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Original Article

Production of ethyl ester from crude palm oil by two-step reaction using continuous microwave system

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Abstract

The esterification of free fatty acids (FFA) in vegetable oils with alcohol using an acid catalyst is a promising method to convert FFA into valuable ester and obtain a FFA-free oil that can be further transesterified using alkali bases. In this work, the direct esterification reaction of FFA in crude palm oil to ethyl ester by continuous microwave was studied and the effects of the main variables involved in the process, amount of catalyst, reaction time and the molar ratio oil/ alcohol, were analyzed. The optimum condition for the continuous esterification process was carried out with a molar ratio of oil to ethanol 1:6, using 1.25%wt of H_2SO_4/oil as a catalyst, microwave power of 78 W and a reaction time 90 min. This esterification process shows that the amount of FFA was reduced from 7.5%wt to values around 1.4 %wt. Similar results were obtained following conventional heating at 70°C, but only after a reaction time of 240 min. The esterified crude palm oil is suitable to perform the transesterification process. Transesterification of the esterified palm oil has been accomplished with a molar ratio of oil to ethanol of 1:8.5, 2.5%wt of KOH as a catalyst, a microwave power of 78 W, and a reaction time of 7 min. In addition, the problem of glycerin separation was solved by mixing 10%wt of pure glycerin into the ethyl ester to induce the glycerin from the reaction to separated. This two-step esterification and transesterification process provided a yield of 78%wt with an ester content of 97.4%wt. The final ethyl ester product met with the specifications stipulated by ASTM D6751-02.

Keywords: microwave, biodiesel, ethanol, crude palm oil

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1. Introduction

Due to the increase in the price of the petroleum and environmental concerns about pollution coming from the car gases, biodiesel is becoming a developing area of high concern. Biodiesel has been defined as “the alkyl esters” of long chain fatty acids derived from renewable feedstock (e.g. vegetable oils and animal fats). Biodiesel can be used as a substitute or an additive to diesel fuel. Compared to fossil-based diesel fuel, biodiesel possesses many advantages such as renewability, biodegradability, non-toxicity, a 65 %wt–reduction in carbon monoxide production, a 90%wt–reduction in sulfur dioxide production, and a 50%wt–reduction in unburnt hydrocarbon emission (Alamu *et al.*, 2008).

Vegetable oil remains the major feedstock for biodiesel production. Animal fat and waste cooking oil have also been used. Soybean (US), rapeseed (Europe), and oil palm (South-East Asia) (Peterson *et al.*, 1999), to mention a few have been successfully used as renewable vegetable oil sources to generate biodiesel with superior qualities over the petroleum-based fuels. Oil palm has a high-yield crop with the lowest price trade among the other vegetable oils.

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Therefore, it has the potential as the main source of biodiesel (Tan et al., 2007). The extracted oil from oil palm is known as Crude Palm Oil (CPO), which consists of more than 90%wt of triglyceride and 3-7%wt of free fatty acid (FFA). The main fatty acids are palmitic and oleic acids. These feedstock often contain significant quantities of free fatty acids and water, which make them unsuitable for existing homogeneous alkaline-catalyzed processes. Neutralization of FFA can be carried out by the addition of excess alkali, but this leads to the formation of soaps and to post–reaction separation problems. Thus, a preferred pretreatment process for CPO is an esterification process with alcohol, which changes FFA to esters and commonly uses a strong liquid acid catalyst, such as sulfuric acid. When the FFA content is lower than 2.0%wt, it was suitable for the raw material in transesterification process to produce biodiesel. Alcohols such as methanol and ethanol are the most frequently employed. Although the use of different alcohols presents some differences with regard to the reaction kinetics, the final yield of esters remains more or less the same (Issariyakul *et al.*, 2007). Therefore, selection of the alcohol is based on cost and performance considerations. Ethanol can be produced from agricultural renewable resources, thereby attaining total independence from petroleum-based alcohols. Also, ethanol, as an extraction solvent, is preferable to methanol because of its much higher dissolving power for oils. For this cause, ethanol is often used as an appropriate alcohol for the esterification of vegetable oils.

