Pork lard conversion to biodiesel using a microchannel reactor

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Abstract-Biodiesel was synthesized from pork lard via transesterification using a microchannel reactor. To investigate the effects of operating parameters, including reaction temperature (55-65 °C), residence time (5-20 s), methanol-to-oil molar ratio (4.5:1 to 9:1), and catalyst concentration (0.7-1.3 wt%), a series of full factorial experiments with a complete replicate were conducted. Results were statistically analyzed using MINITAB with the significance level of 0.05. A quadratic model was proposed for the prediction of %FAME from the specified operating conditions. High %FAME was obtained at low residence time due to the small size of droplets in the microchannel reactor. Evidence of droplets supported the presence of mass transfer limitation in this system. The optimal operating conditions provided %FAME of 95.41% were as follows: methanol-to-oil ratio of 6 : 1, temperature of 65 °C, residence time of 5 s, and KOH concentration of 1.3%w/w.

Keywords: Biodiesel, Microchannel Reactor, Pork Lard, Transesterification

INTRODUCTION

The increase of the human population and the energy demand has contributed to the deterioration in the natural resources and environment. It is predicted that extensive use of fossil fuel will continue to dominate the energy market worldwide in the foreseeable future. Global concerns over the energy (in)security, as petroleum resources are rapidly depleted, have stimulated numerous efforts in research and development focusing on renewable energy (biofuel, wind energy, solar energy, ocean waves, etc.) in order to avert the looming energy crisis. Biodiesel is one of the bioenergy supplies that holds a great potential to reduce petroleum consumption especially for logistics and agricultural machinery.

The simplest form of biodiesel can be produced from vegetable oil for direct use or blending with diesel. The mixing ratio is important for preventing gum formation from oxidation of free fatty acid during storage or from polymerization in the combustion chamber [1]. Micro-emulsion is another method that can reduce viscosity of vegetable oil by using an emulsifier to stabilize the colloidal system of immiscible liquids. This technique provides biodiesel properties similar to petro-diesel oil with lower cetane number and heating value [2]. Thermal cracking (550-850 °C) of large-molecule substance with/without catalyst can also produce biodiesel. Potential raw material includes vegetable oil, animal fat, and natural acid oil. Liquid product is a wide range of alkanes, alkenes aromatics, carboxylic acids, etc. Major drawbacks of this method are high investment cost and small yields [3]. Among several methods for producing biodiesel, the most viable route in terms of product quality and simplicity of system operation is transesterification, which is the reac-

tion between vegetable oils or animal fats with alcohol in the presence of catalyst (immobilized enzyme, KOH, MgO, H₂SO₄, etc.) resulting in ester and glycerol [4]. Potential feedstock includes palm oil, coconut oil, soy bean oil, castor oil, sunflower oil, jatropha oil, etc. By and large, palm oil/jatropha oil and methanol are the raw materials for biodiesel production using alkaline catalyst. Using edible oil as feedstock can cause instability in the food industry and potentially lead to high prices of biodiesel. Moreover, the shortage of raw materials for the production of edible oil during certain periods may aggravate the food security situation. In order not to interfere with the human food security, non-edible oils have been considered as suitable feedstock for biodiesel production. Nowadays, pork lard is generally perceived to be the potential cause of vascular disease and heart disease. It is not used as much in food preparation; therefore, the price of pork lard is relatively low compared to vegetable cooking oil. We used pork lard as feedstock for biodiesel synthesis. In theory, the required molar ratio between oil and alcohol is 1:3 in order to produce three molecules of ester and one molecule of glycerol [5]. However, due to chemical equilibrium limitation, much higher amounts of alcohol are generally used.

The conventional process of biodiesel production via transesterification consists of at least four major steps including mixing of feedstock and catalyst, allowing for chemical transformation, phase separation between biodiesel and glycerol, removing catalyst, water, and other impurities in biodiesel. After phase separation in which glycerol is removed, a large amount of water is necessary to wash the resulting biodiesel in order to eliminate catalyst and alcohol. In the conventional process, there is a physical limitation of large-scale mixing of aqueous and organic phases that hinders the chemical transformation. Together with chemical equilibria of this reacting system, it is common to use excess alcohol far beyond the stoichiometric requirement (50-300% excess) in order to achieve the design performance. Consequently, the required size of the reactor vessel

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and the corresponding residence time are large, resulting in high energy consumption rate for mixing of reagents and for maintaining the reacting fluid at a desired temperature. Alleviating these limitations by means of reaction engineering is necessary for developing a highly efficient biodiesel production process.

