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Reaction Kinetics of Transesterification Between Palm Oil and Methanol under Subcritical Conditions

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Abstracts

The objective of this work was to evaluate transesterification kinetics for biodiesel production from palm oil under subcritical conditions. Experimental investigation was carried out with palm oil and methanol at molar ratio of 46:1, temperatures between 150-200°C and pressure around 140-190 atm in a 400 ml batch reactor. The biodiesel products were analyzed by gas chromatography – mass spectrometry. Area percentage method was used to estimate the methyl esters in the product. Zero- and first-order kinetic models were developed. Apparent activation energy was estimated to be in the range of 91-105 kJ/mol. The reaction rate equation was best approximated by the first order kinetic model with pre-exponential factor of 1.57 x 10^9.

Key words: Biodiesel; Critical fluids; Kinetic equations; Methyl esters; Palm oil; Renewable energy

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INTRODUCTION

The crisis of petroleum shortage stimulates the use of alternative renewable biofuels like biomass, biogas and biodiesel. Biodiesel has been accepted as a good alternative fuel for diesel. Biodiesel has become a common fuel today because it possesses many good characteristics such as renewability and biodegradability. World biodiesel production was reported to increas from 2.3 billion liters in 2004 to 17 billion liters in 2009 (Timilsina and Shrestha, 2011). In Thailand, the government plans to have biodiesel production at 8.5 million liters per day by 2012 (Zhou and Thomson, 2009). Many researchers are interested in biodiesel, particularly in the production technique.

To produce biodiesel, vegetable oils or animal fats with short chain primary alcohols such as methanol or ethanol are used as feedstock. Triglycerides in oils or fats can have chemical reaction with the alcohols under suitable conditions. This reaction is called transesterification. The reaction products are glycerin and biodiesel. If methanol is used, the biodiesel is a mixture of fatty acids methyl esters. Various techniques of biodiesel production are available today. These are catalytic (Lin et al., 2009; Hou et al, 2007), enzymatic (Hama et al., 2008), reactive distillation (Simasatitkul et al., 2011) and non-catalytic techniques (Diasakou et al., 1998; Kusdianna and Saka, 2001; He et al., 2007). Catalytic technique is commonly used in industrial sectors today. However, non-catalytic technique has been demonstrated in laboratory that it is a simplest process and suitable for low grade feedstock that has high content of free fatty acids and water. To scale up from laboratory to a useful application, unknowns with regards to the reaction must be disclosed. For example, the kinetics data should be obtained. Tab. 1 shows literature

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review on kinetic studies of transesterification. So far, few data was available for the non-catalytic technique.

The earliest study was conducted by Diasakou et al. (1998). Mechanisms of the methanolysis reaction of triglycerides to methyl esters were proposed. These mechanisms were three-steps, as shown in eq. 1.

$$\text{three steps:}\quad \begin{aligned} TG + MeOH &\xrightarrow{k_1} DG + ME \\ DG + MeOH &\xrightarrow{k_2} MG + ME \\ MG + MeOH &\xrightarrow{k_3} G + ME \end{aligned} \qquad (1)$$

where TG = triglycerides, MeOH = methanol, G = glyceride, ME = methyl esters, DG = diglycerides, MG = monoglycerides, respectively. Diasakou et al. (1998) studied the methanolysis of soybean oil and assumed that; 1) all steps were irreversible reaction because of high content of methanol involved; 2) each reaction was the 1st order with respect to the major reacting agent. Firstly, triglycerides are converted to diglycerides, then to monoglycerides, and finally converted to methyl esters. In each step, a molecule of methanol is consumed to yield a molecule of biodiesel. Kusdiana and Saka (2001) simplified the three-step Diasakou's mechanisms to one step as:

$$\text{one step:}\quad TG + 3MeOH \xrightarrow{k} G + 3ME \qquad (2)$$

Kusdiana and Saka (2001) used rapeseed oil in excessive methanol and assumed that the reaction was irreversible as well. The reaction order was assumed to be the first order with respect to the triglycerides concentration. The reaction rate (r) was given in terms of the rate constant (k) and the disappearing of triglycerides concentration over a period of time was given as:

$$r = -\frac{dC_{TG}}{dt} = -kC_{TG}$$

Table 1
Literature Survey on Kinetics Study of One Step, Non-catalyst Methanolysis Reaction

