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SCIENCE

REMOVAL OF FREE FATTY ACID IN CRUDE *JATROPHA CURCAS* OIL USING FERRIC SULPHATE FOR BIODIESEL PRODUCTION

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Abstract

Application of *Jatropha curcas* oil has been found in various fields including energy. It is well known that quality of biodiesel produced depends partly on raw material quality. In biodiesel production, high free fatty acid content in crude *Jatropha curcas* oil resulting from inappropriate storage and handling, is one of the most considerable problems. Hence, the aim of this research was to optimize the condition for removal of free fatty acid in crude *Jatropha curcas* oil by esterification using ferric sulphate as the catalyst. The reaction variables, such as methanol to crude *Jatropha curcas* oil molar ratios (6:1-15:1), ferric sulphate amount (1-2.5 wt%), temperature (45-70^{O}C), and reaction time (1-3 h), were applied. It was found that the condition having the methanol to crude *Jatropha curcas* oil molar ratio of 9:1, ferric sulphate at 1.5 wt%, the reaction temperature at 65^{O}C, and the reaction time at 2 h was successful for reducing the amount of free fatty acids in crude *Jatropha curcas* oil to below 1 wt%, and yielded 84% of esterified oil. This optimum condition was also applied for improving quality of crude rubber seed oil with high free fatty acid content. In addition, more than 90% of free fatty acid could be removed resulting in high quality of crude rubber seed oil. When this esterified oil was employed in biodiesel production, it gave high quality of biodiesel whose properties met the Thai biodiesel (B100) specifications.

Keywords: Free fatty acids, crude *Jatropha curcas* oil, ferric sulphate, esterification

Introduction

Presently, attention on biodiesel has increased as a result of the rising energy demand, leading to continuously increased prices of petroleum-derived fuel, and impact awareness of fossil fuel use on environmental pollution. Biodiesel is a promising alternative fuel that gives several environmental benefits, such as renewable, better quality of exhaust gas with low levels of greenhouse gas emissions, and rapidly biodegradable and completely non-toxic (Pramanik, 2003; Rashid and Anwar, 2008).

Biodiesel, consisting of of mono-alkyl esters of long chain fatty acids, can be produced by transesterification, where triglycerides in

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vegetable oils, animal fats, or waste cooking oils react with a short chain alcohol, mostly methanol or ethanol, in the presence of a catalyst. However, methanol is widely used because of its low cost (Jain and Sharma, 2010). Several reports indicated that homogeneous base catalysts such as sodium hydroxide or potassium hydroxide, commonly used in industries, yield higher conversion rates under mild reaction conditions compared with acid catalysts (Marchetti *et al.*, 2007; Demirbas, 2008; Ramos *et al.*, 2009; Endalew *et al.*, 2011). However, base-catalyzed transesterification requires a control of the amounts of free fatty acid and moisture in raw materials to be less than 1.0 wt% and 0.1 wt%, respectively (Meher *et al.*, 2006; Berrios *et al.*, 2007; Lu *et al.*, 2009). This is because high levels of free fatty acid in the reaction can promote saponification between free fatty acid and base catalysts, converting the acid into soap which disturbs separation of biodiesel from glycerine, thereby decreased yields of biodiesel. Canakci and Gerpen (2001) found that unrefined oil containing high amount of free fatty acid (6.66 wt%) can give a yield of biodiesel only 86 wt%.

Various edible and non-edible oils have been used to produce biodiesel depending on availability of the plants in each local area. In Thailand, palm oil has been massively produced and used as a main raw material for edible oil-based production. However, the increasing demand of palm oil might contribute to shortage of the raw material for food sector. In this regard, the use of other potential non-edible oils for biodiesel production deserves a consideration. These include the oil from *Jatropha curcas* which is also widely cultivated in Thailand for production of oil, and the extracted oil from rubber seed, which is also a by product from rubber plantation.

