CHAPTER III

EXPERIMENTS

This chapter consists of experimental systems and procedures which are used in this study. The chapter is divided into four sections. The materials and apparatus for alkyl esters production are explained in section 3.1. The procedures and methodology of esterification reaction of palm fatty acid distillate (PFAD) with various alcohols are described in section 3.2. The analytical tests for analyzing alkyl esters quality are shown in section 3.3. Finally, the details of experimental error are illustrated in section 3.4.

3.1 Material and Apparatus

3.1.1 Raw material

The raw material in this study is palm fatty acid distillate (PFAD) which was obtained from refinery process of crude palm oil.

3.1.2 Chemicals

The details of all chemicals used in the experiments are shown in Table 3.1.

3.1.3 Apparatus for testing properties

The details of all apparatus that used for testing physical and chemical properties of alkyl esters and reactant are shown in Table 3.2.

3.1.4 Experimental apparatus

A reflux set: The reflux set consists of an Erlenmeyer flask, an oil bath, hot plate and magnetic stirrer, magnetic bar, temperature controller, thermocouple and condenser.

An Erlenmeyer flask with magnetic bar equipped with a reflux condenser. It was immersed into a constant temperature oil bath which put on a hot plate and magnetic stirrer in order to provide agitation. The experimental setup is shown in Figure 3.1.

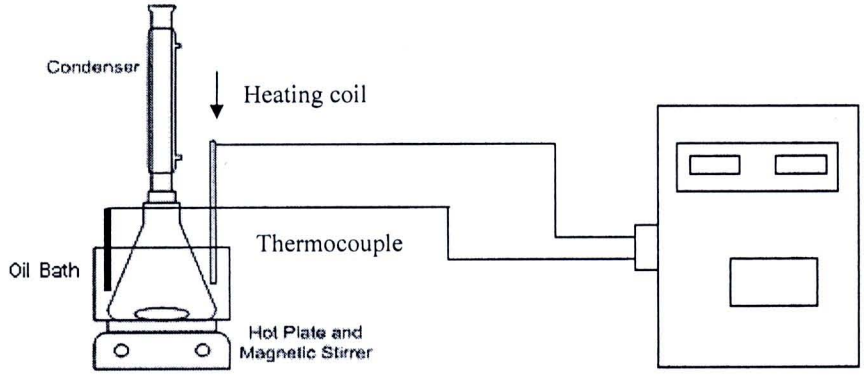

Figure 3.1 The experimental apparatus setup for esterification reaction

Table 3.1 The chemicals used in the experiments

Chemical	Purify	Supplier
Hexanol	98.0%, AR Grade	UNILAB
4-methyl-2-pentanol	98.0%, AR Grade	ALDRICH
Cyclohexanol	98.0%, AR Grade	BDH
Octyl alcohol	-	Thai Oleochemicals Co.,Ltd.
Lauryl-myristyl alcohol	-	Thai Oleochemicals Co.,Ltd.
Cetyl-stearyl alcohol	-	Thai Oleochemicals Co.,Ltd.
Sulfuric acid	98.08%, AR Grade	Mallinckrodt Baker
Phenolphthalein	AR Grade	LABCHEM
Sodium hydroxide	AR Grade	UNILAB
Potassium hydroxide	98.0%, AR Grade	Ajax Finechem
Toluene	AR Grade	Ajax Finechem
Isopropanol	99.8%, AR Grade	QEeC™
Hydrochlolic acid	35.4%, AR Grade	BDH
Ethanol	95.0%, AR Grade	Fisher Scientific

Table 3.2 The apparatus used in the properties test of alkyl esters

Apparatus	Model	Manufacturer
Automatic density meter	DMA 4500	Anton Parr
Automatic viscometer	CAV 2200	Cannon
Automatic pour point	CPP5Gs	ISL
Automatic flash point	APM-7	ISL
Precision colorimeter	PFX 990	Lovibond
Copper corrosion bath	-	Stanhope seta

3.2 Experimental procedure

This section consists of alkyl esters production step and properties verification. The main objective of this thesis is alkyl esters production as lubricating base oil. Therefore, the production step of alkyl esters is carefully handled. The diagram of the production procedure of alkyl esters via esterification reaction is shown in Figure 3.2.

3.2.1 Palm fatty acid distillate analysis

The palm fatty acid distillate was analyzed the kinematic viscosity (ASTM D445), pour point (ASTM D97), flash point (ASTM D92), acid value (AOCS Cd-3D-63) and saponification value (AOCS Cd-3B-76) in order to check its properties based on density at 60 °C.

