

Optimization of biodiesel production process in a continuous microchannel using response surface methodology

Majid Mohadesi, Babak Aghel[†], Mohammad Hassan Khademi, and Sasan Sahraei

Department of Chemical Engineering, Faculty of Energy, Kermanshah University of Technology, Kermanshah, Iran
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Abstract—We assessed the biodiesel production process in a continuous microchannel through preparation of a heterogeneous catalyst (CaO/MgO) from demineralized water plant sediment. This mixed oxide catalyst was used for transesterification of rapeseed oil as feedstock by methanol to produce biodiesel fuel at various conditions. A microchannel, utilized as a novel reactor, was applied to convert rapeseed oil into biodiesel in multiple steps. The effects of the process variables, such as catalyst concentration, methanol to oil volume ratio, *n*-hexane to oil volume ratio, and reaction temperature on the purity of biodiesel, were carefully investigated. Box-Behnken experimental design was employed to obtain the maximum purity of biodiesel response surface methodology. The optimum condition for the production of biodiesel was the following: catalyst concentration of 7.875 wt%, methanol to oil volume ratio of 1.75 : 3, *n*-hexane to oil volume ratio of 0.575 : 1, and reaction temperature of 70 °C.

Keywords: Heterogeneous Catalyst, Microchannel, Rapeseed Oil, Biodiesel, Response Surface Methodology

INTRODUCTION

Due to the increasing price of crude oil, environmental pollution, increased demand for energy, global warming due to the emission of greenhouse gases, and concerns about fossil fuel depletion, more attention has been paid to alternative fuels such as biodiesel [1-3]. Biodiesel is produced via transesterification of renewable resources such as vegetable oils or animal fats using an alcohol, most commonly methanol and a catalyst [4]. Biodiesel has many benefits: it is non-toxic, biodegradable, and renewable, has a low level of carbon dioxide emission, and can be used in most diesel engines without any modification [5,6].

Depending on chemical conditions, the transesterification reaction can be carried out by acidic [7], alkali [8,9] or enzyme [10,11] catalysts, which has many disadvantages such as the difficulty in transesterification of triglycerides with high free fatty acid (FFA) level and high energy consumption [12].

Currently, because of the general advantage of easy separation from the reaction medium and reusability, different types of heterogeneous catalysts are used for biodiesel production [13,14]. However, catalyst activity and the associated costs are the major problems hindering the mass production of biodiesel [15]. Many researchers have investigated many types of alkaline metal oxide catalysts for biodiesel production [16-18]. Among these catalysts, calcium oxide has largely attracted attention as a catalyst for the production of biodiesel, which is due to the exuberance of its natural sources, its low cost, high level of activity, and need for moderate reaction conditions [19].

Nakatani et al. [20], who studied the transesterification reaction,

used a combusted oyster shell as catalyst. They performed the calcination process at various temperatures and observed that above 700 °C the XRD patterns of combusted oyster shells were similar to the XRD patterns of CaO. It showed that reaction time and catalyst concentration were the most important factors affecting biodiesel purity. The results indicated that the optimum condition was achieved at a time of 5 h, and catalyst concentration of 25 wt%.

In another study, Methanolysis of sunflower oil using activated CaO as catalyst was studied by Granados et al. [20]. They revealed that after 100 min the conversion rate was 94% in the reaction conditions of 60 °C, molar ratio of methanol to oil of 13 : 1, and catalyst to oil of 3 wt%. Moreover, they concluded the catalytic activity was the result of using both heterogeneous sites (basic sites at the surface) and homogeneous roots (due to the leaching of the active species of the activated CaO in methanol).

In general, transesterification of vegetable oils by alcohols happens in batch processes, which are very time-consuming. To solve this problem, nowadays many techniques have been proposed to be used as a process intensifier for biodiesel production. However, these tools not only should maintain a high quality of biodiesel, but also should have many characteristics such as shorter reaction time and low energy consumption. Among the newly introduced methods, ultrasonic irradiation, microwave, static mixer, and microchannel have been applied frequently [22-25].

Santana et al. [26] investigated the effect of micromixer with static elements on transesterification of sunflower oil by ethanol and sodium hydroxide catalyst. As they observed, at a reaction condition of ethanol/oil molar ratio of 9 and catalyst concentration of 1% at 75 °C there was more than 91.53% conversion of transesterification reaction.

Alenezi et al. [27] considered the use of a downflow liquid contactor reactor (DLCR) for improving biodiesel production from sunflower oil. A yield of 96.5% was achieved in the presence of

[†]To whom correspondence should be addressed.

E-mail: b.aghel@kut.ac.ir, babakaghel@gmail.com

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0.43 wt% NaOH, molar ratio of methanol to sunflower oil of 4.5 : 1 at 40 °C reaction temperature in the very short time of 2.5 min.

