

# Production of Biodiesel via In-Situ Supercritical Methanol Transesterification

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## 1. Introduction

Most energy that the world is using is derived from unrenovable fossil fuel that has a great impact on environments (Warabi et al., 2004). The demand of fossil fuels is increasing very rapidly and it is estimated that the remaining world reserves will be exhausted by the year 2020, with the current rate of consumption. There is an urgent need to seek for an alternative fuels to substitute the diesel due to gradual depletion of world crude oil reserves. Research is, therefore oriented for alternative energy. Biomass is one of its candidates, because biomass energy has some advantageous in reproduction, cyclic and carbon neutral properties (Warabi et al., 2004). Biodiesel fuel is one example of biomass energy, and it is generally made of methyl esters of fatty acids produced by the transesterification reaction of triglycerides with methanol with the help of a catalyst (Clark et al., 1984). Alcoholysis of vegetable oils produces fatty acids alkyl esters that are excellent substitutes for conventional fossil diesel fuels (Selmi and Thomas, 1998; De et al., 1999). The viscosity of alkyl esters is nearly twice that of diesel fuel instead of 10–20 times as in the case of neat vegetable oil (Rathore and Madras, 2007).

The use of such edible oil to produce biodiesel is not feasible in view of big gap in the demand and supply of such oils in the country for dietary consumption. Increased pressure to augment the production of edible oils has also put limitations on the use of these oils for production of biodiesel (Sinha et al., 2008). Therefore, biodiesel is actually competing limited land availability with the food industry for the same oil crop. Thus, instead of arable land being utilized to grow food, it is now being used to grow fuel. This will then raise the price of edible oil making the biodiesel produced economically unfeasible as compared to petroleum-derived diesel. In order to overcome this issue, many researchers have begun searching for cheaper and non-edible oils to be used as alternative feedstock for biodiesel production (Kansedo et al., 2009). Few sources have been identified such as waste cooking oil (Wang et al., 2006; Chen et al., 2009) and oils from non-edible oil-producing plants such as *Jatropha curcas* (Heller, 1996; Herrera et al., 2006; Tiwari et al., 2007; Berchmans and Hirata, 2008; Chew, 2009), *Pongamia pinnata* (Meher et al., 2006; Naik et al., 2008; Pradhan et al., 2008), *Calophyllum inophyllum* (Sahoo et al., 2007), cottonseed (Demirbas, 2008; Qian et al., 2008; Rashid et al., 2009), rubber seeds (Ikwaagwu et al., 2000; Ramadhas et al., 2005) and tobacco seeds (Usta, 2005; Veljkovic et al., 2006). Obviously, developing nations have to focus their attention on oils of non-edible nature, which are cheaper (Sinha et al., 2008). In Malaysia, *Jatropha curcas* L. (JCL), could be utilized as a source for production of oil and can be grown in large scale on non-cropped marginal lands and waste lands.

JCL oil is obtained only after going through the following steps: collection of fruit from the trees, separation of seed from the hull, seed drying (Chew, 2009), oil pressing and filtration. Pressing oil from the kernel yields kernel cake (40-50%) and crude oil (50-60%). At present, in the majority of cases oil is generally pressed directly from the seed without separating the kernel and shell. This method produces seed cake (70-75%) and crude oil (25-30%) (Chew, 2009). Much of the un-extractable oil still remains in the seed cake; hence better ways of extracting the oils are needed. Among the extraction techniques reported in the literature include the use of Soxhlet extraction method (Castro and Ayuso, 1998; Ayuso and Castro, 1999; Szentmihalyi et al., 2002; Darcia and Castro, 2004), aqueous enzymatic oil extraction (Rosenthal et al., 1996; Sharma and Gupta, 2006; Jiang et al., 2010) and enzyme assisted three phase partitioning (Shah et al., 2004; Gaur et al., 2007). Some of these extraction methods, however, required a longer extraction time (Chew, 2009). Nowadays, many researchers (Papamichail et al., 2000; King et al., 2001; Cao and Ito 2003; Machmudah et al., 2008) turns to supercritical extraction techniques which is relatively rapid because of the low viscosities and high diffusivities associated with supercritical fluids.

Transesterification is the general term used to describe the important class of organic reactions where an ester is transformed into another ester through interchange of the alkoxy moiety. Several aspects, including the type of catalyst (alkaline, acid or enzyme), alcohol/vegetable oil molar ratio, temperature, purity of the reactants (mainly water content) and free fatty acid content have an influence on the course of the transesterification. In the conventional transesterification of fats and vegetable oils for biodiesel production, free fatty acid and water always produce negative effects, since the presence of free fatty acids and water causes soap formation, consumes catalyst and reduces catalyst effectiveness, all of which result in a low conversion (Demirbas, 2007). In addition to that, more catalyst is required to neutralize free fatty acids of oil with higher free fatty acids content (Kusdiana and Saka, 2004). Thus, the catalytic processes have a high production cost and are energy intensive. One primary problem is due to the vigorous stirring required for the mixing of the two-phase mixture of oil and alcohol. Another problem is the separation of catalyst after the reaction (Madras et al., 2004). Therefore, non-catalytic transesterification has been investigated.

