



# Investigating the Effect of Catalyst and Deep Eutectic Co-solvent on Purity of Biodiesel Produced from Linseed Oil

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## ABSTRACT

Nowadays, due to environmental concerns and the reduction in fossil fuel resources, biodiesel is being considered as a renewable alternative or supplement to fossil diesel. In this research, the transesterification method was used to produce biodiesel from low-cost and non-edible linseed oil in the presence of commercial calcium oxide as the catalyst. In addition, the time-consuming and costly calcination step was omitted and a deep eutectic co-solvent of choline chloride/glycerol (2:1) was used to activate the catalyst. The catalyst characterization was studied using X-ray diffraction (XRD) and Fourier transform infrared (FTIR). To ensure the formation of fatty acid methyl esters (FAME), the purity of FAME in the produced biodiesel was calculated using gas chromatography method. Then, the effect of two reaction parameters of the catalyst and the DES concentration on the FAME purity was investigated at a constant temperature of 65 °C, stirring speed of 500 rpm, reaction time of 180 minutes and the methanol to oil molar ratio of 9.5:1. Finally, the optimal catalyst and the DES concentration were obtained as 10% and 14%, respectively. The effect of temperature and time on the FAME purity was investigated and a kinetic study of the reaction suggested a pseudo-first-order reaction with a rate constant of  $0.0311 \text{ min}^{-1}$  at 65 °C and activation energy of 22.45 kJ/mol.

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## NOMENCLATURE

$A$	Peak area ( $\text{m}^2$ )	$V_{is}$	Internal standard volume (ml)
$A_{is}$	Internal standard peak area ( $\text{m}^2$ )	$C_{is}$	Mass of the sample (mg)
$C_{is}$	Internal standard concentration (mg/ml)	$m_{sample}$	Internal standard concentration (mg/ml)

## INTRODUCTION

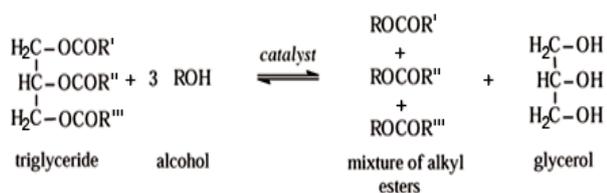
Due to the decrease in fossil fuel resources, increasing fuel demand, and environmental concerns about greenhouse gases, fossil fuels are being replaced with clean and renewable energy. Biodiesel has many advantages over fossil diesel, such as renewability, high cetane number, high combustion efficiency and non-toxicity that emits no  $\text{SO}_x$  or extra amounts of  $\text{CO}_x$  [1]. These advantages make biodiesel a suitable alternative or supplement to fossil diesel [2].

Biodiesel is a combination of mono-alkyl esters of long-chain fatty acids that is usually produced via the

transesterification method. In transesterification reactions, 1 mole of triglyceride reacts with 3 moles of short-chain alcohol in the presence of a catalyst and forms 3 moles of fatty acid methyl esters (FAME, known as biodiesel) and 1 mole of glycerol as the reaction by-product [3, 4]. The stoichiometry of a transesterification reaction is presented in Figure 1.

Calcium oxide (CaO) is a low-cost basic heterogeneous catalyst with high catalytic active sites that leads to high reaction yields under moderate reaction conditions [5]. Calcium oxide is quickly deactivated after a few minutes of exposure to air and a layer of calcium hydroxide and calcium carbonate covers its surface.

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**Figure 1.** Stoichiometry of a transesterification reaction

Therefore, it is necessary to activate the catalyst before the transesterification process [6]. Calcination is one of the conventional methods of activating CaO in which the inactive layers on the catalyst surface decompose by increasing the temperature up to about 900. However, it is a time and energy-consuming method which increases the operational costs of the process [7].

The usage of some kinds of solvents such as deep eutectic solvents can dissolve the inactive layers of CaO and increase the reaction yield due to their high solubility properties. Deep eutectic solvents (DES) with lower cost, low toxicity, non-flammability and other advantages over organic co-solvents can be a suitable alternative to organic solvents and increase transesterification yield. [8]. Deep eutectic solvents are the combination of an organic halide salt with an organic agent in a certain molar ratio that forms a compound with a lower melting point [8].

