

Optimization of biodiesel production from *Jatropha curcas* L. oil via alkali-catalyzed methanolysis

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Abstract: In this study, crude *Jatropha curcas* oil was used as feedstock for biodiesel production by alkali-catalyzed methanolysis. The reaction in the presence of NaOH as catalyst was carried out to investigate the optimum conditions and to study the effects of variables on the reaction. These variables included methanol-to-oil molar ratios of 4:1-10:1, catalyst concentrations of 0.25-2.0% w/w of oil, reaction temperatures of 32-60°C, and reaction times of 5-40 minutes. The methyl ester content in the product was analyzed by gas chromatography (GC). The optimum conditions for *Jatropha curcas* biodiesel production were a methanol-to-oil molar ratio of 6:1, a catalyst concentration of 1.0% w/w of oil, a reaction temperature of 60°C, and a reaction time of 40 minutes. The methyl ester content under these optimum conditions was 98.6% w/w, and all of the measured properties of the *Jatropha curcas* biodiesel met the Thai biodiesel (B100) specifications and international standards EN 14214:2008 (E) and ASTM D 6751-07b, with the exception of lower oxidation stability. The results reveal that all of the reaction variables in this study had positive effects on the reaction.

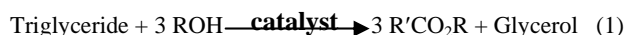
Keywords: Biodiesel · *Jatropha curcas* oil · Methanolysis · Methyl ester.

1. Introduction

In today's world, petroleum is clearly the most important energy source, providing more than half the world's power, as well as being a basic material used in the manufacture of fertilizers, synthetic fibers, plastics, and synthetic rubber [1-2]. Demand is ever-increasing worldwide, yet petroleum resources are finite [1] and non-renewable [3]. Concerns about dwindling supplies, its unstable and rising cost, and environmental problems have motivated researchers more extensively to seek alternative, renewable energy sources.

Among these alternative sources, vegetable oils have gained considerable attention since they are derived from renewable resources, can be domestically produced, and are not as harmful as petroleum to the environment [4]. However, the use of these oils is restricted to diesel engines, since they contain free fatty acids (FFAs), phospholipids, sterols, water, odorants, and other impurities [1]. In addition, vegetable oil's high viscosity, about 11-17 times higher than diesel fuel [1], affects the flow properties of the fuel, such as spray atomization, consequent vaporization, and air-fuel mixing in the combustion chamber, leading to an adverse effect on the combustion process [5].

To overcome these drawbacks, the oil has to be chemically modified. One way is by transesterification, which is the most common process to reduce the high viscosity of triglycerides [1]. This reaction can be represented by



Transesterification, also called alcoholysis, of triglycerides produces fatty acid alkyl esters (biodiesel), which can be used as fuel in diesel engines, and glycerol (glycerin) [1,6]. If methanol is used as alcohol in this process, it is called methanolysis [1]. An important feature of this process is that the unreacted methanol can be recovered and the glycerol can be used as a valuable co-product. The recovered glycerol is also valuable in the soap, pharmaceutical, and cosmetic industries [7]. This reaction consists of a sequence of consecutive reversible reactions where triglycerides are converted to diglycerides, monoglycerides, and finally, to glycerol [8,9].

Transesterification is a very slow reaction which can be accelerated by the presence of a catalyst, such as a strong acid or base [1,6]. Base catalysis is much faster and has lower corrosion than acid catalysis [6,10-11], and so, is most used

commercially [6]. Suitable alcohols used in this reaction include methanol, ethanol, propanol, butanol, and amyl alcohol. The most common is methanol, due to its low cost and its physical and chemical advantages [6]. Alkaline catalysts used for transesterification include sodium and potassium hydroxides, carbonates, and alkoxides [6,9]. The usual alkalis are sodium and potassium hydroxides [12-13].