Supercritical fluid extraction using polar solvent such as methanol as an extraction solvent is highly potential extraction technique to be used whereby high yield of oil can be achieved within a shorter time (Hawash et al., 2009). Further, at supercritical state, the solvent solubility increased dramatically, and the extracted oil is relatively low in impurities (Tan et al., 2009). However, there is no details on the maximum crude biodiesel yield can be obtained related to the in-situ supercritical methanol transesterification direct from the seeds.

In situ transesterification differs from the conventional reaction in the sense that the oil-bearing material contacts acidified alcohol directly instead of reacting with purified oil and alcohol. That is, extraction and transesterification of the seed powder proceed within the same process, with alcohol acts as an extracting solvent as well as esterification reagent (Fukuda et al., 2001). In situ transesterification (Harrington and Evans, 1985; Marinkovic and Tomasevic, 1998; Kildiran et al., 1996; Hass et al., 2004), a biodiesel production method that utilizes the original agricultural products instead of purified oil as the source of triglycerides for direct transesterification, eliminates the costly hexane extraction process and works with virtually any lipid-bearing material. It could reduce the long production system associated with pre-extracted oil and maximize alkyl ester yield. The use of reagents and solvents is reduced, and the concern about waste disposal is avoided. This process reduces the cost of final product as this process has less number of unit operations. It is the best non-renewable source of energy with good environmental impact and easy recovery.

Thus, this study contributes in terms of design, development and improvement of the in-situ supercritical methanol transesterification of biodiesel production via high-pressure high-temperature batch-wise reactor system. In this study, biodiesel is generated directly from JCL seeds using methanol at different solvent critical states.

## 2. Materials and methods

### 2.1 Sample preparation

The *Jatropha curcas* L. (JCL) fruits were obtained with cooperation from the Plantation Unit of Universiti Teknologi MARA Perlis, Malaysia. JCL fruits were cleaned and de-hulled to separate the hull from the seeds. The seeds were then dried in an oven at 105 °C for 35 min (Akbar et al., 2009). The JCL seeds were ground using grinder and sieved through progressively finer screen to obtain particle sizes ( $d_p$ ) of < 1 mm (Augustus et al., 2002). Sieving was accomplished by shaking the JCL powder in a Endecotts Shaker Model EFL2 for about 30 min and finally stored in a tightly-capped plastic container. The seeds need to be dried and ground in order to remove surface moisture content to obtain constant weight and weaken or rupture the cell walls to release oil for extraction, respectively (Akpan, 2006).

### 2.2 In-situ supercritical methanol transesterification

A batch type reactor at supercritical methanol was used for in-situ supercritical methanol transesterification of biodiesel from JCL seeds. The in-situ transesterification was carried out at temperatures and pressures ranging from 180 – 300 °C and 6 – 18 MPa, respectively. After a leak-check test, the reactor was pressurized with nitrogen to the desired pressure and heated to reaction temperature at a rate of 5 °C/min. After reaching desired temperature, the reaction was held for periods of 5 – 35 min. A JCL seeds-to-methanol ratio (1:15, 1:20, 1:30, 1:40 and 1:45 w/v) was also investigated. After each reaction, the vessel was removed from the heater and placed into a cold water bath to quench the reaction and depressurized to ambient pressure. The extracted product was discharged from the reactor and was vacuum-filtered on a Buchner funnel and the filter cake was washed with methanol. The extracted products from the in-situ transesterification were allowed to settle and separated into two phases in 500 ml separating funnel. It took about 30 min to separate into two phases, i.e., the top phase consists of the biodiesel (fatty acid methyl ester) and the lower phase consists of the glycerol and other minor components. The schematic diagram of the experimental apparatus of the batch-wise extraction system is shown in Fig. 1.

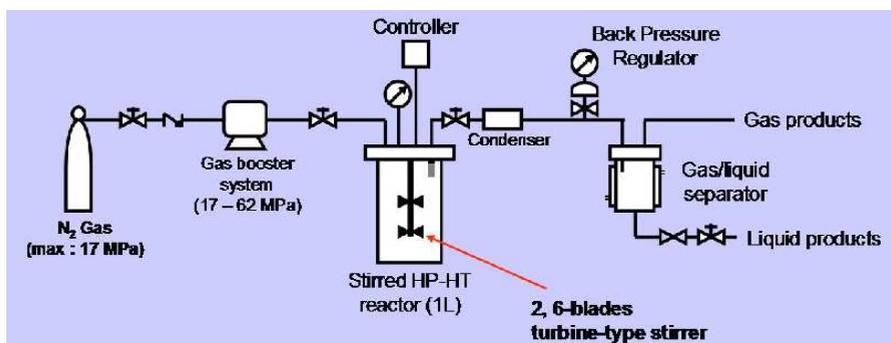


Fig. 1. Schematic diagram of batch-wise extraction system.