Employed in pharmaceutical industry, petrochemical industry, and environmental remediation, microreactor technology offers several advantages for process intensification over the conventional ones [6,7]. Microchannel or microreactor is a tiny channel with a diameter of less than 1 mm for the mixture of reacting fluid to undergo chemical conversion within. It provides small pressure drop and a rapid transfer of heat and matter that can help decrease the required residence time to achieve high conversion. Therefore, this technology has a high potential for high performance biodiesel production. The improved reaction rate can also potentially lessen the methanol-to-oil ratio resulting in reduced load for separation and recycling of methanol as well as the energy consumption in the production process. Once this technology is fully and efficiently implemented for large-scale biodiesel production, the use of biodiesel will be more widespread as the production cost is substantially reduced.

Recent studies of biodiesel production using various designs of microchannel reactor have shown promising improvement in the reaction rate [8,9]. It has been demonstrated that, for biodiesel synthesis from sunflower oil using 4.5 wt% KOH as catalyst with the molar ratio of 23.9:1 at 60 °C, a microchannel reactor with 0.8 mm diameter and the length of 300 mm provided almost 100% FAME within 100 seconds [9]. Avellaneda and Salvado studied the effect of a micromixer on the production of biodiesel from used cooking oil in a system of coiled tubular reactors operating at 60 °C. The reactor system was placed in an ultrasonic water bath. A combination of T-mixer without ultrasonic activation was found most effective, yielding 89% FAME with the reaction time of 13 minutes (75 minutes for a batch process), methanol-to-oil molar ratio of 6:1, and 0.6 wt% of NaOH [10]. Different types of micromixer (T, J, RIMM, SIMM-V2) were used in a system of biodiesel synthesis from cottonseed oil in order to study the effect on %FAME. RIMM exhibited outstanding performance due to more intensified mixing compared to that obtained for other mixers [11]. Even shorter reaction time was reported for biodiesel production from soybean oil using NaOH catalyst in a zigzag microchannel reactor. At 56 °C with methanol-to-oil molar ratio of 9:1 and 1.2 wt% of NaOH, 99.5% FAME was obtained in 28 seconds [8]. Biodiesel synthesis in a microreactor has also been applied for high acid value oils using acid-catalyzed reactions in a two-step process. High methanol-to-oil ratio of 30 with 3 wt% H₂SO₄ and seven minutes of reaction time were used in the first step to convert fatty acid at 100 °C. The second step is transesterification with the methanol-to-oil ratio of 20, 3 wt% H₂SO₄ and 5 minutes of reaction time at 120 °C. FAME yield of 99.5% was achieved after the second step [12]. However, this application has not been thoroughly investigated and still requires much further study. Biodiesel production involves aqueous homogeneous catalyst and bio-oil which form two-phase flow of reacting mixture without any emulsifier. This two-phase mixture has some specific characteristics which are necessary to be investigated in microchannels.

We investigated the effects of operating conditions including the amount of catalyst, reaction temperature, residence time, and methanol-to-oil molar ratio on the performance of biodiesel synthesis from pork lard in a microtube reactor. Statistical analysis was applied in order to classify significant effects and suggest the optimal operating conditions that can be further used for large scale synthesis in multi-microchannel reactor.

MATERIALS AND METHOD

1. Materials

Pork lard used in all experiments was purchased from Nontaburi Market, Thailand. Pretreatment of pork lard for biodiesel production included water removal by heating at 107 °C for 2 h followed by vacuum filtration using filter paper (Whatman, Grade 1 Circles, 110 mm). Methanol (AR-grade) was purchased from Merck. Potassium hydroxide (AR-grade) was purchased from Quality Reagent Chemical Product. Other solvents and reagents such as 2 propanol (AR-grade) were obtained from Merck and phenolphthalein was purchased from Laboratory Chemical.