Alkali-catalyzed transesterification is affected by such variables as free fatty acids (FFA) and moisture contents, methanol-to-oil ratio, catalyst concentration, reaction temperature, and reaction time [1,3,6,8,14]. Several researchers suggest that the FFA content of the oil should be less than 1% w/w [15-16], and also stress that all starting materials be substantially anhydrous [1] due to the complications from soap-formation if the oil has an FFA content of more than 1% w/w [16]. This soap would cause an increase in the viscosity of the reactants or cause the formation of gels and interfere with the reaction, thus lowering the yield of biodiesel. Moreover, the soap would interfere with the separation of the biodiesel from the glycerin layer [1,3]. The literature indicates that the presence of water in the oil has a worse effect on transesterification than does the presence of FFA [17]. As can be seen in Equation 1, the stoichiometric ratio for transesterification requires three moles of alcohol per mole of triglyceride to yield three moles of fatty acid alkyl esters and one mole of glycerol. However, since this is a reaction at equilibrium, most of researchers have found that additional alcohol was required to drive the reaction close to completion [1,6]. Catalyst concentration is another important variable affecting the reaction. Encinar et al. (1999) reveal that the ester conversion was 0% after 120 minutes when alkali-catalyzed transesterification of refined *Cynara cardunculus* L. oil was carried out without a catalyst (0% NaOH) [8]. Another important variable is the reaction temperature which depending on the oil being used [3,8,14,18]. The reaction can be carried out at different temperatures, even at low temperatures such as 25°C [8] and 32°C [14], but temperatures above 60°C should be avoided since that would tend to accelerate the saponification of the glycerides by the alkaline catalyst before the completion of the transesterification [18]. The results of many researchers show that alkali-catalyzed transesterification proceeds very quickly (within 10 minutes) after which the methyl ester content continually increases to the maximum values [3,8,19]. In Thailand, biodiesel is produced mainly from palm oil, and is used as a

blend B5 (5 vol.% biodiesel and 95 vol.% conventional diesel). However, in recent years *Jatropha curcas* oil, which is non-edible, has gained much attention as a feedstock for biodiesel production. The literature indicates that the fatty acid methyl ester of *Jatropha curcas* was one of the 26 fatty acid methyl esters of oils that were most suitable for biodiesel [20], which led to an increase in revenues for agriculturists as well as the creation of new jobs.

The study of biodiesel production from *Jatropha* oil has been conducted by several researchers, but with differing production processes, optimum conditions, and methyl ester yields [11,15,21-22]. These differences depend on the quality and characteristic of oil feedstock. The minimum methyl ester content of the Thai biodiesel (B 100) and EN 14214:2008 (E) specification is 96.5% w/w; therefore, it was necessary to investigate the optimum conditions for biodiesel production from *Jatropha curcas* oil which provide a methyl ester content higher than 96.5% w/w. In addition, these conditions have to be suitable for *Jatropha curcas* cultivated in Thailand. These are important reasons to make further studies of biodiesel production from crude *Jatropha curcas* L. oil by alkali-catalyzed methanolysis. The objectives of this study were: (a) to investigate the optimum reaction conditions and (b) to study the effects of the variables on the reaction.

2. Experimental

2.1 Materials

All chemicals used in the experiments, such as methanol, sodium hydroxide, and n-heptane, were of analytical reagent (AR) grade. The methyl esters, such as methyl heptadecanoate, methyl esters of palmitic, palmitoleic, stearic, oleic, and linoleic acids, were of GC reference standards.

2.2 Extraction of oil

The *Jatropha curcas* seed is small, black in color, and ellipsoid, with an average size of about 1.7-1.9 cm long and 0.8-0.9 cm thick. The weight of 100 seeds is about 69.8 g. The *Jatropha curcas* oil was mechanically extracted from the seeds using a screw press, and then allowed to settle until the impurities precipitated. The extracted oil was filtered with a suction by using nylon mesh filter cloth with mesh opening of 5 microns. 100 kg of *Jatropha curcas* seeds provided on average of 25 L of extracted oil which was clear, viscous, and yellowish in color.