### 2.3 FAMES analysis

The FAMES analysis was quantified by Agilent Technologies 6890N with HP-5 5% Phenyl Methyl Siloxane capillary column (30 m by 320  $\mu\text{m}$  by 0.25 mm) and a flame ionization detector. Methyl heptadecanoate (10.0 mg; internal standard) was dissolved in 1 ml hexane to prepare the standard solution. Approximately 100 mg crude methyl ester was dissolved in 1 ml standard solution for GC analysis (Hong, 2009). Approximately 1  $\mu\text{l}$  sample was injected into the GC at an oven temperature of 210  $^{\circ}\text{C}$  with Helium as the carrier gas. The GC oven was programmed at 210  $^{\circ}\text{C}$ , isothermally for 15 min. the FAMES content was calculated by use of the Equation 1:

$$C = \frac{\sum A - A_{IS}}{A_{IS}} \times \frac{C_{IS} \times V_{IS}}{m} \times 100\% \quad (1)$$

#### Where:

$\sum A$  = total peak area of methyl ester

$A_{IS}$  = peak area of internal standard (methyl heptadecanoate)

$C_{IS}$  = concentration of the internal standard solution, in mg/ml

$V_{IS}$  = volume of the internal standard solution used, ml

$m$  = mass of the sample, in mg

### 2.4 Biodiesel properties

The biodiesel was characterized by its density, viscosity, high heating value, cloud and pour points and flash points according to ASTM standards.

## 3. Results and discussion

### 3.1 Effect of temperature

The effect of temperature on percent of FAMES yields from JCL seeds were investigated. The parameters were fixed at 12 MPa of pressure, 1:40 (w/v) of seeds-to-methanol ratio, 30 min of reaction time and at varying temperatures of 180, 200, 240, 280 and 300  $^{\circ}\text{C}$ . The results of in-situ supercritical methanol on percent of FAMES yields from JCL seeds at various temperatures are shown in Table 1. For simplification, the data are also plotted in Fig. 2.

Temperature ( $^{\circ}\text{C}$ )	Yields (%)					Others
	FAMES	Methyl Palmitate	Methyl Oleate	Methyl Linoleate	Methyl Stearate	
180	63.9	10.3	27.9	22.1	3.6	36.1
200	76.0	13.4	34.6	23.2	4.8	24.0
240	90.3	16.2	36.4	31.1	6.6	9.7
280	97.9	18.1	39.5	33.2	7.1	2.1
300	90.9	16.3	36.6	31.3	6.7	9.1

<sup>1</sup>conditions: 12 MPa, 30 min and 1:40 (w/v) seeds-to-methanol ratio.

Table 1. In-situ supercritical methanol transesterification<sup>1</sup> results from JCL seeds at various temperatures on percent of FAMES yield and its contents.

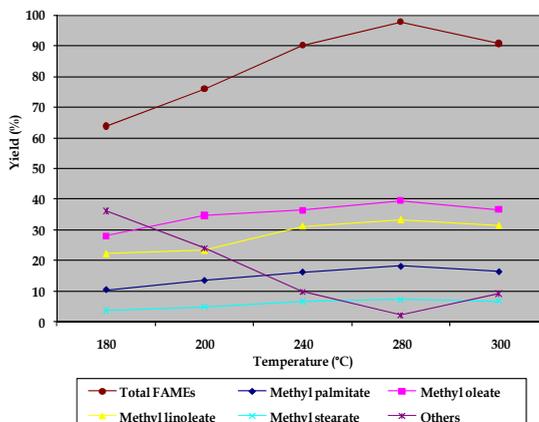


Fig. 2. In-situ supercritical methanol transesterification results from JCL seeds at various temperatures on percent of FAMES yield and its contents.

From Table 1, the results indicate that the percent of FAMES yields obtained at temperatures of 180 to 300 °C were in the range of 63.9 – 97.9%. The saturated FAMES content of the seed samples are low, which is between 10.3 – 18.1% for methyl palmitate and 3.6 – 7.1% for methyl stearate. Meanwhile, the content of unsaturated FAMES, methyl oleate and methyl linoleate are considerably higher at 27.9 – 39.5% and 22.1 – 33.2%, respectively. It should be noted that the critical temperature of methanol is at 240 °C and therefore, the conditions at 180 – 200 °C, 240 and >240 – 300 °C represent subcritical, supercritical and postcritical states of the medium, respectively.

At 180 °C, which is the lowest temperature of investigation, low yields of FAMES (63.9%) were obtained. This observation might be due to the subcritical state of methanol or the instability of the supercritical state of methanol. It was observed that by increasing the reaction temperature to supercritical conditions had a favorable influence on the yield of ester conversion (Demirbas, 2008). Similar results have been reported by Cao et al., (2005), Madras et al., (2004) and Bunyakiat et al., (2006) on soybean oil, sunflower oil and coconut oil, respectively.

Apparently, by increasing the reaction temperature from 200 to 280 °C, the conversion increases significantly with FAMES yields increased from 76.0 – 97.9%. The higher conversions observed in the supercritical state can be attributed to the formation of a single phase between alcohol and oil (Madras et al., 2004). Under supercritical conditions, the solubility parameter of alcohol reduces and was close to the solubility parameter of oil (Han et al., 2005). According to Petchmala et al., (2008), the increase in temperature causes the polarity of methanol to decrease, as a result of the breakdown of the hydrogen bonding of methanol, leading to an increased in the solubility of fatty acids in methanol. The complete solubility occurs as the temperature approaches the mixture critical temperature, at which point the reaction mixture became homogeneous and reaction took place rapidly. In addition, higher temperature contributed to the decomposition of cell walls, and as a result crude biodiesel yield was increased (Machmudah et al., 2007).

