# CHAPTER IV
# RESULTS AND DISCUSSION

In this chapter, experimental results will be presented. The results of all experiments will be presented due to the parameters involved in the esterification and the transesterification such as methanol concentration and using concentrated sulfuric acid and potassium hydroxide as catalysts. In this study, the experiments will be categorized into five sections.

4.1 Characterization of palm fatty acid and palm stearin.

4.2 The effect of reaction time in preliminary study using acid or base catalyst on transesterification and esterification.

4.3 Effect of potassium hydroxide concentration on transesterification.

4.4 Effect of sulfuric acid concentration on esterification.

4.5 Effect of excess methanol from its stoichiometric ratio.

## 4.1 Characterization of palm fatty acid and palm stearin

Palm fatty acid and palm stearin, obtained from palm oil refinery are called Palm Fatty Acid Distillate (PFAD) and Refined Bleached Deodorized (RBD). Palm fatty acid and palm stearin used in this study were characterized for their properties. The results are shown in Table 4.1.

**Table 4.1.** Properties of Palm fatty acid and Palm stearin

| Properties | Palm fatty acid | Palm stearin |
|---|---|---|
| Density at 60°C,(g/ml) | 0.8691 | 0.8705 |
| Viscosity at 40°C,($mm^2/s$) | N/D | N/D |
| Acid Value,(mg KOH/g) | 147.5 | 0.16 |
| Saponification Value,(mg KOH/g) | 203.36 | 193.54 |
| %Free Fatty Acid | 98.82 | 0. 31 |
| Mean molecular Weight,(g/mole) | 272.42 | 831.72 |

Both palm fatty acid and palm stearin are in solid phase at 40°C so their viscosity can not be obtained.

The molecular weight of palm fatty acid and palm stearin were calculated based on the fatty acid composition or saponification value. Chemical analysis of palm fatty acid and palm stearin expressed in free fatty acid content contain the advantage of giving an impression of the acidity as a percentage; its drawback is the need for knowing the mean molecular weigh. Our analysis shows that there is small amount of fatty acid in palm stearin while there is also small amount of non-fatty acid in palm fatty acid.

This general information was confirmed by analyzing the fatty acid composition of the palm fatty acid and palm stearin using gas chromatography. The results are shown in Table 4.2. It shows that the major fatty acid components for palm fatty acid and palm stearin are palmitic acid, oleic acid, stearic acid and linoleic acid.

Palm fatty acid and palm stearin were used in this study. Methyl esters products were analyzed by gas chromatography. Fatty acid in triglycerides can be determined in term of methyl esters. The results are shown in Table 4.2.

## 4.2 Effect of reaction time in preliminary study using acid or base catalyst on transesterification and esterification

In preliminary studies the experiments are divided into 2 parts in transesterification and esterification of palm stearin and palm fatty acid used as raw material with methanol using potassium hydroxide and sulfuric acid as catalyst, respectively. The reaction was carried out at 60ºC and atmospheric pressure. The effect of reaction time on concentration of methyl ester (wt %) was investigated. The reaction time was selected as the result of reaction rate.

The production of methyl ester was the reaction by esterification and transesterification from palm fatty acid and palm stearin. The samples are analyzed for their composition by gas chromatographic analysis techniques. The outputs are used to calculate weight percentage concentration of methyl ester (wt %). The obtained concentration of methyl esters was totally calculated (wt %) as a function of time with related operative conditions are reported in Figures 4.1 and 4.2. In order to calculate the purity of product samples, the percentages of methyl esters are defined as the following equations. The concentration of methyl esters is defined as a ratio of weight of methyl esters, which was determined by using GC, to the weight of sample as shown in Equation 4.1.

$$\text{Concentration of methyl ester (wt \%)} = \frac{\text{weight of methyl esters (determined by GC) x100}}{\text{weight of sample}} \quad (4.1)$$

**4.2.1 Effect of reaction time on transesterification**

The transesterification reaction of triglyceride with methanol was carried out in a batch reactor by using potassium hydroxide as a catalyst.

The reaction conditions for transesterification reaction are as follows: 100%mol excess of methanol from its stoichiometric ratio, 1% KOH on the basis of the palm stearin, reaction temperature of 60°C and 120 minutes.

As shown in Figure 4.1, concentrations of methyl ester are almost constant. It indicates that transesterification of part palm stearin sections its equilibrium conversion within 40-60 minutes and concentration of methyl ester at appropriately 80% was obtained. Based on this study, a section time of 60 minutes was used in subsequent study.

Potassium hydroxide (KOH) can act as effective catalysts for the transesterification of the triglyceride (palm stearin) if the free fatty acid (FFA) content is less than 1%. From 3 experiments at this condition are obtained at the concentration of methyl esters average (wt %) as a function of time shown that in Table 4.2. In addition, gas chromatography analysis confirmed the results of transesterification showed in Figure 4.1.

**Table 4.2** Results of concentration of methyl ester (wt%) from 3 experiments

| Time.(min.) | Concentration of methyl ester total (wt%) | | | |
|---|---|---|---|---|
| | Ex.No.1 | Ex.No.2 | Ex.No.3 | Average |
| 0 | 0.00 | 0.00 | 0.00 | 0.00 |
| 10 | 80.19 | 87.55 | 67.54 | 78.43 |
| 20 | 85.47 | 94.28 | 85.25 | 88.33 |
| 30 | 76.62 | 93.27 | 81.11 | 83.67 |
| 40 | 77.11 | 90.00 | 73.93 | 80.35 |
| 50 | 78.11 | 88.10 | 69.36 | 78.52 |
| 60 | 72.85 | 86.66 | 81.20 | 80.24 |
| 75 | 76.39 | 85.04 | 77.53 | 79.65 |
| 90 | 71.32 | 84.91 | 78.44 | 78.23 |
| 105 | 77.56 | 81.85 | 78.22 | 79.21 |
| 120 | 77.57 | 80.02 | 79.20 | 78.93 |