2.3 Equipment

The reactions were carried out in a 250 mL three-necked flat-bottom flask equipped with a reflux condenser (to reduce the loss of methanol by evaporation), thermometer, and a stopper to add the catalyst solution. The reaction mixture, 100 mL in volume, was heated and stirred by a hot plate with a magnetic stirrer.

2.4 Experimental conditions

The alkali-catalyzed methanolysis of the crude *Jatropha curcas* oil was carried out by using sodium hydroxide as catalyst

at atmospheric pressure. The reaction mixture was well-stirred at a constant stirring speed of 600 rpm [3,19] for all test runs. The experiments were planned to investigate the optimum conditions and to study the effects of the variables on the reactions, such as methanol-to-oil molar ratio, catalyst concentration, reaction temperature, and reaction time. In the present study, the reaction was conducted at different methanol-to-oil molar ratios (4:1, 6:1, 8:1, and 10:1), catalyst concentrations (0.25, 0.5, 1.0, 1.5, and 2.0% w/w of oil), reaction temperatures (32, 40, 50, and 60°C), and reaction times (5, 10, 20, 30, and 40 minutes).

2.5 Experimental procedure

The crude *Jatropha curcas* oil, methanol, and sodium hydroxide were used in amounts established for each experiment. The oil was initially charged into the reactor, and then preheated to the desired temperature. In order to maintain the catalytic activity, the solution of sodium hydroxide in methanol was freshly prepared so that prolonged contact with the air would not diminish the effectiveness of the catalyst through interaction with moisture and carbon dioxide [1,3]. The catalyst solution was added to the preheated oil after which the reaction was timed. After the reaction, the mixture was allowed to settle under gravity for 24 hours in the separatory funnel. Two layers were formed: the upper layer consisted of methyl ester, methanol traces, residual catalyst, and other impurities, whereas the lower layer consisted of glycerin, excess methanol, catalyst, and other impurities.

After separation from the glycerin layer, the methyl ester layer was purified by washing with hot distilled water at 60°C until the washing water had a pH value similar to that of distilled water. The hot distilled water-to-crude methyl ester ratio was 1:1. To prevent the possibility of losing the methyl ester due to emulsion formation, the washing was done gently. Next, any remaining water was removed from the methyl ester layer at 70°C by vacuum rotary evaporator. Finally, the methyl ester content of the products was analyzed by using the standard method, EN 14103. The averages of the results are presented in Figs. 1-3. The optimum of each variable was determined by considering the methyl ester content, which was higher than the minimum required by Thai biodiesel (B100) and EN 14214:2008 (E) specifications (96.5% w/w), while the other variables remained constant. After an optimum was attained, this value was kept constant, while the optimum of the next variable was determined.

3. Results and Discussion

3.1 Characterization of crude *Jatropha curcas* oil

Crude *Jatropha curcas* oil was used without having undergone any further refining for use as a biodiesel feedstock. Its properties were established to ascertain suitability for biodiesel production and to determine a suitable production process for this feedstock. Characteristics, determined by standard methods, of the fatty acid composition, density, viscosity, FFA and water contents in the oil are shown in Table 1.

Table 1. Properties of crude *Jatropha curcas* oil.

Property	Method	Crude <i>Jatropha curcas</i> oil
Fatty acid composition (%) ^a	AOAC (2005), 963.22, 969.33	
(i) Palmitic acid (C16:0)		13.77
(ii) Stearic acid (C18:0)		6.77
(iii) Oleic acid (C18:1)		41.68
(iv) Linoleic acid (C18:2)		35.55
Density at 15°C, kg/m ³	ASTM D 4052-96	918.6
Free fatty acid content (as oleic acid), % w/w	AOAC (2000), 940.28	0.52
Kinematic viscosity at 40°C, mm ² /s	ASTM D 445-06	34.84
Water content, % w/w	AOAC (1990), 984.20	0.1250

^a Other fatty acids (myristic, arachidic, behenic, lignoceric, palmitoleic, erucic and linolenic acids) were presented in amounts of < 1%.