Reference	n	m	k_1 (s-1)	T (°C)	P (atm)	k_0	E_a (kJ/mol)
Cheng et al. (2008)	1.5	0	0.01374	250	100-160	12.45	28.9
			0.02366	265	100-160		
			0.02585	280	100-160		
			0.02923	310	100-160		
Song et al. (2008)	0.96	1.05	no data	200-400	over 400	4.3×10^5	105.3
Joelianingsih et al. (2008)	1	0	0.0034	250	1	4.208	30.8
			0.0051	270	1		
			0.0056	290	1		
He et al. (2007)	1	0	0.000119	210	280	0.00194	11.2
			0.000125	220	280		
			0.000133	230	280		
He et al. (2007)	1	0	0.000366	240	280		
			0.000422	250	280		
			0.000525	260	280	170.8	56.0
			0.000782	270	280		
			0.000881	280	280		
Kusdiana and Saka (2001)	1	0	0.0002	200	70	3.33	38.4 [a]
			0.0003	230	90		
			0.0007	270	12		
Kusdiana and Saka (2001)	1	0	0.0071	300	14	145.57	47.1[b]
			0.0178	350	19		
			0.0249	385	65		
			0.0503	431	90		
			0.0803	487	105		

$r = kC_{TG}^{m}C_{MeOH}^{n}$, r = reaction rate, k = rate constant, k_1 = apparent rate constant, C_{TG} = triglycerides concentration, C_{MeOH} = methanol concentration, m = reaction order with respect to triglycerides concentration, n = reaction order with respect to methanol concentration, k_0 = pre-exponential factor, E_a = activation energy, R = gas constant = 8.314J/mole.K, T = absolute temperature, T= temperature at the k_1, P = pressure at the k_1, (c) = in a bubble column reactor.

where C_{TG} was the triglycerides concentration. A term "unmethyl esterified" compound (uME) was defined by Kusdiana and Saka (2001) as "a mixture of triglycerides, diglycerides, monoglycerides and unreacted free fatty acids". The reaction equation (2) may be written as:

$$uME + x\ MeOH \xrightarrow{k} G + x\ ME \qquad (3)$$

The reaction rate, in terms of the uME was:

$$r = -\frac{dC_{uME}}{dt} = -kC_{uME}$$

where C_{uME} is the concentration of the uME, and k is rate constant which constant respected with temperature. The apparent rate constants (k_1) were estimated to be

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between 0.0002 – 0.0803 s^{-1} for temperature 200 – 487°C. The activation energies were estimated by Sawangkeaw et al. (2010) to be 38.4 and 47.1 kJ/mol for 200 – 270°C and 300 – 487°C, respectively. Based on the one-step mechanism, He et al. (2007), Cheng et al. (2008), Song et al. (2008), and Joelianingsih et al. (2008) performed theirs investigations on soybean oil, peanut oil, refined, bleached and deodorized palm oil, and palm oil, respectively. Various apparent activation energies were found to be in a range of 11.2 – 105.3 kJ/mol.

There are two approaches in non-catalytic technique; supercritical and near critical methanol. Under supercritical conditions, oil and methanol become single phase (homogeneous) due to the fact that supercritical methanol has a low dielectric constant (Kusdiana and Saka, 2001; Deslandes et al., 1998). This phenomena speed up the reaction rate. Therefore, the reaction can be completed within a very shot period. The drawback of supercritical methanol is that it requires high strength equipment to withstand high pressures and temperatures involved. On the other hand, subcritical conditions showed a slower reaction rate, taking longer to complete. Two recently studies (Yin et al., 2010; Patil et al., 2010) demonstrated that the reaction rates under subcritical methanol could be increased by adding small amount of catalyst or co-solvent. These studies were carried out with soybean oil and Camelina Satviva oil, respectively. Kinetic data, however, was not reported.