The general heating system for biodiesel production uses heating coils to heat the raw material. Using microwave for preparative chemistry, it is often possible to accelerate the rate of reactions, to reduce the reaction time, and by all this reduce the energy consumption (Mazzocchia *et al.*, 2004; Hernando *et al.*, 2007). By this, it has been proved possible to prepare biodiesel rapidly and with good conversions by microwave heating.

In this paper, we describe the production of biodiesel from CPO, which is abundant in Thailand. Since CPO has a high FFA content, thus a two-step esterification and transesterification process has been developed. In the first step of the process, the FFA content of the CPO was reduced from 7.5%wt to below 2%wt. The product from the first step was used as the raw material for the second step. In both steps ethanol was used as an alcohol and a microwave heating system was used to facilitate the production of biodiesel in a short time.

2. Experimental Methods

2.1 Materials

Crude palm oil and ethanol (99.5%) were acquired from the Specialized R&D Center for Alternative Energy from Palm Oil and Oil Crops (Thailand). Phenolphthalein used for indicator was analytical reagent grade from Labchem (PA, USA). Potassium hydroxide (95%) and sulfuric acid (98%) were commercial grade.

2.2 Microwave system

All microwave tests were conducted with a Toshiba, model ER-D33SC compact microwave oven working at 2.45 GHz with a power of 1,100 W. Reactions were performed in 1.5 L glass reactor in the microwave oven for continuous experiments.

2.3 Experimental procedures and analysis

2.3.1 Measurement of the microwave power output

Commercial microwave ovens often deviate from their specified power output. The actual microwave power output could be determined by measuring the rate of temperature increase of a known quantity of water placed in the oven. The power is calculated from two variations of this method (Daewoo Electronics Co., Ltd). The method requires loading 1,000 mL of water in a cylindrical glass vessel in the center of the microwave oven. The water temperature is measured before turning on the microwave. As soon as the oven shuts off, the final water temperature is measured. The microwave power output P in W is calculated from Equation 1:

$$P = \frac{4187 \times \Delta T}{t} \tag{1}$$

where P is the microwave power output (W), DT is difference between the initial and final temperature (°C) and t is the heating time (s).

2.3.2 Continuous esterification process

The apparatus used is shown in Figure 1. The continuous esterification process was started by the feeding of CPO and sulfuric acid (0-1.75%wt of H_2SO_4/oil) in ethanol solution (molar ratio of oil/ethanol 1:1 to 1:8) into the mixing Tank 1. The mixture was overflowed into the Reactor 1 which was the glass tube in microwave oven for esterification process at 78 W for 15-120 min. The esterification reaction was carried out with the studied initial condition. After that it was flowed into separation tank to separation the esterified palm oil from the ethanol.

2.3.3 Continuous transesterification process

The transesterification process was carried out with the product form the esterification process and KOH (1-3 %wt of KOH/oil) dissolved in ethanol (molar ratio of oil/ ethanol 1:4.5 to 1:10.5) and mixed in mixing Tank 2. The mixture was overflowed into the Reactor 2, which was the glass tube in microwave oven for transesterification process at 78 W for 1-10 min. At the end of the reaction, 0-20%wt of pure glycerin was added, which resulted in the formation of an upper phase consisting of ethyl esters and a lower phase

