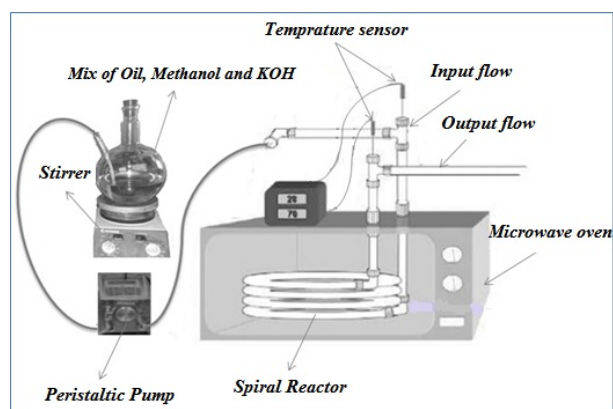


Figure 2. Experimental setup for biodiesel production using a microwave

2.2.1. Fuel property measurement methods

For measuring the density and viscosity of biodiesel, the device has been turned on and kept for a few moments to reach the test temperature ($40\text{ }^{\circ}\text{C}$). About 4 ml of biodiesel sample was injected into the device slowly and initiated the test by pressing the start key. The viscosity and density are reported at a setting temperature after 2 minutes.

The flash point of biodiesel was measured with a closed cup method. The initial and final testing temperatures had to be entered to the device and the start key was pressed to initiate the test. When the device reaches the initial temperature, a message is given by the device for filling the sample into the cup and pressing the run key by the device. One ml of the sample is poured into the

container and the test begins. Ignition of the device is carried out after every 5 °C increase of the sample temperature. The initial temperature was set at 101 °C on the device and with the device temperature rise the ignition flash point was set. Flash point was determined by raising the temperature and the ignition.

For measuring the cloud point of biodiesel manually, the sample is placed into the device and experimenting is started with decreasing the temperature at a constant rate. The sample is continuously monitored by optical detectors and as soon as a cloud is observed on the biodiesel sample the temperature is recorded. Molecular weight of the castor oil has been calculated by using the following Equation;

$$M_{W_{oil}} = 3 \times \sum (M_{W_i} \times X_i) + 38.049 \quad (1)$$

in which, M_{W_i} is the molecular weight of the fatty acids in the oil and X_i is the fatty acid compositions in the oil.

2.2.2. GC analysis

The samples were analyzed for fatty acid methyl esters (FAMES) formation at a predetermined time interval by Gas Chromatograph. The analysis conditions were: detector FID 250 °C, Helium (Purity 99.99) as carrier gas, split flow 50ml/min. Temperature planning of the column was set according to EN 14103 standard. At the first column, temperature was 60°C and fixed for 2 min then reached 210°C with the temperature gradient of the 10°C/min and immediately reached 230°C with the temperature gradient of 5°C/min and was kept at this temperature as the final temperature for 10 min. The percentage of FAMES yield was calculated using Eq. (2). The percentages of free fatty acids in the samples were determined using stock solution (methyl heptadecanoate and n-heptane) [18].

$$FAME\% = \frac{\sum A - A_{EI}}{A_{EI}} \times \frac{C_{EI} \times V_{EI}}{m} \times 100 \quad (2)$$

$\sum A$: Total peak area from the methyl ester in C_{14} to that of C_{24} ;

A_{EI} : Peak area corresponding to methyl heptadecanoate;

C_{EI} : Concentration of the methyl heptadecanoate solution (mg/ml);

V_{EI} : Volume of the methyl heptadecanoate solution (ml);

m: Mass of the sample (mg);

2.3. Statistical analysis

In the present work, the three levels and four factors with Box Behnken experimental designs were used to investigate and validate the process parameters affecting the biodiesel production. The Design Expert (Trial version, Stat-Ease, Inc., USA) was used for regression and graphical analyses of the obtained data.

Biodiesel yield was selected as a response. Range and levels of the investigated variables are listed in Table 2.