Various parameters play important roles in optimization of the transesterification process, such as alcohol to oil molar ratio, residence time, catalyst concentration, and reaction temperature. Several studies on transesterification of rapeseed oil using response surface method (RSM) have recently been published [28-30].

Sendzikiene et al. [31] obtained a 60.1% yield of butyl ester after 9.8 h at 39 °C using Lipozyme RM IM (6%) and a 4.5 butanol and oil molar ratio for enzymatic transesterification of model waste rapeseed oil containing a high FFA content (with 4% acid value) with butanol in two steps.

In another study, Mostafaei et al. [32] tried to optimize the ultrasonic assisted continuous production of biodiesel using RSM from waste cooking oil. According to their experimental results, irradiation distance of 75 mm, probe diameter of 28 mm, ultrasonic amplitude of 56%, vibration pulse of 62%, and flow rate of 50 ml/min were reported as optimal variables to obtain a yield of 91.6% and an energy consumption of 102.8 W.

Due to insolubility of alcohols and oil phases at ambient temperature and pressure in the transesterification reaction, many studies have investigated many ways, such as the use of co-solvents, to enhance system solubility [33-35]. This study aimed to apply RSM and optimize the operational conditions in a microchannel used for biodiesel synthesis via in-situ transesterification of rapeseed oil by methanol and hexane as co-solvents.

METHODS

1. Materials

Rapeseed oil was purchased from a local vender. The composition of oil was tested and the results are shown in Table 1. The acid value was determined using ASTM D644 method and it was 0.16% in rapeseed oil. In addition, the saponification value and average molecular weight of this oil were 186 mg KOH/g oil and 861 g/mol, respectively. The demineralized (DM) water plant sediment was calcined and used as catalyst. All the chemicals used in the experiments such as methanol 99.8%, methyl laurate (methyl dodecanoate) >99.7% and *n*-hexane 95% (extra purity) were purchased

Table 1. Composition data of rapeseed oil used in this study

Fatty acid composition	wt%
Myristic (C14:0)	0.09
Palmitic (C16:0)	10.63
Palmitoleic (C16:1)	0.09
Margaric (C17:0)	0.09
Margaroleic (C17:1)	0.05
Stearic (C18:0)	5.15
Oleic (C18:1)	24.53
Lenoleic (C18:2)	51.80
Linolenic (C18:3)	6.40
Arachidic (C20:0)	0.40
Behenic (C22:0)	0.40
Erucic (C22:1)	0.37

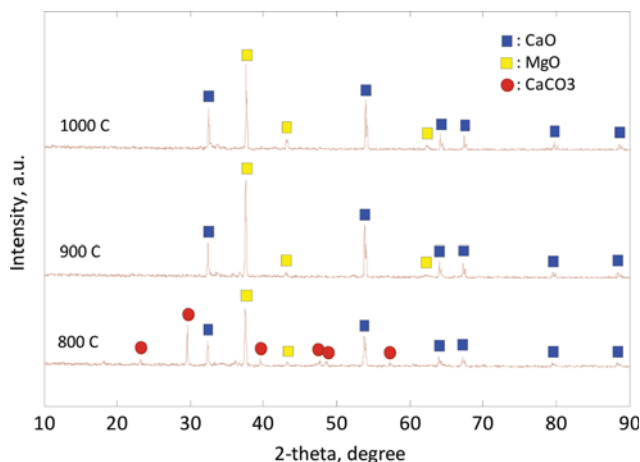


Fig. 1. XRD patterns of DM water plant sediment as a catalyst calcined at temperatures of 800, 900 and 1,000 °C for 2 h.

from Sigma (St. Louis, MO). Double distilled water was used in the operational procedure.

2. Preparation of Catalyst

The DM water plant sediment as catalyst was prepared and then was dried, calcined, and finally cooled. Initially, the sediment was dried at 110 °C for 20 h to remove adhered moisture and volatiles. Afterward, it was milled by mortar and pestle and then fine particles (125-250 μm) were sieved. Next, the collected particles were calcined in furnace at 900 °C for 2 h to decompose the metal carbonate into its corresponding metal oxide with an increasing temperature rate of 5 °C/min. Lastly, the catalysts were kept in desiccators prior to activity testing. As shown in Fig. 1, high calcination temperatures were used to convert DM water plant sediment to CaO and MgO; it is a method reported by Ngamcharussrivichai et al. [36].

