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Synthesis of biodiesel by two-step transesterification from crude *jatropha curcus* L.oil using ultrasonic irradiation assisted

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Abstract

Usually, the crude *Jatropha curcas* L. oil has very high free fatty acid (FFA) which obstructs the transesterification reaction. As a result it provides low yield of biodiesel production. The objective of this research is to investigate the optimum conditions for biodiesel production from crude *Jatropha curcas* L.oil with methanol by ultrasonic irradiation (at 40 kHz frequency and 400 Watt) assisted, using two-step catalyzed method. In the first step, the reaction was carried out in the presence of sulfuric acid as an acid catalyst. The product was then further transesterified with potassium hydroxide in the second step. The effects of different operating parameters such as molar ratio of reactants, catalyst quantity, operating temperature, and time of reactants have been studied with the aim of process optimization. In the first step, the optimum conditions for the acid catalyzed esterification process have been obtained as ratio of methanol to oil as 15% w/w, catalyst concentration of 3% w/w, temperature as 30ºC and reaction time as 20 minutes. For the second step, the optimum conditions for the base catalyzed transesterification process have been obtained as ratio of methanol to oil as 15% w/w, catalyst concentration of 1% w/w, temperature as 30ºC, and reaction time as 40 minutes. It has been observed that the mass transfer and kinetic rate enhancements were due to the increase in interfacial area and activity of the microscopic and macroscopic bubbles formed. The efficacy of using ultrasonic irradiation has been compared with conventional mechanical stirring method. The results indicated that the reaction time is much lower in the case of ultrasonic irradiation and high conversion as compared to stirring method.

Keywords: Biodiesel, Two-step transesterification, Crude *jatropha curcus* L. oil, Ultrasonic irradiation

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1. Introduction

Diesel fuel has been widely use in industry and in automobiles for over a century. But as petroleum price continue to rise, diesel supply becomes scarce and concern for environment grows, scientists have investigated considerable effort in searching for renewable substitutes of diesel fuel. An alternative fuel should be easily available, environment friendly and techno-economically competitive. The esters of vegetable oils or animal fats appear to be most promising alternative. Today, methyl or ethyl esters of fatty acid are used as substitutes to diesel fuel under the name of biodiesel. Biodiesel fuel have many advantages over diesel fuels: produce less smoke and particle, have higher cetane number, produce low carbon monoxide (CO) and hydrocarbon (HC) emission, are renewable, biodegradable and non-toxic (Akarwal and Phaveatare, 2004; Raheman, 2007).

Generally, biodiesel is produced by means of transesterification process. The transesterification is the reaction of triglyceride with an alcohol to from esters and a byproduct, glycerol (Encinar et al., 2007). At present, biodiesel has to compete economically with diesel fuels in order to survive in the market. The major problem is the costs of the raw material. Biodiesel obtained from neat vegetable oil is costly compared to the diesel fuels. It is reported approximately 60-95% of total cost of biodiesel production arises from the cost of raw materials (Cetinkaya and Karaosmanoglu, 2004; Zhang et al., 2003). There has been a renewed focus on the way to minimize the cost of biodiesel production. The problem can be approached in two differences way: by finding cheap source of raw materials or by intensifying the synthesis process by using novel reactor (Vishwanath et al., 2008). In the case of finding cheap source of raw materials, one way of reducing the biodiesel production cost is to use the less expensive feedstock containing fatty acid such as non edible oils, animal fats and waste cooking oil (Veljikovic et al, 2006; Murugesan et al., 2009; Leung and Guo, 2006). Therefore, crude *jatropha curcus* L.oil is promising alternative to neat vegetable oil due to its reduced raw material cost.

Currently, two-step transesterification reaction of biodiesel produce from crude *jatropha curcus* L.oil by the conventional technical based on use of mechanical stirring. The researchers have reported different parameters for two-step transesterification process. In this the acid catalyzed esterification process used very longer reaction time approximately 2-5 hours to reduce free fatty acid content (Lu et al., 2009). The base catalyzed transesterification process required 1-3 hours for making ester and the conversions of triglyceride were observed to be in the range of 50-90% (Chitra et al., 2009; Berchmans and Hirata, 2009). Because of the lower rates of synthesis have been conventional stirring method attributed to mass transfer limitation due to heterogeneous conditions existing during the reaction (Vishwanath et al., 2008).