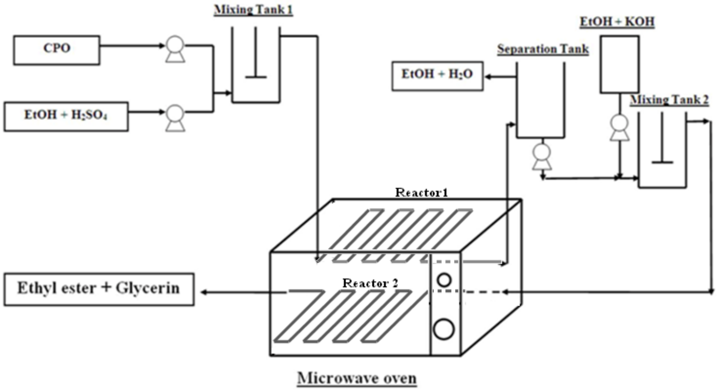

Figure 1. The apparatus used in the continuous experiments

containing glycerin. After separation of the layers by sedimentation in a separatory funnel for 30 min, the ethyl esters were purified by washing with hot water. The washed ethyl esters were dried at 120°C for 20 min.

2.3.4 Analysis of the reaction products

Free fatty acid content was determined by titration (AOCS Ca 5a-40). The glycerin content of the ethyl esters after the separation processes was measured by titration (TIS 1593-2541). The purified ethyl esters were analyzed by gas chromatography (GC) using an Agilent 5890 gas chromatograph with a flame ionization detector and a Stabilwax column of length 30 m, film thickness 0.25 µm, and i.d. 0.25 mm. Fatty acid components of the ethyl esters were identified by gas chromatographymass spectrometry (GC-MS) using the HP 5890 gas chromatograph coupled to an HP 5972 mass-selective detector. The density at 15°C was measured by the hydrometer method (ASTM D1298), the acid value was determined by titration (ASTM D664), the glycerin content was determined by titration (BS 5711) and the water content was determined by the Karl-Fischer method. The flash point was determined by means of a Pensky-Martens closed tester (ASTM D93) and the viscosity was measured with a viscometer (ASTM D445). The cloud point (ASTM D2500) and the pour point (ASTM D97) were determined with pour and cloud point testers, the 95% distillation was determined with appropriate distillation equipment (ASTM D86). The ash content was measured with a furnace (ASTM D874), the copper corrosion was measured with a copper corrosion tester (ASTM D130) and the sulfur content was measured with X-ray fluorescence spectrometry (ASTM D5453).

3. Results and Discussion

Crude palm oil (CPO) consists of triglycerides containing three fatty acids linked by ester linkages to a glycerin moiety. The fatty acids vary in their carbon chain lengths and in the numbers of double bonds. The fatty acid composition of CPO was determined by GC-MS and the results are shown in Table 1. It can be seen that most of the values fall within the ranges published in the literature (Wing-Keong *et al.*, 2003; Choo *et al.*, 2005).

Table 1. Fatty acid composition of crude palm oil.

Fatty acid	Fatty acid composition (wt.%)		
	CPO (Choo *et al.*, 2005)	CPO (Wing-Keong *et al.*, 2003)	CPO this work
Lauric	0.3	0.2	-
Myristic	0.8	1.1	0.9
Palmitic	44.3	42.8	47.6
Palmitoleic	0.2	0.1	-
Stearic	5.0	3.8	2.5
Oleic	39.1	41.1	41.0
Linoleic	10.1	10.3	8.0
Linolenic	0.1	0.3	-
Arachidic	-	0.3	-
Other (Unknown)	-	-	2.3

Table 2. Comparison of microwave efficiency.

Microwave power input (W)	Microwave power output (W)	
	Experiment	Calculate at 70% of microwave power output
110	78	77
330	240	231
550	389	385
770	554	539
1100	798	770

3.1 Measurement of the microwave power output

Microwave power output in this experiment was calculated by indirectly measuring the temperature rise of water; the results are shown in Table 2. Microwave power output estimated from these measurements was 70% of the microwave power input. The measured microwave outputs from these experiments were similar to that reported by Barnard *et al.* (2007) who found that microwave power output was approximately 60% of the microwave power input.

3.2 Esterification process

The objective of this process was to lower the FFA contents of the CPO from its initial level of 7.5 %wt. Important variables affecting the FFA content in the esterification process were the molar ratio of oil to ethanol, the amount of acid catalyst, and the reaction time.

