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## Abstract

Production of fatty acid methyl ester (FAME) from palm fatty acid distillate (PFAD) having high free fatty acids (FFA) was investigated in this work. Batch esterifications of PFAD were carried out to study the influence of: including reaction temperatures of 70–100 °C, molar ratios of methanol to PFAD of 0.4:1–12:1, quantity of catalysts of 0–5.502% (wt of sulfuric acid/wt of PFAD) and reaction times of 15–240 min. The optimum condition for the continuous esterification process (CSTR) was molar ratio of methanol to PFAD at 8:1 with 1.834 wt% of H<sub>2</sub>SO<sub>4</sub> at 70 °C under its own pressure with a retention time of 60 min. The amount of FFA was reduced from 93 wt% to less than 2 wt% at the end of the esterification process. The FAME was purified by neutralization with 3 M sodium hydroxide in water solution at a reaction temperature of 80 °C for 15 min followed by transesterification process with 0.396 M sodium hydroxide in methanol solution at a reaction temperature of 65 °C for 15 min. The final FAME product met with the Thai biodiesel quality standard, and ASTM D6751-02.

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*Keywords:* Oleic acid; Palmitic acid; Taguchi method; Biodiesel

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## 1. Introduction

There is growing interest in biodiesel (fatty acid methyl ester or FAME) because of the similarity in its properties when compared to those of diesel fuels [1]. However, production cost of the biodiesel is not economically competitive with petroleum-based fuel according to relatively high cost of the lipid feedstocks, which are usually edible-grade refined oils. With low-cost lipid feedstocks containing high amount of free fatty acids (FFA), conventional biodiesel production by transesterification with alcohol using base catalyst is not appropriated. A two-step process is then proposed [2–7]. The first step of the process is to reduce FFA content in vegetable oil by esterification with methanol and acid catalyst. The second step is transesterification process, in which triglyceride (TG) portion of the oil reacts with methanol and base catalyst to form ester and glycerol. The acid catalyst is generally sulfuric acid [8,9] while the base catalyst is usually sodium or potassium hydroxide [10]. Product from the

reactions is separated into two phases by gravity. The FAME portion is then purified by water washing process to meet the biodiesel fuel standards.

With tobacco seed oil, the FFA level was reduced from about 17 wt% to less than 2 wt% in 25 min at reaction temperature of 60 °C and molar ratio of 18:1 of methanol to oil in the first step. In the second step, the maximum yield of 91% FAME was obtained in 30 min at molar ratio of methanol to triglycerides of 6:1, KOH amount of 1 wt% and a reaction temperature of 60 °C [4]. FAME production from a high content of FFA waste cooking oil (WCO) catalyzed by sulfuric acid increased rapidly within 1–6 h and then dropped down. When the molar ratio of methanol to oil exceeded 16, WCO conversion increased rapidly. In addition, WCO conversion increased with the amount of sulfuric acid up to 4 wt% [5].

Crude palm oil (CPO) esterification was carried out at temperature of about 80 °C for 30 min in a fixed bed reactor. The esterification reaction products were then transferred to the transesterification section which consisted of two stirred tank reactors. In the both reactors, the transesterification reaction was carried out at about 70 °C for 30 min to achieve >98% conversion [11].

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The works done previously for production of biodiesel from high FFA content feedstock were limited to the amount of FFA less than 40 wt% [3–9]. In this work the potential of palm fatty acid distillate (PFAD), a by product from production of consumable palm oil, with FFA content of 93 wt% to be used as feedstock for a continuous production of biodiesel was studied.

## 2. Materials and methods

### 2.1. Chemical

PFAD was obtained from Chumporn Palm Oil Industry Public Company Limited. It consists of 93 wt% FFA (45.6% palmitic, 33.3% oleic, 7.7% linoleic, 3.8% stearic, 1.0% myristic, 0.6% tetracosenoic, 0.3% linolenic, 0.3% ecosanoic, 0.2% ecosenoic, and 0.2% palmitoleic acid) and the rest are triglycerides, diglycerides (DG), monoglycerides (MG) and traces of impurities. All chemicals including 99% methanol (MeOH), 98% sulfuric acid ( $H_2SO_4$ ), and 99% sodium hydroxide (NaOH) are commercial grade.