At 300 °C, the percent of FAMES (90.9%) yields were slightly decreased. This observation was due to the decomposition of polyunsaturated methyl esters and unreacted triglycerides in postcritical methanol at severe high temperature (Tan et al., 2009). This finding was further supported by Xin et al., (2008) who suggested that the favorable reaction temperature adopted

in supercritical methanol method should be lower than 300 °C. Reaction temperature at above 380 °C is insuitable for transesterification reaction because the oil and methyl esters tend to decompose at the highest rate. Furthermore, Kusdiana and Saka's (2001) pointed out that saturated and unsaturated FAMES undergo side reactions such as thermal decomposition and dehydrogenation reactions at temperature >400 °C and >350 °C, respectively. In these experiments, the temperature used was lower than that of Kusdiana and Saka's work and the side reactions did not occur since the temperature was below 300 °C. Furthermore, at 300 °C, a strong burning smell of the extract was detected. Hence, at this point, there is no reason to further increase the extraction temperature beyond 280 °C.

### 3.2 Effects of pressure

The results of in-situ supercritical methanol transesterification on percent of FAMES yields from JCL seeds at various pressures are shown in Table 2. For simplification, the data are also being plotted and is shown in Fig. 3. The temperature was fixed at 280 °C based on the maximized yield conditions from the previous experiment.

Pressure (MPa)	Yields (%)					
	FAMES	Methyl Palmitate	Methyl Oleate	Methyl Linoleate	Methyl Stearate	Others
6	80.6	13.1	41.0	20.4	6.1	19.4
8	95.6	15.7	47.1	26.3	6.5	4.4
12	97.9	18.1	39.5	33.2	7.1	2.1
16	93.5	16.0	38.4	32.1	7.0	6.5
18	92.5	16.0	38.6	31.1	6.8	7.5

<sup>1</sup>conditions: 280 °C, 30 min and 1:40 (w/v) seeds-to-methanol ratio.

Table 2. In-situ supercritical methanol transesterification<sup>1</sup> results from JCL seeds at various pressures on FAMES yield and its contents.

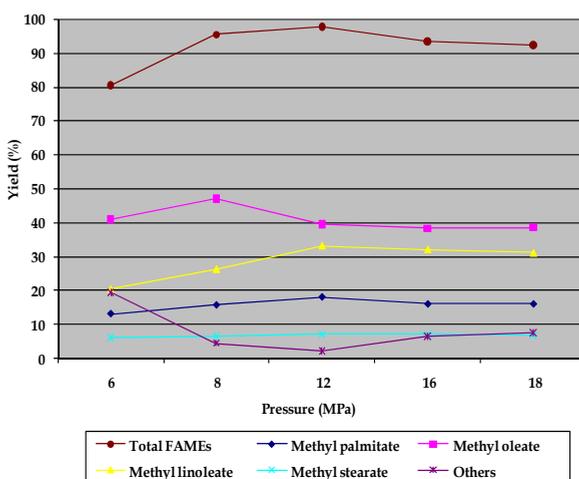


Fig. 3. In-situ supercritical methanol transesterification results from JCL seeds at various pressures on FAMES yield and its contents.

From Table 2, the results indicated that the percent of FAMES yields obtained at temperatures of 280 °C and pressures of 6-18 MPa was in the range of 80.6 – 97.9% with maximum yields at 12 MPa. The saturated FAMES content of the seed samples are low, which is between 13.1 – 18.1% for methyl palmitate and 6.1 – 7.1% for methyl stearate. Meanwhile, the content unsaturated FAMES, methyl oleate and methyl linoleate are considerably higher at 38.6 – 47.1% and 20.4 – 33.2%, respectively.

At the lowest pressure of 6 MPa, FAMES yields are only 80.6%, but increases to 97.9% when the pressure are increased to 12 MPa. The high FAMES yields achieved at 12 MPa, which is slightly above the critical pressure of methanol (8.09 MPa), might be due to the increase in solvent power of methanol with increasing pressure.

Further, increasing the pressure to 18 MPa, the FAMES yield decreases slightly to 92.5%. After the pressure increased to a specific level, the increase of pressure does not cause an obvious improvement in the FAME yield (He et al., 2007). This phenomenon might be due to the maximum solubility and/or hydrogen donor ability of the solvent that has been achieved regardless of high pressure employed.

As the pressure of the system increased, the solubility parameter of the methanol decreased and is close to the solubility parameter of the oil, thus forming a single phase between the alcohol and the oil. Based on these results, it can be seen that the fact that both temperature and pressure play an important role that contributes to high extraction yield, with the later being more prominent. Based on these results, it can be seen that the fact that both temperature and pressure play an important role that contributes to high yield, with the later being more prominent.

### 3.3 Effects of reaction time

Table 3 and Fig. 4 shows the effect of reaction time on percent of FAMES yields from JCL seeds using in-situ supercritical methanol transesterification. The reaction conditions were fixed based on maximum yields at optimized conditions discussed previously, i.e. 280 °C of temperature and 12.7 MPa of pressure.

