

CHAPTER 4

RESULTS AND DISCUSSION

The two-stage process (esterification followed by transesterification) was adopted for converting MCPO into biodiesel because of its high FFA contents (acid value around 8–14 %wt) and un-degumming. This chapter debates results, the kinetics and the modeling of the two-stage process of biodiesel production from MCPO, which was shown the methodology in the previous chapter, as follows:

4.1 Pre-Experiment for Two-Stage Process of Biodiesel Production from MCPO

Results of the pre-experiment on the two-stage process of MCPO processing (the amount of methanol, the speed of stirrer, the amount of catalyst in esterification and in transesterification process, the water and H₂SO₄ separation, purification methods, and the reaction time) are presented and discussed below. Every condition of the pre-experiment was fixed before starting the experiment on the two-stage process of MCPO.

4.1.1 The Amount of Methanol (MeOH)

MeOH, the main reagent, was used in esterification (FFA treatment) and in transesterification (TG, DG, and MG conversion). The excess methanol (a 20:1 molar ratio of methanol to FFA) was used under the condition as shown in Table 3.1. Then the remaining MeOH in the unwashed MCPO solution was analyzed by the Science Equipment Centre, PSU, Hat Yai, Songkhla, Thailand as shown in Figure E.1 of APPENDIX E. It was found that a 16:1 molar ratio of methanol to FFA was used in the first-stage. This molar ratio probably included the loss of volatile methanol, thus the optimum methanol molar ratio, which used in the first stage might be varied from a 4:1 to 16:1 molar ratio of methanol to FFA.

Equation 2.13 indicated that a 3:1 molar ratio of methanol to TG was needed to complete transesterification stoichiometrically. Previous works found that transesterification required a higher ratio to achieve the reaction. Therefore, a 6:1 molar ratio of methanol to TG was used to convert vegetable oil into biodiesel by transesterification (Ma, *et al.*, 1998). Although, some methanol still remained in the first stage of the two-stage process, it was not enough for converting internal compounds of the first stage solution into biodiesel. Therefore, the range of the molar ratio of MeOH to TG for converting TG, DG, and MG into biodiesel of this thesis was in the range of a 3:1 to 9:1 methanol to TG molar ratio.

4.1.2 The Speed of Stirrer

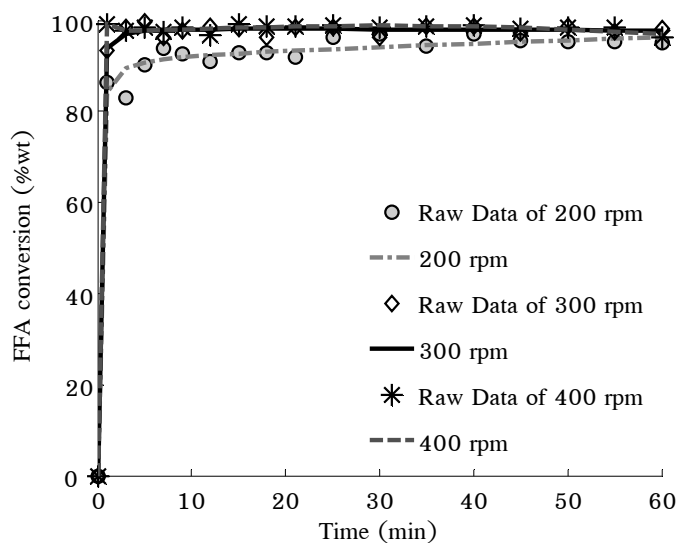


Figure 4.1 The effect of stirring speed on FFA conversion in MCPO by using a 20:1 molar ratio of methanol to FFA at a temperature of 60 degree Celsius, catalyzed by 10 %wt H_2SO_4 of FFA

The reacting mixture formed two immiscible layers of oil and methanol. Thorough the mixing of two layers was necessary for the reaction to proceed (Thaweensinsoha *et al.*, 2005 and Thaweensinsoha, 2006). When the esterification reaction was performed for a long time, there were no significant differences due to the variation of the stirrer speed for reducing FFA over the range 200, 300, and 400 rpm

($N_{RE} = 1,680, 2,520, \text{ and } 3,360$, respectively). However, the conversion of FFA rose to more than 90 %wt in 30 seconds when the speed of the stirrer of this experiment was 300 and 400 rpm as shown in Figure 4.1. There was not much effect when the speed of stirrer was increased from 300 rpm to 400 rpm. Therefore, a 300 rpm speed of stirrer was found to be suitable for using to reduce FFA in MCPO.

As the second stage, transesterification was investigated by converting the first stage production to biodiesel and glycerol. May (2004) found that there was no significant difference when the speed of the stirrer for producing biodiesel by transesterification was varied from 150 to 600 rpm. In addition, a speed of stirrer at 300 rpm ($N_{RE} = 6,200$) was preferred to produce biodiesel from soybean oil by Nouredini, *et al.* (1997). Accordingly, the speed of stirrer of 300 rpm (N_{RE} (Approximately) = 2,520) was also used in the transesterification experiments though the Reynolds Number of the stirring speed in this experiment was less than the Reynolds Number of Nouredini's the stirring speed.

4.1.3 The Amount of Catalyst

H_2SO_4 and NaOH were used as catalyst in the two-stage process of MCPO processing. Various concentrations of them were investigated before fixing for all esterification and transesterification reactions.

4.1.3.1 The Amount of H_2SO_4

H_2SO_4 was used as catalyst in the esterification and varied as 5, 10, and 15%wt of FFA. Effects of varying % H_2SO_4 on FFA reduction were investigated. After analysing solutions with TLC/FID, it was found that both 10 and 15% wt of FFA on a 10:1 molar ratio of MeOH to FFA at 60 degree Celsius could convert FFA into ME more than 90 %wt in a first minute as shown in Figure 4.2. There was not much effect when the catalyst concentration was increased from 10 to 15 %wt of oil.

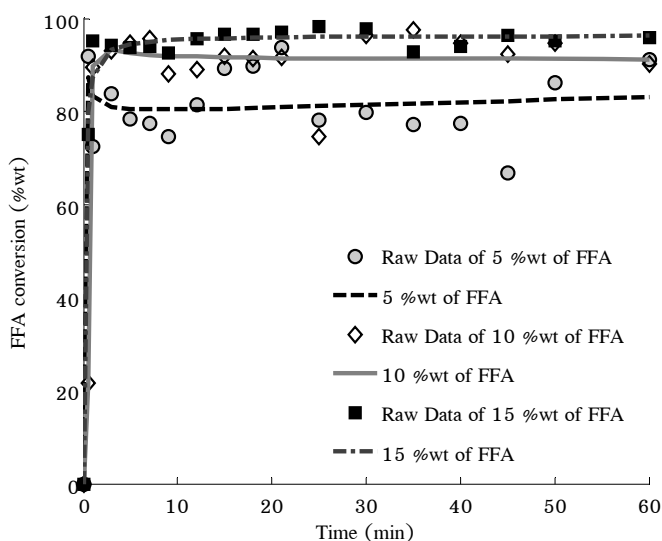


Figure 4.2 The effect of the amount of H_2SO_4 on FFA conversion in MCPO by using a 10:1 molar ratio of methanol to FFA, at temperature of 60 degree Celsius, and a speed of stirrer of 300 rpm

However, raising the amount of catalyst to 15 %wt of FFA was comparable with 10 %wt of FFA. Consequently, 10 %wt of FFA was found to be suitable for reducing FFA (8–14 %wt) in MCPO in the methodology of esterification.

4.1.3.2 The Amount of NaOH

Concentrations of NaOH at 0.3, 0.6, and 0.9 %wt of TG were used as catalyst in transesterification. Analytical values (ME) from TLC/FID indicated that both of 0.6 and 0.9 %wt of TG under a 6:1 molar ratio of MeOH to TG at 60 degree Celsius could give 95 %wt conversion as the oily solution from the first-stage process into ME in 30 seconds as shown in Figure 4.3. It was no significant difference when catalyst was increased from 0.6 to 0.9 %wt of TG. Consequently, the amount of NaOH at 0.6 %wt of TG was found to be suitable for converting the oily solution into methyl ester.