3.2.2 Esterification of palm fatty acid distillate with various alcohols using sulfuric acid as catalyst

3.2.2.1 Palm fatty acid distillate preparation

Palm fatty acid distillate which as a reactant in this study was prepared with heating it up to 100 °C and stirring using a magnetic stirrer for 30 minutes to get rid of the water.

3.2.2.2 Alkyl esters preparation

The alkyl esters in each sample were prepared from palm fatty acid distillate via esterification reaction with the different types of alcohol. Those are hexanol, 4-methyl-2-pentanol, cyclohexanol, octyl alcohol, lauryl - myristyl alcohol and cetyl - stearyl alcohol. In preparation, the amount between palm fatty acid distillate and each alcohol were set as 100% excess from stoichiometric ratio (2:1). Moreover, the amount of sulfuric acid as a catalyst in this reaction was also set as 3% wt. of palm fatty acid distillate. The amount of various alcohols and sulfuric acid in each experiment were

calculated that is shown in Appendix B. The reaction time for esterification reaction is 3 hours in each experiment. Therefore, the factor affecting of alkyl esters preparation in this study is the types of alcohol (hexanol, 4-methyl-2-pentanol, cyclohexanol, octyl alcohol, lauryl - myristyl alcohol and cetyl - stearyl alcohol).

The details of procedures are as follow: First, 300 grams of the prepared palm fatty acid distillate was filled into an Erlenmeyer flask (1000 ml). Then, mixing of sulfuric acid (3% by weight of palm fatty acid distillate) and various alcohol (hexanol, 4-methyl-2-pentanol, cyclohexanol, octyl alcohol, lauryl - myristyl alcohol and cetyl - stearyl alcohol) in homogeneously phase are put into the Erlenmeyer flask which existed with palm fatty acid distillate and heated up to desired temperature with a reflux condenser to avoid each alcohols losing while reaction occurred. The reaction temperature is 130°C. The mixture was mixed vigorously using magnetic stirrer with a reflux condenser set. Leave reaction continue until reaching the desired time (3 hours) for esterification reaction. Then, the sample should be separated by funnel at room temperature for 1 hours in order to divide the solutions into two layers (the upper layer is the solution of alkyl esters and the lower layer is the solutions of water) and separate from each other.

After that, the upper solution (alkyl esters) and lower solution (water) was weighed and boiling to remove the residual alcohols by vacuum distillation. However, after the time passed for 3 hours, the reaction maybe not completed. Therefore, the incompletely alkyl esters product should be re-esterification again by following the experiment procedure until the reaction was completed.