Subcritical methanol has lower temperature and pressure than supercritical one. It is more attractive to industrial sectors because it is easier to manage. Moreover, the transesterification reaction has smaller activation energies, implying that it would require less energy input to overcome the reaction barrier. Kinetics data associated with subcritical methanol reactions are essential for biodiesel production plant design. But, relevant work was rarely found in the existing literature, particularly for palm oil. This oil has great potential as a major source of feedstock of biodiesel for many countries, especially Indonesia, Malaysia and Thailand.

Therefore, in this work, kinetics of the transesterification reaction in converting palm oil to methyl esters under the non-catalyst subcritical methanol condition was investigated. The aims were to find the apparent rate constants, the activation energy, and the reaction rate equation of this reaction.

1. MATERIALS AND METHODS

The investigation was carried out with a refined palm olein and an industrial grade methanol as input reactants. Details on their properties are given in Tab. 2 below.

Table 2
Raw Materials Used

Reactant	Molecular mass	Density @ 25°C	Grade	Supplier
Palm oil	852	0.89 g/ml	Edible grade	Morakot Industry Co. Ltd, Thailand
Methanol	32	0.79 g/ml	Industrial grade (purity: 99.8 % wt)	O.V. Chemical & Supply, Thailand

The transesterification reaction took place in an autoclave with a sample-releasing facility that allowed samples to be released periodically as the reaction progress. The experimental setup is shown in Fig.1. Design and testing of this autoclave can be found in our previous report (Permsuwan et al., 2011). Methanol and palm oil at molar ratio of 46:1 was loaded into the autoclave at full capacity (methanol 265 ml, palm oil 144 ml). The autoclave was installed in an oven with a 2-kW electrical heater. Autoclave and oven temperatures were equalized at the beginning of the experiment (about 25°C). All connectors were fastened to make sure that they were all perfectly sealed. The heater was switched on in which the oven and autoclave temperatures were gradually increased to the set point (200°C for the autoclave). The autoclave pressure was increased in according to the thermodynamic effect. Once the pressure reached the pre-set value of 190 atm, a sample of about 2 ml was released into a container where the pressure was dropped to about 140 atm. A new container was then replaced for next ejection of sample. By this technique, 10 to 12 samples were collected in separated containers. They were later stored at ambient condition before analysis.

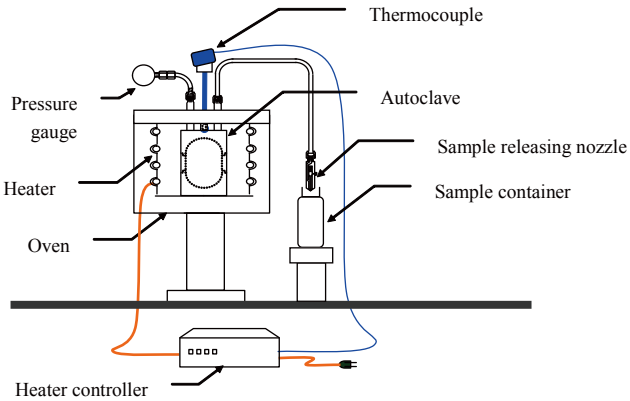

Figure 1
Experimental Setup

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Table 3
GC-MS Setting for Analysis of Biodiesel