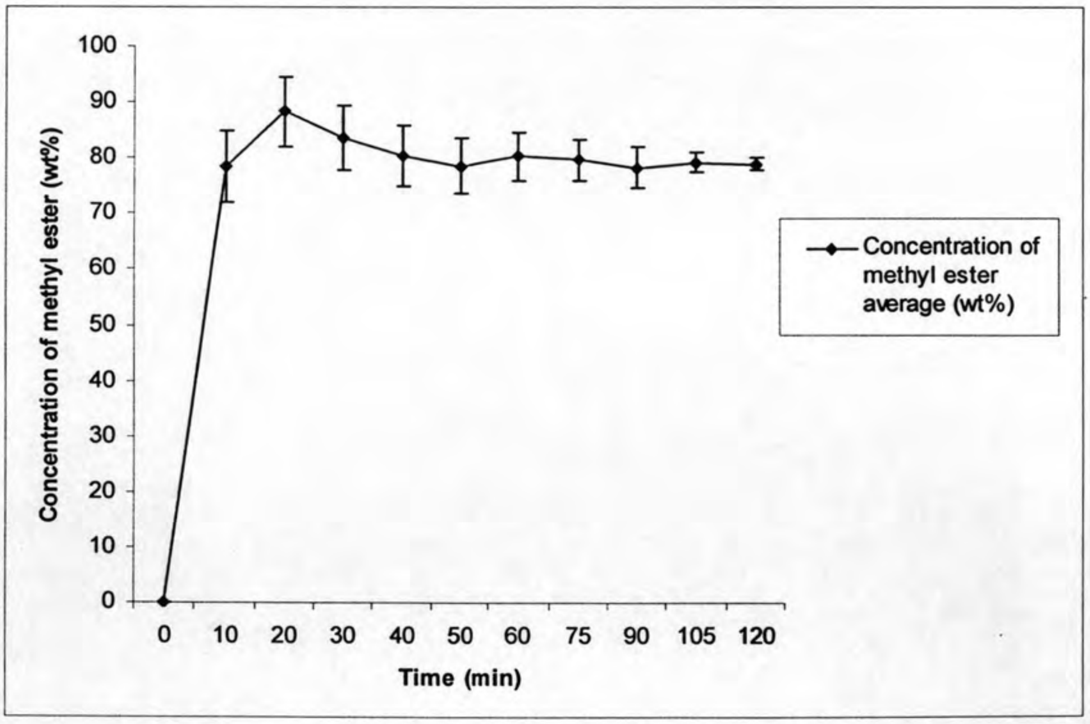


**Figure 4.1** Effect of reaction time at 1% KOH, temperature of 60°C and 100%mol excess of methanol from its stoichiometric ratio.

The results of the transesterification in Figure 4.1 can be observed, the reaction was very fast in the first stage. After 40-120 min, the concentration of methyl ester (wt %) are in the range between 78.23-80.35% for KOH. The reaction seems to be equilibrium. The highest concentration of methyl esters obtained from this experiment was 88.33% for KOH. These results are similar to those found in the literature.

Common alkaline catalysts are well-known for transesterification reaction of triglyceride. The rate of the alkaline-catalyzed transesterification reaction is fast, compared to those using acids, and it is reported that the rate could be as high, compared to those using an acidic catalyst. Although the transesterification reaction uses an alkaline catalysts process gives high concentration of methyl ester (wt %).

Chonsaranon (2006) investigated using sodium hydroxide 0.3-0.5 wt% as catalyzed for transesterification of palm stearin. The reaction time was between 15 and 45 minutes at 60°C. We found that the percentage of methyl ester was reduced with increasing reaction time at 15-20 min. After 20 minutes to 45 minutes the reaction time was equilibrium.

Several researches have confirmed that potassium hydroxide and sodium hydroxide are the effective homogeneous catalysts for transesterification. For the experiment, the transesterification of crude palm and palm stearin with ethanol uses sodium hydroxide as a catalyst. The result also agreed with the

Udomsab (2005) studied the optimal condition to transesterification of crude palm oil and palm stearin with ethanol using sodium hydroxide as a catalyst. The optimal condition to transesterification crude palm oil and palm stearin were similar as follows: 12:1 molar ratio of ethanol/oil, 75°C of reaction temperature, 1% weight of sodium hydroxide and 60 minute reaction time. At this condition, the ethyl ester conversion of crude palm oil and palm stearin were 87.79 and 94.42% weight respectively.

### 4.2.2 Effect of reaction time on esterification

The esterification reaction of fatty acid with methanol was carried out in a batch reactor by using sulfuric acid as a catalyst. The reaction conditions for esterification reaction are as follow: 100%mol excess of methanol from its stoichiometric ratio, 1% $H_2SO_4$ on the basis of the palm fatty acid, reaction temperature of 60°C and 120 minutes.

As shown in Figure 4.2, concentrations of methyl ester are almost constant. It indicates that esterification of part palm fatty acid section it equilibrium conversion within 60 minutes and concentration of methyl ester appropriate 50% was obtained. Based on this study, a section time of 60 minutes was used in subsequent study.

From Figure 4.2, concentration of methyl ester (wt %) increases slightly with increasing time approach maximum concentration of methyl ester 52.25%. The concentrations of methyl ester occur less because the reaction temperature runs at 60°C were the low temperature in esterification. However, the concentration of methyl ester was higher than 52.25% the requirement of high catalyst concentration and high temperature. From 3 experiments at this condition are obtained concentration of methyl esters average as a function of time showed that in Table 4.3. Acid on this study, a reaction time of 60 minutes was used in subsequent study. The reaction was using the long time because the reaction was slowly when using acid catalyst. The advantages of using acid catalyst are insensitive to FFA content, esterification can be performed simultaneously and removal of the catalyst is easy. In addition, gas chromatography analysis confirmed the results of esterification shown in Figure 4.3.