TABLE 2. Experimental range and levels of the independent variables

| Variables | Symbol coded | Range and levels | | |
|------------------------------|--------------|------------------|-----|------|
| | | -1 | 0 | +1 |
| Reaction time (s) | X_1 | 60 | 120 | 180 |
| Methanol/oil molar ratio | X_2 | 6:1 | 9:1 | 12:1 |
| Microwave power (W) | X_3 | 200 | 400 | 600 |
| Catalyst concentration (%wt) | X_4 | 0.5 | 1 | 1.5 |

Factors were reaction time (X_1), methanol/oil molar ratio (X_2), power of microwave (X_3), and catalyst concentration (X_4). Reaction time levels were 60 and 180 seconds, Methanol/oil molar ratio levels were 6:1 and 12:1, power of microwave levels were 200 and 600, Catalyst concentration levels were 0.5 and 1.5% by weight of oil. Central values (zero level) for the experiments were: 120 seconds for reaction time, 9:1 for methanol/oil molar ratio, 400 watt for power of microwave and 1.0% for catalyst concentration. Once the experiments were performed, the response variable (biodiesel yield) was fitted a second order model in order to correlate the response variable to the independent variable. The general form of the second degree polynomial equation is as follows [19]:

$$Y = b_0 + \sum_{i=1}^k b_i X_i + \sum_{i=1}^k b_{ii} X_i^2 + \sum_{i>j}^k \sum_j b_{ij} X_i X_j + \varepsilon \quad (3)$$

where i and j are the linear and quadratic coefficients, respectively, b is the regression coefficient, k is number of the factors studied and optimized in the experiment, and ε is the random error.

3. RESULTS AND DISCUSSION

3.1. Regression surface analysis and analysis of variance

Non-coded values of the reaction parameters and the responses for the experiments are shown in Table 3. The second order polynomial equation based on the coded values that was obtained using multiple regression analysis of the experimental data is presented as;

$$\begin{aligned} Yield (\%) = & 84.59 - 2.65X_1 - 4.97X_2 + 3.73X_3 + 11.41X_4 \\ & + 2.21X_1X_2 + 0.84X_1X_3 + 6.58X_1X_4 - 4.45X_2X_3 \\ & - 8.39X_2X_4 + 8.61X_3X_4 - 8.19X_1^2 - 3.32X_2^2 \\ & - 6.80X_3^2 - 15.49X_4^2 \end{aligned} \quad (4)$$

here, Y is the response (yield of FAME), and X_1 , X_2 , X_3 and X_4 are the values in the coded form of the studied variables.

TABLE 3. Box-behnken design matrix for four variables and the response

| Run | Run | | | | Run |
|-----|----------|-------------|-----------|------------------------|-------|
| | Time (s) | Molar ratio | Power (W) | Catalyst concentration | |
| 1 | 1 | 1 | 1 | 1 | 71.23 |
| 2 | 2 | 2 | 2 | 2 | 67.68 |
| 3 | 3 | 3 | 3 | 3 | 78.17 |
| 4 | 4 | 4 | 4 | 4 | 60.37 |
| 5 | 5 | 5 | 5 | 5 | 70.64 |
| 6 | 6 | 6 | 6 | 6 | 70.61 |
| 7 | 7 | 7 | 7 | 7 | 42.08 |
| 8 | 8 | 8 | 8 | 8 | 41.92 |
| 9 | 9 | 9 | 9 | 9 | 71.22 |
| 10 | 10 | 10 | 10 | 10 | 85.19 |
| 11 | 11 | 11 | 11 | 11 | 75.46 |
| 12 | 12 | 12 | 12 | 12 | 63.67 |
| 13 | 13 | 13 | 13 | 13 | 58.39 |
| 14 | 14 | 14 | 14 | 14 | 52.11 |
| 15 | 15 | 15 | 15 | 15 | 90.62 |
| 16 | 16 | 16 | 16 | 16 | 80.08 |
| 17 | 17 | 17 | 17 | 17 | 65.43 |
| 18 | 18 | 18 | 18 | 18 | 91.21 |
| 19 | 19 | 19 | 19 | 19 | urity |
| 20 | 20 | 20 | 20 | 20 | 85.82 |
| 21 | 21 | 21 | 21 | 21 | 66.09 |
| 22 | 22 | 22 | 22 | 22 | 87.43 |
| 23 | 23 | 23 | 23 | 23 | 71.55 |
| 24 | 24 | 24 | 24 | 24 | 84.69 |
| 25 | 25 | 25 | 25 | 25 | 56.72 |
| 26 | 26 | 26 | 26 | 26 | 70.34 |
| 27 | 27 | 27 | 27 | 27 | 65.56 |
| 28 | 28 | 28 | 28 | 28 | 81.71 |
| 29 | 29 | 29 | 29 | 29 | 83.29 |