3.2.1 Effect of molar ratio of oil to ethanol

The amount of ethanol required for the esterification process was analyzed in terms of its molar ratio with respect to the oil. The effect of molar ratio on the %wt of the FFA content is shown in Figure 2. One percent by weight of H_2SO_4/oil was used as the catalyst, the microwave power was set to 78 W, and the reaction time was 1 h. The molar ratio of oil to ethanol was varied from 1:1 to 1:8, whereupon the optimum molar ratio was found to be 1:6. A further increase in the molar ratio resulted in only a minor reduction in the FFA content. This result was in good agreement with that of Marchetti *et al.* (2008) reported that the optimum molar ratio of oil to ethanol was 1:6 for reduced FFA in refined sunflower oil by esterification process.

3.2.2 Effect of varying the amount of acid catalyst

The amount of acid catalyst used in the process also affects the %wt of FFA content. The amount of acid catalyst was varied in the range 0-1.75 %wt of H_2SO_4/oil under reaction conditions of a 1:6 molar ratio of oil to ethanol, a reaction time of 1 h, and a microwave power of 78 W. The effect of the amount of catalyst on the %wt of FFA content is shown in Figure 3. It was found that the esterification reaction barely occurred without the catalyst. An appropriate amount of acid catalyst was found to be 1.25 %wt of H_2SO_4/oil as this gave the lowest %wt of FFA content. Little decrease in the FFA content was seen when the amount of catalyst was increased beyond 1.5 %wt of H_2SO_4/oil. Also, the addition of excess sulfuric acid could produce a product that

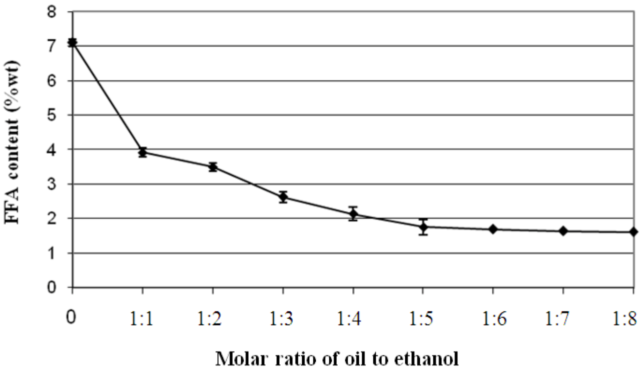

Figure 2. Effect of the molar ratio of oil to ethanol on %wt of FFA content after 1 h of reaction, with 1%wt H_2SO_4/oil and a microwave power of 78 W.

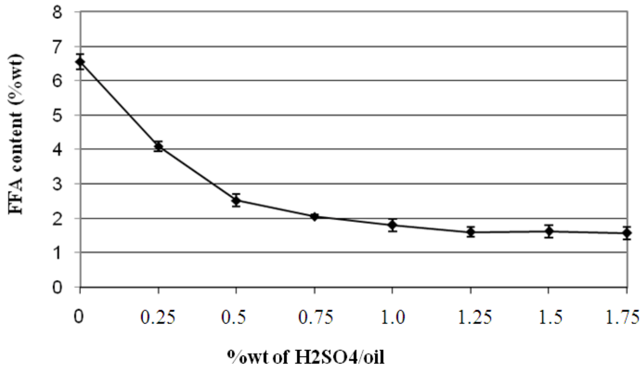

Figure 3. Effect of acid catalyst quantity on %wt FFA content with a 1:6 molar ratio of oil to ethanol after a reaction time of 1 h at a microwave power of 78 W.

was darker in color. This result was in good agreement with that of Ghadge and Raheman (2005), who reported that the optimum the amount of H_2SO_4/oil ratio 1 %wt of H_2SO_4/oil for reduced FFA in Mahua oil by esterification process.