Troter et al. [9], action in the presence of choline chloride/glycerol cosolvent and reported the production of FAME in the initial minutes of the reaction which was attributed to the formation of calcium diglyceroxide and calcium methoxide in the preheating step. Also, Huang et al. [8], used choline chloride/glycerol cosolvent in the transesterification of rapeseed oil in the presence of commercial CaO and reached the maximum reaction yield in the reaction temperature of 65 under optimal reaction conditions as follows: catalyst concentration of 8.07%, methanol to oil molar ratio of 14.28:1 and cosolvent concentration of 10.74%.

In this work, the effect of the catalyst and the cosolvent concentration was investigated in the transesterification of non-edible linseed oil in the presence of commercial calcium oxide. Also, the deep eutectic cosolvent of choline chloride/glycerol (2:1) was used to activate the catalyst. Also, the catalyst characterization of X-ray diffraction (XRD) and Fourier transform infrared (FTIR) were conducted before and after the reaction.

## MATERIAL AND METHODS

### Materials

Linseed oil was purchased from a local market in Tehran, Iran. Glycerin (99%), calcium oxide (99.9%), choline

chloride (99%) and methanol (99.9%) were supplied from Merck.

### Preparation of deep eutectic cosolvent

First, choline chloride was dried in an oven at 70 °C for 72 hours to remove adsorbed moisture. Then, the dried choline chloride and glycerin with a molar ratio of 2:1 were mixed and stirred at a temperature of 70 °C at 500 rpm for 20 minutes until the choline chloride particles were dissolved in glycerin and a transparent homogeneous solution was obtained [10].

### The experimental procedure of biodiesel production

First, methanol, CaO and DES were poured in a double-neck 100 ml flask and stirred for 20 minutes at 65°C at 500 rpm with a magnetic stirrer to activate the catalyst and to preheat the solution. Then 30 grams of linseed oil was preheated to 65 °C and added to the reaction flask. By adding the oil, the reaction started and continued for 180 minutes. A thermometer was used to control the temperature inside the flask and a condenser was used to reflux the evaporated methanol to the flask. After the completion of the reaction, the resulting mixture was given time to allow the catalyst particles to settle down. After separating the precipitated catalyst, the solution was placed in a centrifuge at 4000 rpm for 15 minutes to separate the remaining catalyst particles. The resulting solution was placed in an oven at a temperature of 70°C so that the methanol in it evaporates and the solution inside becomes two-phase. The upper phase is rich in fatty acid methyl ester (FAME) and the lower phase is rich in glycerol. To separate the remaining methanol and glycerol, the upper phase was washed 3 to 4 times with distilled water at 60 °C and finally placed in an oven at 100°C for 4 hours until the water in it evaporated completely and the biodiesel was completely transparent. To evaluate the effect of temperature and time on the FAME purity, transesterification was carried out in three reaction temperatures of 55, 60 and 65. Also, the relation between yield and time was studied by investigating the product composition every 30 minutes at 60, 90, 120 and 180 minutes of the reaction.

## RESULTS AND DISCUSSION

### Properties of the produced biodiesel

The properties of the produced biodiesel were evaluated and compared with ASTM D6751 [11, 12] and other studies as shown in Table 1. As can be seen in Table 1, the biodiesel properties met the requirements of ASTM D6751 standards. The higher pour point of the biodiesel could be due to the unsaturated fatty acids in the biodiesel. Also, higher properties such as density, viscosity and the flash point could be due to the high molecular mass of biodiesel than petrodiesel [13]. However, by comparing

**Table 1.** Properties of the produced biodiesel in comparison with ASTM D6751

Property	Biodiesel (this study)	ASTM D6751	Petro diesel	Bahadorian et. al [14]
Density @25 (g/cm <sup>3</sup> )	33.70	0.86-0.90	0.83	0.879
Kinematic viscosity @25 (mPa s)	14.30	1.6-6	2.04	4.55
Acidity (mg KOH/g)	0.35	0.8 max.	0.4	0.37
Pour point (°C)	41.10	0 max.	-16	-15
Cloud point (°C)	-8	18 max.	-12	-6
Flash point (°C)	105	31 min.	77	170

results with the other study it can be concluded that the properties are better according to the ASTM.