3. Catalyst Characterization

The DM water plant sediment was identified by X-ray diffraction (XRD) with Cu K α X-ray as a source. The intensity data were recorded over 2-theta from 10 to 90° with a 0.6° step. Fig. 1 shows the powder XRD pattern of the calcined catalysts at 800, 900 and 1,000 °C for 2 h. The calcination temperature of 900 °C was identified as the best temperature. In this temperature all calcium and magnesium carbonates were converted to calcium and magnesium oxides (see Fig. 1). Natural calcium carbonate has a low activity; hence a high reaction temperature was required to achieve a conversion rate above 95%. But calcium oxide has high basic strength ($H_{-}=26.5$), high activity and long service lifetime [36]. Elemental analysis of the catalyst was performed using X-ray fluorescence analysis (Spectro Xepor 03 plus), that presented in the DM water plant

Table 2. Amount of the main elements in fresh and used demineralized water plant sediment catalyst based on XRF analysis

Element	wt%	
	Fresh	Five times used
Ca	50.85	49.72
Mg	9.34	9.08

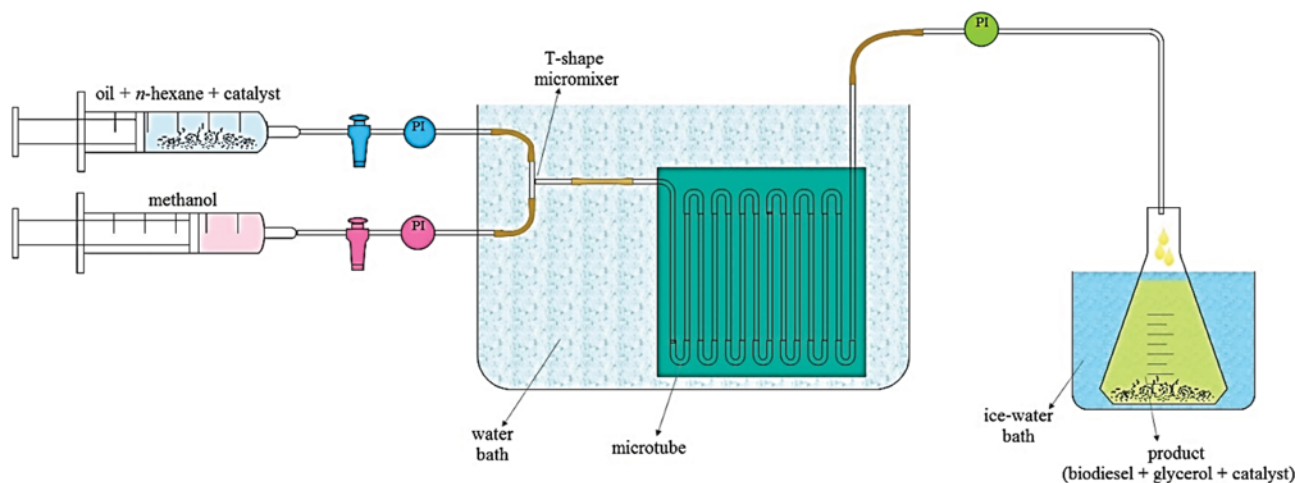


Fig. 2. Schematic diagram of the continuous biodiesel production system.

sediment after being calcined for 2 h. According to the results of The XRF analysis, the amounts of Ca and Mg in this catalyst (calcined DM water plant sediment at 900 °C for 2 h) were 50.85 and 9.34 wt%, respectively. These elements existed in CaO and MgO. The main elements found in the catalyst are presented in Table 2.

4. Methods and Experimental Procedure

The transesterification reaction was in a microreactor equipped with microchannel, syringe pump, and water bath. The schematic diagram of the microreactor system is shown in Fig. 2.

Before the process was started, in order to remove all food residues, the rapeseed oil was filtered using a fine screen cloth and then heated at a temperature of 120 °C for 1 h to remove adhered water. The microreactor was made from plexiglass and had two parts including a T-shaped three-way junction and a serpentine microchannel with an internal diameter of 0.8 mm. Due to the high viscosity of rapeseed oil, the specific weights of catalyst were poured into oil+n-hexane container. Considering the desired ratios of the oil, n-hexane and methanol were measured and then specific weights of catalyst were poured into oil+n-hexane container and subsequently mixed with oil+n-hexane to prepare oil+n-hexane solutions.

Using two syringe pumps (Son Pump 350), the mixture of rapeseed oil+n-hexane+catalyst and methanol was separately injected into a microreactor at different flow rates. The methanol and rapeseed oil+n-hexane+catalyst were mixed at a T-shaped junction which was connected to the serpentine microchannel and the mixture passed through it.