2. Experimental Apparatus

Fig. 1 shows a schematic diagram of the entire reactor system for biodiesel synthesis in a microtube reactor. It consisted of two high performance liquid chromatography (HPLC) pumps; one for the feed of pork lard and another one for the solution of potassium hydroxide dissolved in methanol. The two feed streams flowed through PTFE tubing and were joined in a T-mixer, which has a diameter of 0.508 mm. A microchannel reactor made of PTFE with an inside diameter of 0.508 mm and the length of 1.2 m was connected to the outlet of the T-mixer. The reaction temperature was maintained by means of circulating hot water in the annulus around the reactor. The methanol-to-oil molar ratio was controlled by adjusting the flow rates of the two feed streams. The reactor was connected with another microchannel with cold water flowing through the annulus in order to terminate the reaction. At the outlet, the product mixture was collected in a flask placed in an ice-water bath. The methyl ester layer was gravitationally separated from the glycerin layer over night. After the removal of glycerol, the product was washed with deionized water and dried at 107 °C for 30 min.

To statistically analyze the main and interaction effects, we employed a full factorial experimental design (with a complete replicate) to study the effects of four parameters including KOH con-



Methanol + catalyst

Fig. 1. Experiment setup for biodiesel synthesis in a microchannel reactor.

centration (0.7, 1.0, and 1.3 wt%), reaction temperature (55, 60, and 65 °C), methanol-to-oil molar ratio (4.5:1, 6:1, and 9:1), and residence time (5, 10, and 20 s).

3. Biodiesel Production from Pork Lard by Batch Reactor

The purpose of this experiment is to make a comparison between biodiesel synthesized in a conventional batch reactor and that with a microchannel reactor. The experiment was carried out in a glass flask with a magnetic stirrer and electric heater. The magnetic stirrer was set at 200 rpm to provide vigorous agitation. The methanol-to-oil molar ratio, reaction temperature, and catalyst concentration were the same as the optimal conditions for the microchannel reactor. In this batch reactor, the transesterification of pork lard was allowed for 60 minutes before the reaction flask was cooled by means of ice water. The methyl ester was gravitationally separated from glycerol in a funnel. After the removal of glycerol, the product was washed with deionized water and dried at 107 °C for 30 min.

4. Statically Analysis

An analysis of variance (ANOVA) with 95% confidence level was carried out to study the main and interaction effects of the system parameters, including the molar ratio of methanol-to-oil, catalyst amount, reaction temperature, and residence time. A model for prediction the %FAME will be proposed according to regression analysis. The assumptions of ANOVA are as follows.

a. The dependent variables or residuals are normally distributed.

b. The average of residuals is equal to zero.

c. Constant variance of residuals.

d. All residuals are mutually independent values.

5. Determination of Methyl Ester Content

The methyl ester was analyzed by gas chromatography (GC) (Model GC - 2010, Shimadzu, Japan) according to EN 14103 standard. Methyl Heptadecanoate (C17:0) with 95% purity was used as standard. Eq. (1) was used to determine the methyl ester content.

$$\% FAME = \frac{(\sum A) - A_{us}}{A_{us}} \times \frac{C_s \times V_s}{W} \times 100$$
(1)

Where ΣA =summation of areas under the peaks corresponding to

 C_{14} to C_{24} A_{us} =area under the curve of C_{17} C_s =concentration of C_{17} (mg ml⁻¹) V_s =amount of C_{17} used (µL) W=weight of product (mg)

6. Determination of Biodiesel Properties

Our synthesized biodiesel was subject to various standard testing procedures for viscosity (EN 14214), acid value (EN14214), density (EN14214), flash point (EN14214), cloud point (ASTM D6751) and pour point (ASTM D6751). These properties were compared with the regulation imposed by the Department of Energy, Ministry of Energy, Thailand.

RESULTS AND DISCUSSION

1. Analysis of Pork Lard

Prior to biodiesel synthesis experiments, the pork lard was pretreated by filtering out solid particles using vacuum filtration with paper filter. The compositions of pork lard were analyzed by gas chromatography (GC). The main fatty acid composition (by weight)



Fig. 2. Normal probability plot of residuals (response is %FAME).

was as follows: Oleic=41.46%, Palmitic=22.29%, Linoleic=15.69% and Stearic=11.34%. The molecular weight of the pork lard used in this research was 831.924 g/mol. The acid value, density, and viscosity at at 40 °C of pork lard were 1.12 mgKOH g⁻¹, 831.39 g cm⁻³ and 39.18 cts, respectively.