**Table 4.3** The results of concentration of methyl ester (wt%) from 3 experiments

| Time. | Concentration of methyl ester total (wt%) | | | |
|---|---|---|---|---|
| (Min) | Ex.No.1 | Ex.No.2 | Ex.No.3 | Average. |
| 0 | 0.00 | 0.00 | 0.00 | 0.00 |
| 10 | 30.96 | 38.44 | 30.06 | 33.15 |
| 20 | 37.64 | 43.36 | 30.76 | 37.25 |
| 30 | 42.87 | 51.06 | 33.34 | 42.42 |
| 40 | 45.83 | 54.87 | 33.38 | 44.69 |
| 50 | 45.64 | 56.72 | 38.56 | 46.97 |
| 60 | 45.42 | 55.74 | 49.98 | 50.38 |
| 75 | 47.59 | 55.13 | 52.07 | 51.60 |
| 90 | 50.82 | 58.52 | 47.41 | 52.25 |
| 105 | 51.72 | 53.76 | 49.65 | 51.71 |
| 120 | 52.11 | 54.05 | 48.81 | 51.65 |

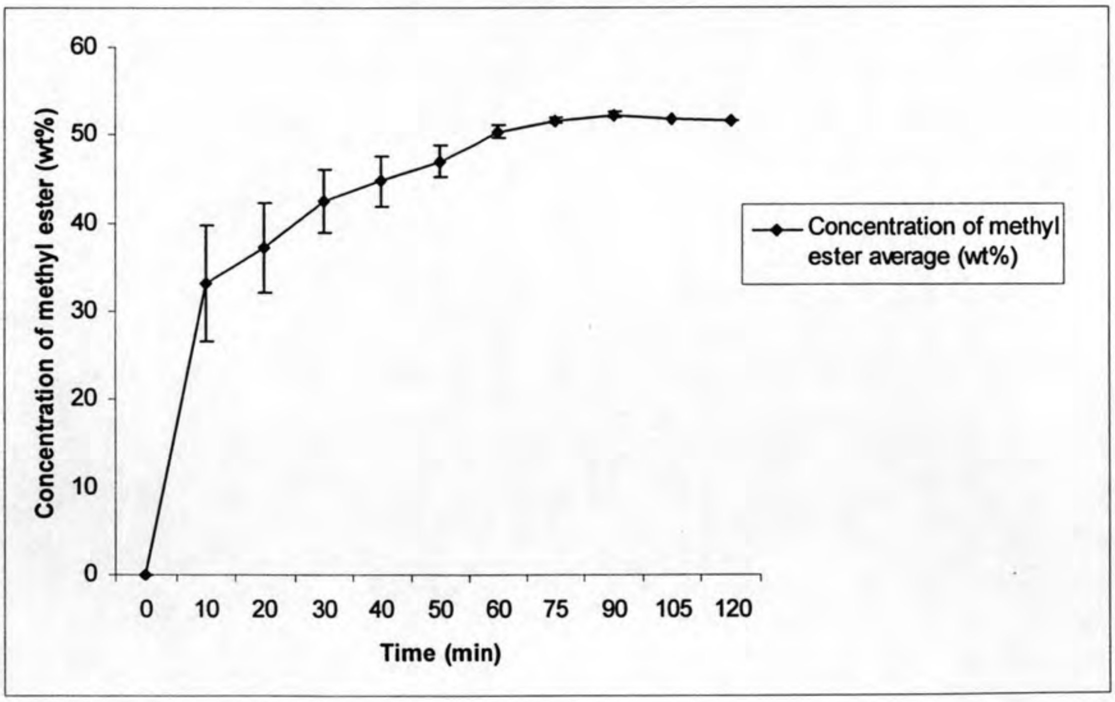


**Figure 4.2** Effect of reaction time at 1% $H_2SO_4$, temperature of 60°C and 100% mol excess methanol from its stoichiometric ratio.

The results of the esterification can be seen in Figure 4.2. The reaction was increased in the start reaction. The highest percentage of methyl esters obtained from this experiment was 52.25% for sulfuric acid ($H_2SO_4$).

Although the esterification reaction using an acids catalysts process give high percentages of methyl esters when using free fatty acid (FFA) is the reactants.

Since acid catalysis is relatively fast for converting the free fatty acid to methyl esters, it is used as a pretreatment for high free fatty acid feedstocks.

**For acid catalysts**

Although acid catalysts can be used for transesterification they are generally considered to be too slow for industrial processing. Acid catalysts are more commonly used for the esterification of free fatty acids. Acid catalysts include sulfuric acid and phosphoric. The acid catalyst is mixed with methanol and then this mixture is added to the free fatty acids or a feedstock that contains high levels of free fatty acids. Acids catalyst systems are characterized by slow reaction rate and high alcohol: TG requirements (20:1 and more). Generally, acid catalyzed reactions are used to convert free fatty acid to esters, or soaps to esters as a pretreatment step for high free fatty acid freedstocks. Residence times from 10 minutes to about 2 hours are reported.

**For base catalysts**

The most commonly used catalyst materials for converting triglycerides to methyl ester are potassium hydroxide, sodium hydroxide, and sodium methoxide. Most base catalyst system use triglyceride as a feedstock. If the triglyceride, it contains small amounts less than 2% of free fatty acids that will form soaps that will form soaps that will end up in the glycerin.

## 4.3 Effect of potassium hydroxide concentration on transesterification

One important variable affecting the percentage methyl ester is the amount of catalyst. Several researches have confirmed that KOH is one of the effective catalysts for this reaction. In the characterization of palm stearin , it showed that palm stearin contain a low percentage of free fatty acid less than one percentage. The effect of KOH concentration (0.5% and 1.0% by weigh) on concentration of methyl ester (wt%) was investigated. The conditions were kept constant 100%, 300% mol excess of methanol from its stoichiotric ratio at 60°C for reaction time of 1 hour. The results are shown in Table 4.4.

The Effect of this parameter in the transesterification of palm stearin is shown in Figure 4.3. It also shows that 0.5% KOH and 1.0% KOH as catalyst was also sufficient for the reaction. When increase the catalyst would then affect the percentage methyl ester production. The effect of the amount of KOH in the transesterification of palm stearin is shown in Figure 4.5.