In recent years, numerous pieces of research have documented the potentials of non-edible *Jatropha curcas* oil for conversion to biodiesel. However, appropriate storage and handling of *Jatropha curcas* oil are required to avoid changes in its properties, particularly free fatty acid contents (Berchmans and Hirata, 2008). As mentioned earlier, high amounts of free fatty acid and moisture have great effects on base-catalyzed transesterification in biodiesel production. Therefore, to use *Jatropha curcas* oil with high free fatty acid (> 1.0 wt%) as a raw material for biodiesel, the free fatty acid can be removed by esterification using an acid catalyst. Sulfuric acid, compared to other acid catalysts, is the most commonly used catalyst in the reduction of free fatty acid including *Jatropha curcas* oil (Tiwari *et al.*, 2007; Berchmans and Hirata, 2008) because of its high conversion rate and low cost. However, effluent disposal, loss of catalyst from the reaction, and high equipment cost due to corrosiveness of the acid are apparently problematic issues of using this acid (Gan *et al.*, 2010). Therefore, heterogeneous acid catalysts such as resins, zeolites, and ferric sulphate seem to be the best choice. In our opinion, ferric sulphate is an interesting type of catalyst for esterification of free fatty acid, taking into account its high activity (Gan *et al.*, 2010; Patil *et al.*, 2010; Zhang *et al.*, 2010), and a simple catalyst removal method. Besides, cost of the chemical is considerably low when compared to that of other catalysts. However, the use of ferric sulphate for catalyzing *Jatropha curcas* oil containing high free fatty acid contents has rarely been reported in open literature.

In this work, ferric sulphate was introduced to catalyze the esterification of free fatty acid in crude *Jatropha curcas* oil with methanol. The effects of methanol to oil molar ratio, catalyst amount, reaction time, and reaction temperature on the esterification reaction were investigated to optimize the condition for reducing free fatty acid contents to below 1.0 wt%. In addition, the above condition will also be further applied to crude rubber seed oil in order to reduce the free fatty acid contents. By using this method reported herein, the quality of crude oils can be significantly improved, making it possible to produce high quality biodiesel via base-catalyzed transesterification.

Materials and Methods

Materials

The *Jatropha curcas* seeds were purchased from a local company in Pathum Thani Province, Thailand in the beginning of 2008. The crude oil was extracted using a screw press after the seeds were kept for a long period of time. After allowing impurities to precipitate, the extracted oil was filtrated using filter paper. The crude rubber seed oil was obtained from local. The properties of the crude *Jatropha curcas* oil and crude rubber seed oil are shown in Tables 1 and 2, respectively. The crude oils were stored in a sealed container at room temperature to avoid an increase in moisture and free fatty acid contents.

Ferric sulphate, potassium hydroxide, and sodium hydroxide were purchased from Ajax Finechem and methanol was purchased from Merck. All chemicals and reagents were of analytical grade.

Methods

Esterification of Free Fatty Acid

The esterification was conducted at different methanol to oil molar ratios (6:1, 9:1, 12:1, and 15:1), catalyst amounts (1, 1.5, 2, and 2.5 wt% based on the weight of crude *Jatropha curcas* oil), reaction temperatures (45, 55, 65, and 70°C), and reaction times (1, 2, and 3 h). Noteworthy, ferric sulphate was dried in an oven at 105°C for 2 h prior to use.

In a 250 ml three-necked flat-bottom flask equipped with a reflux condenser, thermometer, and a stopper, a mixture of required amount of methanol and dried ferric sulphate was heated and stirred at a constant rate of 700 rpm (Gan *et al.*, 2010). After reaching the desired temperature (65±2°C), 40 g of crude *Jatropha curcas* oil was added and the reaction timing was started immediately. The reaction was constantly stirred and heated until the required reaction time was obtained. The mixture was settled overnight in a separatory funnel to allow complete separation between esterified oil, methanol, and ferric sulphate (Zhang *et al.*, 2010). On the next day, the esterified oil in the lower layer was filtered with a filter paper (No. 2, Whatman) to separate the catalyst, yielding clear yellow oil. Then, the oil was heated at 110±2°C to remove the small remaining amount of the methanol and water. The determination of free fatty acid was carried out immediately after the esterification process using AOAC Official Method 940.28. The conversion of free fatty acid was calculated from the equation below

$$\%\ \text{FFA conversion} = \frac{\text{A - B}}{\text{A}} \times 100$$

where A is the initial concentration of free fatty acid (wt%) in the crude *Jatropha curcas* oil and B is the final concentration of free fatty acid in the esterified oil.