The transesterification reaction involves two immiscible phase. The less-dense phase has the catalyst dissolved in the alcohol, whereas the other contains the oil or fat. The reaction between these species can occur in the interfacial region between the liquids, as catalysts are essentially insoluble in the oil phase. Vigorous mixing is required to

increase area of contract between the two phases (Colucci et al., 2005). It is know that ultrasonic irradiation is a useful tool for emulsification of immiscible liquids. In this work the transesterification of crude *jatropha curcus* L.oil was carried out applying ultrasonic wave. Ultrasonic irradiation causes cavitation of bubbles near the phase boundary between the alcohol and oil phase. The collapse of the cavitation bubbles disrupts the phase boundary and causes emulsification, by ultrasonic jest that impinge one liquid to another. The cavitation may also lead to the localized increase in temperature at the phase boundary enhancing the transesterification reaction (Ji et al., 2006; Hanh et al., 2008). Neither agitation nor heating are required to produce biodiesel by ultrasonic application because of the formation of micro jets and localized temperature increase.

The objective of this study was undertaken to optimize the experimental conditions for maximum biodiesel production from acid-base catalyzed transesterification process using an ultrasonic irradiation mixing technique. The main gold was to develop a two-step transesterification process for the production of biodiesel from crude *jatropha curcus* L.oil. It with high content of free fatty acids (FFA.) cannot be directly used in base catalyzed transesterification process because FFA reacts with base catalyst to from soap, resulting in serious emulsification and separation problems. Therefore oils used in base catalyzed transesterification reactions should be contain no more than 3% of free fatty acid. In special attention was paid to optimize the first step of the process for reducing the free fatty acid content of crude *jatropha curcus* L.oil to below 3%. The second attention was focus to optimize the reaction condition for weigh of catalyst to oil, weigh ratio of methanol to oil, reaction temperature and reaction time.

2. Experimental procedures

2.1 Reagents and Materials

The *Jatropha curcus* L. seed in this study was collected from the local area, Ubonratchathani Province, Northeast Thailand. The *Jatropha curcus* L. seed was obtained by grinding the seeds and extracted by hydraulic press machine. The oil was filtrated to remove solid impurities. The fatty acid composition (%) of crude *jatropha curcus* L.oil was showed in table 1. This oil contained 12.5% of free fatty acid corresponding to an acid value of 25 mg KOH/g, was determined by acid value titration. The chemicals used in experimental including sulfuric acid (H_2SO_4), potassium hydroxide (KOH), and methanol were purchased from Merck Chemical Company (Germany). The purities of H_2SO_4, KOH, and methanol were greater than 98%, 95%, and 99.8%, respectively.

Table 1 *Fatty acid composition of crud jatropha curcus L.oil (Berchmans and Hirata, 2009).*

Fatty acid	Formula	Structure[a]	wt%
Myristic	$C_{14}H_{28}O_2$	14:0	0-0.1
Palmitic	$C_{16}H_{32}O_2$	16:0	14.1-15.3
Palmitoleic	$C_{16}H_{30}O_2$	16:1	0-1.3
Stearic	$C_{18}H_{36}O_2$	18:0	3.7-9.8
Oleic	$C_{18}H_{34}O_2$	18:1	34.3-45.8

[a] xx:y indicates xx carbons in the fatty acid chain with y double bonds.

2.2 Equipment Setup

The ultrasonic reactions were performed using KCME-KORN model AK-NANO/BIO systems 400 UL, with total power of 400 watt. A schematic diagram of the ultrasonic reactor is shown in Figure 1.