### 2.2. Studies of significant esterification parameters in batch process

The esterification process was carried out in 250 ml screw-capped bottles. Preheated PFAD was firstly poured into the bottles following by MeOH and  $H_2SO_4$ . The bottles were immersed in an oil bath at designed temperature and time. Operating parameters for esterification process including reaction temperatures in the range of 70–100 °C, molar ratios of MeOH to PFAD in the range of

0.4:1–12:1, quantity of  $H_2SO_4$  catalysts in the range of 0–5.502 wt% and reaction times in the range of 15–240 min were investigated. Production mixture was poured into the separating funnel and then allowed to settle into two phases. A bottom FAME-layer was separated and purified by water washing process before being analyzed for its compositions by a thin layer chromatography (TLC). The optimum condition for batch esterification process was determined by Taguchi method [12] and then used for an initial condition in the continuous esterification process.

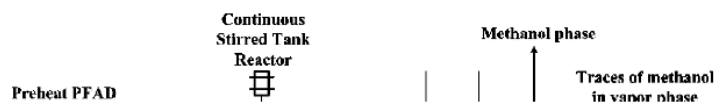
### 2.3. The continuous esterification process

Preheated PFAD and  $H_2SO_4$  (1.834 wt%) in MeOH solution were fed into the continuous stirred tank reactor (CSTR). The effect of molar ratios of MeOH to PFAD in the range of 6.5:1–9.5:1 on continuous production of FAME was investigated at reaction temperatures of 70 and 75 °C under its own pressure for the set time interval of 60 min.

A schematic diagram of a continuous unit for production of biodiesel is shown in Fig. 1. The unit mainly consists of a 22.4 L stainless-steel CSTR and three 30 L stainless-steel separators.

### 2.4. Purification processes

The reaction product was transferred to cooling water unit and then to a separator. It was separated into two phases for 100 min (a good separating time). The top phase contained excess MeOH and water formed during the reaction, while the FAME phase was at the bottom. MeOH



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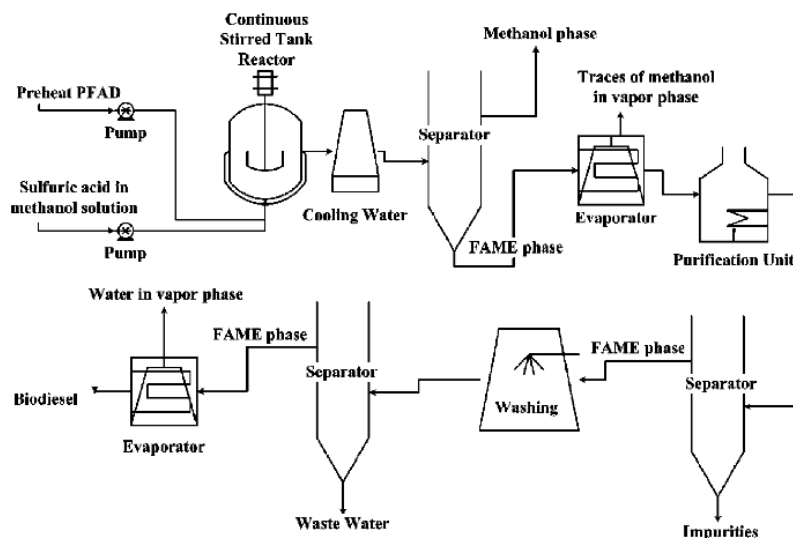
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was purified by rectification and then reused as starting material.

The FAME phase was taken off at the bottom and passed into evaporator to remove traces of methanol. The FAME still had residual FFA about 1.4 wt%, requiring further purification.

The purification was investigated by using three different methods as follows.