Column name : Alltech AT-1MS
Column stationary phase coating agent: 100% dimethylpolysiloxane
Column dimensions: long x inside diameter x film thickness = 30 m x 0.25 mm x 0.25 µm
MS Quadrupole & MS Source Temperature : 150 & 230 °C
Injector Temperature: 250 °C
Oven temperature program:
130 °C on initial, hold 2 min,
200 °C (5 °C/min)
250 °C (10 °C/min), hold 10 min
Carrier gas: Helium (1.0 ml/min)

For analysis of the biodiesel products, hexane was used as a solvent for dilution the sample. The mixture of hexane and a drop of sample was injected into a gas chromatograph-mass spectrometer (Hewllet Packard GC-MS model 53-973 EI). Details of column and analysis conditions are in Tab. 3. Content of the methyl esters in the samples were quantified by using the area percent method.

2. KINETICS OF TRANSESTERIFICATION

From eq. 2, one mole of triglycerides reacts with three moles of methanol yield one mole of glycerin and three moles of methyl esters. The reaction rate (r) can be expressed as;

$$r = -r_{TG} = \frac{-r_{MeOH}}{3} = -r_G = \frac{r_{ME}}{3} \tag{4}$$

$$r = -\frac{dC_{TG}}{dt} = -\left(\frac{1}{3}\right)\frac{dC_{MeOH}}{dt} = \frac{dC_G}{dt} = \left(\frac{1}{3}\right)\frac{dC_{ME}}{dt} \tag{5}$$

where r = reaction rate, $-r_{TG}$ = rate of disappearance of triglycerides, $-r_{MeOH}$ = rate of disappearance of methanol, r_G = rate of appearance of glycerine, r_{ME} = rate of appearance of methyl esters, C_{TG} = the concentration of triglycerides, C_{MeOH} = the concentration of methanol, C_G = the concentration of glycerin, and C_{ME} = the concentration of methyl esters, respectively. The reaction rate (r) can be written in terms of rate constant (k) as;

$$r = kC_{TG}^{m}C_{MeOH}^{n} \tag{6}$$

$$\text{If } \frac{dC_{ME}}{dt} = k_p\frac{dx}{dt} \tag{7}$$

where k_p is a proportionality constant, and x is the fraction conversion of methyl esters. From the reaction rate, two different models will be considered.

2.1 Zero Order Model

$r = k$, when, m = n = 0, From eqs. (5), (6) and (7),

$$r = \frac{1}{3}k_p\frac{dx}{dt} \tag{8}$$

$$k_1 = \frac{3k}{k_p} \tag{9}$$

2.2 First Order Model

$r = kC_{TG}^{m}$, when, m = 1, n = 0

$$r = -\frac{dC_{TG}}{dt} = k_1C_{TG} \tag{10}$$

After integration, we obtained $-\ln C_{TG,t} + \ln C_{TG,0} = k_1t$ where C_{TG}, t and C_{TG}, 0 are triglycerides concentration at any time (t) and at the beginning (t = 0), respectively. Applied the concept of the uME, therefore, the C_{TG} was substituted by the C_{uME} to get

$$-\ln C_{uME,t} + \ln C_{uME,0} = k_1t \tag{11}$$

If we defined that

$$C_{uME,t} = C_{uME,0}(1-x) \tag{12}$$

where x is the fraction conversion of the methyl esters, then

$$\ln(1-x) = -k_1t \tag{13}$$

Numerical data of ln(1-x) at any reaction (t) can be made from eq. (13).

3. RESULTS AND DISCUSSION

3.1 Zero Order Model

From the results obtained from subcritical biodiesel production, conversion rate (x) was plotted against reaction time (t), shown in Fig. 2. The empirical relation between the conversion rate and time was found to be