The experiments were performed in duplicate and the average values were presented.

Production of Biodiesels

Jatropha curcas biodiesel was prepared by using methanol to oil molar ratio of 6:1 (Koh and Ghazi, 2011), and 1 wt% of KOH (based on the weight of the esterified oil). While the rubber seed biodiesel was prepared by using the molar ratio of 9:1 (Ramadhas *et al.*, 2005), and 1 wt% of NaOH (based on the weight of the esterified oil) Both reaction systems were carried out for 1 h.

The process started by pre-heating 165 g of crude esterified oil in a 500 ml two necked flat-bottom flask equipped with a reflux condenser to the desired temperature before adding freshly prepared methoxide solution. This solution was prepared in another flask by dissolving sodium hydroxide or potassium hydroxide into methanol until the clear solution was observed. Measurement of the time started at this point. The mixture was vigorously agitated at a constant rate (600 rpm). When the reaction completed, the mixture was allowed to settle under gravity for 24 h in a separatory funnel in which most of methyl ester was in the upper layer and glycerine was mainly in the lower layer. All impurities remaining in the methyl ester mixture were washed out several times with hot distilled water at 60°C until the washing water was neutralized. The methanol and water were further removed from the methyl ester (biodiesel) by vacuum rotary evaporator at 70°C. After cooling down to room temperature, the biodiesel was filtered with a filter paper (No. 2, Whatman) prior to storing it in a dried and tightly closed container. Determination of biodiesel properties were carried out using standard methods such as methyl ester, kinematic viscosity, sulphate ash, water, monoglyceride, diglyceride, triglyceride, and total glycerine.

Results and Discussion

Characterization of Crude *Jatropha curcas* Oil

Table 1 illustrates the chemical and physical properties of the extracted crude *Jatropha curcas* oil investigated by standard methods. The formation of free fatty acid in the *Jatropha curcas* seeds was mainly caused by the hydrolysis of triglyceride with the presence of water. The crude *Jatropha curcas* oil composed of 7 fatty acids containing mainly 16–18 carbon atoms, estimating about 20.2 and 73.4 wt% of saturated and unsaturated fatty acids, respectively, which is in consistent with the previous reports on crude *Jatropha curcas* oil (Berchmans and Hirata, 2008; Jain and Sharma, 2010). Fatty acid types and contents in crude *Jatropha curcas* oil may vary depending on strains and cultivation methods; however, the amounts of unsaturated fatty acids are always higher than the amounts of saturated fatty acids, suggesting that the oil is in an oleic linoleic acid vegetable oil group.

Esterification of Free Fatty Acid

Effect of Methanol to Oil Molar Ratio

To observe the effects of molar ratios on reduction of free fatty acid contents, the experiments were carried out using various methanol to oil molar ratios (6:1-15:1). The results indicated that the molar ratio of methanol to oil has an influence on the conversion of free fatty acid. As seen in Figure 1, higher amounts of methanol promoted free fatty acid conversion. This result is similar to those found by Wang *et al.* (2007) and Gan *et al.* (2010) in the esterification of waste cooking oil with high free fatty acid contents. One explanation is that esterification is reversible. Therefore, an increase in methanol content shifts the equilibrium reaction toward the more product formation. The increased methanol volume also reduced viscosity of the mixture, increasing the mass transfer of the reaction. From the above results, methanol to oil molar ratio of 9:1 is considered to be the optimum ratio, taking into account the reduction of free fatty acid content from 8.6 wt% to below 1 wt% and the relatively small amount of methanol employed, which contributes to a low product cost.