The residence time for all experiments was 10 min. So, taking into account the ratios of methanol to oil and n-hexane to oil, the flow rates were determined. The temperature of the reaction was controlled via immersing the microreactor in a water bath. The product samples were placed in an ice-water flask to terminate the transesterification reaction. At the end of the experiment, each sample was poured into the separation funnel and two distinct layers were formed; the top and the bottom phases, respectively, represented the fatty acid methyl ester (FAME) and glycerol. The FAME layer was further centrifuged to remove the residuals of catalyst and glycerol at 3,000 rpm for 10 min; then it was washed by distilled water three times for 5 min.

The concentration of FAME was determined by using a gas chromatograph (Varian CP3800 Netherland) equipped with a capillary column, DB-WAX (30 m×0.25 mm) and a flame ionization detector (FID). Helium gas as the carrier gas together with an auxiliary gas was used for FID. In the next step, to test each of the samples, 50 µl of the diluted sample (product sample in n-hexane) was dissolved into 50 µl of methyl laurate (C12:0) (internal standard solution). Then 1 µl of the prepared mixture was injected into the gas chromatography (GC) and biodiesel purity was computed. The weight percentage of FAME or biodiesel purity was determined by using the following equation suggested by Wang et al. [37]:

$$\text{Purity} = \frac{\text{area of all FAME}}{\text{area of reference}} \times \frac{\text{weight of reference}}{\text{weight of biodiesel sample}} \times 100 \quad (1)$$

5. Design of Experiments and Optimization

Biodiesel production depends on several variables; to decrease costs and save time, RSM is used to obtain suitable data from the experimental runs. According to the literature, this method has been often used to maximize efficiency and reduce the cost of production [38–40].

In this study, we used Box-Behnken experimental design to find the optimal conditions to maximize the purity of biodiesel in the continuous transesterification reaction. The effects of the main operational variables including catalyst concentration (C), methanol to oil volume ratio (MR), n-hexane to oil volume ratio (HR), and reaction temperature (T) on the purity of biodiesel were investigated. Taking into consideration the results of previous experi-

Table 3. Range and level of variables

Factors	Symbol	Range and levels		
		-1	0	+1
Catalyst concentration (oil based), wt%	C	5.0	7.5	10.0
Methanol to oil volume ratio, vol : vol	MR	1 : 3	1 : 2	2 : 3
n-Hexane to oil volume ratio, vol : vol	HR	0 : 1	1 : 2	1 : 1
Temperature reaction, °C	T	50	60	70

Table 4. Experimental design for four independent variables and the response

Run no.	Manipulated variables				Response P, %
	X_C	X_{MR}	X_{HR}	X_T	
1	1	0	0	-1	51.26
2	0	1	1	0	25.97
3	-1	1	0	0	40.81
4	0	0	0	0	80.69
5	0	0	-1	1	87.54
6	-1	0	0	1	68.21
7	0	1	0	-1	42.69
8	1	1	0	0	61.09
9	1	-1	0	0	29.14
10	-1	0	1	0	12.25
11	-1	0	0	-1	34.18
12	0	0	0	0	86.92
13	0	0	1	1	59.80
14	-1	0	-1	0	42.38
15	0	-1	0	-1	25.97
16	1	0	0	1	90.71
17	0	0	-1	-1	38.92
18	0	-1	-1	0	10.74
19	0	-1	1	0	16.50
20	1	0	-1	0	34.87
21	-1	-1	0	0	6.62
22	0	0	1	-1	24.15
23	0	-1	0	1	41.02
24	0	1	0	1	97.28
25	1	0	1	0	34.18
26	0	1	-1	0	68.74
27	0	0	0	0	82.59

mental studies [41-43], we selected the variables and their levels on the transesterification process.

Table 3 presents the levels and range of each variable at three levels: low (-1), middle (0) and high (+1). At each level, respectively, we had a catalyst concentration (oil based) of 5, 7.5 and 10 wt%, methanol to oil volume ratio of 1 : 3, 1 : 2 and 2 : 3, *n*-hexane to oil volume ratio of 0 : 0, 1 : 2 and 1 : 1 and reaction temperature of 50, 60 and 70 °C. Using this method, 25 tests and two duplicate tests were performed at a central point to determine the errors. Experimental design and the results of the tests are shown in Table 4.

Analysis of variance (ANOVA) is a technique to analyze and define the degree of accuracy of experimental data [44]. Also statistical analysis of the empirical model was performed to evaluate ANOVA. The response variable (FAME %) was fitted by a full quadratic model to assess the relationship between the FAME % and the mentioned variables (catalyst concentration, methanol to oil volume ratio, *n*-hexane to oil volume ratio, and reaction temperature). The form of the model is shown in the following equation:

$$Y = \beta_0 + \sum_{i=1}^4 \beta_i X_i + \sum_{i=1}^4 \beta_{ii} X_i^2 + \sum_{i=1}^3 \sum_{j=i+1}^4 \beta_{ij} X_{ij} \quad (2)$$

where Y is the FAME % (purity of biodiesel), X_i and X_{ij} are the uncoded independent variables, β_0 is the offset term, and β_0 , β_i , β_{ii} , β_{ij} are regression coefficients.