The fatty acid composition of oils is an important factor which affects the performance of biodiesel in an engine [18]; saturated methyl esters have higher cetane values and oxidation stability, but poorer low-temperature flow properties than those of unsaturated ones [23-24]. The subject, crude *Jatropha curcas* oil, consisted of a high proportion of unsaturation, comprised predominantly of oleic and linoleic acids and a lower proportion of saturation, comprised primarily of palmitic acid. In this study, the average molecular weight of the oil was calculated to be 866.13 g.

The crude *Jatropha curcas* oil was approximately 11 times more viscous than diesel fuel, and reduced to a level close to that of diesel after conversion to biodiesel. In this study, the FFA and water contents measured were 0.52% w/w and 0.1250% w/w respectively, thus demonstrating the feasibility of alkali-catalyzed methanolysis of crude *Jatropha curcas* oil.

3.2 Alkali-catalyzed methanolysis

3.2.1 Effects of methanol-to-oil molar ratio and reaction time

The reaction was carried out by varying the methanol-to-oil molar ratio and the reaction time using a catalyst concentration of 1% w/w of oil and a reaction temperature of 60°C. The results are shown in Fig. 1.

It was observed that the alkali-catalyzed methanolysis could occur quickly and the reaction time required for the completion of the reaction. The methyl ester content increased as the methanol-to-oil molar ratio increased. The methyl ester content for a methanol-to-oil molar ratio of 6:1 was 93.1% w/w after 20 minutes, whereas the same content was obtained with a methanol-to-oil molar ratio of 8:1 and 10:1 after 10 and 5 minutes, respectively. This reflected the fact that this reaction was slower with the lower methanol-to-oil molar ratio, whereas shorter time was required for a higher methanol-to-oil molar ratio to get the same methyl ester content. These results were found to concur with those obtained by other researchers [3]. The methyl ester content of 98.6% w/w at methanol-to-oil molar ratio of 6:1 was attained in 40 minutes, which is a value higher than the limits prescribed by Thai biodiesel (B100) and EN 14214:2008 (E) specifications. Although the methanol-to-oil molar ratios of 8:1 and 10:1 provided the higher methyl ester contents in less time, these two ratios should be avoided for economic reasons; thus, a methanol-to-oil molar ratio of 6:1 was selected as being the optimum.

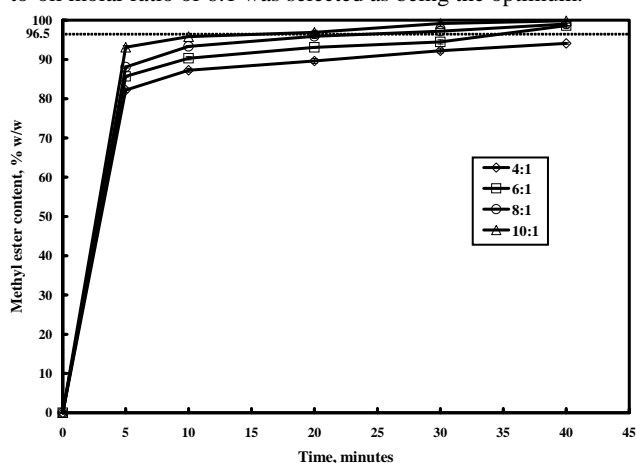


Figure 1. Effects of methanol-to-oil molar ratio and reaction time on methyl ester formation (catalyst concentration of 1% w/w of oil, reaction temperature of 60°C).

In industrial applications, the methanol consumption in the biodiesel production can be minimized by recovering the methanol from the methyl ester and glycerin layers using the distillation equipment. The recovered methanol can then be

returned to the process, resulting in a reduction of both operating costs and environmental impacts.

3.2.2 Effects of catalyst concentration and reaction time

In the present study, NaOH was chosen to catalyze the methanolysis due to its relatively low cost. The experiments were conducted with varying catalyst concentrations and reaction times using a methanol-to-oil molar ratio of 6:1 and a reaction temperature of 60°C. The effects of these variables on the methyl ester formation are shown in Fig. 2.