Analysis of variance (ANOVA) for response surface quadratic model for the FAME content is shown in Table 4. The Fisher F-test ($F_{\text{model}} = 35.01$) with a very low probability value (<0.0001) demonstrates a very high significance for the regression model. To test the fit of the model, the regression equation and

determination coefficient (R^2) were evaluated. In this case, the value of the determination coefficient ($R^2 = 0.9722$) indicates that the sample variation of 2.78% for biodiesel production is attributed to the independent variables. The regression model provided accurate description of the experimental data indicating

successful correlation among the four transesterification process parameters that affect the yield of biodiesel. The value of the adjusted determination coefficient ($\text{Adj.}R^2 = 0.9445$) is also very high to advocate for a high significance of the model [20]. The regression analysis indicates that all the four parameters had significant influence on yield of biodiesel, which was confirmed by the P-values of the analysis. The response surfaces were fitted using process variables that were found to be significant after the analysis. The P-value of the lack of fit analysis was 0.208, which is more than the 0.05 (confidence level is 95%).

Effects of different reaction variables on the response can be studied based on the data listed in Table 5. The table provides the F-values and Prob>F values (or P-value) that indicate the significance of each coefficient. In general, larger F-value and smaller p-value indicate higher significance of the corresponding coefficient [21]. This implies that the variable with the largest effect was the term of catalyst concentration and squared term of catalyst concentration. This great importance of catalyst concentration in conversion to biodiesel was also emphasized in the literature [22]. The linear effect of time reaction is less significant than those of the other three factors. Interactive effect of alcohol to oil molar ratio and catalyst concentration are more significant than other interaction factors, and the quadratic effect of the molar ratio is lower than those of the other three squared factors.

3.2. Interactive effects of variables on reaction yield

The 3D response surface and the 2D contour plots are generally the graphical representations of the regression equation, and the 2D contour plots are presented in Figs. 3-8. Each contour curve represents an infinite number of combinations of two test variables with the other two that maintained at their respective optimum levels.

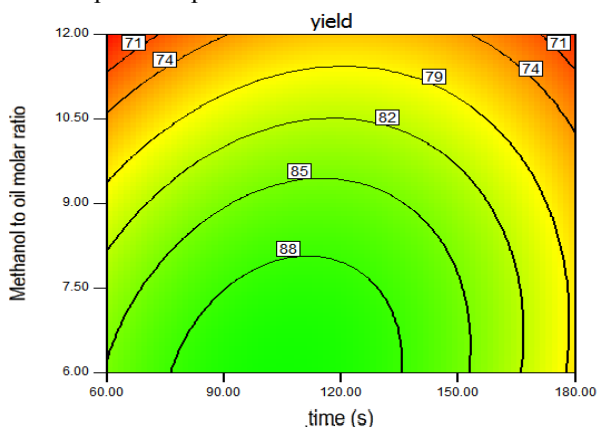


Figure 3. Interaction effects for reaction time and methanol to oil molar ratio on the yield at catalyst concentration of 1.44 wt.% and power of 500 W

From the contour plots, it is easy and convenient to understand the interactions between two factors and also locate their optimum levels.