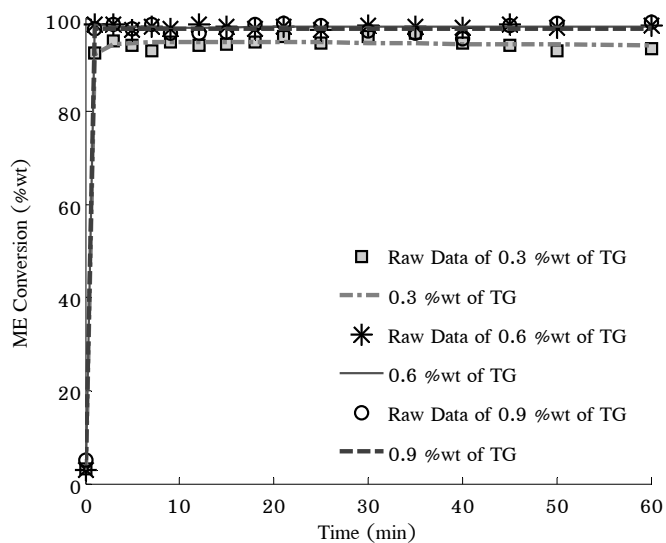


Figure 4.3 The effect of the amount of NaOH on ME conversion in MCPO by using a 6:1 molar ratio of methanol to TG, at temperature of 60 degree Celsius, and a speed of stirrer of 300 rpm

4.1.4 Water and H_2SO_4 Separation

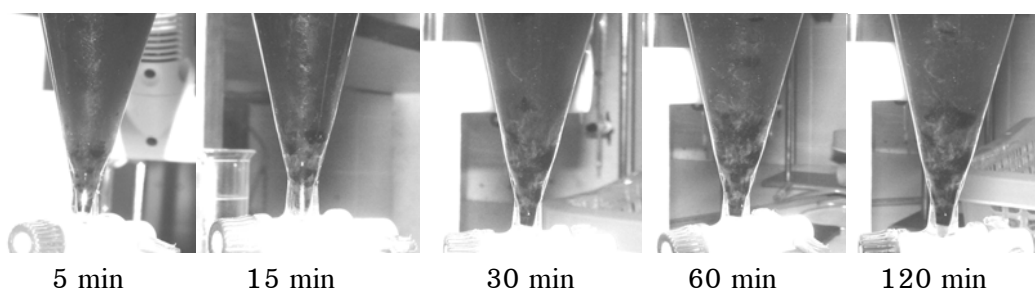


Figure 4.4 Standing time for water and H_2SO_4 separation of first stage process

Usually, final products of esterification were ester and water as shown in Equation 2.11. However, water caused gel in the transesterification stage. In addition, a lot of NaOH was required to achieve neutrality H_2SO_4 in the first stage solution (Thaweensinopha, 2006). Before starting the second-stage process as shown in Figure 4.4, when the reaction in the first-stage process reached completion, it was allowed to

stand to separate water, H_2SO_4 and Some MeOH (black solution), which also dissolved in the aqueous solution. It was found that acid values of each solution were reduced when it was left over a long period. A separating time of about 2 hours was allowed to separate impurities (approximately 5 %vol) from the first-stage solution.

4.1.5 Purification Methods

The reaction to produce ester is reversible. Therefore, when ester was treated with acid or base in aqueous solution, a molecule of ester could be converted to alcohol and an acid compound (hydrolysis) as shown in Equation 2.1 (Brown *et al.*, 2003). In addition, excess methanol in samples interfered with the signal of TLC/FID (Sherma, 2000). Therefore, each sample was purified by reducing impurities in samples before analyzing substances in samples as follows

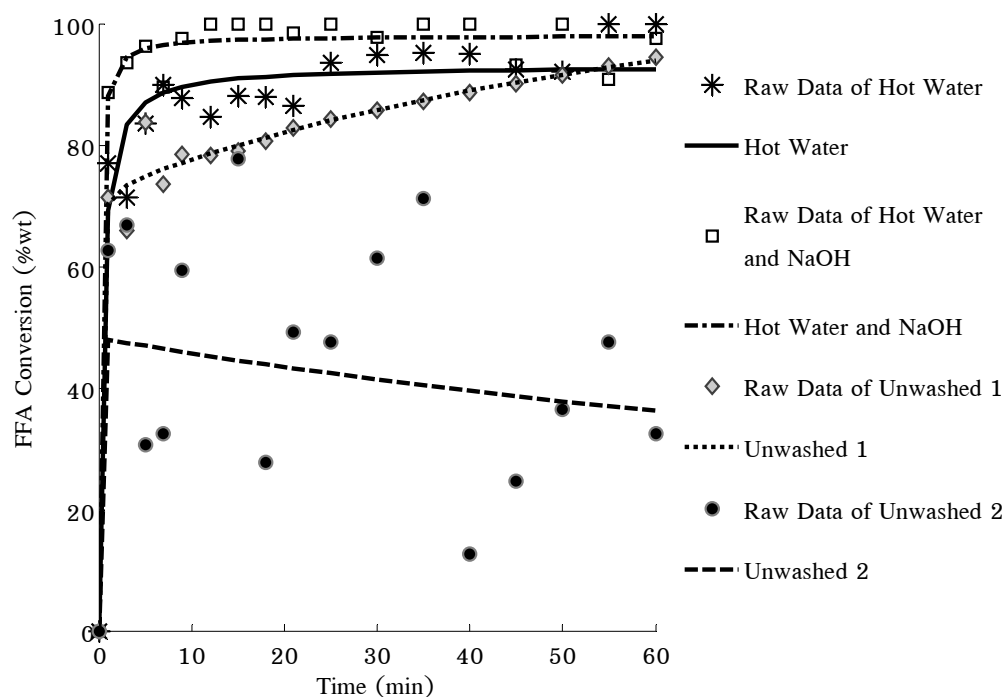


Figure 4.5 The effect of purification method on FFA conversion in MCPO under a 10:1 molar ratio of methanol to FFA, at temperature of 60 degree Celsius, a speed of stirrer of 300 rpm, and catalyzed by 5 %wt H_2SO_4 of FFA

The washing method was selected to purify samples in this experiment because it was an easy method for cleaning a small amount of sample. Results of this experiment indicated that there were significant differences in methods that were used to clean the first-stage solution as shown in Figure 4.5. When solution was washed with hot water and NaOH (strong base), 95 %wt of FFA in solution was converted to soap (Ma and Hanna, 1998). On the other hand, Analyzed results of unwashed solution were unsteady because of the presence of a highly volatile substance (MeOH) in unwashed solutions (Sherma, 2000). For these reasons, every sample from the first stage must be cleaned with hot water only. Moreover, this washing method (only hot water) was preferred for purifying the sample from transesterification (Noureddini, *et al.*, 1997 and Veljković, *et al.*, 2006). Consequently, the washing method with hot water was fixed for cleaning samples of the two-stage process with 250 ml of hot water being used for 3 times before analyzing substances in each sample with the analytical instrument.

4.1.6 Reaction Time

Although the conversion rate of esterification and transesterification reactions increased with the reaction time, eventually, the equilibrium was reached. However, the reaction, which was used to study chemical kinetics, should not be carried out over a long time. Therefore, the reaction was carried out for a short time only.

The pre-experiment showed the rapid conversion of FFA (esterification) and ME (transesterification) in the first minute until an equilibrium was reached as shown Figure 4.1, 4.2, and 4.3. Both of the reactions in the two-stage process should be carried out for a time in order to reach the equilibrium. Therefore, 20 minutes were set as the preferring reaction time in this thesis because it was enough to reach the equilibrium and this reaction time was also preferred to produce biodiesel in experimental of Foon, *et al.* (2004).