Table 1. Properties of crude *Jatropha curcas* oil

Property	Method	Crude *Jatropha curcas* oil
Total acid number, mg KOH/g	ASTM D644-01 (2001)	17.29
Density at 15°C, kg/m^3	ASTM D4052-96 (1996)	917.70
Water content, wt%	AOAC (1990), 984.20	0.09
Kinematic viscosity at 40°C, mm^2/s	ASTM D445-06 (2006)	35.13
Free fatty acid content (as oleic acid), wt%	AOAC (2000), 940.28	8.60
Fatty acid composition, (%)	AOAC (2005), 963.22, 969.33	
(i) Palmitic acid (C16:0)		14.47
(ii) Palmitoleic acid(C16:1)		0.80
(iii) Stearic acid (C18:0)		5.61
(iv) Oleic acid (C18:1n9c)		37.07
(v) Linoleic acid (C18:2n6c)		35.40
(vi) Linolenic acid (C18:3n3)		0.18
(vii) Arachidic acid (C20:0)		0.14

Moreover, the conversion of free fatty acid was largely increased when the molar ratio of methanol to oil was altered from 6:1 to 9:1, and little conversion increase was observed when changed to 12:1 molar ratio. This implies that the appropriate amount of methanol for free fatty acids conversion depended on the starting contents. For the oil containing high free fatty acid contents, high amount of methanol was required for the reaction. In case of the feedstock with less than 5 wt% of free fatty acid, the methanol to oil molar ratio at 6:1 was enough to reduce free fatty acid content into 1 wt%.

Effect of Catalyst Amount

Compared to the reaction with the presence of catalyst, little conversion of free fatty acid was observed in the reaction without catalyst. The esterified oil appeared in a yellow color similar to the crude oil and only small amount of free fatty acid was reduced (from 8.6 wt% to 7.2 wt%). In the absence of catalyst, reduction of free fatty acids was autocatalyzed by the un-dissociated free fatty acid undergoing protolysis, producing activated carboxylic acids which consequently induced esterification (Gan *et al.*, 2010).

The catalytic amounts used in the study were varied between 1 and 2.5 wt%, in which 0.5 wt% of the catalytic amount was increased in every experiment, while the other parameters were kept constant at the 9:1 methanol to oil molar ratio, 65°C and 2 h. Figure 2 demonstrates that degree of esterification increased with increasing amount of the catalyst. At 2.5 wt% of the catalyst amount, free fatty acid conversion reached 93.6%. It was also found that the remaining free fatty acid content was only 0.7 wt% when 1.5 wt% of the ferric sulphate was used. In comparison, work by Wang *et al.* (2007) and Zhang *et al.* (2010) reported that the optimum catalyst content is 2 wt%. Gan *et al.* (2010) also found that an excessive amount of catalyst tends to decrease the free fatty acid conversion.

Effect of Temperature

The other major parameter on esterification is temperature. The results showed that at high temperature, viscosity of the crude oil was decreased, resulting in enhancement of the contact between the oil and methanol phases which supported even distribution of catalyst in the mixture. The clear evidence of the influence of temperature on the reaction is seen when the

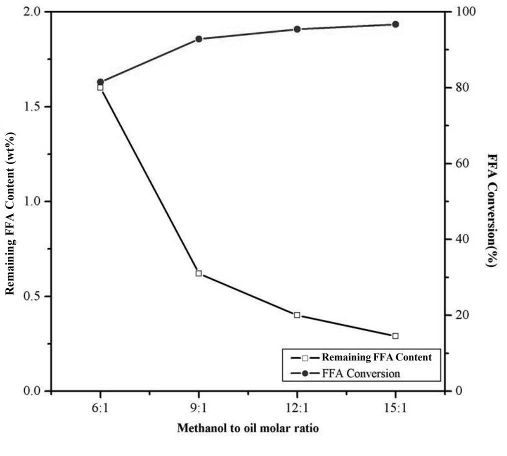

Figure.1. **Effect of methanol to oil molar ratio on remaining free fatty acid content and free fatty acid conversion (using the catalyst amount at 2 wt%, the reaction temperature 65°C, the reaction time 2 h, and the stirring speed at 700 rpm)**