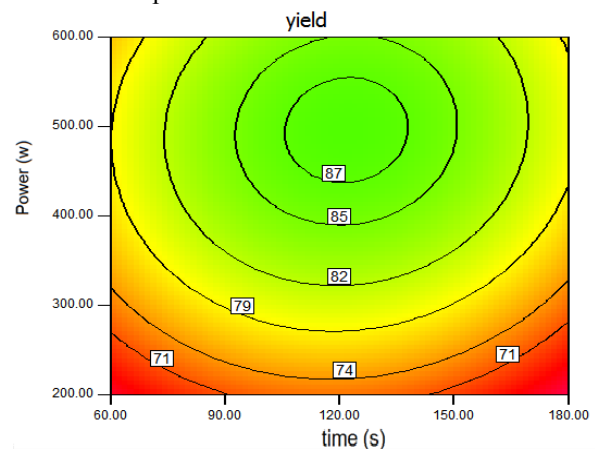


Figure 4. Interaction effects for time of reaction and power of microwave on the yield at catalyst concentration of 1.44 wt.% and methanol to oil molar ratio of 7.12:1

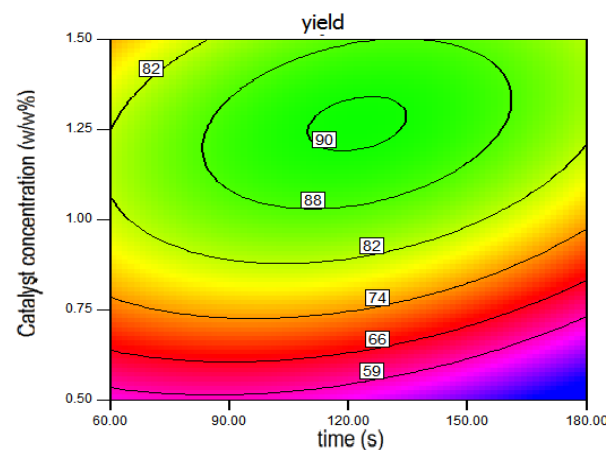


Figure 5. Interaction effects for reaction time and concentration of catalyst on the yield of biodiesel at power of 500 W and methanol to oil molar ratio of 7.12:1

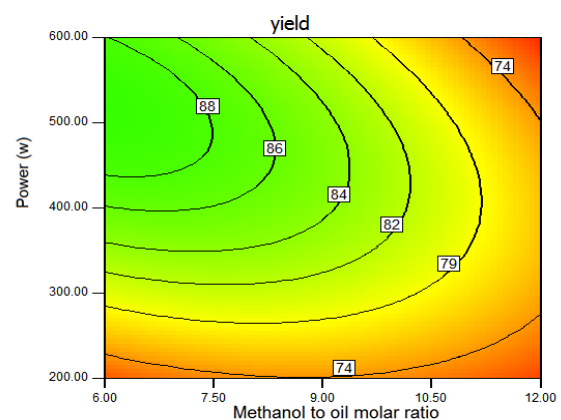


Figure 6. Interaction effects for methanol to oil molar ratio and microwave power on the biodiesel yield at time 120s and catalyst concentration of 1.44 wt.%

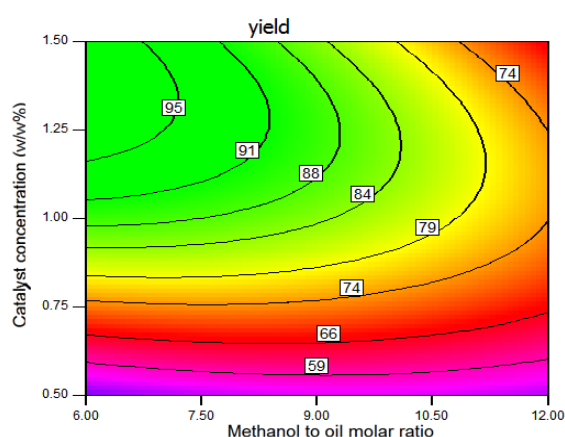


Figure 7. Interaction effect for methanol to oil molar ratio and catalyst concentration on the biodiesel yield at time 120s and power of 500 W