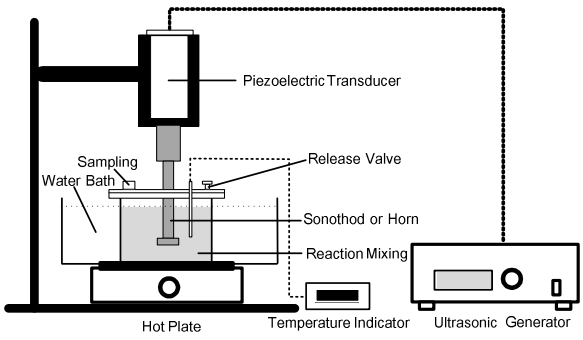

Figure 1 *Schematic diagram of experiment setup used for the ultrasonic irradiation assisted biodiesel production from crude jatropha curcus L. oil*

The ultrasonic reactor consists of a generator, which convert a standard 60 Hz line voltage to a high- frequency electrical power of 40 KHz. This high frequency electrical energy is fed to the piezoelectric transducer, where it is converted to mechanical vibrations of the same frequency. The tip of the horn was submerged 3 cm in the reactor mixture contained in the glass reactor. The temperature of the reaction mixture was controlled by water bath.

2.3 Two-step biodiesel production process and product analysis

The main key factors during transesterification reaction are percentage of the presence of free fatty acid (FFA.) and moisture, because these two favor the side reactions like saponification and lower the yield of ester (biodiesel). Thus, before proceeding transesterification reaction the characterization of raw material must be performed to take the judgment for transesterification reaction, either one or two steps. If free fatty acid presence goes beyond the limit, two-step process, first neutralization of free fatty acid with acid catalyst (esterification) and then transesterification with base catalyst should be applied to get the higher yield of ester. Meher et al. recommended a single-stage transesterification reaction with base catalyst when free fatty acids content are less than 3% (Meher et al., 2006). It was observed that percentages of free fatty acid in the crude *jatropha curcus* L.oil are 12.5%. Therefore, two-step transesterification process, acid catalyzed esterification process and followed by base catalyzed transesterification process, was selected for converting crude *jatropha curcus* L. oil to ester. In all experiments, for a successful reaction the oils must be heated above 100°C for 30 minutes to remove water content and other impurities.

2.3.1 Acid-catalyzed esterification.

On this first step acid catalyzed esterification, the objective of the first step is reduced free fatty acid of the crude *jatropha curcus* L. oil to about 3%. The crude *jatropha curcus* L.oil (150 g.) was poured into reactor. The reaction mixtures consist of crude *jatropha curcus* L. oil, methanol and sulfuric acid (H_2SO_4). The solution of concentration sulfuric acid catalyst were 1%, 2%, 3%, 3.5% and 4% (w/w) of the weight of oil in methanol and then added to the reactor. The ratios of methanol to oil were 10, 15, 20, 25 and 30% (w/w) of the weight of oil. The reaction temperature and reaction time for all process were tested at 30 (room temperature), 40, 50, 65°C and 10, 20, 30, 40, 50 minutes, respectively. After the

reaction, the mixture was allowed to settle for 8 hours or overnight. Then, gumming, methanol, and water fraction at the bottom layer was removing. The acid values and FFA of the product was determined by titration technique and the product was used for the base catalyzed transesterification reaction.

2.3.2 Base-catalyzed transesterification.

In the second step, optimize conditions for KOH concentration, methanol to oil ratio, reaction temperature, and reaction time were investigated. Firstly, the oil products that have been pretreated from the first step were poured into the reactor. KOH pellets were dissolved in methanol before being poured in the reactor containing about 150 g crude *jatropha curcus* L.oil heated up to a desired temperature, which was 30, 40 and 50°C. The reaction was kept at a desired temperature for 5, 15, 25, 40, 50 and 60 minutes. The ratios of methanol to oil varied from 10% w/w to 20% w/w of the weight of oil, while the amount of KOH catalyst was ranging from 0.5 to 2.5% (w/w) of the weight of oil. After the reaction, the mixing was placed in a bath of ice water for stop reaction and it was allowed to settle for 8 hours or overnight. The mixing of KOH and methanol settles at the bottom of the funnel, where as small amount of catalyst, methanol and glycerol are in the upper biodiesel layer. The upper layer is collected for further purification by washing. After final washing and dying, the product yield of biodiesel was determined by equation 1. In this study, product yield is defined as the weight percentage of the final product relative to the weigh of oil at the start (Eq.1).

$$\text{Production yield} = \frac{\text{Weight of product}}{\text{Weight of raw oil}} \quad (1)$$

3. Results and discussion

3.1 Acid-catalyzed esterification

As discussed earlier, in order to avoid the problem of saponification, two-step process was used for the preparation of biodiesel from crude *jatropha curcus* L. oil. In the first step, esterification of FFA in crude *jatropha curcus* L. oil was done using sulfuric acid as a catalyst.

