CHAPTER III

EXPERIMENTS AND ANALYSIS TECHNIQUES

3.1 Experimental apparatus

In this study, a 1000-ml conical flask was used as a reactor for transesterification reaction. The flask equipped with a condenser and thermometer. The reactor was immersed in a water bath; a water bath was place on the hotplate magnetic stirrer for heating and agitating. The system was designed and constructed in order to operate at temperature of 60-65°C. A schematic diagram of the system is shown in Figure 3.1.

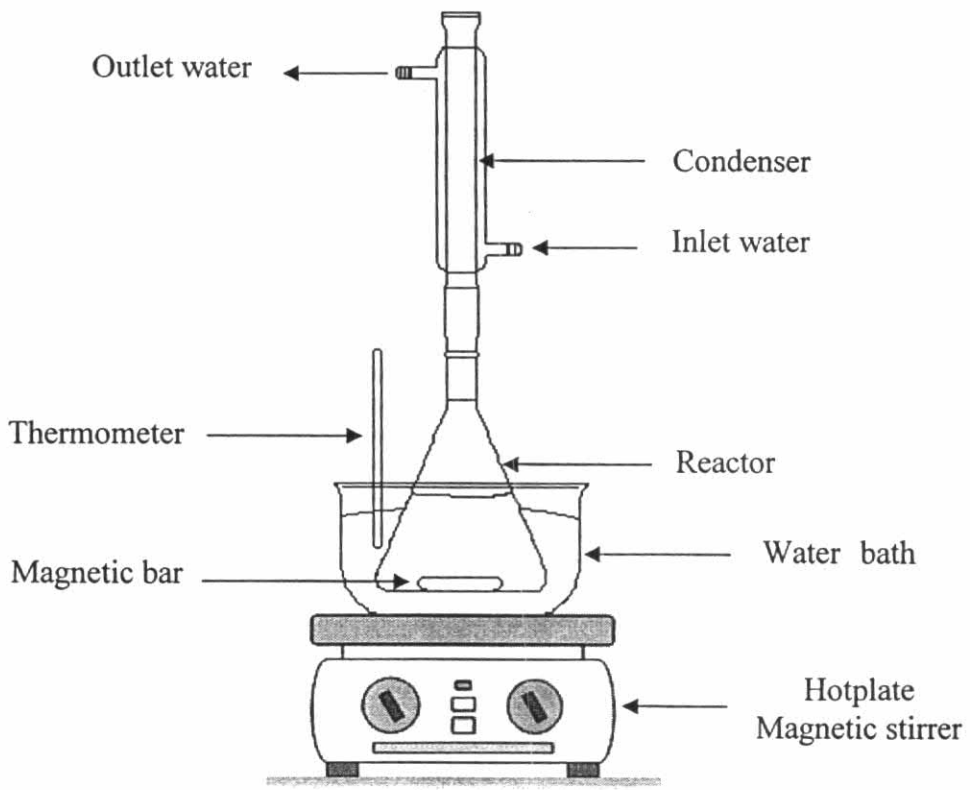

Figure 3.1 Schematic diagram of system

3.2 Materials and chemical reagents

3.2.1 Palm olein, palm stearin, and used palm olein

Vegetable oil used in this studied were palm olein, palm stearin, and used palm olein. Palm olein was purchased from supermaket in Thailand: Oleen brand, Palm oleen CO,

ltd. Palm stearin was obtained from "Verasuwan" biodiesel industry and used palm olein is obtained from frying shop.

3.2.2 Chemical reagents

All chemical reagents used in the experiment are shown in Table 3.1.

Table 3.1 Chemical reagents used in the experiments

Name	Source	Purity
Methanol	-	95% (Commercial grade)
Ethanol	Fisher Scientific	95%
Iso Propanol	Fisher Scientific	99.8%
Toluene	Fisher Scientific	98%
Potassium hydroxide	Ajax Chemicals	85%
Sodium hydroxide	Ajax Chemicals	98%
Sodium methoxide	ACROS ORGANICS	97%
Sulfuric acid	Ajax Chemicals	98%
Hydrochloric acid	J.T. Baker	36.5-38%
Acetone	J.T. Baker	99.7%
Phenolphthalein Indicator	Fisher Scientific	General purpose grade
Bromophenol Blue Indicator	Fisher Scientific	General purpose grade

3.3 Experimental Procedures

3.3.1 Basic catalyzed process

3.3.1.1 Transesterification step

Prior to start the experiment, palm oils feedstocks were obtained to determine free fatty acid content. After that, palm oil was heated to 100°C to reduce the water content. 500-g of palm oils was weighted and put into 1000-ml conical flask, and then a magnetic bar is put into the flask. The magnetic bar was use for agitation during reaction. The flask was connected with condenser and then palm oil was heat to 60°C. Prepared the methanol and catalysts were added into the flask. The stoichometric ratio of methanol to oil for the reaction is 3:1, however 6:1 molar ratio

of methanol to oil was used in this study. The excess of methanol shifts the equilibrium to the right side. The concentrations of basic catalyst as potassium hydroxide at 0.5 and 1.0 percent based on weight of oil were used. The reaction was starting at 60°C and stirring about 600 rpm for 2 hours.