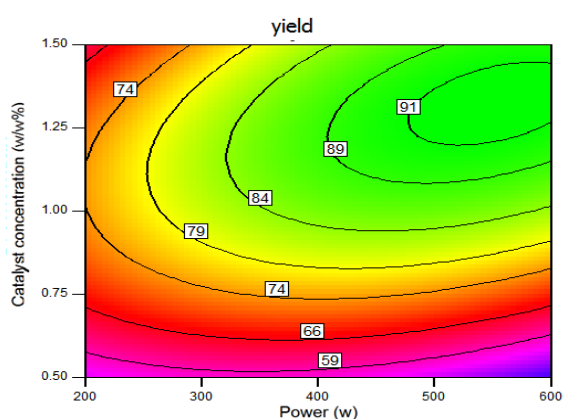


Figure 8. Interaction effects for microwave power and concentration of catalyst on the yield of biodiesel at time 120s and methanol to oil molar ratio of 7.12:1

Fig. 3 shows the interaction between reaction time and reactants molar ratio. The biodiesel yield decreases with increasing the methanol to oil molar ratio as illustrated in the figure. This is due to the fact that with increasing the methanol to oil molar ratio, excess alcohol is dissolved in glycerol that is produced by the reaction and therefore caused reverse of the reaction and decreasing the biodiesel yield [2].

For these conditions the optimal region of operation would be a reaction time of 100 seconds with the molar ratio slightly greater than the 6:1. The interactive effect between reaction time and power of microwave, while the other parameters kept at their optimum values as shown in Fig. 4, for this state the optimal region of operation would be a reaction time around 120s with the microwave power of about 490 W. Similarly for Fig. 5, in catalyst concentration about 1.24 wt.%, time 120s and the other optimum parameters, the production yield is in optimal point. As illustrated in Fig. 6, optimal region is molar ratio of 6:1, the power microwave slightly greater than 500 W and other parameters in optimum point. In Fig. 7, optimal point occurs at molar

ratio of 6:1, catalyst concentration of slightly greater than 1.30 wt.% and remaining parameters in optimum point. As shown in Fig. 8, the optimal point is catalyst concentration of 1.25 wt.%, microwave power of 550 W and the rest of parameters in the optimum point. As indicated in Figs. 4 and 6, an increase in the microwave power causes an increase in the biodiesel yield. The results showed that increasing the microwave power leads to an increase in the temperature of the microwave cavity. Since the overall transesterification is an endothermic and reversible reaction, an increase in the power causes an increase in the reaction rate initially and then increasing the temperature which results saponification of the reaction and leads to a decrease in the efficiency [7].

As shown in the view at contour curves of Figs. 3-8, and interaction parameters listed in Table 5. the elliptical nature of the contour plot in reaction yield indicates that the interaction of two parameters is significant, and the circular nature of the contour plot reveals that there are no significant interaction effects between two parameters on the reaction yield [23].

3.3. Optimization of FAME yield

Transesterification of castor oil was optimized for obtaining the highest FAME yield. The response surface analysis indicated that the predicted optimum FAME yield of castor oil transesterification was 94.68% at methanol to oil molar ratio of 7.12:1, catalyst concentration 1.44 wt.%, reaction time 120 seconds, and microwave power of 500 W. An additional experiment was carried out to validate the optimization result obtained by the response surface analysis. The theoretical predicted FAME yield was 94.68% by the response surface analysis which is compared with the observed experimental value of 92.15% with an error of 2.74%. The error was considered small as the observed values are within the 5% level of significance. The standard deviation obtained from the ANOVA table is used to derive the confidence intervals. Therefore, the optimum condition for FAME yield was at 94.68% at methanol to oil molar ratio of 7.12, 1.44 wt.% catalyst concentration, reaction time of 120s, and microwave power of 500 W.