The results from this experiment of the effect increase amount of catalyst for palm stearin is shown in Figure 4.3. As can be see in figure 4.3 when the KOH concentration was increased from 0.5% to 1.0 % wt, it was found that the concentration of methyl ester (wt%) at temperature of 60°C increased from 72.16% to 86.91% and 77.94% to 94.44% when 100% mol, 300% mol excess of methanol from its stoichiometric ratio, respectively. The optimum conditions were 300% mol excess of methanol from its stoichiotric ratio, 1.0% KOH, 60 min and temperature of 60°C which gave maximum concentration of methyl ester 94.44%.

**Table 4.4** The results of concentration of methyl ester (wt%) from palm stearin at temperature of 60°C and 60 min

| % mol excess of methanol from its stoichiometric ratio | %KOH | Concentration of methyl ester tatal (wt%) |
|---|---|---|
| 100 | 0.5 | 72.16 |
| 300 | 0.5 | 77.94 |
| 100 | 1.0 | 86.91 |
| 300 | 1.0 | 94.44 |

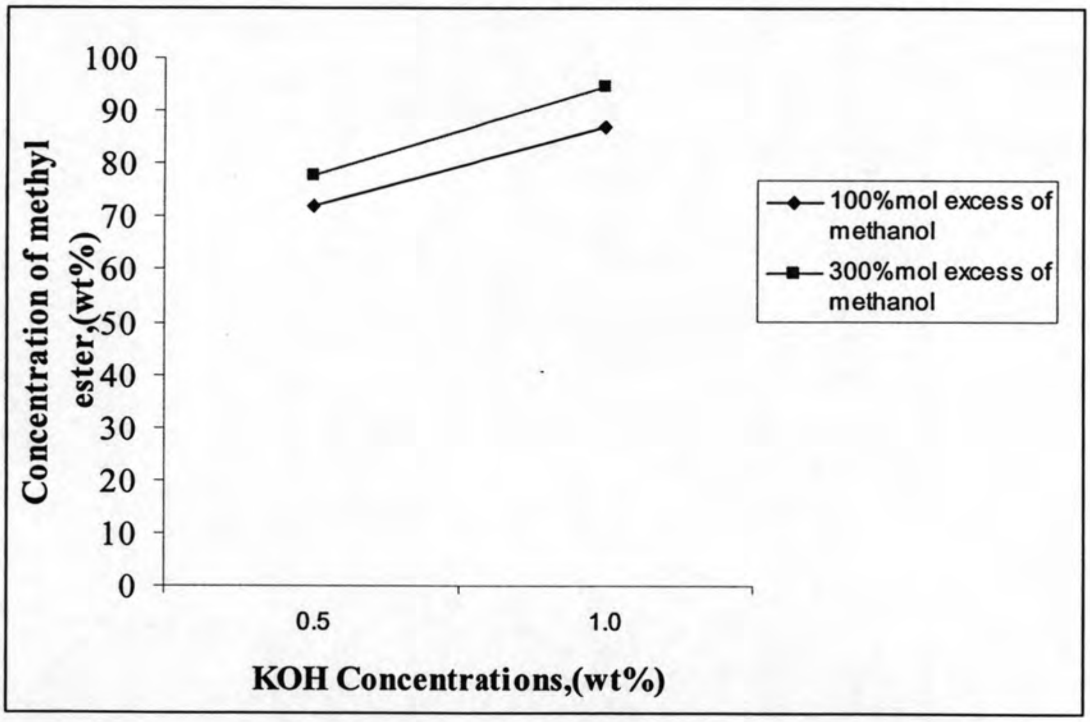


**Figure 4.3** Effect of the amount of catalyst.

Udomsap (2005) studied amount of catalyst for transesterification of palm stearin using sodium hydroxide as a catalyst. The condition of reaction are 12:1 methanol: oil molar ratio at 75°C and 60 min reaction time. The reaction can be catalyzed by sodium hydroxide 0.5, 1, 1.5 and 2 percent on the basis of the palm stearin at 75°C for 60 min. They found that the optimum amount of NaOH should be 1% based on the weight of oil at 12:1 methanol: oil molar ratio was obtained concentration of methyl ester 94.42%.

Laoprasert (2002) studied amount of catalyst for transesterification of used cooking oil using potassium hydroxide as a catalyst. The optimum conditions were 25% MeOH, 1.0% KOH, 30°C for reaction time of 30 min and the maximum yield was 91.87%.

### 4.4 Effect of sulfuric acid concentration on esterification

The effect of $H_2SO_4$ concentration (0.5 and 1.0% by weigh) on % methyl ester was investigated. The conditions were kept constant at 60°C for reaction time of 1 hour. The results are shown in Table 4.5.

It also shows that 0.5% $H_2SO_4$ and 1.0% $H_2SO_4$ as catalyst was also sufficient for the reaction. Increasing the catalyst would then affect the concentration of methyl ester (wt %). The effect of the amount of $H_2SO_4$ in the esterification of palm fatty acid is shown in Figure 4.4.

The results from this experiment of the effect increase amount of $H_2SO_4$ catalyst for palm fatty acid is shown in Figure 4.4. As can be see in figure 4.4 when the $H_2SO_4$ concentration was increased from 0.5% to 1.0%, it was found that the concentration of methyl ester (wt %) at 60°C. From relation between concentration of methyl ester production to amount $H_2SO_4$ can be see amount $H_2SO_4$ 0.5-1.0 percentage by weigh make the concentration of methyl ester (wt%) production are increased from 45.50% to 49.25% and 46.19% to 55.73% when 100, 300%mol excess of methanol from its stoichiotric ratio, respectively. The optimum conditions were 300% mol excess of methanol from its stoichiotric ratio, 1.0% $H_2SO_4$, 60 min and 60°C which gave maximum concentration of methyl ester (wt%) 55.73%.