### Catalyst characterization

XRD patterns of the catalyst were recorded before and after the transesterification as shown in Figure 2. As can be seen, strong peaks at  $2\theta = 32.4^\circ, 37.2^\circ, 53.1^\circ, 65^\circ$  and  $67.5^\circ$  are relevant to the CaO structure (in accordance with JCPDS No. 00-037-1497). Also, Ca(OH)<sub>2</sub> with peaks at  $2\theta = 18.2^\circ, 34.24^\circ$  and  $50.89^\circ$  and CaCO<sub>3</sub> with peaks at  $2\theta = 29.54^\circ, 43.14^\circ$  and  $47.54^\circ$  are formed due to the hydration of CaO during the catalyst collection step [15]. From Figure 2, new peaks appeared at  $2\theta = 10.34^\circ, 21.34^\circ, 24.4^\circ$  and  $26.49^\circ$  indicates the formation of calcium diglyceroxide. Calcium diglyceroxide, the active basic compound with a high catalytic activity, is the result of combination of CaO with the glycerol.

FTIR spectra of the catalyst before (a) and after the reaction (b) are shown in Figure 3. The peak of  $535.20\text{ cm}^{-1}$  is the stretching vibration of CaO in the catalyst. Also, the peak at  $3642.28\text{ cm}^{-1}$  belongs to the stretching vibration of O – H in Ca(OH)<sub>2</sub>. The peaks of  $1419\text{ cm}^{-1}$  and  $875\text{ cm}^{-1}$  belong to the carbonate group. New bands in the catalyst structure after the reaction demonstrates the formation of calcium diglyceroxide (in accordance with

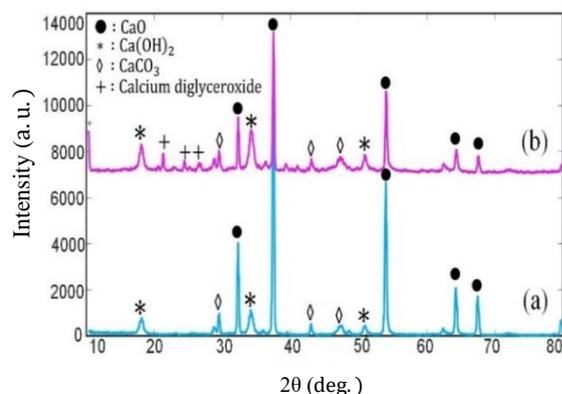
the XRD result). New peaks of  $2878.69\text{ cm}^{-1}$  and  $2850.33\text{ cm}^{-1}$  belong to the symmetric stretching vibration of C – H in the –CH<sub>3</sub> and –CH<sub>2</sub> groups respectively and the broad peaks of  $3566.7\text{ cm}^{-1}$  and  $3212.25\text{ cm}^{-1}$  indicate the O – H stretch.

### Effect of the parameters on the FAME purity

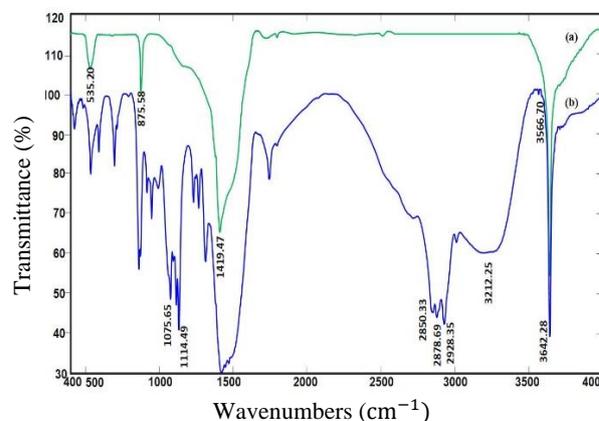
In order to investigate the performance of the DES in catalyst activation, the transesterification reaction of linseed oil was conducted in the presence of commercial CaO catalyst and choline chloride/glycerol (2:1) as the cosolvent under the constant temperature of  $65^\circ\text{C}$ , stirring speed of 500 rpm, the reaction time of 180 minutes and methanol to oil molar ratio of 9.5:1. Also, using the DES was the only method of activating the catalyst. To ensure the formation of methyl ester and to calculate its purity in the resulting product, a gas chromatography device (Clarus-Perkin Elmer, USA) was employed. The purity of methyl ester was calculated using the gas chromatography results by Equation (1) [16].

$$FAME\% = \frac{\sum A - A_{is}}{A_{is}} \times \frac{C_{is}V_{is}}{m_{sample}} \times 100 \quad (1)$$

where  $\sum A$  is the total area under the FAME peaks,  $A_{is}$  is the area under the methyl nonadecanoate peak,  $C_{is}$  is the



**Figure 2.** XRD patterns of the catalyst (a) before transesterification; (b) after transesterification



**Figure 3.** FTIR patterns of the catalyst (a) before transesterification ; and (b) after transesterification

concentration of the methyl nonadecanoate in mg/ml,  $V_{is}$  is the volume of the methyl nonadecanoate in ml and  $m_{sample}$  is the mass of the sample in mg.