4.2 Two-Stage Process of Biodiesel Production from MCPO

The characteristic concentration of substances in esterification and transesterification, and the effect of molar ratio of methanol to oil and the reaction temperature on the two-stage process are discussed as follows:

4.2.1 Esterification

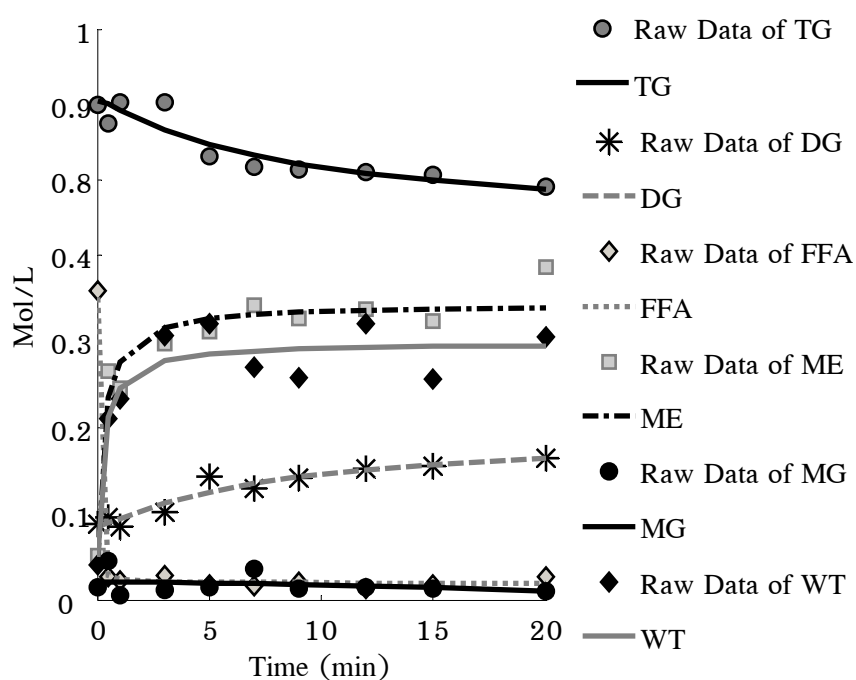


Figure 4.6 Variations of the reaction mixture composition during esterification of MCPO by using a 10 %wt H_2SO_4 of FFA and a 10:1 molar ratio of methanol to FFA at 60 degree Celsius

Reactants and products concentration for the treatment of FFA in MCPO under conditions of a 10 %wt H_2SO_4 of FFA and a 10:1 molar ratio of methanol to FFA at 60 degree Celsius were shown in Figure 4.6. It could be seen that the FFA concentration decreased in the first 30 seconds and then reduced until equilibrium was reached. The concentration of triglyceride (TG) and monoglyceride (MG) reduced slowly but diglyceride (DG) increased slightly. The rapid formation tendency of methyl ester (ME) and water

(WT) as shown in a sigmoid curve were observed within the first minute and they then increased steadily, primarily due to esterification of FFA.

As for the description of the typical concentration curve of esterification, as well as esterification of the FFA, some MG, DG, and TG were also transesterified. Nevertheless, the comparison between the acid-catalyzed transesterification reaction in Figure 4.6 and the alkali-catalyzed transesterification reaction in Figure 4.7 indicated that acid-catalyzed transesterification had lower significant differences in TG, DG, and MG converting than alkali-catalyzed transesterification. Consequently, in the first stage, the decrease in the FFA content and the rise in ME and WT content were only investigated.

4.2.2 Transesterification

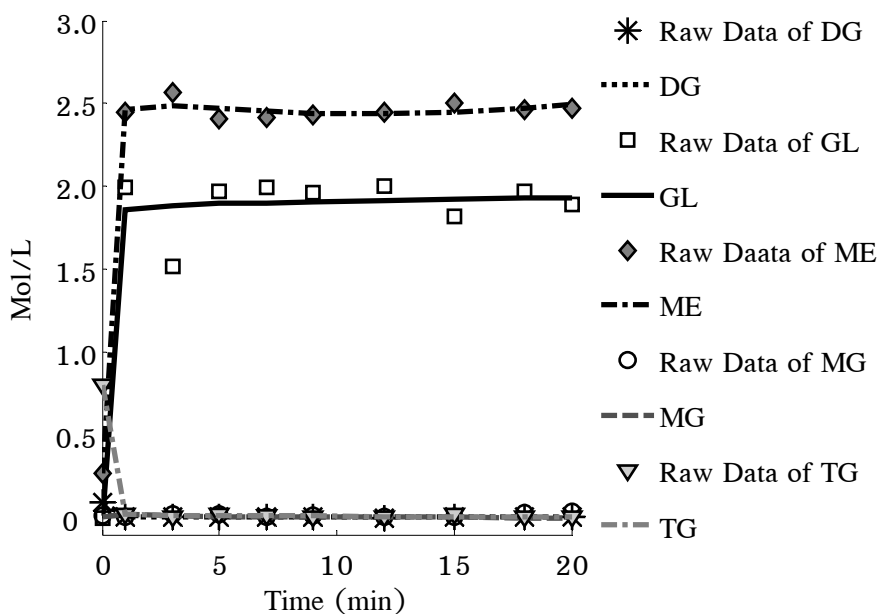


Figure 4.7 Variations of the reaction mixture composition with transesterification of MCPO by using a 0.6 %wt NaOH of TG (a 0.5 %wt NaOH of oil) and a 6:1 molar ratio of methanol to TG at 60 degree Celsius

Figure 4.7 showed the concentration of reactants and products in transesterification of MCPO esterified to reduce FFA. Conditions used a 0.6 %wt NaOH of TG and a 6:1 molar ratio of methanol to TG at 60 degree Celsius. ME and GL concentrations followed sigmoid curves. The rate of the reaction was initially high and then lowered until equilibrium was reached. Both of ME and GL concentrations depend on DG and MG concentrations. On the other hand, in Figure 4.7 that is some evidence of the mass transfer reactions controlling the mechanism of TG disappearance. Subsequently, the TG concentration was reducing within the first minute until equilibrium was reached. DG and MG concentration were also increasing in the first minute. After that their concentrations were decreasing until equilibrium (Foon, et al., 2004).

4.2.3 The Effect of Methanol Ratio on Two-Stage Process

The MeOH ratios are parameters, which have an effect on both of reaction rate, the FFA and the TG conversion. Therefore, the effect of the molar ratio of MeOH to FFA (esterification) and to TG (transesterification) (varied according to the reason in Topic 4.1.1) were investigated by focusing on the objective of this thesis, which was to convert FFA more than 90 % and ME more than 96.5 % under the lower methanol ratio.

Therefore, the FFA conversion under a 4:1, 10:1, and 16:1 molar ratio of methanol to FFA and temperature at 55, 60, and 65 degree Celsius was investigated by fixing the amount of H_2SO_4 , and the speed of stirrer as shown in Figure 4.8. Results indicated that the molar ratio of methanol to FFA influenced on the rate of reaction. In addition, FFA was converted to more than 90 % for high methanol ratios in a half minute. No significant differences were found in FFA reduction on the molar ratio of methanol to FFA of 10:1 and 16:1 and though low methanol ratios were also considered to reduce 8–14 %wt of FFA which are contained in MCPO. Therefore, a 10:1 molar ratio of methanol to FFA was used to decrease FFA (8–14%wt) held in MCPO before starting to produce biodiesel by transesterification.

In addition, the effect of MeOH ratios on the ME conversion in transesterification were also investigated by varying the molar ratio of MeOH to TG at 3:1, 6:1, and 9:1 and at temperature of 55, 60, and 65 degree Celsius with 0.6 %wt NaOH

of TG, and the speed of stirrer at 300 rpm as shown in Figure 4.9. Results indicated that the rate of the reaction was also influenced with the methanol molar ratio. For high methanol ratios, ME were converted to more than 97 % in 5 minutes. Although there were no significant differences in ME that increase when the molar ratio of methanol to TG was changed from 6:1 to 9:1, low methanol ratio (6:1) was also considered to produce biodiesel in this thesis.