$$x = 5.04\cdot10^{-7}t^2 + 3.3\cdot10^{-5}t + 0.106 \tag{14}$$

$$\text{So } k_1 = \frac{dx}{dt} = 1.1\cdot10^{-6}t + 3.3\cdot10^{-5} \tag{15}$$

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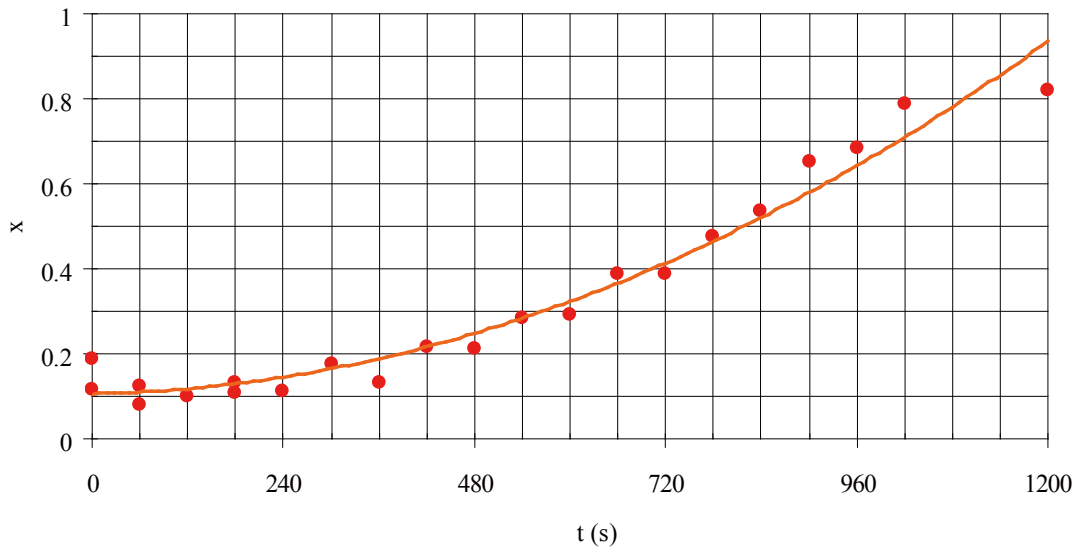

Figure 2
Biodiesel Conversion Rate

Variation of the apparent rate constants (k_1) at any reaction time (t) can be found from eq. (15). And data of reactor temperature (T) at any reaction time (t) was recorded during the experiment. Therefore, an Arrhenius plot can be obtained. It is shown in Fig. 3, where the gradient and the intercept can be estimated. Therefore, the apparent activation energy and the pre-exponential factor were calculated to be 90.9 kJ/mol and 1.18 x 107, respectively.