**Table 4.5** The results of concentration of methyl ester (wt%) from palm fatty acid at temperature of 60°C and 60 min

| % mol excess of methanol from its stoichiometric ratio | % $H_2SO_4$ | Concentration of methyl ester total (wt%) |
|---|---|---|
| 100 | 0.5 | 45.50 |
| 300 | 0.5 | 46.19 |
| 100 | 1.0 | 49.29 |
| 300 | 1.0 | 55.73 |

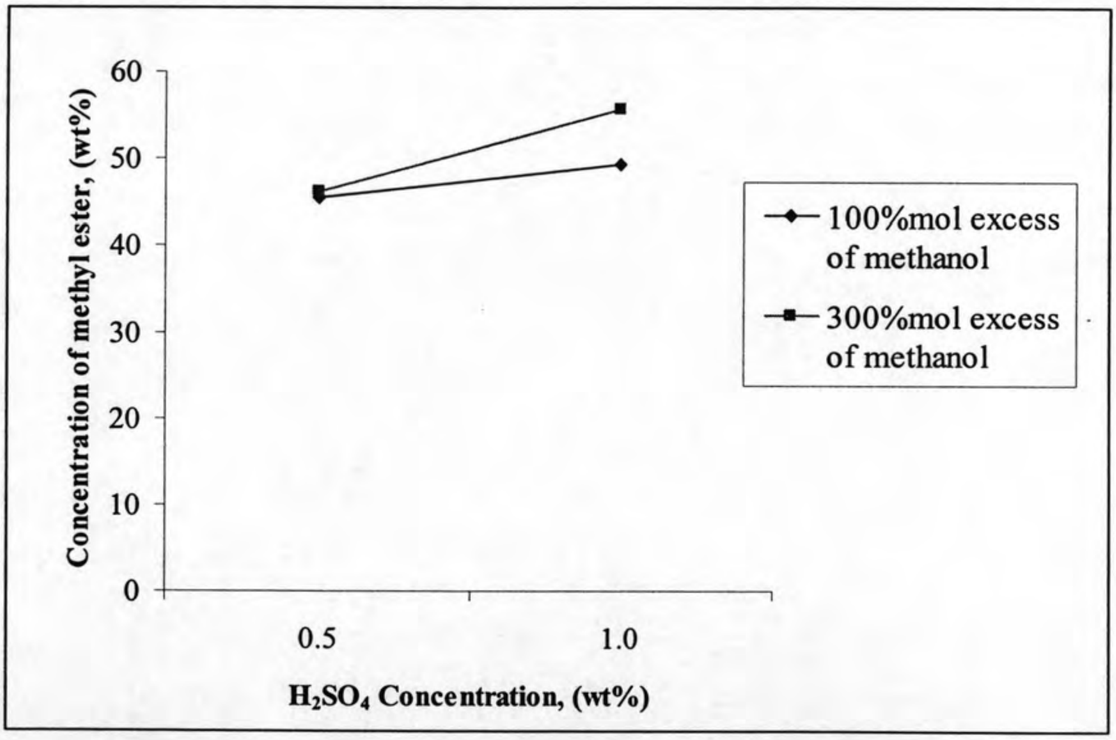


**Figure 4.4** Effect of the amount of catalyst.

Edward et al. (2001) studied the amount of acid-catalyzed (sulfuric acid) which is usually conducted at high temperature. The effect of reaction temperature was investigated at 75, 80 and 95°C using 1-5% $H_2SO_4$ as catalyst and a 40:1 molar ratio of methanol to oil over a 24-h period. The rate of the reaction increased as the reaction temperature increased and the amount of sulfuric acid increased. The amount of sulfuric acid is 1% wt when the result was obtained 52% methyl ester. When the increasing amount of $H_2SO_4$ catalyst found that the increased concentration of methyl ester (wt %) production were 52, 68, 76, 80, 83%, respectively.

The results of the effect of transesterification and esterification, thus, as there was the optimum conditions as same as the concentration of methyl ester, were different because amount of fatty acid of the reactant are different.

### 4.5 Effect of %mol excess of methanol from its stoichiometric ratio

The excess of methanol from its stoichiometric ratio to oil is one of the important factors that affect the concentration of methyl ester (wt %) as well as production. In this study the reaction vary 100% mol, 300% mol excess of methanol from its stoichiometric ratio. When increased excess of methanol higher than 300%mol, as can see concentration of methyl ester was decreased Udomsab (2005) because the catalyst was dilute when mix the methanol.

#### 4.5.1 Acid catalyst

In the transesterification used acids catalysts, 3 moles of methanol for every mole of oil is needed to form 3 moles of methyl esters and one mole of glycerol. Freedman et al. (1984) studied this effect in the transesterification of refined sunflower oil. They noted that at 6:1 molar ratio, a 98% conversion of the ester was obtained. While as the molar ratio decreased to the theoretical ratio of 3:1, the percentage of ester decreased to 82%

The results of the effect of methanol: oil ratio for palm fatty acid: palm stearin is shown in Figure 4.5 and Figure 4.6. This condition was obtained the concentration of methyl esters as a function time of excess methanol from its stoichiotric ratio showed that in Table 4.6 and Table 4.7. The condition use acid catalyst at 0.5%$H_2SO_4$ and 1.0%$H_2SO_4$, respectively. At 300% mol excess methanol from its stoichiotric ratio, the concentration of methyl ester was higher than 100% mol excess of methanol from its stoichiotric ratio and it showed that the concentration of methyl ester (wt %) was maximal.

From experiment can see acid catalyst using with free fatty acid reactant. When increased acid catalyst and excess methanol can see % methyl ester are increased. The optimized variables are high methanol/oil and high acid catalyst at high temperature for long time.

This approach solves the reaction rate problem by using each technique to accomplish the process for which it is best suited. Since acid catalysis is relatively fast for converting the free fatty acid to methyl esters, it is used as a pretreatment for the high free fatty acid feedstocks. Then, when the free fatty acid level has been reduced to 0.5%, or lower, an alkali catalyst is added to convert the triglycerides to methyl esters. This process can convert high free fatty acid feedstocks quickly and effectively. Water formation is still a problem during the pretreatment phase. One approach is to simply add so much excess methanol during the pretreatment that the water produced is diluted to the level where it does not limit the reaction. Molar ratios of alcohol to free fatty acid as high as 40:1 may be needed. If the free fatty acid level is still too high, then additional methanol and, if necessary, acid catalyst can be added to continue the reaction. This process can be continued for multiple steps and will potentially use less methanol than the previous approach.