The results show that the catalyst concentration had a significant effect on alkali-catalyzed methanolysis. The methyl ester content increased with higher catalyst concentrations. This has also been reported by some researchers [3]. However, with the catalyst concentration increased to 2% w/w of oil, it was not possible to achieve methanolysis due to the formation of soap and a gelatinous layer in the reaction mixture. A similar result was also observed by other researchers [25]. In addition, it was found that the reaction scarcely occurred with a catalyst concentration of 0.25% w/w of oil. At a catalyst concentration of 1% w/w of oil, the methyl ester content achieved 98.6% w/w after 40 minutes, which is a figure higher than the limits prescribed by Thai biodiesel (B100) and EN 14214:2008 (E) specifications. Although, the catalyst concentration of 1.5% w/w of oil provided a higher methyl ester content than that of 1% w/w of oil for all reaction times, such a concentration should be avoided for two significant reasons: the cost of the additional NaOH, and the cost of removing the residual catalyst in the methyl ester layer. Moreover, the methyl ester layer obtained from using this catalyst concentration has to be washed with hot distilled water several times in the water washing step, so there is a possibility of losing some biodiesel product to emulsion formation. For these reasons, 1% w/w of oil was considered to be the optimum catalyst concentration.

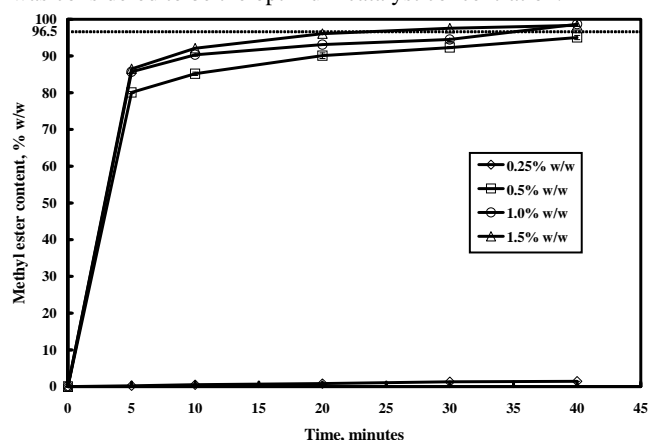


Figure 2. Effects of catalyst concentration and reaction time on methyl ester formation (methanol-to-oil molar ratio of 6:1, reaction temperature of 60°C).

3.2.3 Effects of reaction temperature and reaction time

Fig. 3 shows the methyl ester formation during *Jatropha curcas* oil methanolysis at various reaction temperatures and reaction times. The operating conditions were fixed at a methanol-to-oil molar ratio of 6:1 and a catalyst concentration of 1% w/w of oil.

The results indicate that methanolysis could occur at room temperature (32°C), but it would be incomplete, even if the reaction time were extended to 40 minutes. For the same reaction time, the methyl ester content increased with temperature. These results are in agreement with those obtained by other researchers [3,14]. The increase in the methyl ester content might have been due to the viscosity of the oil decreasing with an elevation of reaction temperature, which resulted in an increase in the solubility of the oil in the methanol, leading to an improvement in the

contact between the oil and the methanol [13,19,25]. Hence, the reaction was faster at a higher temperature. The same methyl ester content was achieved at a lower temperature as well, but the reaction time had to be increased. The methanolysis proceeded to completion in 40 minutes at 60°C, and the methyl ester content of 98.6% w/w was higher than the minimums prescribed by Thai biodiesel (B100) and EN 14214:2008 (E) specifications. Hence, 60°C was selected as the optimum reaction temperature.