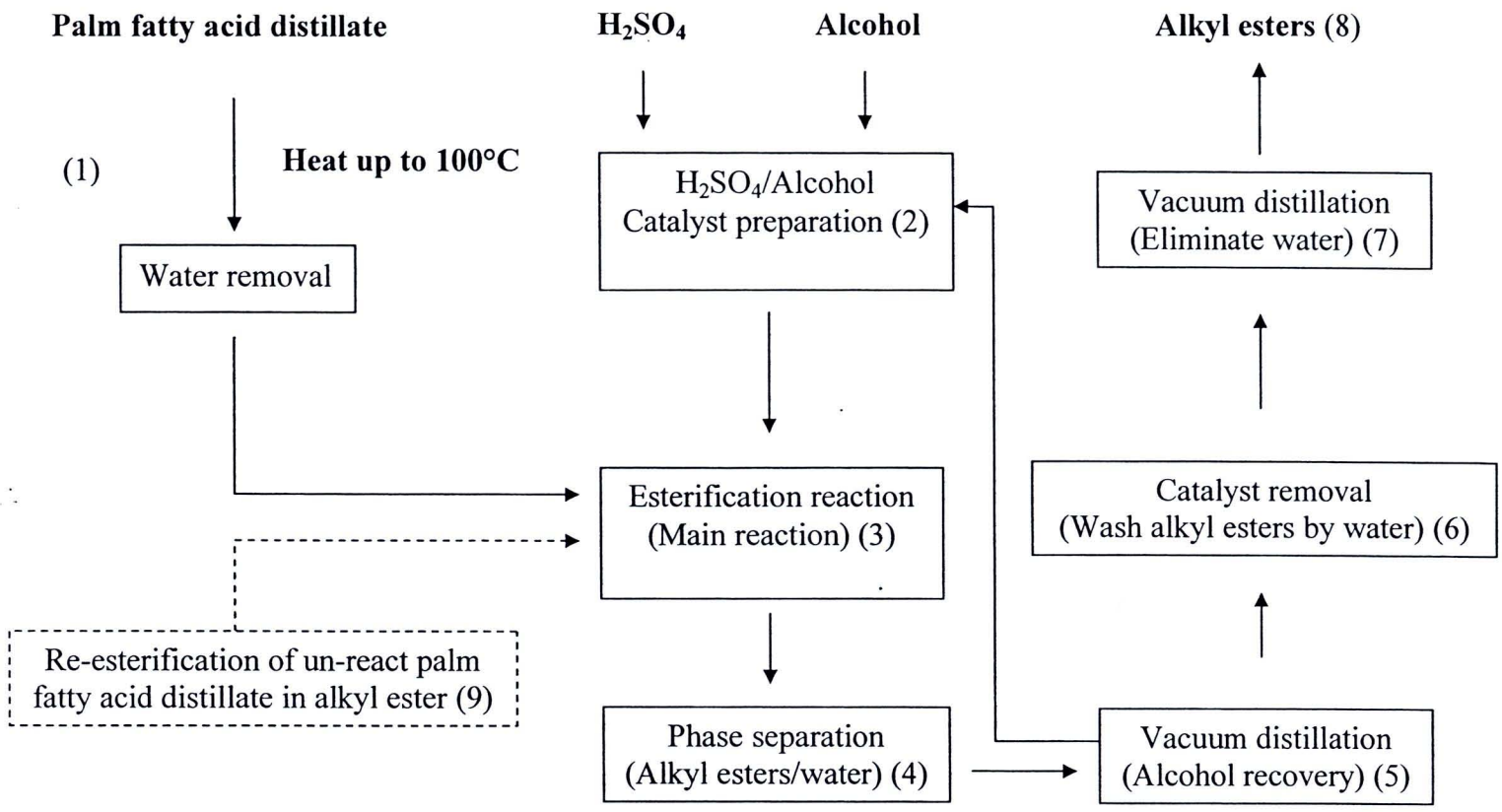

Figure 3.2 The diagram of esterification of palm fatty acid distillate concludes the following: feed stock pretreatment (1), catalyst preparation (2), esterification reaction (3), phase separation (4), alcohol recovery (5), catalyst removal (6), eliminate water (7), alkyl esters product (8) and re-esterification of un-react palm fatty acid distillate in alkyl ester (9)

In the next step, the alkyl ester product was washed with water until pH of the washing water increased to 7 to avoid the product from acidity and remove the catalyst. Finally, the water presence in the product is eliminated in vacuum distillation set before analyzing physical and chemical properties (kinematic viscosity, viscosity index, flash point, pour point, ASTM color, API gravity, weld load and copper strip corrosion). The condition for each experiment was shown in Table 3.3.

Table 3.3 Conditions used for esterification reaction of palm fatty acid distillate with various alcohol

Type of alcohol	**Palm fatty acid distillate (g)**	**Amount of alcohol (g)**	**Amount of sulfuric acid (g)**	**Temperature (°C)**
Hexanol	300	227.0	9	130
4-methyl-2-pentanol	300	227.0	9	130
Cyclohexanol	300	222.6	9	130
Octyl acohol	300	349.9	9	130
Lauryl-myristyl alcohol	300	426.3	9	130
Cetyl-stearyl alcohol	300	583.2	9	130

3.3 The physical and chemical properties characterization

3.3.1 Alkyl esters characterization

In last step, the product (alkyl esters) was analyzed the physical and chemical properties as followed:

1. Kinematic viscosity at 40 and 100°C	by ASTM D445
2. Viscosity Index	by ASTM D2270
3. API gravity	by ASTM D1298
4. Pour point	by ASTM D97
5. Flash point	by ASTM D92
6. Color	by ASTM D1500
7. Four-ball test	by ASTM D2266
8. Copper strip corrosion	by ASTM D130

All physical and chemical properties can indicate the alkyl esters qualification that produces from palm fatty acid distillate with various alcohols.

3.4 Experimental and Analytical error

3.4.1 Experimental error

In this section, the experiments are conducted to verify repeatability, an average, and a standard deviation value of the experiment. Equation (3.5) and (3.6) defined an average value and percent deviation respectively.

$$\text{Average value, } \bar{x} = \frac{\Sigma x}{n} \tag{3.5}$$

$$\text{Percent deviation from average value} = \sqrt{\frac{\Sigma(X-\bar{X})^2}{n-1}} \times 100 \tag{3.6}$$

Some experiments are repeated to study the error that might occur during each experiment of alkyl esters production. The experiment is repeated for 3 times at the same condition to verify the result of experiment. The experiment error of production of alkyl esters was shown in Appendix D.