TABLE 4. Analysis of variance (ANOVA) for the quadratic model

| Sources of variations | Degrees of freedom | Sum of squares | Mean square | F-value | Prob.>F Or P-value |
|-----------------------|--------------------|----------------|-------------|---------|--------------------|
| Model | 14 | 4780.55 | 341.47 | 35.01 | <0.0001 |
| Residual | 14 | 136.56 | 9.75 | - | - |
| Lack of fit | 10 | 116.99 | 11.70 | 2.39 | 0.2080 |
| Pure error | 4 | 19.57 | 4.89 | - | - |
| Total | 28 | 4917.11 | - | - | - |

$R^2 = 0.9722$, $adj R^2 = 0.9445$, C.V = 4.42, Std.Dev = 3.12 .

TABLE 5. The least-squares fit and parameter estimates (significance of regression)

| Model parameters | Parameter estimate | F-value | P-value |
|-------------------------------|--------------------|---------|----------|
| Intercept | 84.59 | - | - |
| X ₁ | -2.65 | 8.62 | 0.0109 |
| X ₂ | -4.97 | 30.38 | < 0.0001 |
| X ₃ | 3.73 | 17.16 | 0.0010 |
| X ₄ | 11.43 | 160.70 | < 0.0001 |
| X ₁ ² | -8.19 | 44.59 | < 0.0001 |
| X ₂ ² | -3.32 | 7.32 | 0.0171 |
| X ₃ ² | -6.80 | 30.76 | < 0.0001 |
| X ₄ ² | -15.49 | 159.66 | < 0.0001 |
| X ₁ X ₂ | 2.21 | 2.00 | 0.1793 |
| X ₁ X ₃ | 0.84 | 0.29 | 0.6002 |
| X ₁ X ₄ | 6.58 | 17.78 | 0.0009 |
| X ₂ X ₃ | -4.45 | 8.12 | 0.0129 |
| X ₂ X ₄ | -8.39 | 28.90 | < 0.0001 |
| X ₃ X ₄ | 8.61 | 30.38 | < 0.0001 |

3.4. Fuel properties of the produced biodiesel

For commercial fuel, the finished biodiesel must be analyzed using analytical equipment to ensure that it meets the international standards. Analytical methods were applied on the final biodiesel product to determine the fuel characteristics and the results were compared with the diesel standards of EN 14214 and are listed in Table 6. As seen from the results shown in Table 6, the properties of produced biodiesel are in accordance with the EN 14214 standards.

TABLE 6. Fuel properties and FAME compositions of biodiesel from castor oil

| Property | Value | EN 14214 |
|--|-------|----------|
| Density (kg/m ³ , 15°C) | 892 | 860-900 |
| Flash point (°C) | 196 | >101 |
| Cloud point (°C) | -23 | - |
| Kinematic viscosity (mm ² /s) | 3.7 | 3.5-5 |
| Fatty acid composition | wt. % | |
| Ricinoleic acid | 89.12 | |
| Linoleic acid (C18:2) | 4.18 | |
| Oleic acid (C18:1) | 1.29 | |
| Stearic acid (C18:0) | 0.87 | |
| Palmitic acid (C16:0) | 0.45 | |
| Linolenic acid (C18:3) | 2.84 | |
| Arachidic acid (C 20:0) | 0.62 | |
| Lignoceric acid (C24:0) | 0.63 | |

4. CONCLUSIONS

Based on the obtained experimental results, it can be concluded that all the variables such as reaction time, molar ratio of methanol to oil, microwave power, and catalyst concentration gave significant effects on the transesterification reaction. However, interaction between the reaction time and molar ratio of methanol to oil, and also interaction between reaction time and microwave power gave the lowest effect on FAME yield. The results indicated that RSM can be used to find the relationships among process variables and responses in an efficient manner using minimum number of experiments. Moreover, the recommended optimum condition of biodiesel production can also be determined via RSM. Based on the results obtained in this research, the combined effect of ultrasonic and microwave can increase the reaction efficiency. Also, the required time for transesterification reaction is decreased compared with conventional method. The important parameters for the reaction are alcohol to oil molar ratio, catalyst concentration, reaction temperature, and microwave power.

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