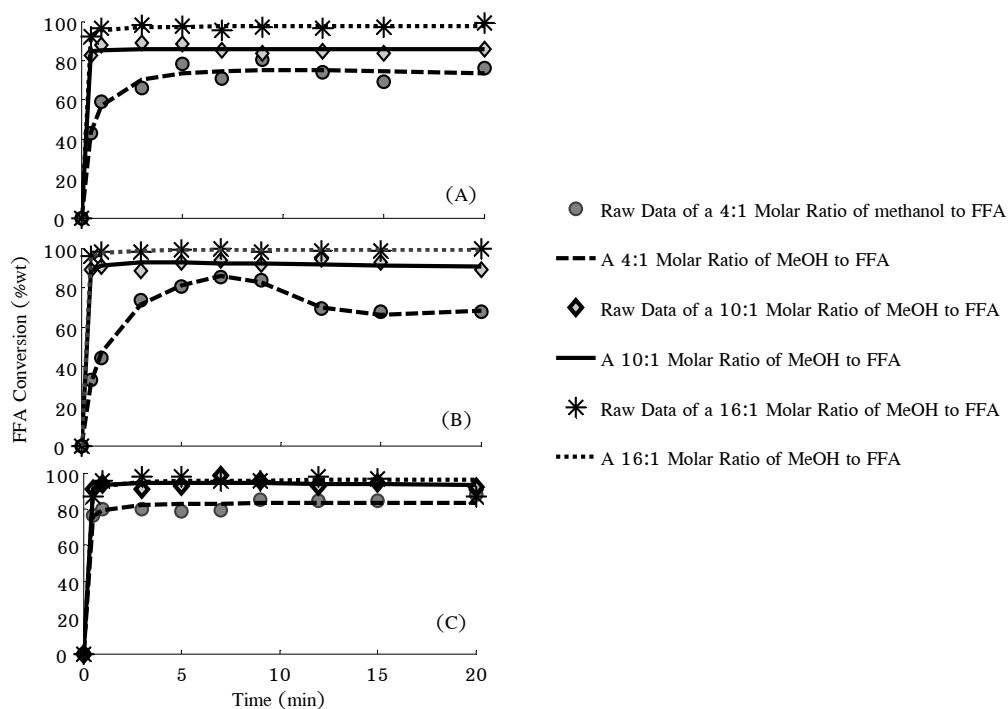


Figure 4.8 The effect of molar ratio of methanol to FFA on FFA concentration in MCPO using 10 %wt H_2SO_4 of FFA and a stirring rate of 300 rpm at 55 (A), 60 (B), 65 (C) degree Celsius, respectively

4.2.4 The Effect of Temperature on Two-Stage Process

Temperature had a major influence on the reaction rate of biodiesel production. Normally, the reaction is carried out nearly at the boiling point of methanol (60–65 degree Celsius) at the atmosphere pressure. Therefore, reaction temperatures at 55, 60, and 65 degree Celsius were used to produce biodiesel using the two-stage process.

According to the thesis's objective, the lower temperature was considered to convert FFA and ME more than 90 % and 96.5 %, respectively. Therefore, the FFA conversion by esterification of MCPO to more than 90 %wt was investigated, which were obtained for 55, 60, and 65 degree Celsius at a molar ratio of methanol to FFA of 10:1 and 16:1 as shown in Figure 4.8. In addition, ME in the subsequent transesterification to more than 97 %wt was also investigated. The conversion results as shown in Figure 4.9 were obtained for 55, 60, and 65 degree Celsius at a molar ratio of methanol to TG of 6:1 and 9:1. The time required for reducing FFA to 1 %wt was 30 seconds and for ME 97 %wt conversion was observed in 5 minutes when the temperature increased from 55 to 60 and 65 degree Celsius. Even though there were no significant differences in the reaction time when the temperature was changed (from 60 to 65 degree Celsius), it had an influence on the reaction rate of the two-stage process. As a result, the reaction temperature of 60 degree Celsius was sufficient for converting high FFA (8–14 %wt) and producing ME to more than 90% and 97%, respectively.

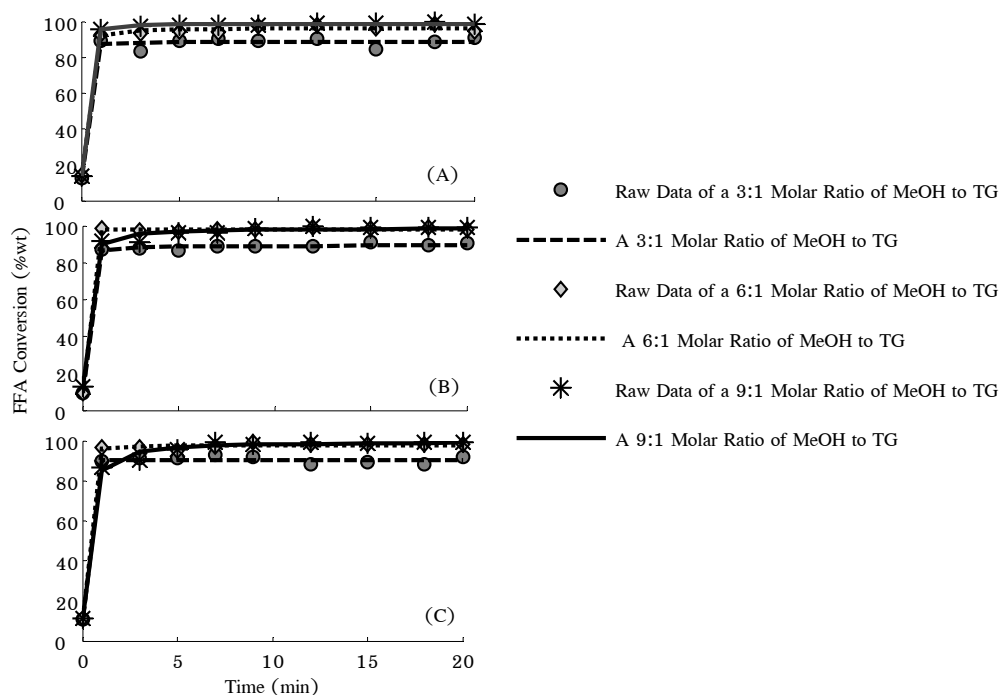


Figure 4.9 The effect of molar ratio of methanol to TG on ME conversion in MCPO using 0.6 %wt NaOH of TG and a stirring rate of 300 rpm at 55 (A), 60 (B), 65 (C) degree Celsius, respectively

4.2.5 Properties of Methyl Ester from MCPO

The biodiesel production from un-degummed and un-deacidified MCPO was carried out with the scale-up bath reactor (80 liter/ bath) under the first-stage process condition of a 10:1 molar ratio of methanol to FFA with 10 %wt H₂SO₄ base on FFA followed by the second-stage process condition of a 6:1 molar ratio of methanol to TG by using a 0.6 %wt NaOH as a catalyst. In addition, both of reactions were fixed the reaction temperature at 60 degree Celsius and the speed of stirrer at 150 rpm, which yielded 95 % conversion of MCPO, had physical and chemical properties given in Table 4.1 and Figure E.5-E.12 of APPENDIX E, which was analyzed by the Science Equipment Centre, PSU, Hat Yai, Songkhla, Thailand.

Table 4.1 The Comparison between properties of methyl ester from MCPO and some requirements of biodiesel qualities and quantities in Thailand

Parameter	Corresponding Value (Biodiesel for Agricultural Engine)	Corresponding Value (Commercial Biodiesel)	CPOME*
Methyl Ester (% wt)	-	≥ 96.5	93.2
Density at 15 °C (kg/m ³)	≥ 860 and ≤ 900	≥ 860 and ≤ 900	878.7
Viscosity at 40 °C (cSt)	≥ 1.9 and ≤ 8	≥ 3.5 and ≤ 5	5.28
Flash point (°C)	≥ 120	≥120	167
Sulphur (%wt)	≤ 0.0015	≤ 0.0010	0.0004
Sulfated Ash (%wt)	≤ 0.02	≤ 0.02	0.0050
Water and Sediment (%vol)	≤ 0.2	≤ 0.050	<0.05
Copper Strip Corrosion	≤ Number 3	≤ Number 1	Number 1
Acid Value (mg KOH/g)	≤ 0.80	≤ 0.50	0.25
Free Glycerin (%wt)	≤ 0.02	≤ 0.02	-
Total Glycerin (%wt)	≤ 1.50	≤ 0.25	1.06

*CPOME is Mixed Crude Palm Oil Methyl Ester

The biodiesel production from MCPO by the two-stage process succeeded in the objectives of producing biodiesel with acceptable qualities and in adequate quantities for using in an agricultural engine. It also met some of requirements for commercial biodiesel in Thailand. The viscosity did not reach in the standard of the commercial biodiesel because the remaining glycerin composed more than 0.25 %wt and the methyl

ester content was less than 96.5 %wt. In addition, although the optimal condition of this thesis was used to produce biodiesel from MCPO by the two-stage process, the difference in the physical of biodiesel reactor was also affected on the biodiesel production. Therefore, MCPO could be converted to biodiesel less than 96.5%wt. Consequently, if it was required to reach the standard of the commercial biodiesel, the process should be further improved. However, it had been shown that un-degummed and un-deacidified MCPO could be used as a raw material for producing biodiesel using the two-stage process and the product could be used as a fuel for agricultural engines.