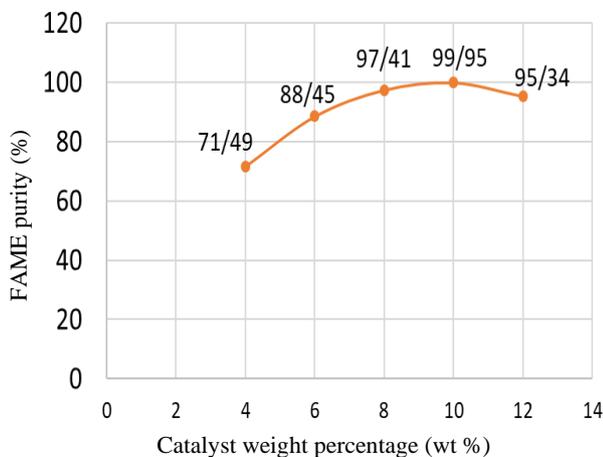
To study the effect of the catalyst concentration on the FAME yield, transesterification reactions were performed in catalyst weight percentages of 4, 6, 8, 10 and 12 wt.%. the results of the FAME purity are shown in Figure 4.

As can be concluded from the figure, by increasing the catalyst concentration up to 10 wt.%, the purity of biodiesel was significantly increased. By further increasing of the catalyst concentration to more than 10%, the FAME purity decreased due to the increased viscosity, mass transfer resistance and accelerated soap formation reactions. increasing the catalyst concentration to above 10%, decreased the FAME purity due to the increased viscosity, mass transfer resistance and accelerated soap formation reactions.

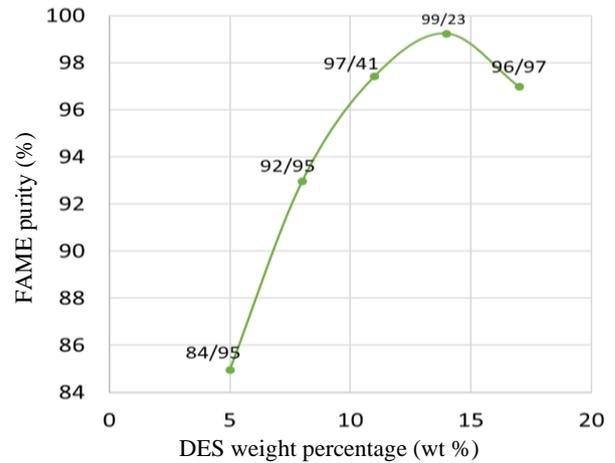
Moreover, the FAME purity results in different DES concentrations (5, 8, 11, 14 and 17 wt. %) are demonstrated in Figure 5. According to the figure, by increasing the DES concentration from 5 to 14 wt. % the FAME purity was increased due to the increased solubility of methanol to oil and the increased contact surface area between methanol and oil. Also, the DES led to the activation of the catalyst and increased the catalyst active sites. However, a further increase in the DES concentration also increased the glycerol concentration in the reaction mixture, leading to the accelerated reverse reaction and resulting in low FAME production.

Also high catalyst concentration can increase the soap formation side reactions and lead to FAME production decrease [17, 18].

Similar results have been reported by Mohadesi et al. [19] who reached the maximum biodiesel purity of 99.24%. under catalyst (calcined cow bone) concentration of 8.5 wt %, an oil to methanol volume ratio of 2.25 vol:vol at a temperature of 63.1 °C. Also, optimum



**Figure 4.** Effect of the catalyst concentration on the FAME purity



**Figure 5.** Effect of the DES concentration on the FAME purity

biodiesel conversion of 99%, occurred at 60 °C with CaO–MgO catalyst reported by Korbag et al. [20].

Also Compared to the transesterification of Salicornia persic oil reported by Shenavaei-Zare et al. [10], in which the maximum yield of 95% was achieved at a temperature of 70 °C, in this study A higher yield was achieved at a lower temperature of 65 °C and at the same time of 180 minutes. According to the research of Gargari and Sadrameli [21], they also studied the effect of several organic cosolvents on the transesterification yield of linseed oil in the presence of CaO and obtained yields of 97.15%, 91.86 % and 89.72%. in the presence of tetrahydrofuran, n-hexane and biodiesel as cosolvent, while in this study, higher yield was achieved in the transesterification of linseed oil and calcium oxide catalyst in the presence of the deep eutectic cosolvent of choline chloride/glycerol (2:1).