2. Statistical Analysis

To systematically evaluate the effects of parameters studied, a statistical analysis was applied. First, the assumptions of ANOVA were verified by the residual histogram of %FAME collected from all experiments as shown in Fig. 2. The majority of data lies on a reference straight line; therefore, this data distribution is approximately normal [13].

The assumptions of ANOVA were also verified by the residual plot as shown in Fig. 3. There is no specific distribution pattern of residuals, suggesting that the average of residuals is zero and the variance of residuals is constant. For this reason, it can be inferred ANOVA can be used for our full factorial experiments.

In this research, the significance level of 95% was used throughout the analysis of variance. By testing the following assumptions, the main and interaction effects on the %FAME obtained from the transesterification of pork lard in a microtube reactor were specified [13].

(i) H₀: $\mu_1 = \mu_2 = \mu_3 = \mu_4$ or all parameters did not affect the %FAME.

(ii) H_1 : at least one of parameters affected the %FAME.

The analysis of variance for %FAME is summarized in Table 1.



Fig. 3. Plot of residuals versus fitted value (response is %FAME).

Source	Degree of freedom	Sum of squares	Mean square	F-value	Prob>F (p-value)
Methanol to oil ratio (r)	2	3829.7	1914.85	264.01	0.000
Temperature (T), °C	2	685.31	342.66	47.24	0.000
Residence time (t), sec	2	2361.58	1180.79	162.80	0.000
Concentration of KOH (c), %w/w	2	3355	1677.50	231.29	0.000
r*T	4	419.59	104.90	14.46	0.000
r*t	4	327.21	81.80	11.28	0.000
r*c	4	662.04	165.51	22.82	0.000
T*t	4	58.18	14.54	2.01	0.099
T*c	4	360.22	90.05	12.42	0.000
t*c	4	316.04	79.01	10.89	0.000
r*T*t	8	41.11	5.14	0.71	0.683
r*T*c	8	355.01	44.38	6.12	0.000
T*t*c	8	145.92	18.24	2.51	0.015
Error	105	761.55	7.25		
Total	161	13678.47			

Table 1. Variance analysis of %FAME

The effect of parameters on %FAME can be identified by p-value. The effect is considered significant if the p-value is smaller than 0.05 and vice versa. The p-values greater than 0.05 are associated with T*t and r*T*t. Therefore, these two effects were dropped out for further analysis. For the main effects, the p-value of r, T, t and c are less than 0.05. Then null hypothesis (H_0) is rejected supporting that all main effects are significant. From Table 1, there are five significant two-parameter interaction effects including r*T, r*t, r*c, T*c, and t*c. Fig. 4 shows the interaction effect between KOH concentration and residence time. For KOH concentration less than 1.0%, increasing KOH concentration improves the reaction rate of transesterification resulting in high %FAME. However, there is a nonlinear pattern for KOH concentration higher than 1.0%. This is due to the saponification of KOH and FFA in pork lard. It appears that decreasing of residence time increased the %FAME. This counterintuitive effect is because higher flow rates were used to provide small residence times. Consequently, the mixing of two input streams was enhanced. This effect will be further examined in section 3.3.

Fig. 5 shows the interaction of methanol-to-oil ratio versus residence time. For the region with small ratios, increasing this ratio



Fig. 4. Contour plot (data means) for %FAME: effect of KOH concentration and residence time.

provided high %FAME as the transesterification reaction is shifted in the forward direction. This trend is reversed when the methanolto-oil ratio is higher than 6 : 1. This is because a large amount of methanol acted as an emulsifier, which improved mixing between



Fig. 5. Contour plot (data means) for %FAME: effect of methanolto-oil ratio and residence time.



Fig. 6. Contour plot (data means) for %FAME: effect of methanolto-oil ratio and temperature.

glycerol and biodiesel phases [14,15]. Therefore, the optimal ratio is 6:1. With large residence time (slow flow rates), the reaction is hindered by poor mixing, which compounded the emulsion effect resulting in lower %FAME compared to that of lower residence times.

Fig. 6 is the interaction plot between methanol-to-oil ratio and reaction temperature. In general, the rate of transesterification is increased by increasing the reaction temperature. However, further increasing temperature beyond 60 °C lowered the %FAME. This is because saponification simultaneously occurred with transesterification at high temperatures. Therefore, the optimal temperature falls in the range of 60-65 °C. For the effect of methanol-to-oil ratio, the ratios beyond 6:1 resulted in reduction in %FAME. This is in line with Fig. 5 as previously explained about the emulsion effect. The reduction in %FAME can worsen when the methanol-to-oil molar ratio and temperatures are too high [16].