4.3 Kinetics of Two-Stage Process

4.3.1 Rate Coefficients and Reaction Orders of Two-Stage Process

Beginning, when rate laws of esterification in equation 2.12 was used to fit into the raw data by varying the overall reaction order in the range of first to fourth order, the results as shown in Equation 4.1 indicated that pseudo-first order with respect to disappearances of FFA and MeOH, and appearances of ME and WT in half order was conform to the raw data more than another one.

$$\frac{d[\text{FFA}]}{dt} = -k_1[\text{FFA}]^{0.5}[\text{A}]^{0.5} + k_2[\text{E}]^{0.5}[\text{WT}]^{0.5} \quad (4.1)$$

After that, Rate laws of the two-stage process in equation 2.14 and 4.1, and raw data in each experiment were used to find rate coefficients and reaction rates of the two-stage process by using curve-fitting tool of MATLAB7 as shown in Table F.1 and F.2 of APPENDIX G. Fitting results indicated that the determination coefficient (R^2) of MATLAB7 was in the range of 0.845 to 0.992. After that, rate coefficients and reaction orders of the two-stage process were calculated by using the Runge-Kutta method and then it compared with raw data as shown in Table F.3 of APPENDIX G. Results did not conform to raw data because it had high the % data error mean (μ) and the standard deviation (σ) in categories of FFA, MeOH, ME, and WT concentration (esterification), or of TG, DG, MG, ME, GL, and MeOH concentration (transesterification) in each time.

Table 4.2 Rate coefficients for esterification reaction

Conditions for Reducing FFA in MCPO (MeOH: FFA; Temperature) (Molar Ratios; Degree Celsius)	Rate Coefficients of Esterification (min ⁻¹)			
	k_1	k_2	μ	σ
4:1;55	1.034	0.918	15	14
4:1;60	1.079	0.963	35	41
4:1;65	1.326	0.975	28	33
10:1;55	0.840	0.680	37	43
10:1;60	1.340	0.682	22	21
10:1;65	1.899	0.684	37	32
16:1;55	0.858	0.379	25	43
16:1;60	0.892	0.380	76	155
16:1;65	0.894	0.455	19	27

Table 4.3 Rate coefficients for transesterification reaction

Conditions for Producing CPOME (MeOH: TG; Temperature) (Molar Ratios; Degree Celsius)	Rate Coefficients of Transesterification (L/mol.min)							
	k_3	k_4	k_5	k_6	k_7	k_8	μ	σ
3:1;55	5.010	4.594	4.442	1.676	5.074	0.080	35	78
3:1;60	5.125	4.972	5.892	1.790	5.524	0.081	17	17
3:1;65	5.160	5.200	5.930	1.799	5.570	0.082	32	47
6:1;55	2.579	0.020	0.600	0.101	0.900	0.021	59	107
6:1;60	2.600	0.248	1.186	0.227	2.303	0.022	41	42
6:1;65	2.620	0.700	1.210	0.400	2.360	0.028	44	80
9:1;55	1.990	0.247	1.130	0.072	0.820	0.014	37	40
9:1;60	2.000	0.510	1.144	0.077	0.824	0.016	38	41
9:1;65	2.119	0.524	1.248	0.082	0.829	0.027	33	36

Subsequently, rate coefficients and reaction orders of the two-stage process were calculated by the Runge-Kutta method again until their rate coefficients and reaction orders had reduced the % data error mean and the standard deviation in categories as shown the results in Table 4.2 and 4.3 (whole population: $\mu = 35$, $\sigma = 62$). Lastly, rate

coefficients and reaction orders of the two-stage process were used to calculate activation energies and to create the Two-Stage Process Modeling.

4.3.2 Activation Energies (E_a) of Two-Stage Process

Activation energy had been equated with a minimum energy that must be possessed by reacting molecules before the reaction will occur (Fogler, 1999). Activation energies were approximated by experimental values of reaction rate coefficients and temperature, which were curve-fitted into the relationship between logarithm k and $1/T$ such as Figure 4.10 and 4.11. The slope of curve was estimated activation energies as shown results in Table 4.4. The results indicated that forward reactions and some backward reactions of the two-stage process had low activation energies except for esterification under a 4:1 and 10:1 molar ratio of methanol to FFA, transesterification under a 3:1 molar ratio of methanol to TG for converting DG to MG and MG to GL and mostly backward reactions had high activation energies.

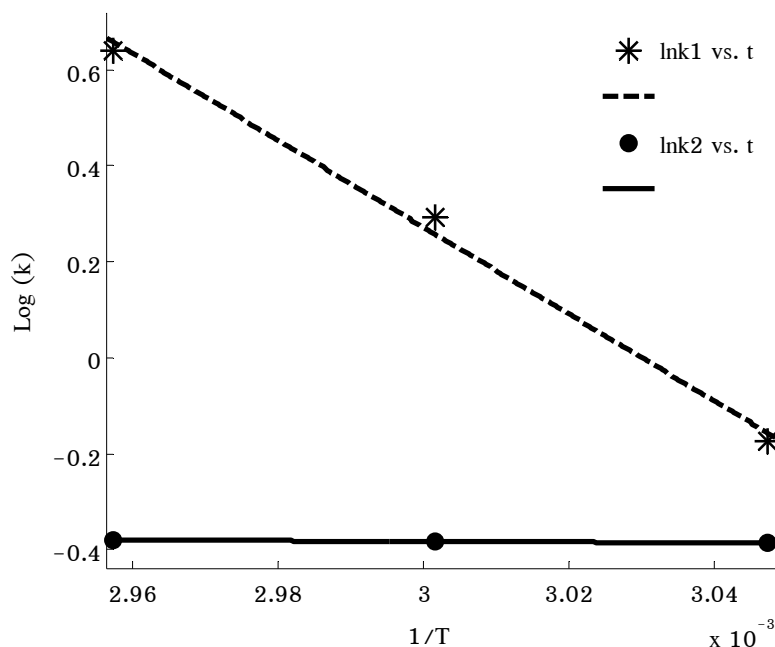


Figure 4.10 The temperature dependency of reaction rate coefficients of esterification at a 10:1 molar ratio of methanol to FFA

On the larger activation energy, the temperature was more sensitive to the rate of reaction (Fogler, 1999) otherwise the rate coefficients were rapidly increasing when the reaction temperature was rising (Boonamnuaivitaya, 2001) as shown the sample in Figure 4.10 and 4.11. In addition, low methanol ratio was used in some conditions of the two stage process that might cause for using high activation energies to convert reactants into products. On the hand other, activation energies of some reactions in the two-stage process were very low because reactions were reacted very quickly. Moreover, the temperature was not sensitive to the rate of reaction.

Although the reaction was rapidly increased and different raw material was used in this investigation, activation energies involved in transesterification (under the condition of a 6:1 molar ratio of methanol to TG) was consistent with experimental values of activation energies of Nouredini et al. (1997) for the reactions in transesterification of soybean oil reported to be in the range of 6,400 to 20,000 cal/mol based on the Arrhenius equation.