3.2.3 Effects of reaction time

Figure 4 shows the effect of reaction time using a microwave power of 78 W on the %wt of FFA content compared with that using conventional heating at 70°C. The reaction times ranged from 20 to 120 min, keeping the other reaction parameters constant (1.25 %wt of H_2SO_4/oil, microwave power output of 78 W and a 1:6 molar ratio of oil to ethanol). Figure 4 shows the %wt of FFA content rapidly decreased within the first 15 min. Thereafter, the %wt of FFA content slowly decreased until the reaction time exceeded 90 min. At this point, the plot of the %wt of FFA content showed a slight upward trend, which may have been due to cleavage of the triglyceride to shorter-chain organic fractions such as FFA (Refaat and El Sheltawy, 2008). A reaction time of 90 min was selected for this study, whereas conventional heating at 70°C required 4 hrs; the latter is in good agreement with the findings of Wang et al. (2007) who employed a reaction time of 4 hrs and a reaction temperature of 95°C for the esterification process. The optimum conditions for the esterification process have been identified as a 1:6 molar ratio of oil to ethanol, 1.25 %wt of H_2SO_4/oil as the catalyst, a reaction time of 90 min and a microwave power of 78 W. At the end of the esterification process, the amount of FFA had been reduced from 7.5 %wt to 1.4%wt which was suitable for the raw material in biodiesel production.

3.3 Transesterification process

A case study of alkaline-catalyzed transesterification was run using esterified CPO that had 1.4%wt of FFA content, a molar ratio of oil to ethanol of 1:4.5 and residual acid catalyst. Important variables affecting the ester content in the transesterification process are the molar ratio of oil to ethanol, the amount of alkaline catalyst, and the reaction time.

3.3.1 Effects of molar ratio of oil to ethanol

The amount of ethanol required for transesterification was analyzed in terms of the molar ratio with respect to triglyceride. Stoichiometrically, the molar ratio of triglyceride to ethanol is 1:3. However, in practice this is not sufficient to complete the reaction. Higher molar ratios are required to complete the reaction at a satisfactory rate (Demirbas, 2002). Esterified CPO has initial molar ratio of oil to ethanol of 1:4.5; however, in practice this also is not sufficient to complete the reaction at a fast enough rate. Again, higher molar ratios are required. It can thus be seen that lower molar ratios require longer reaction periods. The effect of molar ratio on the %wt of ester content is shown in Figure 5. This demonstrates that the %wt of ester content increases with an increasing molar ratio of ethanol. The maximum %wt of ester content is obtained for a molar ratio of oil to ethanol of 1:8.5. Further increases in the molar ratio resulted in only a minor increase in ester content. This result showed that the transesterification of esterified CPO needed a lower molar ratio of ethanol compared with the result of Encinar *et al.* (2007) who employed a molar ratio of oil to ethanol of 1:12 for producing ethyl ester from used frying oil by a transesterification process.

3.3.2 Effects of alkaline catalyst amount

The amount of alkaline catalyst used in the process also affects the %wt of ester content. Tests were performed using varying amounts of alkaline catalyst, in a range of 1-3 %wt of KOH/oil, under reaction conditions of a 1:8.5 molar ratio of oil to ethanol, a reaction time of 5 min and a microwave power of 78 W. The effect of the amount of catalyst on the %wt of ester content is shown in Figure 6. An appropriate amount of alkaline catalyst was found to be 2.5 %wt of KOH/oil, giving the maximum %wt of ester content. Also,

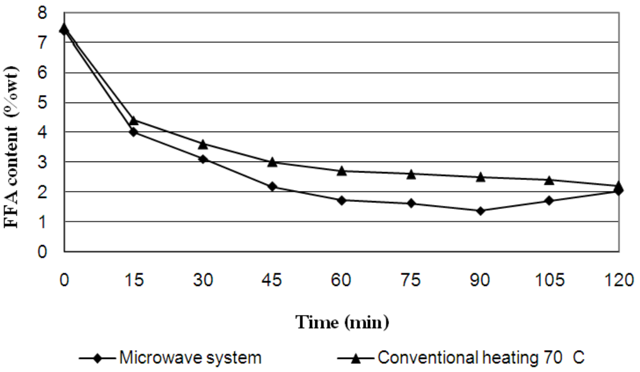

Figure 4. Effect of reaction at a microwave power of 78 W compared to conventional heating at 70°C on %wt of FFA content with 1.25%wt of H_2SO_4/oil and a 1:6 molar ratio of oil to ethanol.