### Kinetic study

To study the kinetics of the reaction, the FAME concentration variation over time and temperature was studied. Assuming the transesterification is an elementary one, the reaction the rate law could be considered as shown in Equation (2) [22, 23].

$$-r_{TG} = \frac{d[TG]}{dt} = k_1[TG][MeOH]^3 - k_2[GLY][FAME]^3 \quad (2)$$

where  $k_1$  and  $k_2$  are the rate constant of the forward and backward reactions, respectively. Also, the brackets show the concentrations. It should be noted that two simplifying assumptions have been considered due to the excessive amount of methanol, 1. Concentration of methanol remains constant during the reaction. 2. the transesterification equilibrium shifted toward FAME production. Thus the reaction was considered irreversible and the reaction rate was simplified to a pseudo-first-order with a rate law shown in Equation (3).

$$-r_{TG} = \frac{d[TG]}{dt} = K[TG] \quad (3)$$

By integrating Equation (3) and considering that  $[TG] = [TG_0](1 - X_{TG})$ , the reaction rate is demonstrated by Equation (4).

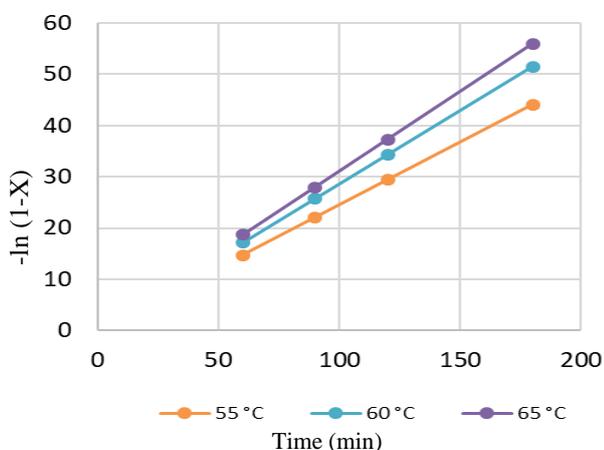
$$\ln(1 - X_{TG}) = -Kt \quad (4)$$

where  $[TG_0]$  and  $[TG]$  are the initial concentration and concentration of triglycerides at any time, respectively. Also,  $X_{TG}$  is the FAME yield. The reaction rate constant ( $K$ ) was obtained by calculating the slope of  $-\ln(1 - X_{TG})$  vs. time plot (Figure 6) at each reaction temperature. It should be noted that the coefficient of determination of the lines in Figure 6 ( $R^2 > 0.9$ ) indicates that the assumptions came true and the reaction follows a pseudo-first-order kinetics. As can be seen in Figure 6, as the temperature increases from 55 °C to 65 °C,  $-\ln(1 - X)$  increases which has a positive relation with the FAME purity and is due to the decreased viscosity of the reaction mixture followed by increased temperature. Then the rate constants at 55, 60 and 65 °C were found as  $0.0245 \text{ min}^{-1}$ ,  $0.0286 \text{ min}^{-1}$  and  $0.0311 \text{ min}^{-1}$ , respectively. Also, positive effect of the decreased viscosity is obviously seen in increasing the reaction rate constant.

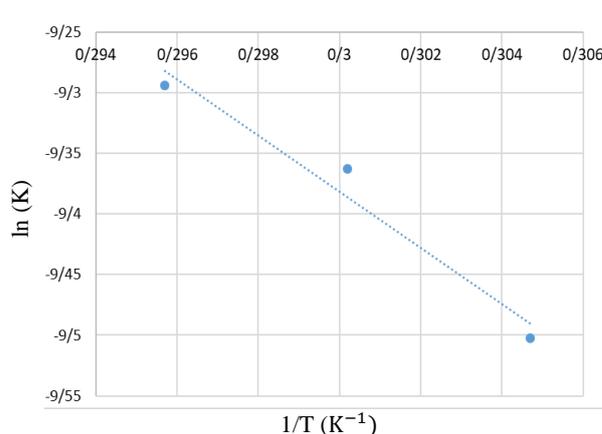
The relation between a reaction rate constant and the temperature is well introduced by Arrhenius law as shown in Equation (5) [24].