6. Catalyst Reusability

The reusability of the catalyst was measured through conducting several runs which represented the capacity to perform the same catalytic activity. After each step of the transesterification reaction, the catalyst was washed completely by using methanol to remove any residue adhered to the surface of the catalyst. The cleaned catalyst was then dried at 90 °C for 12 h. The catalyst was reused in the experiments for five times.

RESULTS AND DISCUSSION

1. Response Variance Analysis

Using the least square of error method, which was performed on all data, a quadratic model for biodiesel purity (P) was obtained, which is shown in Eq. (3):

$$P = 81.15 + 8.07X_C + 17.22X_{MR} - 9.20X_{HR} + 18.95X_T + 7.36X_CX_{HR} - 12.13X_{MR}X_{HR} + 9.88X_{MR}X_T - 21.19X_C^2 - 26.08X_{MR}^2 - 27.40X_{HR}^2 \quad (3)$$

where X_C , X_{MR} , X_{HR} and X_T are catalyst concentration (oil based) in wt%, methanol to oil volume ratio in vol:vol, *n*-hexane to oil volume ratio in vol:vol and reaction temperature in °C, respectively. The observed values of biodiesel purity compared with the values predicted by the quadratic model are shown in Fig. 3. The predicted biodiesel purity values were obtained by using Eq. (3). The R^2 and R^2_{adj} of biodiesel purity model were 0.98 and 0.97, respectively. Table 5 shows the results of ANOVA test performed for biodiesel purity model. Total degrees of freedom was 26, and regression and residual error degrees of freedom were 16 and 10,

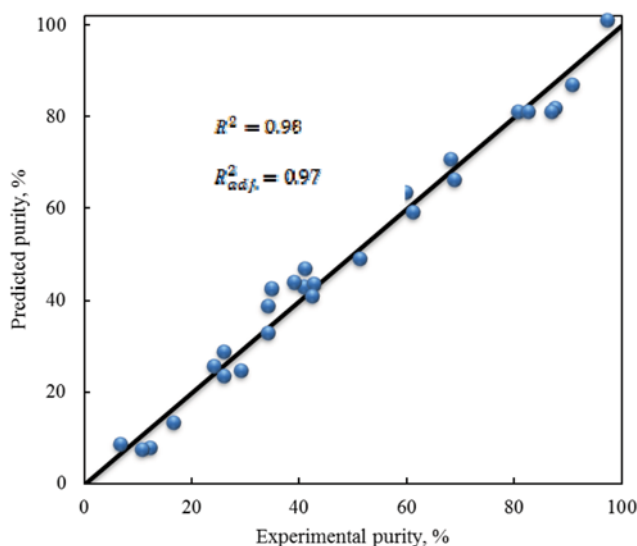


Fig. 3. Comparing the observed and predicted values for purity using rapeseed oil, methanol, *n*-hexane, and DM water plant sediment as a catalyst and at different catalyst concentrations, methanol to oil volume ratios, *n*-hexane to oil volume ratios, and reaction temperatures (the residence time was 10 min in all the experiments).

Table 5. Standard deviation analysis (ANOVA) for quadratic purity model

Sources	SS	DF	MS	F-value	P-value	Degree of significance
Model	18461.05	10	1846.10	82.55	<0.0001	highly significant
X_C	780.85	1	780.85	34.92	<0.0001	highly significant
X_{MR}	3556.62	1	3556.62	159.04	<0.0001	highly significant
X_{HR}	1014.58	1	1014.58	45.37	<0.0001	highly significant
X_T	4308.85	1	4308.85	192.68	<0.0001	highly significant
$X_C X_{HR}$	216.68	1	216.68	9.69	0.0067	highly significant
$X_{MR} X_{HR}$	588.79	1	588.79	26.33	0.0001	highly significant
$X_{MR} X_T$	390.85	1	390.85	17.48	0.0007	highly significant
X_C^2	2693.57	1	2693.57	120.45	<0.0001	highly significant
X_{MR}^2	4080.74	1	4080.74	182.48	<0.0001	highly significant
X_{HR}^2	4503.05	1	4503.05	201.37	<0.0001	highly significant
Residual error	357.80	16	22.36			
<i>Lack - of - fit</i>	337.41	14	24.10	2.36	0.3368	not significant
<i>Pure error</i>	20.39	2	10.20			
Total	18818.84	26				

respectively, which are shown in Table 5.