The first method was neutralization with sodium hydroxide in methanol (NaOH–MeOH) solution at a reaction temperature of 80 °C. NaOH (4 wt%) was dissolved in MeOH and then mixed with FAME. The mixture was heated to its reaction temperature and let the reaction to carry out for 15 min. Both neutralization and transesterification reactions took place at the same time.

The second method was carried out using 3 M of sodium hydroxide in water (NaOH–H<sub>2</sub>O) solution using the same conditions as the first one. In this case there are no transesterification reactions only the formation of soaps, which were removed by 2 wt% salt (NaCl) addition (2 g of salt/100 g of FAME phase).

The third method was neutralization with 3 M of NaOH–H<sub>2</sub>O solution at a reaction temperature of 65 °C for 15 min. The neutralized product was settled and the soap phase was then removed. After that the neutralized FAME phase was transesterified at a temperature of 80 °C under its own pressure for 15 min with 0.396 M of NaOH–MeOH solution.

The product obtained from the purification step was settled in a separator. The FAME phase was separated and washed with water at temperatures of 25–90 °C to remove impurities. The solution was settled for water separation and finally the residual water was evaporated.

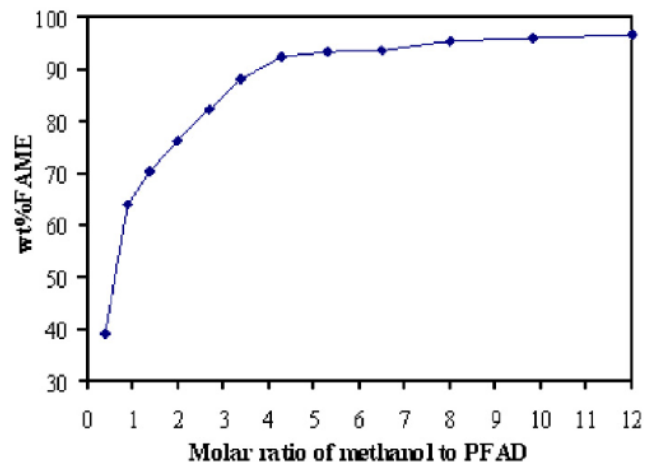


Fig. 2. Effect of molar ratio of methanol to PFAD on FAME content at 90 °C, 4 h and 1.834 wt% of H<sub>2</sub>SO<sub>4</sub> under its own pressure.

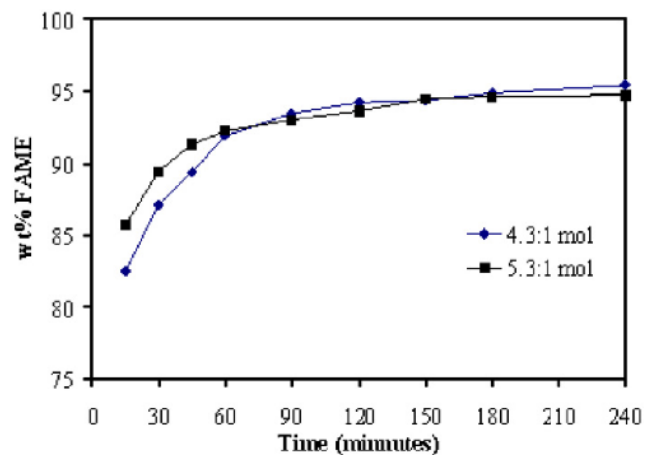


Fig. 3. Effect of reaction time on FAME content at 90 °C, 4.3:1 and 5.3:1

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### 2.5. Analysis of FAME

The compositions of the reaction mixture samples were determined by a thin layer chromatography equipped with flame ionization detector (TLC–FID) using an Itronscan MK-6s with Chromarods type S-III quartz rod (Mitsubishi Kagaku Iatron). One microliter of the reaction medium, diluted in hexane at appropriate dilution, was spotted onto chromarods. Firstly, chromarods were immersed in a solvent mixture of hexane:diethyl ether:formic acid (50:20:0.3 v/v) until the solvent reaching to 8 cm (approximately 15 min). Secondly, the chromarods were immersed in a solvent mixture of benzene:hexane (50:50 v/v) for about 35 min (or until the solvent reach to 10 cm). The spotted samples were developed in these two solvent mixtures. The chromarods were then dried at 105 °C for 5 min and scanned with TLC–FID. Scanning was performed using a 160 ml/min of hydrogen flow rate and 20 l/min of air flow rate to produce a chromatogram. The compositions were calculated as wt% based on the peak areas of each component.