The interaction plot between methanol-to-oil ratio and KOH concentration is presented in Fig. 7. The biodiesel synthesis using medium methanol-to-oil molar ratio (6 : 1) resulted in higher %FAME. With excess methanol, the product separation was hindered by the enhanced solubility of glycerol in biodiesel [17]. High KOH concentration also caused saponification (as previously explained).

Fig. 8 shows the interaction plot between KOH concentration and reaction temperature. Higher %FAME could be obtained at high reaction temperature and high catalyst concentration and vice versa.

3. Analysis of Droplet Size

Experimental results suggest that %FAME has a strong correlation with residence time. To further investigate this effect, we analyzed samples obtained from the reactor exit by means of an optical microscope. Fig. 9 shows a comparison of droplets from the following



Fig. 7. Contour plot (data means) for %FAME: effect of methanolto-oil ratio and KOH concentration.

operating conditions: 65 °C, 1.3 wt% KOH, and methanol-to-oil ratio of 6 : 1. Different residence times were adjusted by changing the flow rates of both methanol and oil streams. The system was operated for a certain period prior to taking samples. The flow patterns of the droplets at the exit end of the microchannel were stable. In Fig. 9, it is clear that the droplets are smaller with 5 s compared to other residence times. Consequently, the presence of large interfacial surface areas associated with small droplets allowed mass transfer to proceed rapidly. Note that lowering the residence time involved high pressure drop at the T-mixer. The pressure at the reactor entrance for the residence time of 5, 10, 20 was 70, 100, and 130 psi, $(4.826 \times 10^5, 6.895 \times 10^5, and 8.963 \times 10^5 Pa)$, respectively. In the range of operating conditions investigated, high pressure drop provided small droplets.

4. Regression Analysis

Regression model fitting was performed to correlate the %FAME with the specified operating conditions for the biodiesel synthesis in a microchannel reactor. Among several models tested, the quadratic model was found superior for predicting the %FAME. The model is presented in Eq. (2). The statistical analysis showed that this model is acceptable for prediction with the adjusted determination coefficient (R^2 adj.) of 92.4%.





Fig. 8. Interaction plot (data means) for %FAME: effect of KOH concentration and reaction temperature.



Fig. 9. Microscopic images of droplet size at 5 (i), 10 (ii) and 20 (ii) s.

 Table 2. Variance analysis for the polynomial regression model

			T J		
Source	d. f.	SS	MS	F-value	p-value
Regression	48	12944.36	269.67	41.51	0.000
Error	113	734.12	6.50		
Total	161	13678.47			
100 - 00 experiment		<u>ن</u> م. •		ania e se s	\$ ••
%FAME fro		1			
60 65	70	75 8	80 85	90	95 100
	9/6	FAME from	regression	model	

Fig. 10. Parity plot for %FAME.



Where t (sec) is the residence time, r is the methanol-to-oil ratio, T (°C) is the reaction temperature and c (%w/w) is KOH concentration. The variance analysis was applied to verify the model sufficiency. The analysis of variance for the regression model results are summarized in Table 2. The p-value is less than the significance level of 0.05; therefore, the model can be used to predict %FAME from specified operating conditions. A comparison between experimental and predicted values of %FAME using Eq. (3) is illustrated in Fig. 10. With a small degree of scattering, it is apparent that there is a linear correlation that supports the agreement between observed and predicted values of %FAME.

5. Comparison of Microchannel Reactor and Batch Reactor for Biodiesel Synthesis

The properties of biodiesel obtained from the synthesis in a microchannel reactor under the optimal conditions (methanol-to-oil ratio of 6:1, temperature 65 °C, residence time 5 seconds and concentration of KOH 1.3%w/w) were compared with biodiesel obtained from a batch reactor as shown in Table 3. The operating conditions were the same for both experiments, except that the batch system was operated with the reaction time of 1 h and the batch volume of 50 ml. It is clear that the microchannel system provided much better quality of biodiesel in a much shorter reaction time compared to that of the batch system. For the microchannel system operated under the specified conditions, the product accumulation over the period



Fig. 11. %FAME versus residence time at difference of length of reactor.

of 1 h was 175 ml. In practice, the number of microchannels can be increased for scaling up the production capacity.