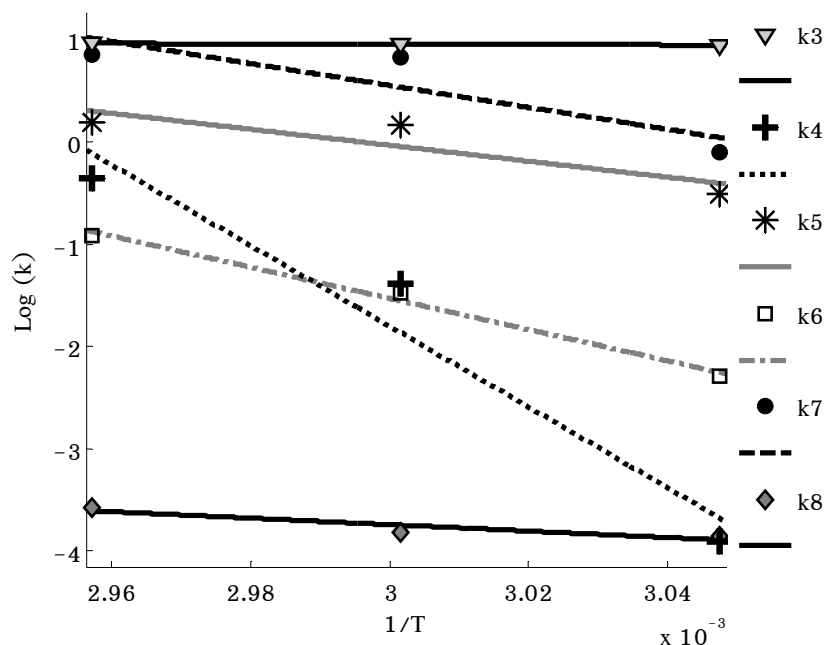


Figure 4.11 The temperature dependency of the reaction rate coefficients of transesterification at a 6:1 molar ratio of methanol to TG

Table 4.4 Activation energies (cal/mol) of the two-stage process at different molar ratios of methanol to oil

Esterification	Activation Energy at Different Molar Ratio of Methanol to FFA (cal/mol)		
	4:1	10:1	16:1
FFA-WT	5,466	17,997	910
WT-FFA	1,332	129	3,519
Transesterification	Activation Energy at Different Molar Ratio of Methanol to TG (cal/mol)		
	3:1	6:1	9:1
TG-DG	283	814	1,379
DG-TG	1,188	1,181	16,659
DG-MG	2,780	9,150	2,182
MG-DG	681	21,142	2,867
MG-GL	897	1,425	241
GL-MG	236	4,020	14,438

4.4 Two-Stage Process Modeling

Two-Stage Process Modeling in Figure 4.12 to 4.24 was used to predict the FFA conversion from MCPO under condition of a 4:1, 10:1, and 16:1 molar ratio of methanol to FFA at 50, 60, and 65 degree Celsius and the methyl ester conversion from the first stage solution under the condition of a 3:1, 6:1, and 9:1 molar ratio of methanol to TG at 50, 60, and 65 degree Celsius.

This model consisted of three parts: the methodology for producing biodiesel by the two-stage process as shown in Figure 4.12 and Figure 4.13, the prediction of the FFA conversion in esterification as shown in Figure 4.13–4.19, and the prediction of the ME conversion in transesterification as shown in Figure 4.20–4.24, which were showed the instruction as follows:

The first instruction for using this model to predicted the FFA reduction in MCPO and the ME production was to fill %wt initial concentrations of substances (FFA, ME, and WT) in esterification part (Figure 4.13). Subsequently, this model converted %wt concentrations of substances into mol/L (Figure 4.14). After that, it showed the number of conditions for reducing FFA in MCPO (Figure 4.15) and then the condition number was selected and the reaction time (min) was filled into the blank (Figure 4.15). Next, the model computed and showed table and curves of the substances concentration (FFA, ME, and WT) in mol/L. Afterward, final outputs were converted the mol/L concentration of substances into %wt (Figure 4.16 and 4.17). If the condition number was not optimization, the model showed the number of the optimized condition and then input it in this model. (Figure 4.17) Subsequently, the model computed and showed table and curves of the substance concentration (FFA, ME, and WT) in mol/L and final output were converted the mol/L concentration of substances into %wt, again (Figure 4.18 and 4.19).

ME production in the optimization condition of esterification part was used in transesterification part. Subsequently, %wt initial concentrations of TG, DG, MG, and GL were filled in the blank (Figure 4.20). After that, they were converted the unit into mol/L by the calculation of this model (Figure 4.21). Next, the number of conditions for producing ME from MCPO was showed and selected by filling in the blank (Figure 4.22). Next, the reaction time was filled in the blank (Figure 4.22). Afterward, the model computed and showed table and curves of TG, DG, MG, ME, and GL concentration in mol/L (Figure 4.23). Finally, final outputs were converted the mol/L concentration of substances into %wt (Figure 4.24).

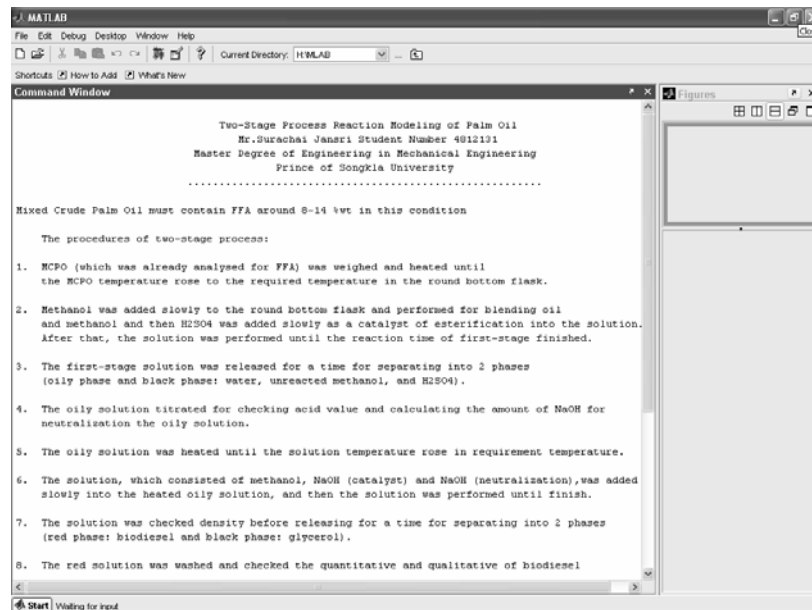


Figure 4.12 The procedure used in the two-stage process part

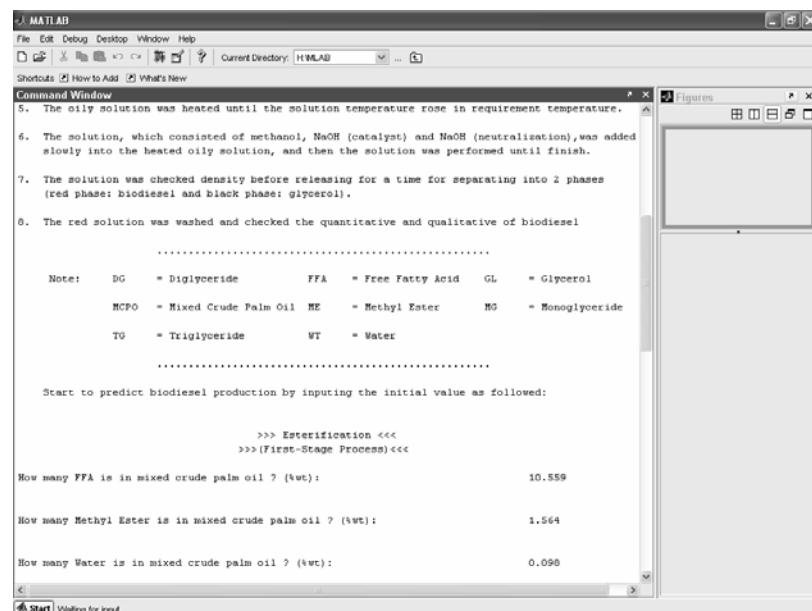


Figure 4.13 Inputting initial concentrations of substances (FFA, ME, and WT) in %wt

```

MATLAB
File Edit Debug Desktop Window Help
Current Directory: H:\MLAB
Shortcuts How to Add What's New
Command Window
>>> (First-Stage Process) <<<

How many FFA is in mixed crude palm oil ? (%wt):          10.559

How many Methyl Ester is in mixed crude palm oil ? (%wt):  1.564

How many Water is in mixed crude palm oil ? (%wt):         0.098

--- Unit Conversion ---

FFAO =
    0.3584
(mol/L)

MEO =
    0.0505
(mol/L)

WTO =
    0.0499
(mol/L)