Reaction time (min)	Yields (%)					
	FAMES	Methyl Palmitate	Methyl Oleate	Methyl Linoleate	Methyl Stearate	Others
5	88.4	15.0	38.5	28.6	6.3	11.6
10	94.2	16.6	40.5	30.6	6.5	5.2
20	96.0	17.2	39.6	32.0	7.2	4.0
30	97.9	18.1	39.5	33.2	7.1	2.1
35	93.1	16.6	38.8	30.8	6.9	6.9

<sup>1</sup>conditions: 280 °C, 12.7 MPa and 1:40 (w/v) seeds-to-methanol ratio.

Table 3. In-situ supercritical methanol transesterification<sup>1</sup> results of JCL seeds at various reaction times on percent of FAMES yield and its contents.

From Table 3 and Fig. 4, the results indicated that the percent of FAMES yields obtained at temperatures of 280 °C, pressures of 12.7 MPa, seeds-to-methanol ratio of 1:40 (w/v) and reaction time of 5 – 35 min was in the range of 88.4 – 97.9% with maximum yields at 30 min.

The saturated FAMES content of the seed samples are low, which is between 15.0 – 18.1% for methyl palmitate and 6.3 – 7.2% for methyl stearate. Meanwhile, the content unsaturated FAMES, methyl oleate and methyl linoleate are considerably higher at 38.5 – 39.5% and 28.6 – 33.2%, respectively.

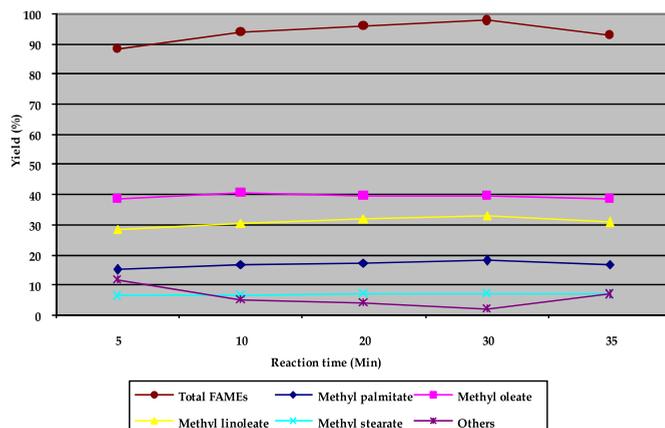


Fig. 4. In-situ supercritical methanol transesterification results from JCL seeds at various reaction times on percent of FAMES yield and its contents.

From the results, it can be seen that the percent of FAMES yields were only 88.4% at 5 min of reaction time. According to Saka and Kusdiana (2001), in the common method, the reaction is initially slow because of the two-phase nature of the methanol/oil system, and slows even further because of the polarity problem even with the help of an acid or an alkali catalyst. However, as described in this work, supercritical method can readily solve these problems because of the supercritical temperature and pressure employed. It can be seen that the conversion was increased in the reaction time ranges between 5 and 30 min with the percent of FAMES yields showed a slight increase in the range of 88.4 – 97.9%.

Further, the results indicated that an extension of the reaction time from 30 to 35 min had leads to a reduction in the FAMES yield (93.1%). This is because longer reaction time enhanced the hydrolysis of esters (reverse reaction of transesterification), resulted in loss of esters as well as causing more fatty acids to form soap (Eevera et al., 2009). Hence, for this process, there is no reason to prolong the reaction time beyond 30 min. Thus, the reaction time of 30 min can be considered as the economic reaction time by considering the percent of crude biodiesel and FAMES yields being achieved.

### 3.4 Effects of seeds-to-methanol ratio

Table 4 and Fig. 5 shows the effect of seeds-to-methanol ratio on percent of FAMES yields from JCL seeds using in-situ supercritical methanol transesterification. The reaction conditions were fixed based on maximized yields at optimized conditions discussed previously, i.e. 280 °C of temperature and 12.7 MPa and 30 min of reaction time with varying seeds-to-methanol ratio of 1:20, 1:30 and 1:40 (w/v).

Seed-to-methanol ratio (w/v)	Yields (%)					
	FAMEs	Methyl Palmitate	Methyl Oleate	Methyl Linoleate	Methyl Stearate	Others
1:15	89.0	15.2	37.4	29.8	6.6	11.0
1:20	94.4	16.9	38.8	31.6	7.1	5.6
1:30	95.9	17.5	38.6	32.3	7.5	4.1
1:40	97.9	18.1	39.5	33.2	7.1	2.1
1:45	97.0	17.4	40.1	32.3	7.2	3.0

<sup>1</sup> conditions: 280 °C, 12.7 MPa, 30 min reaction time.

Table 4. In-situ supercritical methanol transesterification<sup>1</sup> results of JCL seeds at various seed-to-methanol ratios on percent of FAMEs yield and its contents.