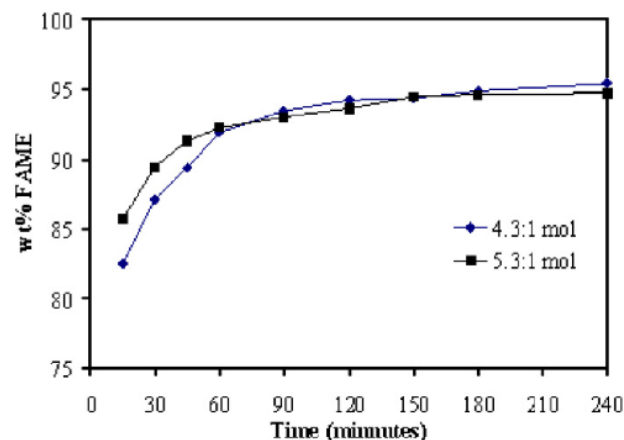


Fig. 3. Effect of reaction time on FAME content at 90 °C, 4.3:1 and 5.3:1 molar ratios of methanol to PFAD and 1.834 wt% of H<sub>2</sub>SO<sub>4</sub> under its own pressure.

## 3. Results and discussion

### 3.1. Batch process

#### 3.1.1. Effect of molar ratio of methanol to PFAD

FAME content of esterification reaction in relation with molar ratio of MeOH to PFAD is shown in Fig. 2. The optimum conversion was achieved at the molar ratio of 4.3:1. Further increase of molar ratio did not significantly increase the amount of FAME.

#### 3.1.2. Effect of reaction time

Fig. 3 shows the effect of reaction time on FAME content at molar ratios of methanol to PFAD 4.3:1 and 5.3:1 with 1.834 wt% of H<sub>2</sub>SO<sub>4</sub>. For both molar ratios, the rapid formation of FAME was observed within the first 90 min. After that the conversion rate was slower and finally reached steady state.

### 3.1.3. Effect of reaction temperature

For all molar ratios the FAME content was increased with temperature ranging from 70 to 100 °C (Fig. 4). However, the conversion rates were reduced in the range of 90–100 °C. The highest studied temperature was 100 °C because there was a chance of loss of methanol and increasing of darkness color of the product at higher temperature.

### 3.1.4. Effect of acid catalyst amount

The amount of acid catalyst used in the process was varied as 0, 0.183, 0.917, 1.834, 2.751, and 5.502 wt% of H<sub>2</sub>SO<sub>4</sub>. It was found that the esterification reaction hardly occurred without catalyst (Fig. 5). An appropriate amount of H<sub>2</sub>SO<sub>4</sub> acid catalyst was 1.834 wt% as it gave maximum amount of FAME. There was no improvement of FAME content with the amount of catalyst higher than 1.834%.

### 3.1.5. Determination of the optimal initial condition for continuous process by Taguchi method

Molar ratio of methanol to PFAD (*R*), reaction temperature (*T*), reaction time (*t*) and catalyst amount

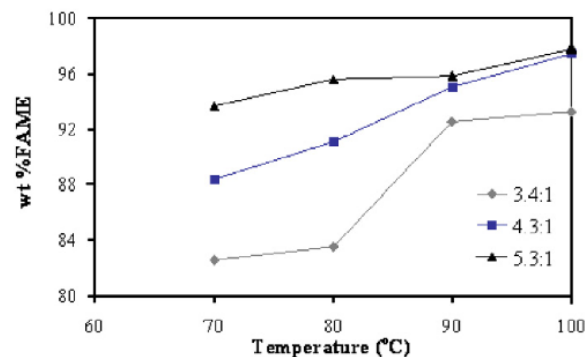


Fig. 4. Effect of reaction temperature on FAME content at 3.4:1, 4.3:1 and 5.3:1 molar ratios of methanol to PFAD, 2 h and 1.834 wt% of H<sub>2</sub>SO<sub>4</sub> under its own pressure.