```

Figure 4.14 Unit conversions for concentration in the esterification reaction (%wt to mol/L)

```

MATLAB
File Edit Debug Desktop Window Help
Current Directory: H:\MLAB
Shortcuts How to Add What's New
Command Window
(mol/L)

The condition for reducing FFA in MCPO

11 = a 4:1 molar ratio of methanol to FFA at 55 degree Celcius
12 = a 4:1 molar ratio of methanol to FFA at 60 degree Celcius
13 = a 4:1 molar ratio of methanol to FFA at 65 degree Celcius
14 = a 10:1 molar ratio of methanol to FFA at 55 degree Celcius
15 = a 10:1 molar ratio of methanol to FFA at 60 degree Celcius
16 = a 10:1 molar ratio of methanol to FFA at 65 degree Celcius
17 = a 16:1 molar ratio of methanol to FFA at 55 degree Celcius
18 = a 16:1 molar ratio of methanol to FFA at 60 degree Celcius
19 = a 16:1 molar ratio of methanol to FFA at 65 degree Celcius

Note:

a 4:1 molar ratio of methanol to FFA = a 1:1 molar ratio of methanol to oil
a 10:1 molar ratio of methanol to FFA = a 2.5:1 molar ratio of methanol to oil
a 16:1 molar ratio of methanol to FFA = a 3.5:1 molar ratio of methanol to oil

What is the condition which was used to reduce FFA in MCPO ? (input number)  11

How long of the reaction time is used to reduce FFA ? (min):                20

```

Figure 4.15 Conditions for reducing FFA concentration in MCPO

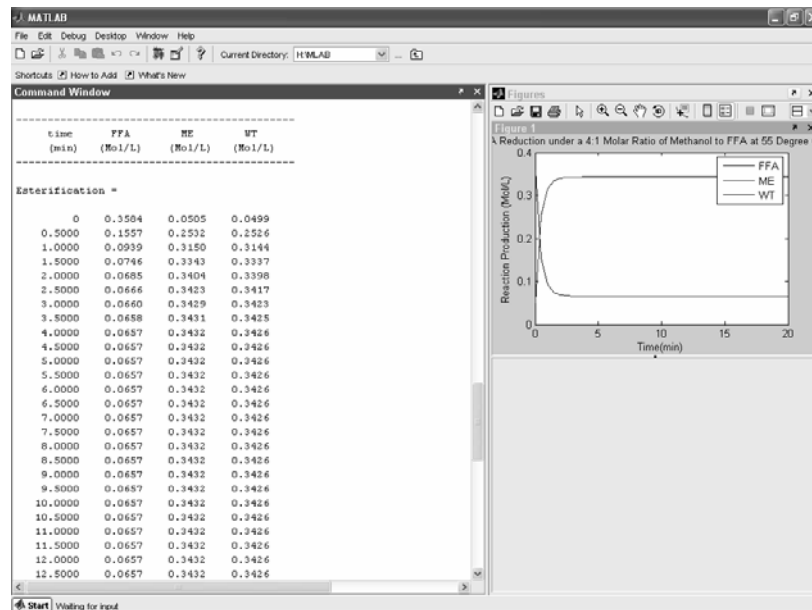


Figure 4.16 The table and curves of the component concentrations (FFA, ME, and WT) in mol/L

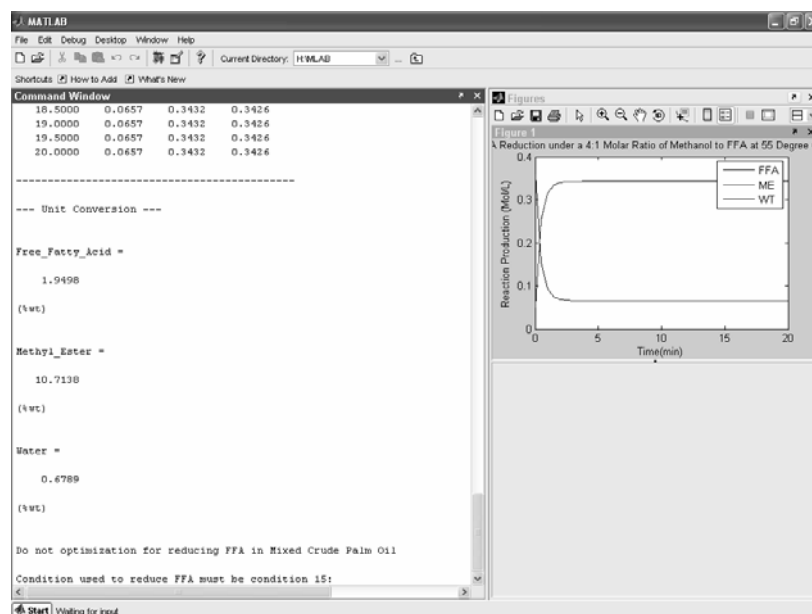


Figure 4.17 Unit conversions for concentration in the esterification reaction (mol/L to %wt)

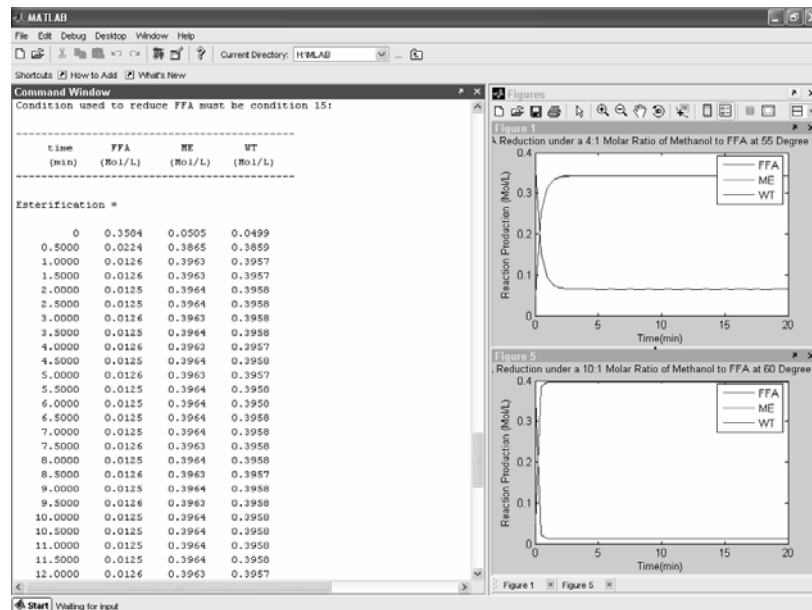


Figure 4.18 The table and curves of FFA, ME, and WT concentrations the optimal condition in mol/L

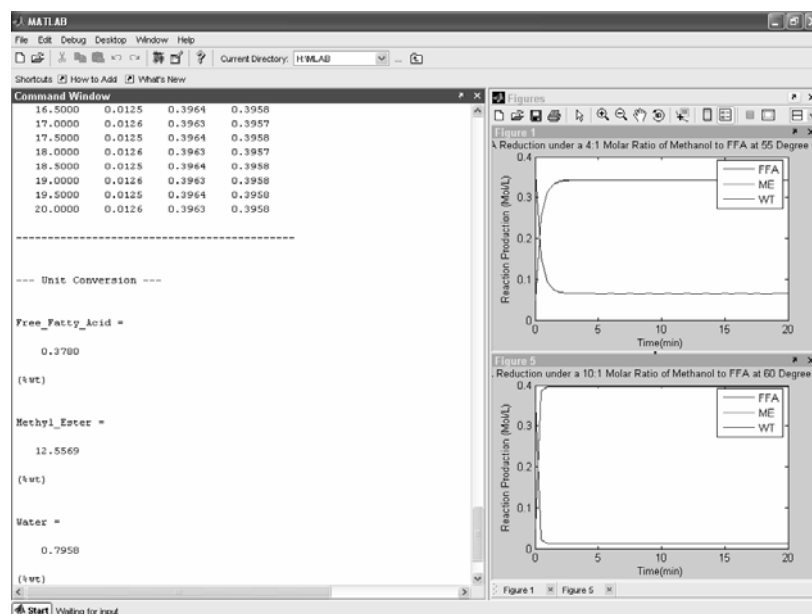


Figure 4.19 Unit conversions for concentration in the esterification reaction (mol/L to %wt) of the optimal condition