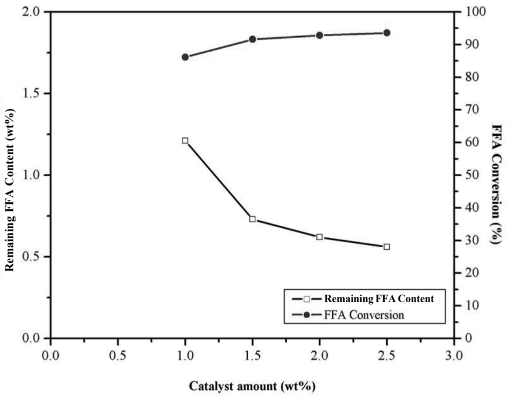

Figure 2. **Effect of catalyst amount on remaining free fatty acid content and free fatty acid conversion (using the methanol to oil molar ratio of 9:1, the reaction temperature at 65°C, the reaction time at 2 h, and the stirring speed at 700 rpm)**

temperature was increased from 45°C to 65°C, improvement of free fatty acid esterification was significantly observed, yielding maximum reduction of free fatty acid. The above trend is similar to that observed by Gan *et al* (2010) in a study of used cooking oil. However, at higher temperature (70°C) which is above the boiling point of methanol, reduction rate of free fatty acid was slightly lower than at the reaction at 65°C. This is due to methanol evaporation from the mixture (Figure 3).

Effect of Reaction Time

Figure 4 shows that conversion of free fatty acid rapidly increased within the first 1 h of the reaction. At the reaction time of 2 h, the amount of free fatty acid was reduced to less than 1 wt%. The above effect is attributed to the fact that a relatively high amount of methanol was used for the esterification. Consequently, the water which is generated as by product from the above reaction is low. However, a small increase in conversion was observed at the longer reaction time (more than 2 h). This can be ascribed to the increase of water content with time. As a result, the equilibrium might shift to the reverse direction, i.e. the hydrolysis of methyl ester. Besides, by increasing the reaction time, more deactivated catalyst can also be possible (Gan *et al.*, 2010).

From the optimization of various parameters on esterification of free fatty acid in crude *Jatropha curcas* oil, the appropriate condition that reduced free fatty acid contents in the oil into 0.7 wt% was the methanol to oil molar ratio at 9 : 1, 1.5 wt% of ferric sulphate catalyst, the reaction temperature of 65°C, and 2 h reaction time, giving a yield of esterified oil at 84.0%. After esterification, the acidic value of the oil reduced from 17.29 mg KOH/g into 1.45 mg KOH/g. One advantage of using heterogeneous catalysis is that water wash is not required in the process, thereby no problem of water remaining in the product. Analysis of water content in the oil found only 0.08 wt%. Therefore, the purity of the crude *Jatropha curcas* oil after esterification process was enough for using it as a raw material in biodiesel preparation with a base catalyst.

Rubber seed oil is another essentially economic plant in Thailand. Compared with crude *Jatropha curcas* oil, properties of crude rubber seed oil are similar to those of Jatropha curcas oil (Table 2). For this reason, the optimal condition obtained from esteri-fication of crude *Jatropha curcas* oil was applied for crude rubber seed oil (with free fatty acid 6.5 wt%). After esterification process, free fatty acid of

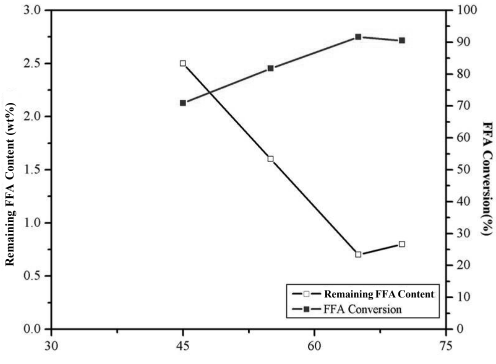

Figure 3. Effect of reaction temperature on remaining free fatty acid content and free fatty acid conversion (using the methanol to oil molar ratio at 9:1, the catalyst amount at 2 wt%, the reaction time at 2 h, and the stirring speed at 700 rpm)