Comparing F -values shown in Table 5, the F -value calculated for the model is larger and has a higher significant level (P -value < 0.0001). P -values less than 0.05 indicate that the model terms are significant. Accordingly, X_C , X_{MR} , X_{HR} , X_B , $X_C X_{MR}$, $X_{MR} X_{HR}$, $X_{MR} X_B$, X_C^2 , X_{MR}^2 , and X_{HR}^2 are significant regression terms. P -values more than 0.1 indicate that the regression terms are not significant (such as X_B^2 , $X_C X_{MR}$, $X_C X_T$ and $X_{HR} X_T$). When there are not many significant model terms (not taking into account those required to support hierarchy, i.e., X_B^2 , $X_C X_{MR}$, $X_C X_T$ and $X_{HR} X_T$), model reduction may improve the model. So, non-significant terms were eliminated from quadratic model and only significant terms remained (see Eq. (3)).

The *lack - of - fit* F -value of 2.36 implies the *lack of fit* is not significant relative to the pure error. There is a 33.68% chance that a *lack - of - fit* F -value this large could occur due to noise. Non-significant *lack - of - fit* is good, i.e., we want the model to fit.

The empirical model for biodiesel purity is plotted in contour diagrams between significant interactions (Figs. 4(a), 4(b) and 4(c)). In Fig. 4(a), a contour diagram of biodiesel purity in terms of X_{MR} and X_{HR} is plotted (coded catalyst concentration and coded reaction temperature are in medium level, in other words $X_C = X_T = 0$). In Fig. 4(b) contour diagram of biodiesel purity in terms of X_C and X_{HR} is plotted ($X_{MR} = X_T = 0$). Moreover, Fig. 4(c) shows the contour diagram of biodiesel purity in terms of X_{MR} and X_T ($X_C = X_{HR} = 0$).

2. Optimization of Biodiesel Purity

As shown in Fig. 4(a), the effect of the interaction between methanol to oil volume ratio of n -hexane to oil volume ratio on the purity of biodiesel is negative. By taking the derivative of Eq. (3) with respect to these two variables (X_{HR} and X_{MR}) and solving a system of two equations in the two variables, these values are obtainable. The other two variables (X_C and X_T) are considered to be zero. At the highest level of purity, the volume ratio of methanol to oil was 1.675 : 3 ($X_{MR} = 0.35$), and the volume ratio of n -hexane to oil was 0.375 : 1 ($X_{HR} = -0.25$). In Fig. 4(a), all variables are fixed other than the methanol to oil and n -hexane to oil volume ratios.

Accordingly, biodiesel purity can be determined by changing these two variables (methanol to oil volume ratio and n -hexane to oil volume ratio). As shown in this figure, by increasing n -hexane ratios up to 0.375 : 1, biodiesel purity increased as well. This trend confirms that the use of n -hexane as a co-solvent leads to a reduction in the use of methanol.

Fig. 4(b) shows a positive interaction between catalyst concentration and volume ratio of methanol to oil. Highest purity was obtained at a catalyst concentration of 7.875% ($X_C = 0.15$) and n -hexane to oil volume ratio of 0.575 : 1 ($X_{HR} = -0.15$). As mentioned above in this figure it is assumed that $X_{MR} = X_T = 0$. As shown in Figs. 4(a) and 4(b), n -hexane to oil volume ratio had an optimal value between 0.375 : 1 to 0.575 : 1 ($-0.25 < X_{HR} < -0.15$). In addition, when the catalyst concentration increased beyond critical amount, a slight decrease was observed in the amount of biodiesel purity (Figs. 4(a) and 4(c)), which was probably due to a problem in mixing DM water plant sediment catalyst and reactants [45].

When n -hexane to oil volume ratio exceeds 0.575 : 1, oil molecules and methanol will be less in contact with each other. So n -hexane is used up to a level that could increase the solubility of oils and methanol. Also as shown in Fig. 4(c), with increasing the temperature, the biodiesel purity increases as well. The optimal amount of methanol to oil volume ratio in this figure is 1.75 : 3 ($X_{MR} = 0.5$).