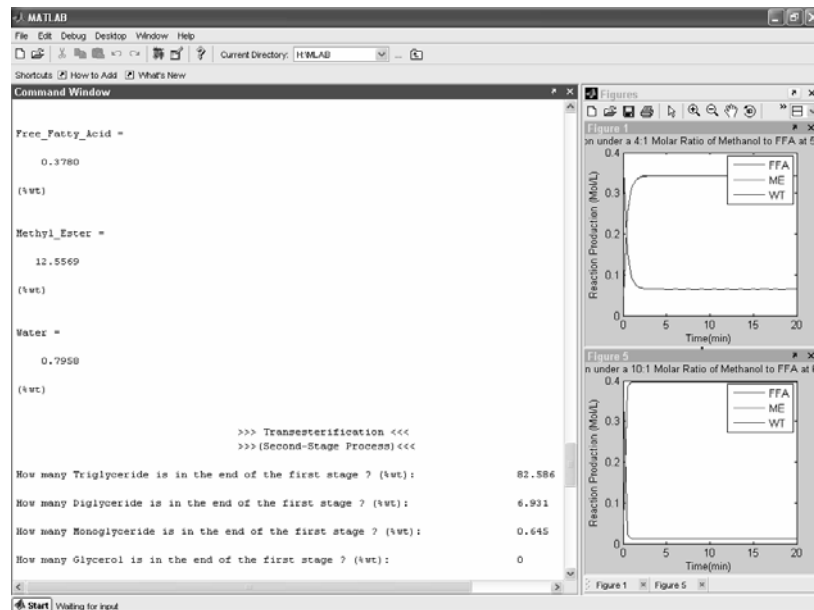


Figure 4.20 Inputting initial concentrations of TG, DG, MG, and GL in %wt

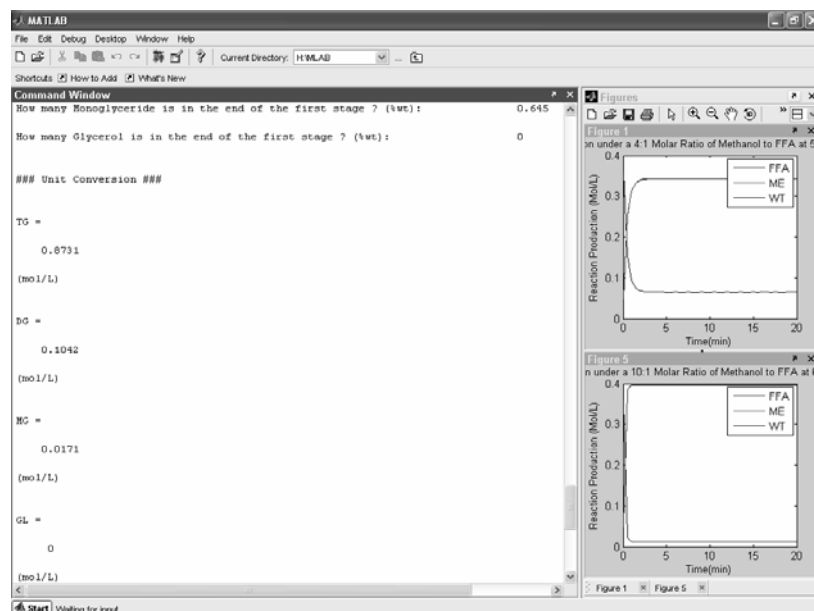


Figure 4.21 Unit conversions for concentrations in the transesterification reaction (%wt to mol/L)

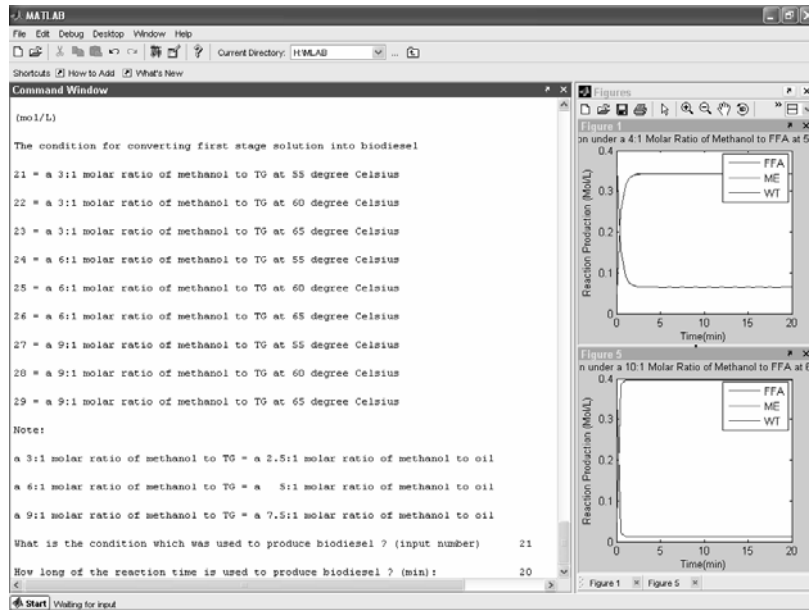


Figure 4.22 Conditions for producing ME from MCPO

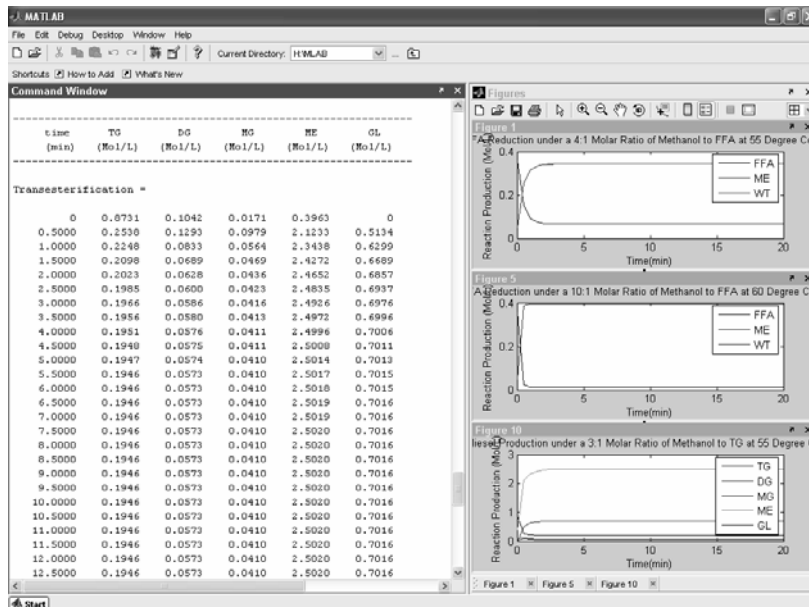


Figure 4.23 The table and curves of TG, DG, MG, ME, and GL concentrations in mol/L

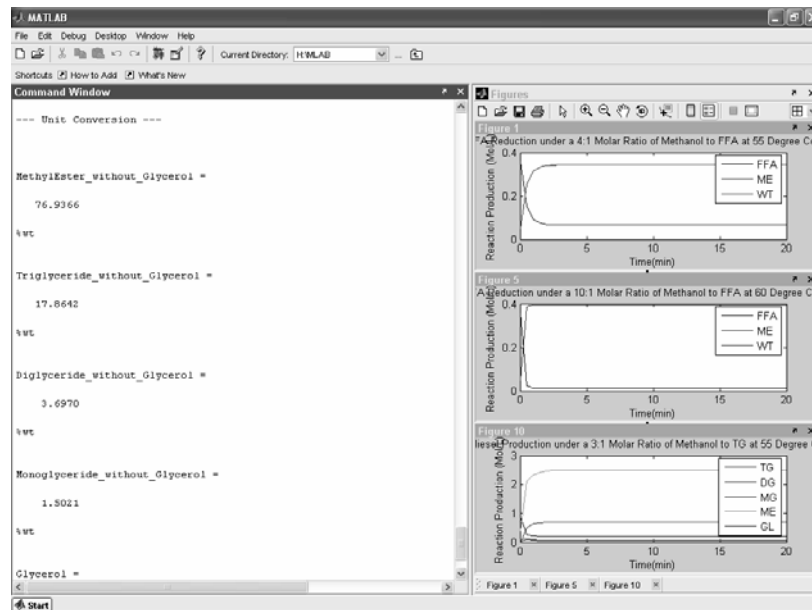


Figure 4.24 Unit conversions for concentrations in the transesterification reaction (mol/L to %wt)