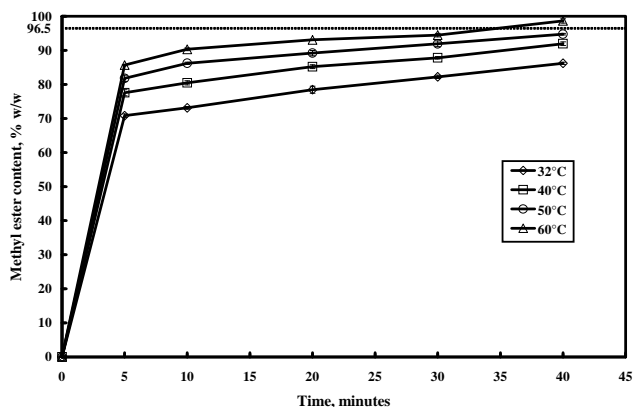


Figure 3. Effects of reaction temperature and reaction time on methyl ester formation (methanol-to-oil molar ratio of 6:1, catalyst concentration of 1% w/w of oil).

In conclusion, the optimum conditions for the alkali-catalyzed methanolysis of crude *Jatropha curcas* oil were: a methanol-to-oil molar ratio of 6:1, a catalyst concentration of 1% w/w of oil, a reaction temperature of 60°C, and a reaction time of 40 minutes. At these optimum conditions, the methyl ester content was 98.6% w/w. The GC chromatogram of *Jatropha curcas* biodiesel is shown in Fig. 4.

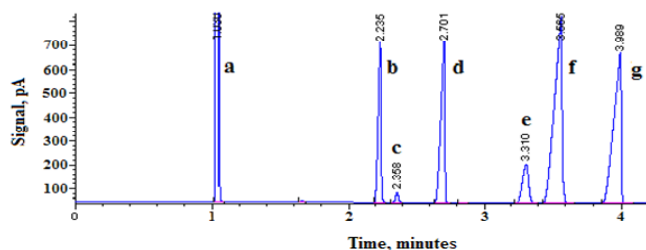


Figure 4. Chromatogram of *Jatropha curcas* biodiesel: (a) n-heptane (b) methyl palmitate (c) methyl palmitoleate (d) methyl heptadecanoate; internal standard (e) methyl stearate (f) methyl oleate (g) methyl linoleate.

3.3 Properties of *Jatropha curcas* biodiesel

The quality of biodiesel is very important for the performance and emission characteristics of a diesel engine. Thus, the *Jatropha curcas* biodiesel produced at the optimum conditions was sent to the Quality Control Division of the Petroleum Authority of Thailand Public Company Limited for quality testing using standard methods. The results are shown in Table 2.

The results indicated that the alkali-catalyzed methanolysis successfully reduced the viscosity of the crude *Jatropha curcas* oil by approximately one-eighth of its initial value. Its viscosity at 40°C (4.328 mm²/s) was slightly higher than that of diesel (3.068 mm²/s); however, it was within the limits of Thai biodiesel (B100), EN 14214:2008 (E) and ASTM D 6751-07b specifications. Pour point (PP) is one of the important parameters associated with engine performance in cold weather conditions. The *Jatropha curcas* methyl ester had the same PP as soybean methyl ester (PP = -1°C) [18], a suitable fuel for use in cold-weather. The flash point of biodiesel is higher than that of diesel (67.5°C) [1], and blending even a small percentage of biodiesel with diesel can increase the flash point of diesel [23]. The *Jatropha curcas* biodiesel produced in this study exhibited a flash point higher than 130°C; therefore, *Jatropha curcas* biodiesel or a *Jatropha curcas* biodiesel-diesel mixture would be safer than pure diesel for storage, transport, and handling. The iodine number is indicative of the degree of the unsaturation of the biodiesel [26]. The degree of unsaturation makes biodiesel susceptible to thermal and/or oxidative polymerization. This may cause problems in the fuel system due to the formation of insoluble product [1]. In this analysis, the iodine number of the *Jatropha curcas* biodiesel obtained was below the maximum specifications. In alcoholysis, monoglycerides and diglycerides are the intermediates of the reaction (monoglycerides and diglycerides of saturated fatty acids tend to crystallize easily from the biodiesel [12]), and their presence, along with triglycerides, adversely affect the fuel's properties, causing engine performance problems such as fuel filter plugging [1]. In this study, only trace amounts of monoglycerides and diglycerides were detected, giving assurance that the *Jatropha curcas* biodiesel could be used in diesel engines without the described engine performance difficulties. Moreover, these values, along with high methyl ester content and 0% w/w of triglycerides indicated the completeness of alkali-catalyzed methanolysis of the crude *Jatropha curcas* oil at the optimum conditions. Finally, considering measured content of the free glycerol, it can be seen that the free glycerol did not remain in the biodiesel product, which demonstrates the efficiency of the glycerol separation and the sufficiency of the biodiesel washing step. In addition, the other measured properties of the *Jatropha curcas* biodiesel also has met the quality requirements, except for lower oxidation stability, according to Thai biodiesel (B100) specifications and international standards EN 14214:2008 (E) and ASTM D 6751-07b.