**Table 4.6** The results of concentration of methyl ester (wt%) from palm fatty acid to palm stearin using 0.5%$H_2SO_4$

| Fatty acid | % mol excess of methanol from its stoichiometric ratio | Concentration of methyl ester total (wt%) |
|---|---|---|
| 0%FFA | 100 | 6.45 |
| 0%FFA | 300 | 8.91 |
| 25%FFA | 100 | 16.68 |
| 25%FFA | 300 | 17.49 |
| 50%FFA | 100 | 18.23 |
| 50%FFA | 300 | 25.63 |
| 75%FFA | 100 | 35.48 |
| 75%FFA | 300 | 38.23 |
| 100%FFA | 100 | 45.50 |
| 100%FFA | 300 | 46.20 |

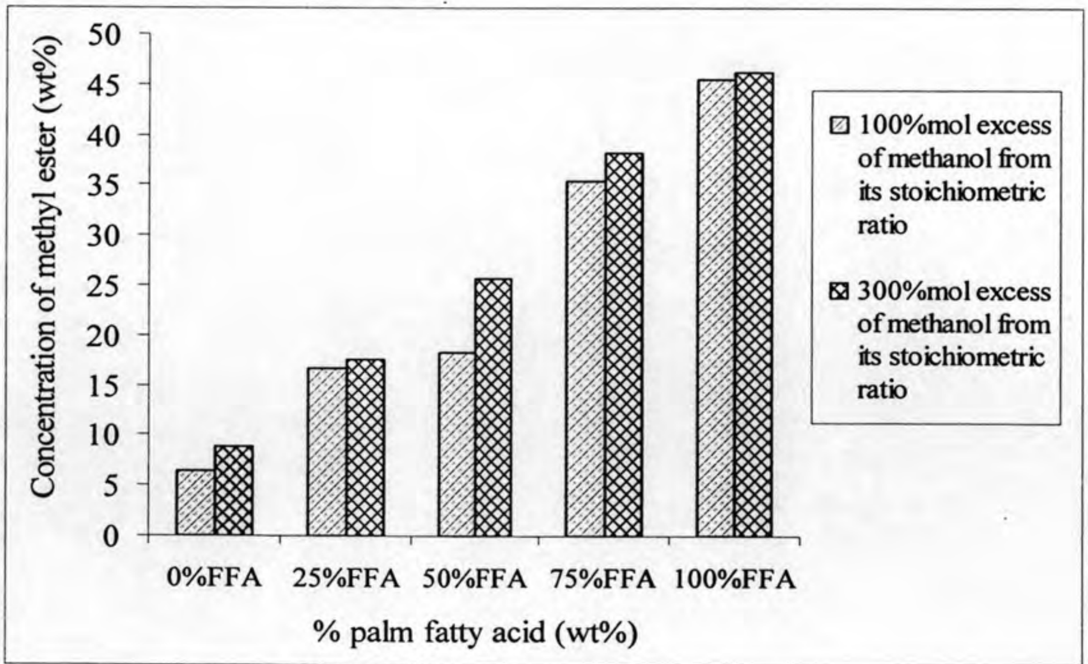


**Figure 4.5** Effect of % mol excess of methanol from its stoichiometric ratio at 0.5%$H_2SO_4$, temperature of 60°C and 60 min

**Table 4.7** The results of concentration of methyl ester (wt%) from palm fatty acid to palm stearin using 1.0%$H_2SO_4$

| Fatty acid | % mol excess of methanol from its stoichiometric ratio | Concentration of methyl ester total (wt%) |
|---|---|---|
| 0%FFA | 100 | 18.49 |
| 0%FFA | 300 | 21.36 |
| 25%FFA | 100 | 25.76 |
| 25%FFA | 300 | 29.15 |
| 50%FFA | 100 | 27.88 |
| 50%FFA | 300 | 33.44 |
| 75%FFA | 100 | 39.86 |
| 75%FFA | 300 | 45.81 |
| 100%FFA | 100 | 48.71 |
| 100%FFA | 300 | 49.29 |