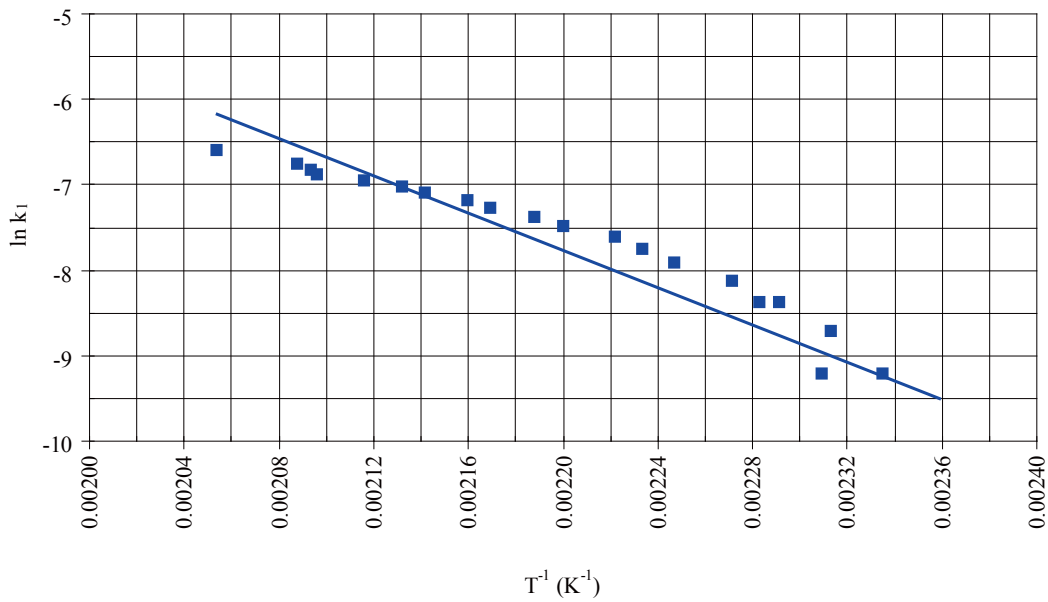

Figure 3
Arrhenius Plot for the Zero Order Model

3.2 First Order Model

Similarly, for the first order model, a plot of ln(1-x) and t was undertaken for 160 and 190°C, respectively, and it is shown in Fig. 4. It can be seen that k_1 = 0.003 and 0.002 at 160 and 190°C, respectively. Therefore, Arrhenius plot for the first order model can be made, shown in Fig. 5. The apparent activation energy (E_a) and the pre-exponential factor were found to be 105 kJ/mol and 1.57 x 109, respectively.

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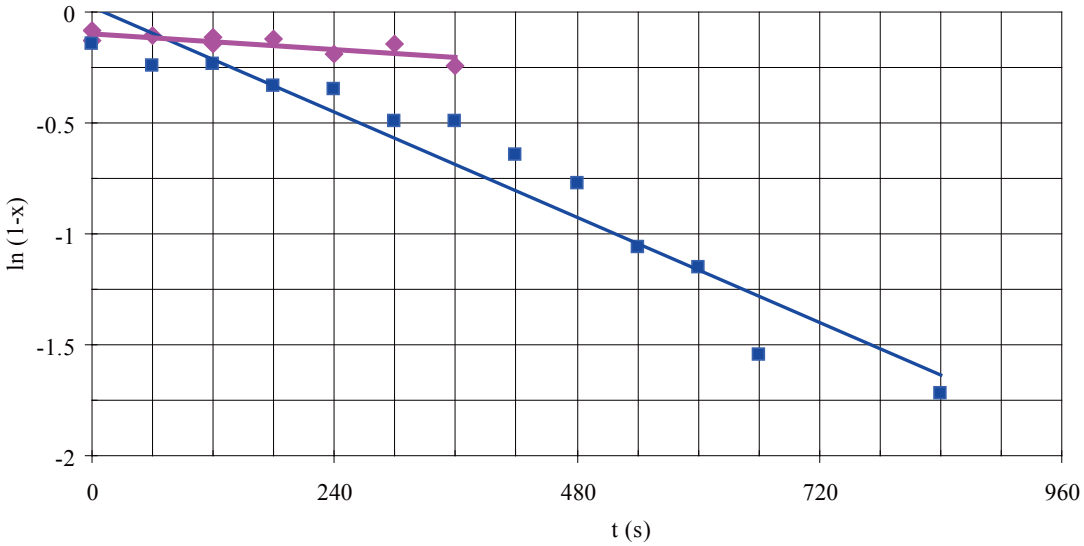