So, considering the results shown in Figs. 4(a) and 4(c), it can be concluded that the optimal methanol to oil volume ratio is between 1.675 : 3 and 1.75 : 3 ($0.35 < X_{MR} < 0.5$). As shown, biodiesel purity can be increased (Figs. 4(a) and 4(c)) through increasing methanol to oil volume ratio up to a critical amount. Exceeding this value would reverse the reactions since transesterification is a reversible reaction. The same trends are reported in other studies [46,47]. Thus, according to Figs. 4(a) and 4(c), $X_C = 0.15$, $0.35 < X_{MR} < 0.5$, $-0.25 < X_{HR} < -0.15$ and $X_T = 1$ are the optimum values for biodiesel production. Fig. 5 shows biodiesel purity calculated at X_{MR} and X_{HR} at $X_C = 0.15$ and $X_T = 1$. According to this figure, the highest efficiency was achieved at $X_{MR} = 0.5$ and $X_{HR} = -0.25$. Using an empirical model, the purity in optimal conditions ($X_C = 0.15$, $X_{MR} =$

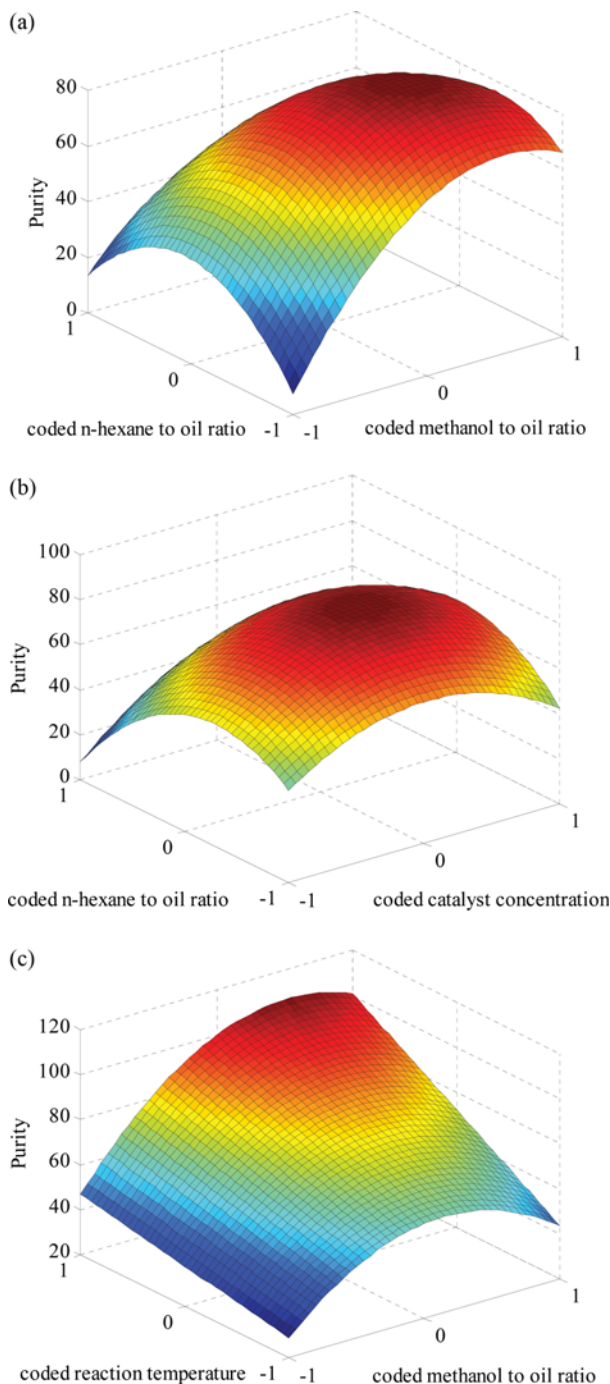


Fig. 4. (a) Effect of methanol to oil volume ratio and *n*-hexane to oil volume ratio on purity of biodiesel using rapeseed oil, methanol, *n*-hexane and DM water plant sediment as a catalyst; catalyst concentration of 7.5 wt%, reaction temperature of 60 °C, and residence time of 10 min. (b) Effect of catalyst concentration and *n*-hexane to oil volume ratio on purity of biodiesel using rapeseed oil, methanol, *n*-hexane and DM water plant sediment as a catalyst; methanol to oil volume ratio of 1 : 2, reaction temperature of 60 °C, and residence time of 10 min. (c) Effect of methanol to oil volume ratio and reaction temperature on purity of biodiesel using rapeseed oil, methanol, *n*-hexane and DM water plant sediment as a catalyst; catalyst concentration of 7.5 wt%, *n*-hexane to oil volume ratio of 1 : 2, and residence time of 10 min.