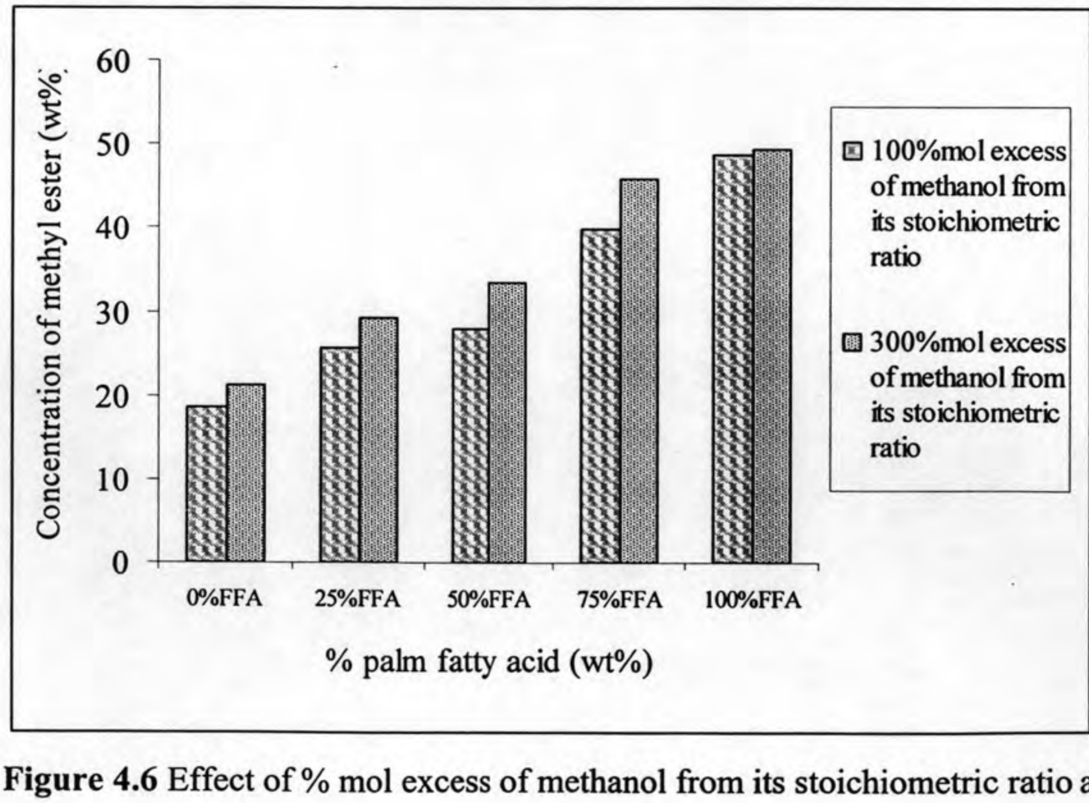


**Figure 4.6** Effect of % mol excess of methanol from its stoichiometric ratio at 1.0% $H_2SO_4$, temperature of 60°C and 60 min.

The comparison of this work with Kulkarni et al. (2006) were made as shown in the transesterification of edible oil using strong acids (Bronsted acids) such as sulfuric and hydrochloric acid which are available in literature reports. The acid-catalyzed transesterification of pure soybean oil was studied by

Canaki et al. (1999). The optimal condition to transesterification were: 6:1 molar ratio of methanol/oil, temperature of 60°C of reaction temperature, 3% weight of sulfuric acid and 96 hours reaction time. The results obtained at high free fatty acid (33% FFA) are conversion of 60%.

Edward et al. (2001) studied three principal variables, molar ratio of methanol to oil, amount of catalyst, and reaction temperature, affecting the yield of acids-catalyzed production of methyl ester from crude palm oil were investigated. The optimized variables, 40:1 methanol/oil (mol/mol) with 5%$H_2SO_4$ (vol/wt) reacted at temperature of 95°C for 9 h, gave a maximum ester yield of 97%.

### 4.5.2 Base catalyst

Common alkaline catalysts (such as NaOH, KOH and $NaOCH_3$) are well-known for the transesterification reaction. The rate of the alkaline-catalyzed transesterification reaction is fast, compared to using acids.

The results of the effect of excess methanol for palm fatty acid: palm stearin is shown in Figure 4.7 and Figure 4.8. There conditions are obtained by using percentage of methyl esters as a function of the excess of methanol from its stoichiometric ratio shown in Table 4.8 and Table 4.9. The condition uses acid catalyst at 0.5%KOH and 1.0%KOH, respectively.

The results from this experiment could be seen when increasing amount of palm fatty acid was found that the concentration of methyl ester (wt %) was decreased. The optimum conditions were 300%mol excess of methanol from its stoichiotric ratio, 1.0%KOH, 60 min, 60°C and 0%FFA which gave maximum concentration of methyl ester (wt%) 94.44%.

**Table 4.8** The results of concentration of methyl ester (wt %) from palm fatty acid to palm stearin using 0.5%KOH

| Fatty acid | % mol excess of methanol from its stoichiometric ratio | Concentration of methyl ester total (wt%) |
|---|---|---|
| 0%FFA | 100 | 72.16 |
| 0%FFA | 300 | 77.94 |
| 25%FFA | 100 | 19.90 |
| 25%FFA | 300 | 19.97 |
| 50%FFA | 100 | 13.38 |
| 50%FFA | 300 | 15.51 |
| 75%FFA | 100 | 9.35 |
| 75%FFA | 300 | 10.50 |
| 100%FFA | 100 | 7.39 |
| 100%FFA | 300 | 7.61 |

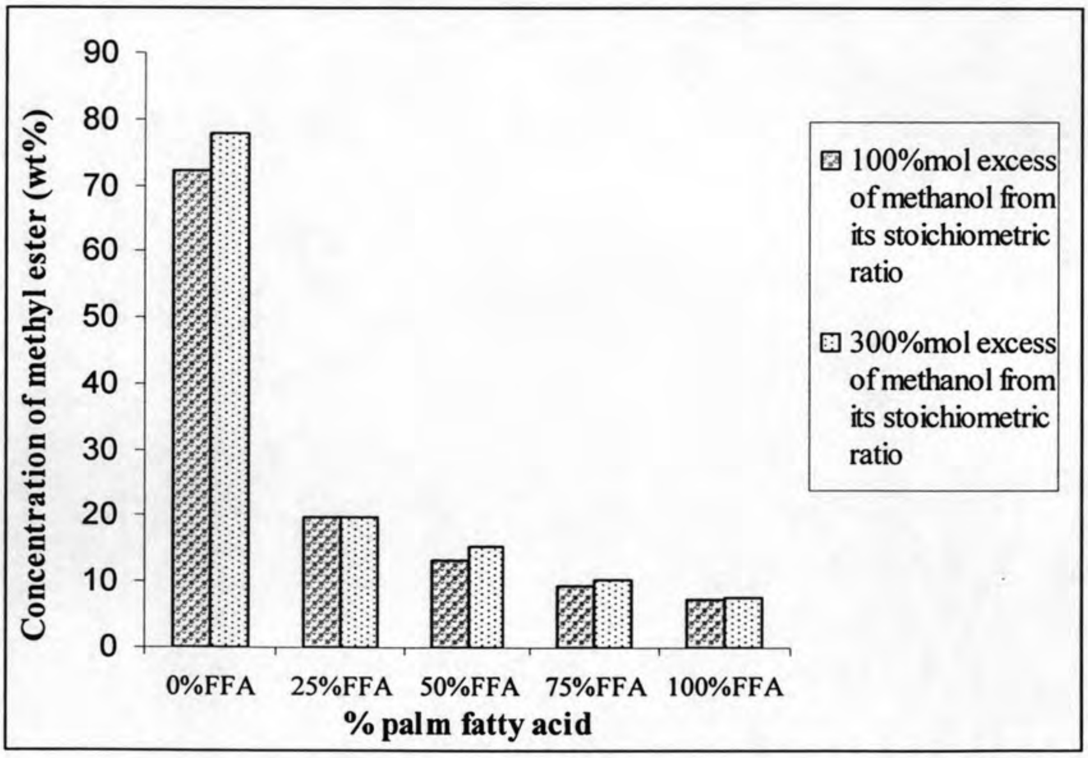


**Figure 4.7** Effect of % mol excess of methanol from its stoichiometric ratio at 0.5%KOH, temperature of 60°C and 60 min.