Figure 4
Plot of ln(1-x) against Time for the First Order Model

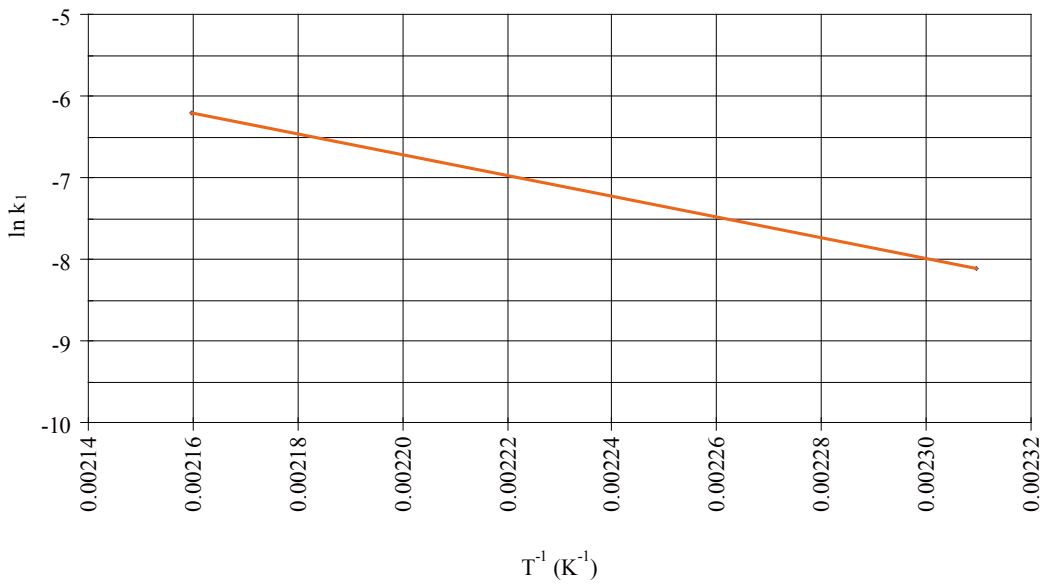

Figure 5
Arrhenius Plot for the First Order Model

3.3 Comparison with Literature

The kinetic data derived from the experiments are summarized in Tabs. 4 and 5. From the experiments carried out with palm oil at 150-200°C, two activation energies were estimated at 90.9 kJ/mol (from the zero order model) and at 105 kJ/mole (from the 1st order model). The values obtained were higher than those reported by Kusdiana and Saka (2001) with E_a = 38.4 kJ/mol for rapeseed oil at 200-270°C, and by He et al. (2007) with E_a = 11.2 kJ/mol for soybean oil at 210-230°C. But, they were comparable with Song et al. (2008)'s reported Ea of 105 kJ/mol for palm oil at 200-400°C. It was apparent that the E_a estimated by the 1st order model was in better agreement than the zero order model. It should be noted that Song et al. (2008) assumed their reaction rates to be second order model with respect to both oil and methanol concentrations. Such a high activation energy of 105 kJ/mol of this study may represent high energy barrier from the effect of the mass transfer limitation. The gradient of the energy curve was reduced as temperature increased. The effect of mass transfer limitation was minimized when oil and methanol were completely homogeneous.

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Table 4
Results of the Apparent Rate Constants (k_1)

Model	k_1 (s-1)	T (°C)	P (atm)
1st order model	0.0414	160	140-190
	0.1107	190	140-190
zero order model	0.0051	150	140-190
	0.0151	160	140-190
	0.0257	170	140-190
	0.0375	180	140-190
	0.0513	190	140-190
	0.0691	200	140-190

When comparing the apparent rate constants with those reported in literature, our values were rather high (see Fig. 6). This may be attributed to the effect of the proportionality constant (k_p) which was included in our k_1, as stated in eq. 9. If the proportionality constant was identified, the apparent rate constants would be lower. It should be noted that that the apparent rate constants of Dasari et al. (2003) were in similar pattern to our findings. This may be contributed to the fact that Dasari et al. (2003) carried out their experiments in glass reactors so that the catalytic effect of the reactor surface was eliminated while this study and others used metal reactors.