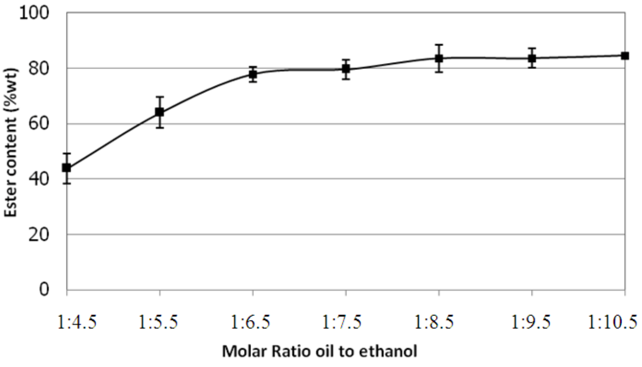

Figure 5. Effect of the molar ratio of oil to ethanol on %wt of ester content after 5 min of reaction, with 1.5%wt of KOH/oil and microwave power of 78 W.

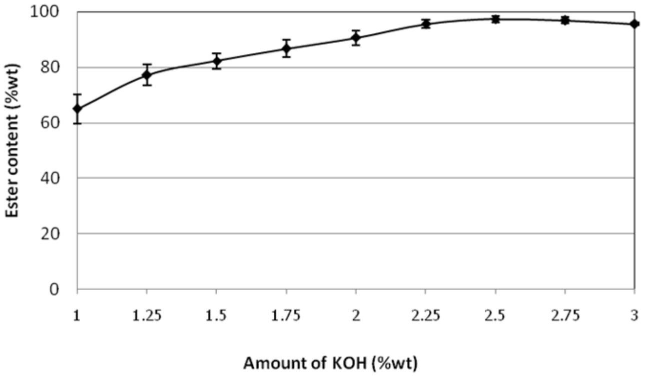

Figure 6. Effect of alkaline catalyst amount on %wt of ester content, using a 1:8.5 molar ratio of oil to ethanol, a reaction time of 5 min and microwave power of 78 W.

the addition of an excess amount of catalyst gave rise to the formation of an emulsion which in turn led to the formation of gels and a decreased % of ethyl ester. This result differed from that of Kulkarni *et al.* (2007) who employed an alkaline catalyst of 0.7 %wt of KOH/oil for producing ethyl ester from used canola oil by a transesterification process. Part of this difference may be attributed to differences in the raw material used, as well as the amount of alkaline catalyst needed to neutralize the acid catalyst in the esterification product (Ghadge and Raheman, 2005).