**Table 4.9** The results of concentration of methyl ester (wt%) from palm fatty acid to palm stearin using 1.0%KOH.

| Fatty acid | % mol excess of methanol from its stoichiometric ratio | Concentration of methyl ester total (wt%) |
|---|---|---|
| 0%FFA | 100 | 86.91 |
| 0%FFA | 300 | 94.44 |
| 25%FFA | 100 | 20.97 |
| 25%FFA | 300 | 23.09 |
| 50%FFA | 100 | 16.74 |
| 50%FFA | 300 | 17.65 |
| 75%FFA | 100 | 10.46 |
| 75%FFA | 300 | 13.16 |
| 100%FFA | 100 | 9.03 |
| 100%FFA | 300 | 10.39 |

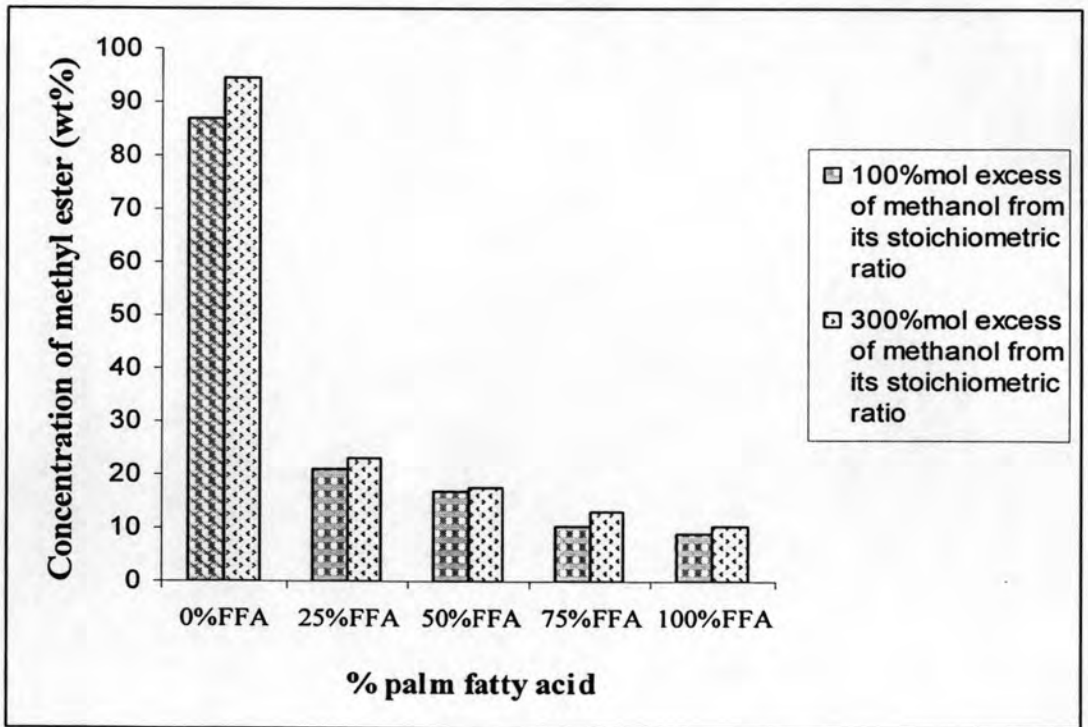


**Figure 4.8** Effect of % mol excess of methanol from its stoichiometric ratio at 1.0%KOH, temperature of 60°C and 60 min.

The application of an alkaline catalyst in the transesterification of palm stearin mix palm fatty acid is somewhat limited, because the FFA in the experiment reacts with the alkaline catalyst (KOH) and forms soap. The soap formed during the reaction prevents the glycerol separation, which drastically reduces the concentration of methyl ester (wt%). The water in the reaction also affects the concentration of methyl ester (wt%) by favoring a saponification reaction. Despite these two serious problems, many researchers have studied the transesterification of palm stearin mix palm fatty acid used an alkaline catalyst.

An alternative approach to the utilization of highly free fatty acid feedstocks is to use a base catalyst to deliberately form soap from the free fatty acid. The soap is recovered, the oil dried, and then used in a conventional base catalyzed system.

One limitation in the alkali-catalyzed process is its sensitivity to the purity of reactants, especially to both water and free fatty acid (FFA). The FFA and water make the use of an alkaline catalyst difficult, because of soap formation and then difficulty in product separation. Freedman et al. have reported that an acid catalyst is insensitive to free fatty acid (FFA) and is better than the alkaline catalyst for triglyceride with less than 1% free fatty acid (FFA). The only disadvantage of an acidic catalyst is a slower reaction rate.

When an alkali catalyst is added to these feedstocks, the free fatty acids react with the catalyst to form soap and water, as shown in Equation 4.2.

$$\underset{\text{Fatty Acid}}{HO-\overset{\overset{O}{\|}}{C}-R} \quad + \quad \underset{\text{Potassium Hydroxide}}{KOH} \longrightarrow \underset{\text{Potassium soap}}{K^{+}\,{}^{-}O-\overset{\overset{O}{\|}}{C}-R} \quad + \quad \underset{\text{Water}}{H_2O}$$

**Equation 4.2** Reaction of fatty acid using base catalyst