(*S*) were selected as independent variables. The levels of independent variables determined from preliminary experiments are given in Table 1. With a three-level-four-factor array, L<sub>9</sub> (3<sup>4</sup>), nine experiments were required as shown in Table 2. The conditions of experiment 7 were chosen as the starting conditions for the continuous esterification.

## 3.2. Continuous process

### 3.2.1. Effect of molar ratio of methanol to PFAD and reaction temperature on the conversion of FAME

It can be seen in Fig. 6 that the FAME conversion was raised when the molar ratio of methanol to PFAD was increased for both reaction temperatures of 70 and 75 °C. Table 3 shows the levels of residual FFA and glycerides with increasing molar ratio of methanol.

Table 1  
Independent variables and levels of L<sub>9</sub> (2<sup>4</sup>) for Taguchi method

Parameters	Symbol	Level 1	Level 2	Level 3
Temperature (°C)	<i>T</i>	65	70	75
Time (min)	<i>t</i>	60	90	120
Molar ratio of methanol to PFAD	<i>R</i>	5.3:1	6.5:1	8.0:1
H <sub>2</sub> SO <sub>4</sub> amount (wt%)	<i>S</i>	0.917	1.834	2.751

Table 2  
Taguchi experiments for determining the initial condition for the continuous esterification process

No.	<i>T</i>	<i>t</i>	<i>R</i>	<i>S</i>	FAME content (wt%)
1	1	1	1	1	84.834
2	1	2	2	2	92.294
3	1	3	3	3	93.902
4	2	1	2	2	93.326
5	2	2	3	1	93.796
6	2	3	1	3	93.318
7	3	1	3	2	93.900
8	3	2	1	3	93.331
9	3	3	2	1	93.857

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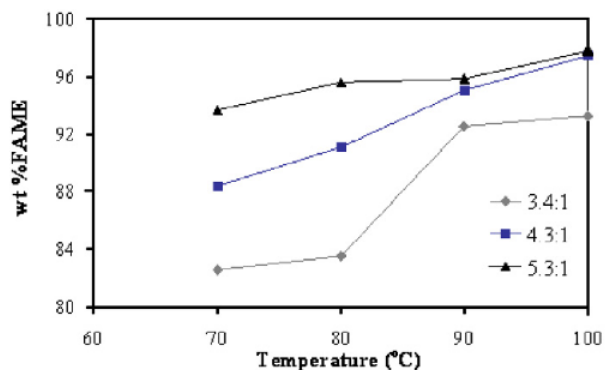


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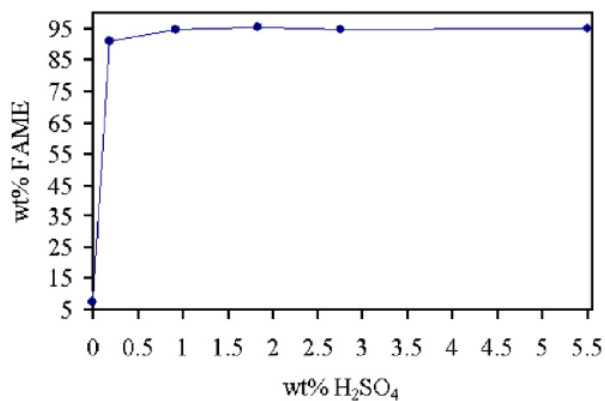


Fig. 5. Effect of acid catalyst amount on FAME content at 90°C, 4.3:1 molar ratio of methanol to PFAD and 2 h under its own pressure.