3.1.1 Effect of sulfuric acid concentration

The concentration of the acid catalyst was the first parameter studies. The effect of sulfuric acid (H_2SO_4) concentration on the acid catalyzed esterification of the crude *jatropha curcus* L. oil was investigated with its concentration varying from 1 to 4% w/w (base on the weight of raw oil). The operation conditions during the whole reaction process were fixed as: reaction temperature of 30°C (room temperature), reaction time of 20 minutes, and ratio of methanol to oil at 20 %w/w. Experiment results (Figure 2) show that the change in acid value and free fatty acid (FFA.) content with catalyst concentration. The FFA was found to be decreased from 12.5 to the minimum value of 2.2% in 20 minutes. The FFA reduced sharply to 3% at 3% w/w of sulfuric acid to oil ratio and then decreased gradually to 2.2% at 4% w/w of sulfuric acid to oil ratio. Increasing the sulfuric acid amount was significant effect to acid value or FFA concentration reduction. There was an optimum quantity of sulfuric acid required to complete the acid catalyzed process of all FFA in crude *jatropha curcus* L. oil. So

the optimum sulfuric acid concentration was selected 3% w/w at FFA concentration be equal to 3%, acid value of 6 mg KOH/g because recommended a single stage transesterification reaction (direct transesterification) with base catalyst when FFAs content are less than 3%.

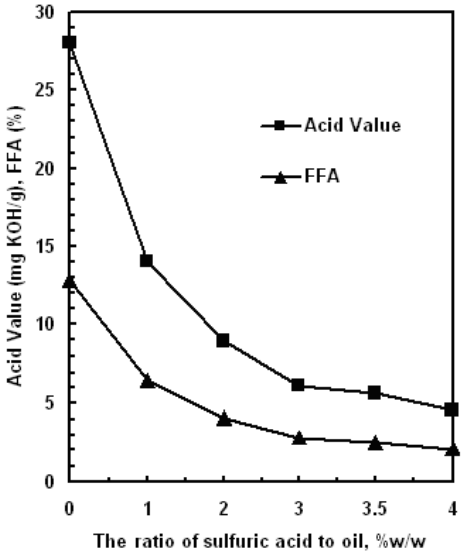

Figure 2 *Influence of sulfuric acid quantity on acid value and FFA of crude jatropha curcus oil (reaction temperature=30°C; methanol to oil ratio 20%w/w; and reaction time=20 minutes)*

3.1.2 Effect of Methanol quantity

The effect of quantity methanol on acid values and FFA of the mixtures after 20 minutes reaction is shown in figure 3. The figure indicates that the acid values and FFA concentration was influence by the quantity of methanol. The FFA was decreased from 12.5 to 1.5% as methanol to oil ratio increased from 10 to 30% w/w. The FFA reduced sharply to 3% at 10% w/w of methanol to oil ratio and then decreased gradually to 1.5% at 30% w/w of methanol to oil ratio. Moreover, it was observer that a high methanol to oil ratio beyond 20% w/w, the excessively added methanol had no significant effect on the FFA and acid value. So the optimum methanol to oil ratio was selected 15% w/w at FFA concentration less than 3%, acid value of 6 mg KOH/g.

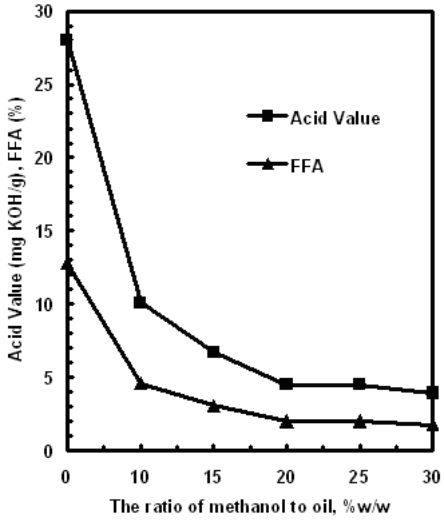

Figure 3 *Influence of methanol quantity on acid value and FFA of crude jatropha curcus L. oil (reaction temperature=30°C; sulfuric acid to oil ratio 3%w/w; and reaction time=20 minutes)*