3.3.3 Effects of reaction times

Figure 7 shows the effects of reaction times on the %wt of ester content. Reaction times ranged from 1 to 10 min, with other reaction parameters remaining constant (2.5 %wt of KOH/oil, a 1:8.5 molar ratio of oil to ethanol and a microwave power of 78 W). The %wt of ester content rapidly increased within the first minute. Thereafter, the %wt of ester content slowly increased until the reaction times exceeded 7 min. Thus, a reaction time of 7 min was selected for this study. Conventional heating at 70°C would require 1 h to achieve the same ester content as that produced by microwave heating with a 7 min reaction time. The similar result was also observed by Issariyakul *et al.* (2007) who employed a reaction time of 1 h and a reaction temperature of 60°C to produce ethyl ester from waste fryer grease by a transesterification process. The present study confirms that a microwave-assisted chemical reaction significantly reduces the reaction time and increases the product yield, as has been mentioned in the literature (Saifuddin and Chua, 2004; Refaat and El Sheltawy, 2008). This result is to be expected, since the changing electrical field activates a very small degree of variance of molecules and ions, leading to molecular friction; therefore, initiation of chemical reactions is possible. Microwave treatment induces greater accessibility of the pertinent bonds, and hence a much more efficient chemical reaction (Azcan and Danisman, 2008). Consequently, microwave irradiation accelerates the chemical reaction, and a high product yield can be achieved within a short time. The obtained results are in good agreement with the findings of Hernando *et al.* (2007) who employed a reaction time of 5 min and used a microwave oven to produce biodiesel from rapeseed oil by a transesterification process. In the present study, the optimum conditions for the transesterification process were identified as a 1:8.5 molar ratio of oil to ethanol, 2.5 %wt KOH/oil as a catalyst, a reaction time of 7 min and a microwave power of 78 W.

At the end of the transesterification process, glycerin from the reaction had not separated from the ethyl ester by gravitation. Pure glycerin was then mixed in the product from transesterification process. This produced the formation of an upper phase consisting of ethyl esters, and a lower phase containing glycerin. Figure 8 shows the results of testing various %wt of pure glycerin added to the ethyl ester in order to reduce its glycerin content. The addition of 2-6 %wt of pure glycerin did not decrease the glycerin content, because the pure glycerin dissolved in the ethyl ester, and so could not induce a separation reaction. But the addition of more than 6 %wt of pure glycerin greatly decreased the glycerin content due to separation from the ethyl ester. The optimum amount of pure glycerin was found to be 10%wt. Adding more than 10%wt pure glycerin had no significant additional

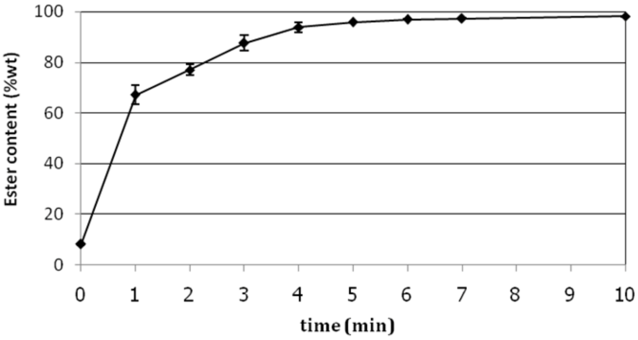

Figure 7. Effect of reaction time on %wt of ester content, using a 1:8.5 molar ratio of oil to ethanol, with 2.5%wt of KOH/oil and microwave power of 78 W.

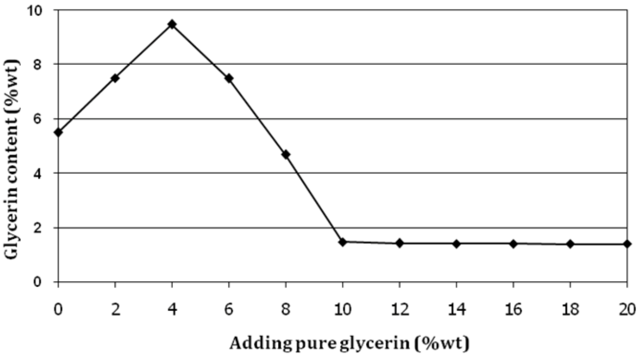

Figure 8. Effect of adding pure glycerin quantity on %wt of glycerin content in ethyl ester.

effect on the glycerin content of the ethyl ester. This result differed from Encinar *et al.* (2007) report, in which 25%wt of pure glycerin was added. Part of the difference may be attributed to the differences in the process used (heating system, molar ratio of oil to ethanol, and type of catalyst).