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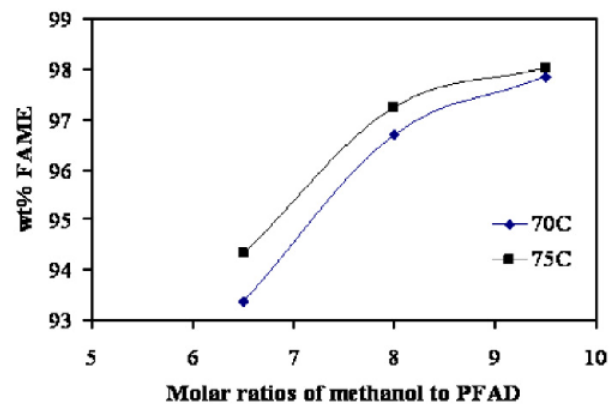


Fig. 6. FAME content as a function of molar ratios of methanol to PFAD (6.5:1, 8:1, and 9.5:1) and reaction temperatures (70 and 75°C).

Therefore, the optimum condition for continuous esterification process to produce FAME was a reaction temperature of 70 °C for 60 min of reaction time with 1.834 wt% H<sub>2</sub>SO<sub>4</sub> (molar ratio of MeOH to PFAD to H<sub>2</sub>SO<sub>4</sub> is 8:1:0.05).

### 3.2.2. Purification processes

The FAME obtained from esterification process still had 1.4 wt% residual FFA, which must be removed to meet the biodiesel standards.

When the FAME was neutralized with NaOH–MeOH solution there was no residual FFA, lower glycerides and higher FAME content product. Hundred percent FAME was obtained using 5.48 ml of 1 M NaOH–MeOH solution. However, this method produced high levels of waste methanol and soap.

When the FAME was neutralized with NaOH–H<sub>2</sub>O solution, the method required higher amount of NaOH than the first method. However, with salt addition removal of soap from water was easily obtained. It was also found that increasing levels of NaOH could increase the amount of FAME and decrease the amount of glycerides. To meet quality standard of biodiesel, 9.13 v/wt% of 3 M NaOH–H<sub>2</sub>O solution (9.13 ml of the solution/100 g of FAME phase) was required and to get 100% FAME 11.87 v/wt% of 3 M NaOH–H<sub>2</sub>O solution was needed (Fig. 7).

Table 3

The amount of residual FFA and glycerides in the product after the continuous esterification process

Molar ratios of MeOH to PFAD	wt%	
	Residual FFA	Glycerides
6.5:1	2.212	4.408
8.0:1	1.402	1.913
9.5:1	0.984	1.166

The FAME phase can also be purified by neutralization with NaOH–H<sub>2</sub>O solution followed by transesterification process. Neutralization with 1.83 v/wt% of 3 M NaOH–H<sub>2</sub>O solution could get rid of all residual FFA; however, the residual glycerides needed to be transformed to FAME by transesterification process. The purity of FAME met quality standard for biodiesel when the amount of 0.396 M NaOH–MeOH solution was 3.85 wt% (3.85 g of solution/100 g of neutralized FAME phase) and to get 100% FAME 4.46 wt%, the 0.396 M NaOH–MeOH solution was required (Fig. 8).

To obtain maximum yield of FAME, neutralization with NaOH–H<sub>2</sub>O solution followed by transesterification process was required.

### 3.2.3. Fuel properties of PFAD biodiesel

The fuel properties of biodiesel obtained in this work are summarized in Table 4. It can be seen that most of its properties are in the range of fuel properties prescribed in the latest Thai and American standards for biodiesel, except cloud point and pour point. PFAD mainly consists of saturated FFA (>49 wt%) which results in high values of cloud point and pour point of PFAD biodiesel. However, a blend of diesel and PFAD biodiesel is possible in practice.

As it is widely known that the color of FAME obtained from acid-catalyzed esterification process is usually black. The process of purifying the FAME to the biodiesel standard was able to remove some of this color and our final product was a brown color.