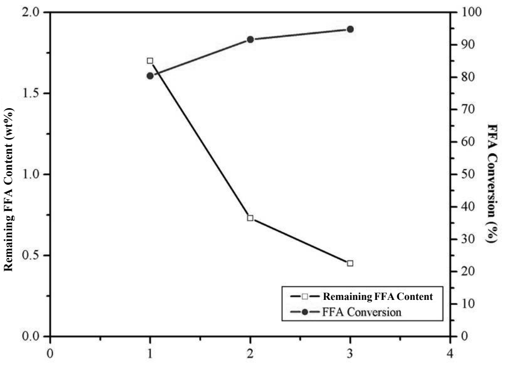

Figure 4. Effect of reaction time on remaining free fatty acid conversion (using the methanol to oil molar ratio at 9:1, the catalyst amount of 2 wt% based on the oil weight, the reaction temperature at 65°C, and the stirring speed at 700 rpm)

crude rubber seed oil was successfully decreased into only 0.5 wt%. Interestingly, esterification of free fatty acid in these two crude oils using ferric sulphate catalyst does not depend on the oil type. In contrast,, the reaction is associated with the initial amount of free fatty acid.

Biodiesel Production

After free fatty acid reduction, the crude *Jatropha curcas* and rubber seed oils were used as feedstock for biodiesel production by base-catalyzed transesterfication. Subsequently, the properties of the biodiesel produced were investigated using various standard methods (Table 3). Considering the contents of monoglyceride, diglyceride, triglyceride, the values indicated the completion of transesterification. The biodiesels had satisfying properties to Thai biodiesel (B100) specifications where the purity of biodiesels was above 96.5 wt% fatty acid methyl esters.

Conclusions

In this study, ferric sulphate showed high catalytic activity of esterification of free fatty acid in crude *Jatropha curcas* oil. After the esterification step, the free fatty acid in the crude *Jatropha curcas* oil (8.6 wt%) can be reduced to be less than 1 wt% under the condition of methanol to oil molar ratio at 9:1, the catalyst amount of 1.5 wt% of ferric sulphate, the reaction temperature at 65°C, and the reaction time of 2 h. The conversion of free fatty acid under the optimal condition was greater than 90%. Furthermore, this condition could be used to effectively reduce free fatty acid in crude rubber seed oil, from 6.5 wt% into lower than

Table 2. Properties of crude rubber seed oil

Property	Method	Crude rubber seed oil
Density at 15°C, kg/m^3	ASTM D4052-96 (1996)	923.4
Water content, wt%	AOAC (1990), 984.20	0.15
Kinematic viscosity at 40°C, mm^2/s	ASTM D445-06 (2006)	37.16
Free fatty acid content (as Linoleic acid), wt%	AOAC (2000), 940.28	6.50
Fatty acid composition, (%)[a]	AOAC (2005), 963.22, 969.33	
(i) Palmitic acid (C16:0)		10.63
(ii) Stearic acid (C18:0)		7.77
(iii) Oleic acid (C18:1n9c)		23.09
(iv) Linoleic acid (C18:2n6c)		41.77
(v) Linolenic acid (C18:3n3)		15.36

[a] Other fatty acids were presented in amounts of < 1%.

Table 3. Biodiesel properties

Property	Method	Limit	*Jatropha curcas* biodiesel	Rubber seed biodiesel
Kinematic viscosity at 40°C, mm^2/s	ASTM D445-06 (2006)	3.5-5.0	4.504	4.886
Sulphate ash, wt%	ASTM D874-00 (2000)	Max 0.02	<0.005	<0.005
Water content, mg/kg	ISO 12937 (2000)	Report	400	400
Methyl ester, wt%	EN 14103 (2003)	Min 96.5	98.6	99.5
Monoglyceride, wt%	EN 14105 (2003)	Max 0.80	0.48	0.52
Diglyceride, wt%	EN 14105 (2003)	Max 0.20	0.10	0.01
Triglyceride, wt%	EN 14105 (2003)	Max 0.20	0.00	0.00
Total glycerine, wt%	EN 14105 (2003)	Max 0.25	0.13	0.13

1 wt%. Compared with homogenous catalyst, utilization of heterogeneous catalyst for esterification can omit the water purification step, subsequently the water content in the esterified oil is not increased. Conclusively, transesterification of reduced-free fatty acid raw materials can be conducted under mild condition and consumed less alcohol to produce high purity biodiesel, which can reduce production cost.

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