3.3.1.2 Separation step

At the end of reaction, the reaction products were carried out to separating funnel. In the separating funnel reaction products was separated into two phases; crude biodiesel, in the upper phase, and crude glycerol, in the bottom. Vacuum distillation was used to remove methanol from both phase of reaction products. Sample were taken from both phase and were kept for analysis to determine the amount of residue catalyst, soap, and methanol. Crude biodiesel was added to the washing step.

3.3.1.3 Washing step

Once the phase separation had been accomplished, crude biodiesel was purification by washing techniques. This technique is a simple technique for biodiesel purification. The procedure for this step is as following; weigh the amount of crude biodiesel to determine the amount of washing water to be used. The weight of washing water is approximately equal to weight of crude biodiesel. After that, the washing water is divided equally into three parts. The washing water was heat to 60-70°C before use. Biodiesel was washed by bubble washing until 6-7 of pH was presented. If the pH of the washed biodiesel is equal 6-7 washing step was finished. Otherwise washing step should be continued to the second time and third time until the pH is at 6-7. At the end of washing step, the amount of washed water is measured. Then sample of washing water should be taken to determine amount of the residue catalysts and amount of soaps. Washed biodiesel was heated to 100°C to evaporate the water content. The amount of the refined biodiesel was measured. The sample was taken to determine amount of residue catalysts and soaps.

3.3.2 Acid catalyzed process

3.3.2.1 Transesterification step

Palm olein, palm stearin and used palm olein were analyzed for free fatty acid content. After that, palm oil was heated to 100°C to reduce water content. 500 g of palm oil was weighted and put into 1000-ml conical flask, then a magnetic bar is put

into the flask. The magnetic bar was use for agitation during reaction. The flask was connected with condenser and then palm oil was heat to 60°C. Prepared methanol and catalysts were added into the flask. The stoichometric ratio of methanol to oil for the reaction is 3:1, however 6:1 molar ratio of methanol to oil was used in this study. The excess of methanol shifts the equilibrium to the right side. The concentrations of acid catalyst as sulfuric acid at 0.5 and 1.0 percent based on weight of oil were used. The reaction was starting at 60°C and stirring about 600 rpm for 2 hours.

3.3.2.2 Seperation step

At the end of reaction, the reaction products were carried out to separatory funnel. In the separatory funnel reaction products was separated into two phases; crude biodiesel, in the upper phase, and crude glycerol, in the bottom. Vacuum distillation was used to remove methanol from both phase of reaction products. Sample were taken from both phase and were kept for analysis to determine the amount of residue catalysts and methanol. Crude biodiesel was added to the washing step.

3.3.2.3 Washing step

Once the phase separation had been accomplished, crude biodiesel was purification by washing techniques. This technique is a simple technique for biodiesel purification. The procedure for this step is as following; weigh the amount of crude biodiesel to determine the amount of washing water to be used. The weight of washing water is approximately equal to weight of crude biodiesel. After that, the washing water is divided equally into three parts. The washing water was heated to 60-70°C before use. Biodiesel was washed by bubble washing until 6-7 of pH was presented. If the pH of the washed biodiesel is equal 6-7 washing step was finished. Otherwise washing step should be continued to the second time and third time until the pH is at 6-7. At the end of washing step, the amount of washed water is measured and defined as waste water. Then sample of washing water should be taken to determine amount of the residue catalysts and amount of soaps. Washed biodiesel was heated to 100°C to evaporate the water content. The amount of the refined biodiesel was measured and the sample was taken to determine amount of residue catalysts.

3.4 Analysis Techniques and Method

Production of biodiesel using basic catalyst always produces some amount of soap. After the transesterification reaction was completed, the residue catalysts and soaps tend to concentrate in the glycerol phase. However, some soap and small amount of catalyst may be left in the biodiesel phase. It can be useful to know amount of soap formed, where the catalyst residue, and how effective the washing process is in removing these two compounds.

A standard method for determine catalysts and soaps can be used to measure the amount of soaps and catalysts. The basic procedure consists of titrating samples of the phase to be analyzed, such as crude glycerol, crude biodiesel, refined biodiesel, and wash water, with a 0.1 normal solution of hydrochloric acid to the phenolphthalein end point. This gives an estimate of the amount of catalysts. Then, a few drops of bromophenol blue indicator were added and the titration continued to the color change for that indicator. This gives an indication of the amount of soap. In the first titration, the hydrochloric acid neutralizes basic catalyst, so when the phenolphthalein indicates that the solution has become neutral, then all of the catalyst has been counted. If the titration is continued, the hydrochloric acid, as a strong acid, begins to split the soap molecules to free fatty acids and salt. When the pH reaches about 4.6, where the bromophenol blue changes color, this indicates that the hydrochloric acid has split all of soap.

To determine residue acid catalyst in sample, after the transesterification reaction is completed, a simple titration procedure can be used to measure the amount of catalyst. The basic consists of the phase to be analyzed with a 0.1 N sodium hydroxide solution to the phenolphthalein end point.