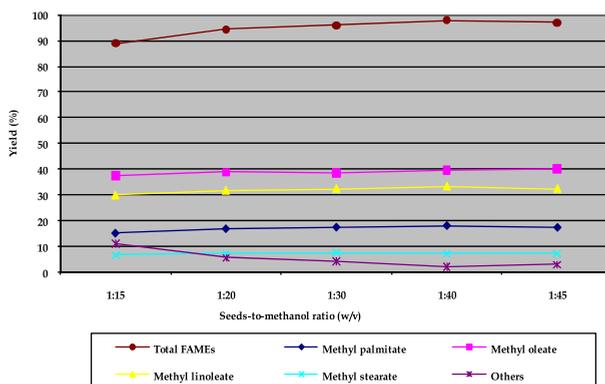


Fig. 5. In-situ supercritical methanol transesterification results of JCL seeds at various seeds-to-methanol ratios on percent of FAMEs yield and FAMEs contents.

From Table 4 and Fig. 5, the results indicated that the percent of FAMEs yields obtained at temperatures of 280 °C, pressures of 12.7 MPa, reaction time of 30 min and at various seeds-to-methanol ratio (1:15 – 1:45 w/v) was in the range of 89.0 – 97.9%, with maximum yields at 1:40 (w/v). The saturated FAMEs content of the seed samples are low, which is between 15.2 – 18.1% for methyl palmitate and 6.6 – 7.5% for methyl stearate. Meanwhile, the content unsaturated FAMEs, methyl oleate and methyl linoleate are considerably higher at 37.4 – 39.5% and 29.8 – 33.2%, respectively.

Obviously, at the lowest seeds-to-methanol ratio of 1:15 (w/v), the percent of FAMEs yields was relatively low (89.0%) and increased with increasing seeds-to-methanol ratio. When the methanol content in the supercritical fluids increased, the percent conversion of methyl ester also increased. The higher methanol content is favorable not only because more molecules of methanol surround the oil molecules but also because it contributes to the lower critical temperature of the mixture. Maximum percent of crude biodiesel and FAMEs yields were

obtained at a 1:40 (w/v) of seeds-to-methanol ratio. This is a significant difference from conventional catalytic reaction for which at least 1 h of reaction time is needed to attain the same yield. In this reaction, an excess of methanol was used in order to shift the equilibrium in the direction of the products (Demirbas, 2007). Kusdiana and Saka (2001) have suggested that higher molar ratios of methanol to oil also result in a more efficient transesterification reaction. The results obtained shows good agreement with previous work, where maximum conversion was obtained for rapeseed oil (Saka and Kusdiana, 2001) at molar ratio of 42:1, for various vegetable oils (Demirbas, 2002; Diasakou et al., 1998; Ma, 1998) and linseed oil (Varma and Madras, 2007) at molar ratio of 41:1 and 40:1, respectively. According to Bunyakiat et al., (2006), when the methanol content in the supercritical fluids increased, the percent of methyl esters conversion also increased.

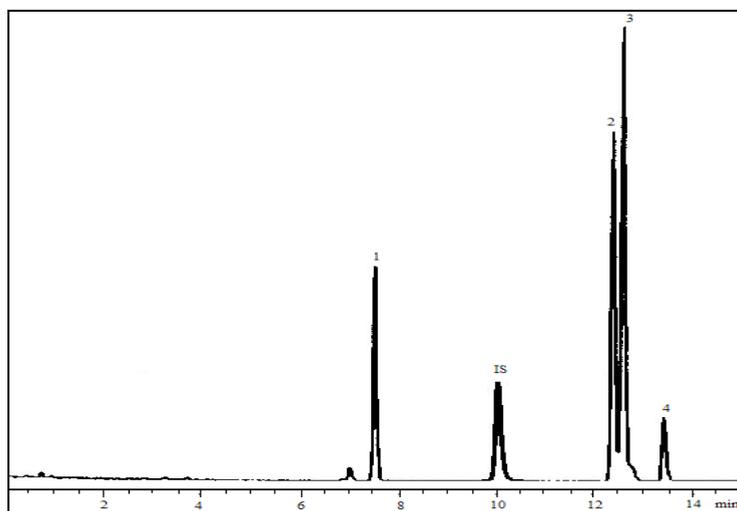
The higher methanol content is favorable not only because more molecules of methanol surround the oil molecules but also because it contributes to the lower critical temperature of the mixture. It can be seen that an increment in seed-to-methanol ratio can enhance biodiesel yield due to higher contact area between methanol and triglycerides. However, when the ratio is beyond 40, the yield of biodiesel begins to decrease substantially. This might be due to the restriction of the reaction equilibrium and difficulties in separating excessive methanol from methyl esters and glycerol, which subsequently lowered the yield of biodiesel (Tan et al., 2009).

Moreover, it was observed that for high seeds-to-methanol ratio added the set up required longer time for the subsequent separation stage since separation of the FAMES layer from the organic layer becomes more difficult with the addition of a large amount of methanol. This is due to the fact that methanol, with one polar hydroxyl group, can work as an emulsifier that enhances emulsion. Operating beyond the optimal value, the ester yield would not be increased but will result in additional cost for methanol recovery (Eevera et al., 2009). Therefore, increasing the seeds-to-methanol ratio is another important parameter affecting the FAMES yield. This report is in line with the results of many investigations based on neat vegetable oils (Freedman et al., 1984; Zhang et al., 2003; Leung et al. 2006, Eevera et al., 2009).