3.1.3 Effect of reaction temperature

As it has been indicated, temperature was varied between 30 and 65°C. In all experiments, a methanol/oil ratio of 20% w/w, sulfuric concentration of 3% w/w, and reaction time of 20 minutes were used. Figure 4 shows that the Influence of reaction temperature on acid value and FFA of crude *jatropha curcus* L. oil. From the figure, it can be seen that the FFA reduced sharply to 2% at 30°C and then decreased gradually to 1.8% at 40°C. Increasing the reaction temperature was no significant effect to acid value or FFA concentration reduction. In summary, considering that the obtained results at 30 and 40°C were very similar,

30°C was selected for used crude *jatropha curcus* L. oil acid catalyst transesterification.

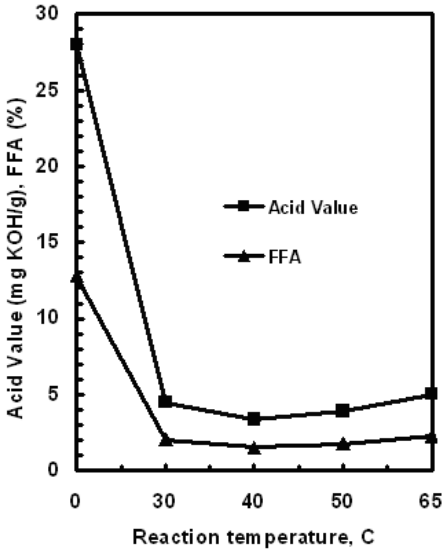

Figure 4 *Influence of reaction temperature on acid value and FFA of crude jatropha curcus L. oil (methanol to oil ratio 20%w/w; sulfuric acid to oil ratio 3%w/w; and reaction time=20 minutes)*

3.1.4 Effect of reaction time

In order to optimize the reaction time, the difference reaction times selected for this study were 10, 20, 30, 40 and 50 minutes. The constant methanol amount of 15%, constant sulfuric acid concentration of 3% and constant temperature of 30°C were maintained with reaction time variations.

Figure 5 shows the effect of reaction time on acid values and FFA. As it can be observed, the FFA reduced sharply to 3.1% at 10 minutes of reaction time and then decreased gradually to 1.1% at 50 minutes of reaction time. Increasing the reaction time was significant effect to acid value or FFA concentration reduction. Then, the optimum reaction time condition was selected 20 minutes of reaction time at FFA concentration less than 2.8%, acid value of 5.6 mg KOH/g.

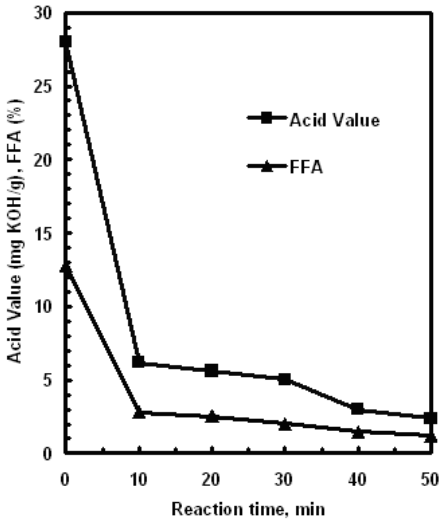

Figure 5 *Influence of reaction time on acid value and FFA of crude jatropha curcus L. oil (reaction temperature=30°C; sulfuric acid to oil ratio 3%w/w; and methanol to oil ratio 15%w/w)*

3.2 Base-catalyzed transesterification

The pretreatment oil produced by the above first step was further transesterified to biodiesel catalyzed by potassium hydroxide.