Table 5
Comparisons of Our Results with Literature

Reference	Kinetics model: $r = k_0 C_{TG}^m C_{MeOH}^n e^{-E_a/RT}$			
	m	n	k_0	E_a (kJ/mol)
This study (zero order)	0	0	5.45×10^8	88.9
This study (1st order)	1	0	1.62×10^5	56.1
Cheng et al. (2008)	1.5	0	12.45	28.9
Song et al. (2008)	0.96	1.05	4.30×10^5	105.3
Joelianingsih et al. (2008)	1	0	4.21	30.8
He et al. (2007)	1	0	1.94×10^{-3}	11.2
He et al. (2007)	1	0	170	56.0
Kusdiana and Saka (2001)	1	0	3.33	38.4[a]
Kusdiana and Saka (2001)	1	0	145	47.1[b]

C_{TG} = triglycerides concentration,
k_0 = pre-exponential factor,
R = universal gas constant = 8.314 J/mole.K,
(a) = for 200-270°C,
C_{MeOH} = methanol concentration,
E_a = activation energy,
T = absolute temperature,
(b) = for 300-487°C

CONCLUSION

Transesterification reaction of palm oil and methanol for biodiesel production under a subcritical condition was investigated at temperature of 150-200 °C and pressure of 140-190 atm. Relevant kinetic data were determined. The apparent activation energies were estimated to be 90.9 and 105 kJ/mol for the zero and fist order models, respectively. The rate equation that was best described the reaction was expressed by the first order model as

$$r = (1.57x10^9)e^{-\left[\frac{105404}{RT}\right]}C_{TG}$$

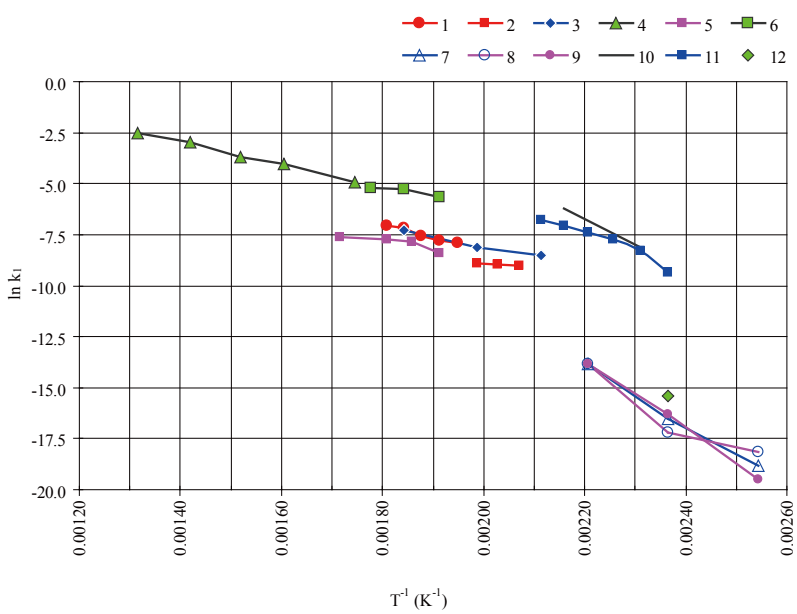

Figure 6
Comparison of Various Arrhenius Plots Between This Study and Those from the Literature

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(1) He et al. (2007): Soybean oil 240-280°C,
(2) He et al. (2007): Soybean oil 210-230°C,
(3) Kusdiana and Saka (2001): Rapeseed oil 200-270°C,
(4) Kusdiana and Saka (2001): Rapeseed oil 300-487°C,
(5) Cheng et al. (2008): Peanut oil 250-310°C,
(6) Joelianingsih et al. (2008): Palm oil 250-290°C,
(7) Dasari et al. (2003): Soybean oil,120-180°C, MeOH:oil=3:1,
(8) Dasari et al. (2003): Soybean oil,120-180°C, MeOH:oil=6:1,
(9) Dasari et al. (2003): Soybean oil,120-180°C, MeOH:oil=12:1,
(10) This work, first order: Palm oil 160-190°C,
(11) This work, zero order: Palm oil 150-200°C,
(12) Dasari et al. (2003): Soybean oil,120-180°C, MeOH:oil=6:1, added 25% biodiesel as co-solvent).

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