$$\ln(K) = -\frac{E_a}{RT} + C \quad (5)$$

where  $E_a$  is the reaction activation energy in J/mol,  $T$  is the temperature in Kelvin and  $R$  is the universal molar gas constant of  $8.314 \text{ J}/(\text{mol K})$ . The activation energy ( $E_a$ ) was obtained using the slope of the  $\ln(K)$  vs.  $1/T$  graph (Figure 7). The activation energy was obtained as  $22.45 \text{ kJ/mol}$ . This finding is close to other transesterification reactions used commercial CaO as the catalyst reported



**Figure 6.**  $-\ln(1 - X)$  vs. time for transesterification of linseed oil at 60, 90, 120 and 180 minutes of the reaction



**Figure 7.**  $\ln(K)$  vs.  $1/T$  for transesterification of linseed oil according to the rate constants at 55, 60 and 65 °C

by Anantapinitwatna et al. [25] (transesterification of triolein, of  $24.8 \text{ kJ/mol}$ ) and transesterification of scrum oil reported by Krishnamurthy et al. [26] (activation energy of  $67.21 \text{ kJ/mol}$ ).

## CONCLUSION

In this research, the effect of the catalyst and DES concentration on the transesterification of linseed oil was investigated in the presence of commercial CaO catalyst and deep eutectic co-solvent of choline chloride/glycerol (2:1). Linseed oil was used as the feedstock because of its low cost and high fatty acid content. Also, the time-consuming and high-cost method of calcination was eliminated and the deep eutectic co-solvent of choline chloride/glycerol (2:1) was used to activate commercial CaO. Also the XRD and FTIR spectra of the catalyst indicates formation of high catalytic activity sites of Calcium diglyceroxide. The FAME purity was obtained utilizing the gas chromatography method, whose high values indicate the proper production of methyl ester. Finally, the maximum FAME purity was reached at constant temperature of 65 °C, stirring rate of 500 rpm, the reaction time of 180 min and methanol to oil ratio of 9.5:1 under optimal reaction conditions as follows: catalyst concentration of 10 wt % and DES concentration of 14 wt %. Also reaction rate constant of  $0.0311 \text{ min}^{-1}$  was achieved at 65 °C and under optimal reaction conditions and activation energy of  $22.45 \text{ kJ/mol}$  was calculated.

## CONFLICT OF INTEREST

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#### Persian Abstract

#### چکیده

امروزه به دلیل مشکلات زیست محیطی و کاهش منابع سوخت‌های فسیلی، سوخت تجدیدپذیر بیودیزل، به عنوان جایگزین و یا مکمل دیزل فسیلی مورد توجه قرار گرفته است. در این پژوهش از روش ترانس استریفیکاسیون جهت تولید بیودیزل از روغن غیر خوراکی و ارزان بزرک در حضور کاتالیست کلسیم اکسید تجاری استفاده شد. همچنین مرحله‌ی زمان و هزینه‌بر کلسیناسیون حذف و برای فعال‌سازی کاتالیست از حلال کمکی یونکتیک عمیق کولین کلراید/گلیسرول (۱:۲) استفاده شد. ساختار کاتالیست با استفاده از پراش اشعه ایکس و تبدیل فوریه فروسرخ مورد مطالعه قرار گرفت. برای اطمینان از تشکیل متیل استر، خلوص بیودیزل تولیدشده به کمک روش کروماتوگرافی گازی محاسبه شد. در ادامه تأثیر دو پارامتر غلظت کاتالیست و حلال کمکی بر خلوص بیودیزل در دمای ثابت ۶۵ درجه سانتی‌گراد، دور همزن ۵۰۰ دور بر دقیقه و زمان واکنش ۱۸۰ دقیقه در نسبت الکل به روغن ۱:۹/۵ مورد بررسی قرار گرفت و غلظت بهینه (با هدف بیشینه خلوص) برای هر کدام به ترتیب، ۱۰ درصد و ۱۴ درصد بدست آمد. اثر دما و زمان بر خلوص متیل استر اسید چرب (بیودیزل) مورد بررسی قرار گرفت و مطالعه سینتیک واکنش، یک واکنش مرتبه اول را با ثابت سرعت  $0.0311$  بر دقیقه در دمای ۶۵ درجه سانتی‌گراد و انرژی فعال سازی  $22/45$  کیلوژول بر مول نتیجه داد.