3.2.1 Effect of reaction temperature

At first, the effect of reaction temperature on the product yield is investigated to understand the optimum condition under the ultrasonic irradiation condition. As it has been indicated, temperature was varied between 30 and 65°C. In all experiments, a methanol to oil ratio of 15% w/w (base on the weight of raw oil), and a KOH concentration of 1.5% w/w were used. Figure 6 shows the relationships between the product yield and reaction time at various reaction temperatures under ultrasonic irradiation condition. As it can be

observed, the final product yield was almost reached in 40 minutes. After 40 minutes the product yield present in the 30, 50 and 65°C runs were 93, 90 and 85%, respectively. Then, the product yield obtained in the 30 and 50°C run were very similar, and the one in the 65°C run was clearly less. An experimental result indicates that at lower temperature the extent of product yield is higher and when the temperature is increased, the product yield decreased which showed temperature had negative influence on methanolysis of crude *jatropha curcus* L.oil. The extent of cavitational effect is dampened at higher operating temperature due to the cavitation may also lead to the localized increase in temperature at the phase boundary of mixing reaction. At higher reaction temperature, there is a chance of loss of methanol. Then, the reaction temperature above boiling point of alcohol is avoided since at high temperature it tends to accelerate the saponification of glycerides by the alkaline catalyst.

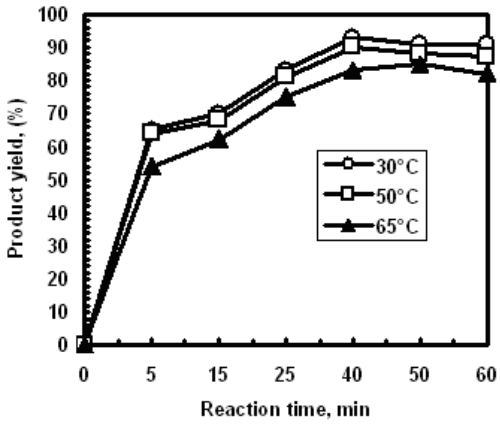

Figure 6 *Effect of reaction temperature on the product yield of biodiesel at 1.5% w/w KOH and methanol to oil ratio 15%w/w)*

3.2.2 Effect of KOH concentration

The catalyst concentration variations adopted in this study were 1%, 1.5% and 2% w/w. Figure 7 shows the relationships between the product yield and reaction time at various catalyst (KOH) concentrations, methanol to oil ratio 15% w/w and 30°C under ultrasonic irradiation condition. It was observed the maximum product yield at 1% w/w KOH was higher than those at other KOH concentration. The maximum product yield of 95% was obtained using 1% w/w KOH concentration in 40 minutes. The results clearly indicated that the optimum concentration of catalyst (KOH) required for effective base catalyzed transesterification was 1% w/w. With the increase in the concentration of catalyst, there was decrease in the product yield. This was due to the formation of soap in presence of high amount of catalysts, which increased the viscosity of the reactants and lowered the product yield.

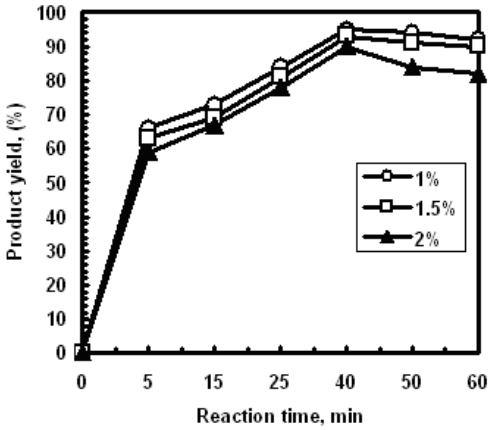

Figure 7 *Effect of catalyst concentration on the product yield of biodiesel at reaction time 30°C and methanol to oil ratio 15%w/w*

3.2.3 Effect of Methanol quantity

The experiments were carried out varying the methanol/oil ratio between 10% w/w and 20 % w/w, KOH concentration of 1% and reaction temperature 30°C. Figure 8 shows the relationships between the product yield and reaction time at various methanol to oil ratio under ultrasonic irradiation condition. As it can be observed, with methanol/oil ratio of 10% w/w, the product yield was obtained 90% after 40 minutes. The product yield increased as the methanol to oil ratio increased, with the best results (96%) being for methanol/oil ratio of 15% w/w. nevertheless, a later increased of methanol/oil ratio 20% w/w does not produce and increased in the product yield, since a lower value is obtained (94%) because for higher methanol/oil ratio, the separation of ester from glycerol layer becomes difficult and hence methanol/oil ratio of 15% w/w can be considered as an optimum operating ratio.