## 4. Conclusion

A process for the production of biodiesel from relatively low cost PFAD a residual product from the refining of crude palm oil has been evaluated. The final product is a light brown material meeting the requirements of the Thai biodiesel standard. A range of methanol to PFAD ratios



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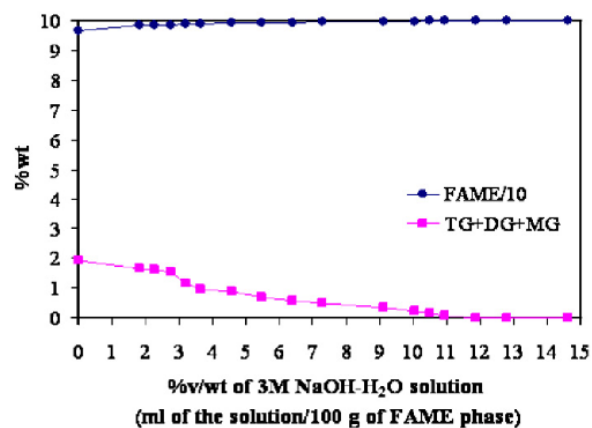


Fig. 7. FAME and glycerides content in the product after neutralization with 3 M sodium hydroxide in water solution at 80 °C and atmospheric pressure for 15 min.

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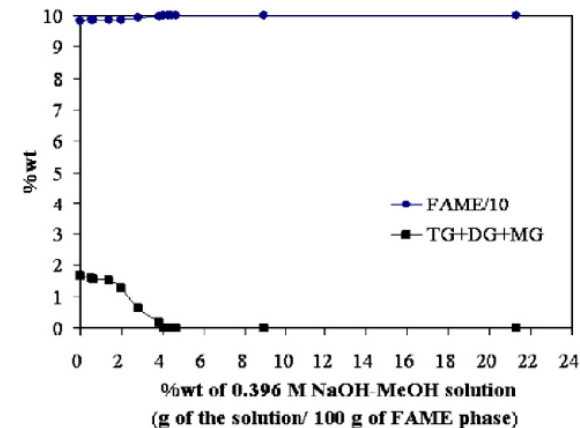


Fig. 8. FAME and glycerides content in the neutralized product after transesterification process with 0.396 M sodium hydroxide in methanol solution at 80 °C under its own pressure for 15 min.

				Thai standard	ASTM D6751-02
Density at 15 °C	kg/m <sup>3</sup>	ASTM D4052	879.3	860–900	870–900
Viscosity at 40 °C	mm <sup>2</sup> /s	ASTM D445	4.865	3.50–5.00	1.9–6.0
Flash point	°C	ASTM D93	181	120 min	130 min
Cloud point	°C	ASTM D2500	15	–	–3 to 12
Pour point	°C	ASTM D97	14	–	–15 to 10
Distillation 95%	°C	ASTM D86	335	360 max	360 max
Water content	wt%	ASTM D6304	0.03	0.05 max	0.03 max
Ash content	wt%	ASTM D874	0.0066	0.02 max	0.02 max
Carbon residue	wt%	ASTM D4530	0.07	–	–
Acid value	mg KOH/g	ASTM D664	0.33	0.50 max	0.80 max
Copper corrosion	Number	ASTM D130	1	1 max	3 max
Ester content	wt%	TLC	99.480	96.5 min	–
Triglyceride	wt%	TLC	0.000	0.2 max	–
Diglyceride	wt%	TLC	0.058	0.2 max	–
Monoglyceride	wt%	TLC	0.462	0.8 max	–

<sup>a</sup>PFAD biodiesel from neutralization with 1.83 v/wt% of 3 M NaOH–H<sub>2</sub>O solution at 65 °C for 15 min followed by transesterification process using 3.85 wt% of 0.396 M NaOH–MeOH solution at 80 °C for 15 min.

and acid catalyst concentrations were established that would produce a high-quality product in reasonable CSTR residence times, and at lower temperatures which we considered to be a more economic solution.

### Acknowledgment

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