3.4 Fuel properties

The transesterification of esterified palm oil was carried out using KOH as the catalyst. When ethanol was used, the glycerol was not entirely separated and hence the amount of ethyl ester recovered was as low as 78%wt with respect to the CPO for this reaction. Issariyakul *et al.* (2004) also obtained 62%wt of ethyl ester from waste fryer grease. The purified ethyl ester was analyzed by GC-MS. Figure 9 shows the GC-MS trace of the ethyl esters from this experiment, which indicates 97.4% of ethyl esters. The fuel properties of ethyl ester in comparison with those of methyl ester standards are shown in Table 3. It can be seen that most of its salient properties fall within the limits prescribed by the American standards for methyl ester.

4. Conclusions

The production of ethyl ester from CPO, a high %wt of FFA feedstock, under microwave assistance has been investigated. It has been found that microwave irradiation facilitates the synthesis of ethyl ester from triglycerides within a few minutes. A two-step esterification and transesterification process has thus been developed to convert the high-FFA oils into ethyl ester. For the studied esterification process, the amount of FFA was reduced from 7.5%wt to around 1.4%wt under optimum conditions of a molar ratio of oil to ethanol of 1:6, 1.25%wt H_2SO_4/oil as catalyst, a reaction time of 90 min, and a microwave power of 78 W. The optimum conditions for the transesterification process as a 1:8.5 molar ratio of oil to ethanol, 2.5%wt KOH/oil as a catalyst, a reaction time of 7 min and a microwave power of 78 W. The problem of glycerin separation was solved by mixing 10 %wt of pure glycerin into the ethyl ester to induce the glycerin from the reaction to separated. This two-step transesterification process yielded 78%wt CPO ethyl ester; its ester content was 97.4%wt and its fuel properties were within the limits prescribed by American standards for methyl ester.

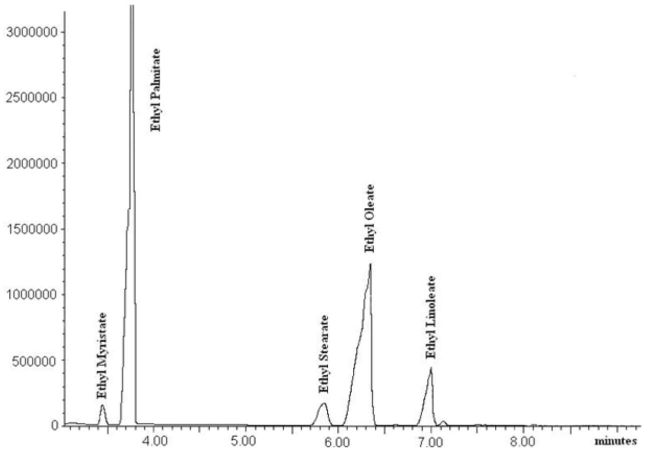

Figure 9. GC-MS chromatogram of ethyl esters.

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Table 3. Properties of ethyl ester in comparison with diesel and methyl ester standards

Properties	Unit	Test method	This work	ASTM D 6751-02*
Acid value	mg KOH/g	ASTM D664	0.33	0.80 max
Ash content	wt.%	ASTM D874	0.0066	0.02 max
Cloud point	°C	ASTM D2500	8	-3 to 12
Copper corrosion	No.	ASTM D130	1	3 max
Density at 15°C	kg/m^3	ASTM D4052	879.8	870-900
Distillation 95%	°C	ASTM D86	337	360 max
Ester content	wt.%	EN 14103	97.4	-
Flash point	°C	ASTM D93	160	130 min
Pour point	°C	ASTM D97	6	-15 to 10
Sulfur	wt.%	ASTM D5453	0.0002	0.001 max
Viscosity at 40°C	mm^2/s	ASTM D445	5.57	1.9-6.0
Water content	wt.%	ASTMD6304	0.03	0.03 max

*Standard for methyl ester (Adopting ASTM D 6751-02 for the methyl ester to the ethyl ester) (Shell Comments on National for Biodiesel Discussion Paper, 2003)

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