### 3.5 Biodiesel characterization

The biodiesel obtained through the one-step supercritical methanol extraction and transesterification in-situ process in this experiment was dark yellow in color. Compositions of samples were analyzed by GC. Figure 6 shows the total ion current chromatogram of the biodiesel. Furthermore, Table 5 shows the names, structure and compositions of *Jatropha curcas* L. FAMES.

Fig. 6 depicts the gas chromatographic evaluation of the FAMES produced over the course of reaction. The methyl esters analyzed by GC appear in the retention time of less than 15 min in the chromatograms. The weight percentages were similar for all of the variables condition; temperature, pressure, reaction time and seeds-to-methanol ratio of in-situ transesterification, as suggested by Carrapiso et al., (2000) that transesterification was random. The average saturated FAMES content of the seed samples are low: 18.1% for methyl palmitate (C17:0) and 7.1% for methyl stearate (C19:0). The average content of the unsaturated FAMES, methyl oleate (C19:1) and methyl linoleate (C19:2) are considerably higher at 39.5 and 33.2%, respectively which are comparable to the fatty acid composition in crude JCL oil feedstock. Depending on the origin, either oleic or linoleic acid content is higher. In this case, the seed oil belongs to the oleic or linoleic acid group, to which similar to the majority of vegetable oils (Carrapiso et al., 2000).



IS: Internal standard (Methyl heptadecanoate)

Fig. 6. Total ion current chromatogram of the biodiesel.

Peak No.	Name	Wt%
1	Methyl Palmitate	18.46
2	Methyl Oleate	40.41
3	Methyl Linoleate	33.91
4	Methyl stearate	7.22

Table 5. Names, structure and compositions of *Jatropha curcas* L. FAMES.

### 3.5.1 Biodiesel characterization

Vegetable oil methyl esters, commonly referred to as “biodiesel” are prominent candidates as alternative Diesel fuels. Biodiesel is technically competitive with or offers technical advantages compared to conventional petroleum Diesel fuel. The vegetable oils, as alternative engine fuels, are all extremely viscous with viscosities ranging from 10 to 20 times greater than that of petroleum Diesel fuel (Demirbas, 2003). The purpose of the transesterification process is to lower the viscosity of the oil. In this study, in-situ supercritical methanol transesterification for production of biodiesel from *Jatropha curcas* L. (JCL) seeds was generate via 1000 ml high-temperature high-pressure batch-wise reactor system in an absence of catalyst. The reaction conditions were conducted at 280 °C of temperature, 12.7 MPa of pressure, 30 min of reaction time and 1:40 of seeds-to-methanol ratio at 450 rpm of stirring rate. Samples of the biodiesel obtained from the in-situ experiment were determined using reference methods published by American Society for Testing and Materials (ASTM) D6751. In order to ensure that it can be used in diesel engine without any modification, the properties of biodiesel produced from this in-situ transesterification reaction was comparable with fuel properties of No. 2 Diesel. Fuel

properties of No. 2 Diesel, JCL biodiesel and ASTM D6751 derived biodiesel standards is shown in Table 6 for comparison.

Properties	No.2 Diesel <sup>a</sup>	JCL biodiesel <sup>b</sup>	ASTM D6751 <sup>a</sup>	JCL Biodiesel (This study)
Specific gravity	0.85	0.86 to 0.87	0.87 to 0.90	0.87
Kinematic viscosity @ 40 °C (cSt)	1.9 to 4.1	4.23 to 5.65	1.9 to 6	5.27
Cloud point (°C)	-19 to -8	8 to 10.2	Report	-2.06
Pour point (°C)	-34 to -10	4.2 to 6	-15 to 10	0
Flash point (°C)	51 to 85	130 to 192	130 min	100
Calorific value (MJ/kg)	45.0 to 45.3	38.5-42.7	41.0	39.3

<sup>a</sup>Demirbas, (2008); Encinar, (2005); Vyas, (2009)

<sup>b</sup>Ghadge and Rehman, (2005); Vyas, (2009); Sahoo and Das, (2009)

Table 6. Fuel properties of No. 2 Diesel and JCL biodiesel.