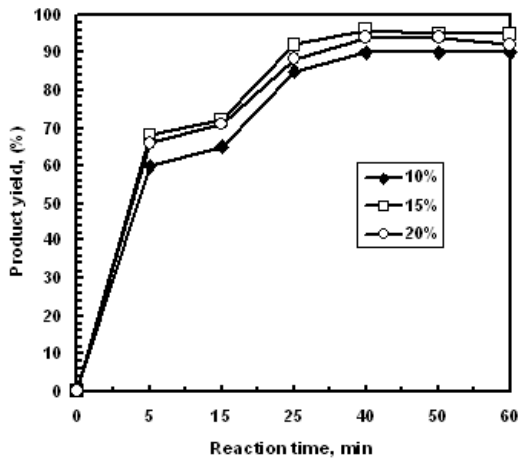

Figure 8 *Effect of methanol quantity on the product yield of biodiesel at 1.5%w/w KOH and reaction time 30°C*

3.3 Comparison between ultrasonic cavitation and mechanical stirring

A comparison of the effect of ultrasound in the transesterification reaction was carried out at the optimal operating conditions (methanol to oil ratio = 15% w/w, catalyst amount = 1% w/w and reaction temperature = 30°C).

Figure 9 shows the product yield obtained applying the ultrasonic cavitation process and the conventional mechanical stirring process. It can be seen from the figure that ultrasonic cavitation results in 96% conversion whereas the conventional stirring method results in lower extent of conversion 83% over similar time operation as 40 minutes. This can be attributed to the fact that as the reaction is mass transfer controlled, the micro level turbulence generated due to cavitation bubbles results into the higher availability of the interfacial area and hence higher conversion. Moreover the figure 9 indicated that use of ultrasonic irradiation enhances the limiting equilibrium conversion product yield from about 91% in 60 minutes to about 96% in 40 minutes of reaction time. Thus, the use of ultrasonic irradiation to be in both enhances the rate of reaction as well as shifting the equilibrium and resulting in higher product yield.

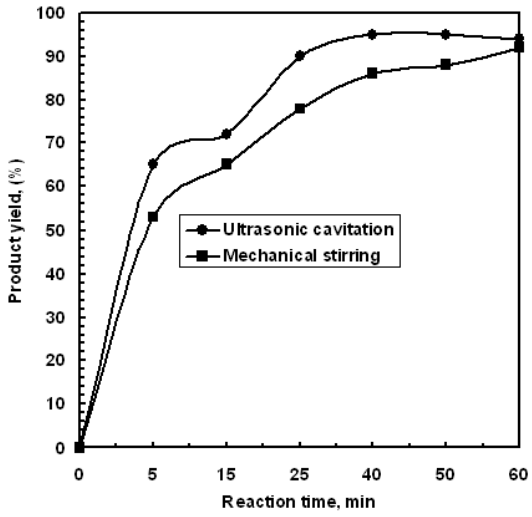

Figure 9 *Effect of ultrasonic irradiation and conventional mechanical stirring on product yield.*

4. Conclusions

In summary, the two-step process coupled with ultrasonic irradiation condition is an efficient method and time saving for biodiesel production from crude *jatropha curcus* L.oil with high free fatty acid. A two-step transesterification process was selected to improve the product yield. The first step was acid catalyzed esterification process, which could reduced the FFA level of crude *jatropha curcus* oil less than 3%, The second step, base catalyzed transesterification process gave more than 90% of product yield. The optimum condition for production of methyl ester (biodiesel) under the ultrasonic irradiation condition was follow as: the first step process was obtained at methanol/oil ratio of 15% w/w, catalyzed concentration of 3% w/w (by the weigh of oil), reaction time of 20 minutes and temperature of 30 °C (room temperature). While, the second step process was obtained at methanol/oil ratio of 15% w/w, catalyzed concentration of 1% w/w (by the weigh of oil), reaction time of 40 minutes and temperature of 30°C (room temperature). Finally, comparison with conventional mechanical stirring method has enabled to clearly illustrate the role of ultrasonic irradiation in inducing an effective emulsification and mass transfer so that the rate of ester formation is significantly enhanced.

Acknowledgments

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