6. Effect of Residence Time to %FAME

One of the key parameters for operating a chemical reactor is residence time, which can be adjusted by either changing the reactant feed flow rate or the length of the reactor. To study these two effects on the %FAME, a series of experiments were conducted with different lengths of microchannel and flow rates. The operating conditions were as follows: methanol-to-oil ratio of 6:1, temperature of 65 °C, and KOH concentration of 1.3%w/w. The residence time was varied from 5 to 20 s for two microchannels with different length: 120 cm and 240 cm. Fig. 11 shows that %FAME obtained from the longer microchannel was lower than that of the short one. For the same flow rate, at 5 s for the upper curve and at 10 s for the lower curve, the short microchannel yielded higher %FAME of 94.51%. The same observation is more evident for cases of 10 s/120 cm and 20 s/240 cm. This counter-intuitive result can be attributed to the mass transfer effect. We observed a relationship between pressure and residence time as represented in Fig. 12. For a fixed length of microchannel, the pressure increased with increasing flow rate. This affected the droplet size as shown in the photos embedded in Fig. 12 for each case (taken from the outlet of microchannel). It is apparent that the droplet size exhibited a strong dependence on pressure. Comparing between B and F, where the same flow rates were used, shows that %FAME was lower for the longer microchannel despite the slightly higher pressure in the longer microchannel. The same comparison can be observed for cases A and E. This is because the droplets generated for case F were larger than those of case B, possibly due to the agglomeration of small droplets as they propagated through the microchannel. As mentioned previously, the transesterification rate was affected by the interface mass transfer. Consequently, the reaction was slower in the longer microchannel.

Table 3. Performance of biodiesel synthesis: microchannel vs batch

Dopator	Time	0% FAME	Acid value	Viscosity	Standard	
Reactor	(sec)	701 AIVIE	(mgKOH/g)	(cst)	Viscosity	Acid value
Microchannel	5	95.41	0.4250	4.1976	3.5-5	0.5 max
Batch	3600	71.97	0.4675	4.5065		



Fig. 12. Droplet size and pressure at difference length of reactor (a), (d) 5 seconds (b), (e) 10 seconds and (c), (f) 20 seconds.

Ta	bl	e 4.	Pro	perty	of	bio	diese	l fr	om	micro	channel	reactor

Property	Biodiesel from microchannel reactor	Standard	Method
%FAME	95.41	96.5 min	EN14013
Viscosity at 40 °C (cst)	4.1976	3.5-5	ASTM D 445
Pour point (°C)	5	(-15) - 10	ASTM D 6751
Cloud point (°C)	7	(-3) - 12	ASTM D 6751
Flash point (°C)	166	120 min	ASTM D 93
Acid value (mg KOH/g)	0.425	0.5 max.	ASTM D 664
Density at 15 °C (kg/m3)	872	860-900	ASTM D 1298

7. Properties of Biodiesel

Our synthesized biodiesel was analyzed for basic properties as summarized in Table 4. The operating conditions were as follows: methanol-to-oil ratio of 6:1, temperature of 65 °C, residence time of 5 s, and KOH concentration of 1.3%w/w. %FAME of biodiesel from microchannel reactor was 94.51% probably due to the free fatty acid content in the pork lard. Other properties were compatible with ASTM for biodiesel.

CONCLUSION

Pork lard was transesterified in a microchannel reactor with KOH as homogeneous catalyst. A complete factorial design of experiments was used to specify operating conditions in terms of reaction temperature, molar ratio of methanol-to-oil, residence time, and catalyst concentration. Statistical analysis was performed to identify significant effects. High reaction temperatures caused a decrease in %FAME due to promoted saponification, a side reaction, and phase change of methanol. High molar ratio of methanol-to-oil resulted in emulsifying effect. The residence time was found to be inversely correlated with the %FAME. The optimal conditions that yielded 95.41% of FAME were methanol-to-oil ratio of 6 : 1, temperature of 65 °C, residence time of 5 s, and KOH concentration of 1.3%w/w. A quadratic model was proposed for the accurate prediction of %FAME in terms of parameters studied. The counter-effects of high pressure and agglomeration of droplets have been demonstrated.

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