The properties of biodiesel produced from this in-situ supercritical methanol transesterification were comparable with fuel properties of commercial No. 2 Diesel. It was found that specific gravity of JCL biodiesel was 0.87 g/cm<sup>3</sup> and it falls between the ASTM D6751 ranges. Fuel injection equipment operates on a volume metering system, hence a higher density for biodiesel results in the delivery of a slightly greater mass of fuel (Demirbas, 2005). The kinematic viscosity was 5.27 cSt. Among the general parameters for biodiesel the viscosity of FAMES can go very high levels and hence it is important to control it within an acceptable level to avoid negative impacts on fuel injector's system performance (Murugesan et al., 2009). The flash point was determined to be at 100 °C. Since biodiesel has a higher flash point than diesel, it is a safer fuel than diesel. Addition of a small quantity of biodiesel with diesel increases the flash point of diesel which can result in improved fire safety for transport purpose (Lu et al., 2009) and it is safer to store biodiesel-diesel blends in comparison to diesel alone (Sahoo et al., 2009). Meanwhile, the pour point was measured to be 0 °C which was slightly higher than that of No. 2 Diesel fuel. This might be due to the presence of wax, which begins to crystallize with the decrease in temperature. This finding was agreed with Vyas et al., (2009) and Raheman and Ghadge, (2007). The problems of higher pour point of JCL biodiesel could be overcome by blending with diesel. The cloud point was reported to be -2.06 °C. The cloud point depends upon the feedstock used and must be taken into consideration if the fuel is to be used in cold environments (Fernando et al., 2007). The calorific value of JCL biodiesel was 39.3 MJ/kg, which was almost 88% of the calorific value of diesel (44.8 MJ/kg). The lower calorific value of JCL is because of the presence of oxygen in the molecular structure, which is confirmed by elemental analysis also. Furthermore, the presence of oxygen in the biodiesel helps for complete combustion of fuel in the engine. These findings were also agreed by Sinha et al., (2008). Therefore, they could be excellent substitutes and blends of No. 2 diesel fuel.

#### 4. Conclusions

Based on the findings, it can be concluded that temperature is an important property in this in-situ process. As the temperature increased, the crude biodiesel and FAMES yields also increased. The crude biodiesel and FAMES of the yields reached a maximum (59.9 and 97.9,

respectively) at 280 °C and then decreased with increasing temperature. The loss was caused by thermal decomposition, dehydrogenation and other side reactions. For the effect of pressure, the crude biodiesel and FAMES yield increased with increasing pressure. Above 12 MPa, no improvement of both yields was observed. The optimum pressure was thus fixed at 12.7 MPa in this experiment. For the effect of reaction time, it can be seen that the conversion was increased in the reaction time ranges between 5 and 30 min, and thereafter reduced as a representative of the equilibrium conversion. The excess reaction time did not promote the conversion but favors the reverse reaction of transesterification which resulted in a reduction in the ester yield. The optimal FAMES yield was found to be 97.9% in 30 min. For the effect of seeds-to-methanol ratio, the maximum crude biodiesel and FAMES yields were obtained at a 1:40 of seeds-to-methanol ratio. It can be seen that an increment in seed-to-methanol ratio can enhance biodiesel yield due to higher contact area between methanol and triglycerides. However, when the ratio is beyond 40, the yield of biodiesel begins to decrease substantially.

The merit of this method is that this new process just requires a single process, where the normal oil extraction process can be avoided. In addition, because of non-catalytic process, the purification of products after transesterification reaction is much simple, compared to the common method. Therefore, this new process can offer an alternative way to convert the fruits directly to methyl esters by a simpler-shorter production process.

## 5. Acknowledgment

The authors acknowledge the scholarship fund provided by Ministry of Science, Technology and Innovative under the National Science Fellowship (NSF), Universiti Teknologi MARA and Fundamental Research Grant Scheme (FRGS grant no: 600-RMI/ST/FRGS 5/3/Fst (2/2009)) for their financial support.

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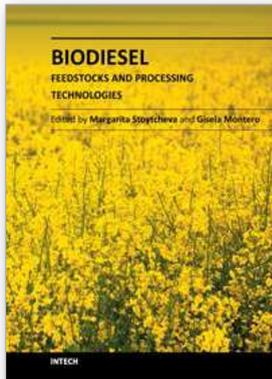
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## **Biodiesel - Feedstocks and Processing Technologies**

Edited by Dr. Margarita Stoytcheva

ISBN 978-953-307-713-0

Hard cover, 458 pages

**Publisher** InTech

**Published online** 09, November, 2011

**Published in print edition** November, 2011

The book "Biodiesel: Feedstocks and Processing Technologies" is intended to provide a professional look on the recent achievements and emerging trends in biodiesel production. It includes 22 chapters, organized in two sections. The first book section: "Feedstocks for Biodiesel Production" covers issues associated with the utilization of cost effective non-edible raw materials and wastes, and the development of biomass feedstock with physical and chemical properties that facilitate its processing to biodiesel. These include Brassicaceae spp., cooking oils, animal fat wastes, oleaginous fungi, and algae. The second book section: "Biodiesel Production Methods" is devoted to the advanced techniques for biodiesel synthesis: supercritical transesterification, microwaves, radio frequency and ultrasound techniques, reactive distillation, and optimized transesterification processes making use of solid catalysts and immobilized enzymes. The adequate and up-to-date information provided in this book should be of interest for research scientist, students, and technologists, involved in biodiesel production.

### **How to reference**

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Asnida Yanti Ani, Mohd Azlan Mohd Ishak and Khudzir Ismail (2011). Production of Biodiesel via In-Situ Supercritical Methanol Transesterification, *Biodiesel - Feedstocks and Processing Technologies*, Dr. Margarita Stoytcheva (Ed.), ISBN: 978-953-307-713-0, InTech, Available from:

<http://www.intechopen.com/books/biodiesel-feedstocks-and-processing-technologies/production-of-biodiesel-via-in-situ-supercritical-methanol-transesterification>

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