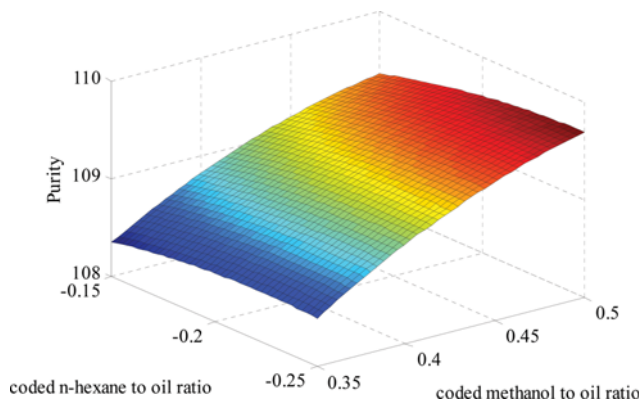


Fig. 5. Effect of methanol to oil volume ratio and *n*-hexane to oil volume ratio on purity of biodiesel using rapeseed oil, methanol, *n*-hexane and DM water plant sediment as a catalyst; catalyst concentration of 7.875 wt%, reaction temperature of 70 °C, and residence time of 10 min.

Table 6. Properties of optimum biodiesel with ASTM D6751

Property	Biodiesel	ASTM D6751
Density, g/cm ³	0.876	0.86-0.90
Kinematic viscosity, mm ² /s	4.556	4-6
Pour point, °C	5	-15 to 10
Cloud point, °C	7	-3 to 12

0.5, $X_{HR}=-0.25$ and $X_T=1$) was equal to 109.70%. By testing the purity of biodiesel was obtained in optimum conditions achieved 99.31%.

To show the advantage of the microchannel in biodiesel production some studies are summarized in Table 7. Various reactors with different operation conditions cannot be compared directly, but by comparing the purity and the order of residence time it can be concluded that the purity in the microchannel is quite higher while the residence time is lower than in conventional reactors.

3. Biodiesel Properties

The properties of the best produced biodiesel ($X_C=0.15$, $X_{MR}=0.5$, $X_{HR}=-0.25$ and $X_T=1$) were measured. These values are presented in Table 6 and are compared with ASTM D6751. According to this table, biodiesel properties are at standard ranges.

4. Catalyst Reusability

Finally, we studied catalyst reusability. The catalyst was reused at the optimum conditions (catalyst concentration of 7.875 wt%, methanol to oil volume ratio of 1.75 : 3, *n*-hexane to oil volume ratio of 0.575 : 1, and reaction temperature of 70 °C). Fig. 6 shows the results of catalyst reusability. After reusing catalyst for five times, the purity was reduced from 99.31% to 95.12%. Due to the short reaction time, less calcium oxide was extracted and thus little decrease was achieved in the amount of efficiency. The amount of Ca and Mg after reusing catalyst for five times was 49.72 and 9.08 wt%, respectively (see Table 2).

CONCLUSIONS

DM water plant sediment and rapeseed oil were successfully

Table 7. Comparison between obtained purity in different kinds of reactors

Reactor	Oil	Co-solvent	Catalyst	Residence time, min	T, °C	FAME, %
Tubular [48]	Rapeseed	Ethyl acetate	Triethylamine	145	280	85.00
Batch reactor [33]	Palm	Tetrahydrofuran	CaO	90	65	98.50
Present work	Rapeseed	<i>n</i> -Hexane	Cao/MgO	10	70	99.31

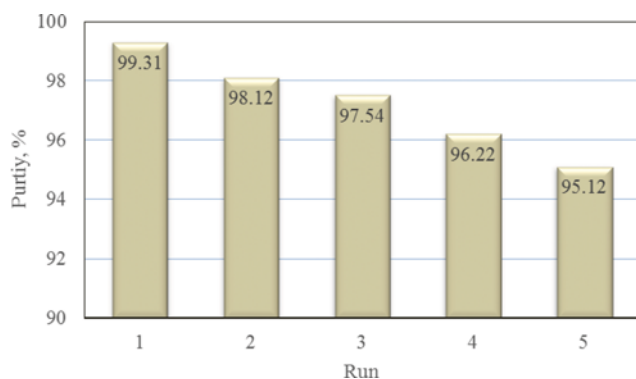


Fig. 6. Reusability of catalyst at optimal condition (catalyst concentration of 7.875 wt%, methanol to oil volume ratio of 1.75 : 3, *n*-hexane to oil volume ratio of 0.575 : 1, and reaction temperature of 70 °C) at residence time of 10 min.

utilized as a low-cost feedstock to produce biodiesel within a micro-reactor via transesterification at various operational conditions. According to the experimental results, the highest biodiesel purity obtainable by the model and experiment was 109.70% and 99.31%, respectively. Optimization of transesterification reaction parameters revealed that catalyst concentration of 7.875 wt%, methanol to oil volume ratio of 1.75 : 3, *n*-hexane to oil volume ratio of 0.575 : 1, and reaction temperature of 70 °C formed the optimal reaction condition. Mean relative error (MRE) of model with experimental data was 10.24%. Furthermore, the use of a microreactor undoubtedly assisted in achieving this remarkable result in less than 10 min reaction time. In addition, the reusability of the catalyst was good at least for five times and reusable.

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