Table 2. Properties of *Jatropha curcas* biodiesel.

Property	Method	Jatropha biodiesel	Biodiesel specification		
			Thai specification	ASTM D 6751-07b	EN 14214:2008 (E)
Viscosity at 40°C, mm ² /s	ASTM D 445-06	4.328	3.50-5.00	1.9-6.0	-
Flash point, °C	ASTM D 93-02a	> 130	Min. 120	Min. 130	-
Carbon residue, % w/w (on 10% distillation residue)	ASTM D 4530-00	0.06	Max. 0.30	Max. 0.050	-
Copper strip corrosion (3 h at 50°C)	ASTM D 130-94	1a	Max. No. 1	Max. No. 3	-
Acid number, mg KOH/g	ASTM D 664-01	0.27	Max. 0.50	Max. 0.50	-
Oxidation stability, 110°C, hours	EN 14112	3.4	Min. 6.0	Min. 3.0	Min. 6.0
FAME content, % w/w	EN 14103	98.6	Min. 96.5	-	Min. 96.5
Water content, mg/kg	EN ISO 12937	480	Max. 500	-	Max. 500
Iodine value, g iodine/100g	EN 14111	62	Max. 120	-	Max. 120
Monoglyceride content, % w/w	EN 14105	0.3727	Max. 0.80	-	Max. 0.80
Diglyceride content, % w/w	EN 14105	0.0429	Max. 0.20	-	Max. 0.20
Triglyceride content, % w/w	EN 14105	0	Max. 0.20	-	Max. 0.20
Free glycerol, % w/w	EN 14105	0	Max. 0.02	-	Max. 0.02
Total glycerol, % w/w	EN 14105	0.07	Max. 0.25	-	Max. 0.25
Density at 15°C, kg/m ³	ASTM D 4052-96	880.3	860-900	-	-
Pour point, °C	ASTM D 5950-02	-1	Report	-	-
Gross heat of combustion, MJ/kg	ASTM D 240	39.6	Report	-	-

4. Conclusion

This study demonstrates that biodiesel can be produced successfully from crude *Jatropha curcas* oil by alkali-catalyzed methanolysis. The optimum conditions were a methanol-to-oil molar ratio of 6:1, a catalyst concentration of 1% w/w of oil, and a reaction temperature and reaction time of 60°C and 40 minutes, respectively. Produced under these conditions, the methyl ester content of the *Jatropha curcas* biodiesel achieved 98.6% w/w, and all of the measured properties of *Jatropha curcas* biodiesel complied with the limits prescribed by Thai biodiesel (B100) specifications and international standards EN 14214:2008 (E) and ASTM D 6751-07b, with the exception of lower oxidation stability. The results indicate that all of the reaction variables in this study had positive effects on the reaction. However, it was not possible to achieve methyl ester formation by using a catalyst concentration of 2% w/w of oil, due to soap formation. Due to the low price of sodium hydroxide, the short reaction time, and high methyl ester content obtained, these optimum conditions can be used in large-scale production to reduce the cost of production.

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