

CHAPTER III

EXPERIMENTAL AND ANALYTICAL TECHNIQUES

This chapter is explained the experimental and analytical techniques for study about demulsification of oil – in – water emulsion by electric field.

3.1 Experimental Techniques

The experiments are operated in an acrylic rectangular horizontal vessel in size wide 3.0 cm height 2.5 cm length 8.0 cm. The electrodes are made by stainless steel plate size wide 2.5 cm length 12 cm. The electrodes are fixed in parallel with distance between electrodes 2.0 cm. The direct current (DC) (Model GW-6060D) is used as electric field in this experiment. The experimental set up for measurement of demulsification is shown in figure 3.1.

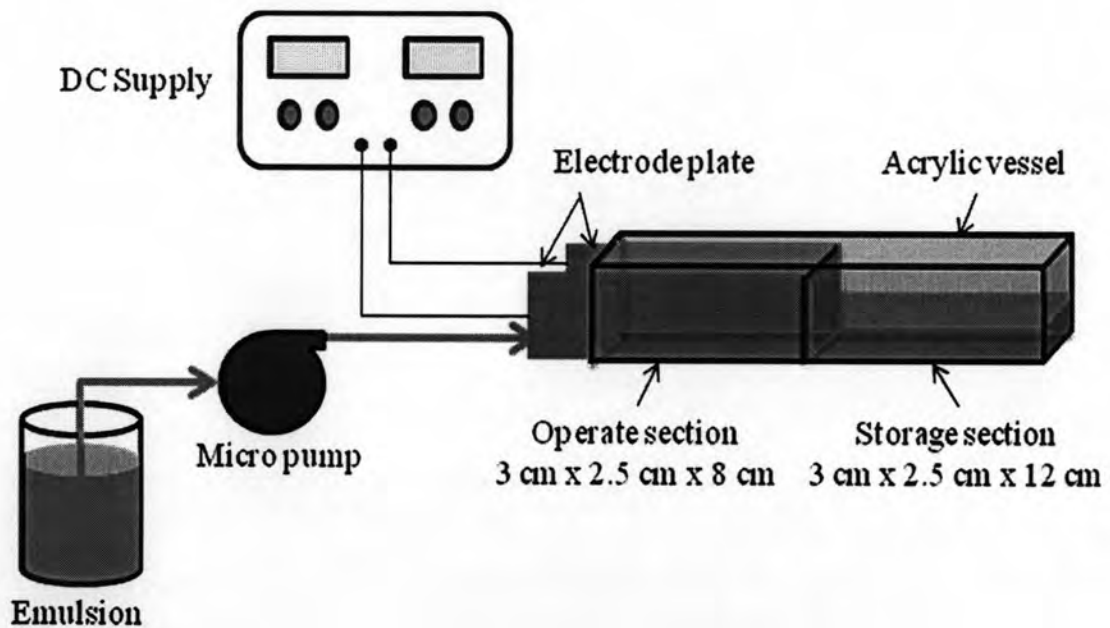


Figure 3.1 Schematic diagrams of the experimental apparatus

The oil – in – water emulsion was prepared by homogenizing the mixture of crude palm oil and deionized water. The homogenizing procedure takes time about 3 minute. Sodium sulfate (Na_2SO_4) as electrolyte must be added to deionized water at concentration 3 mmol/l before homogenizing. Emulsions had 2%wt of crude palm oil in water. There was a feedstock. In batch system, emulsions was poured to the vessel. A sample was taken at specific time. The experiments were operated at voltage 0, 2, 4, 6, 8 and 10 V/cm. In continuous system, emulsions were passed through the vessel by pump at specific flow rate. The overflow of emulsions from operated section was come in storage section. A sample was taken 10 ml every 5 minutes from the bottom of storage section for oil content analysis. The remain of emulsions in storage section after taking a sample was drained, and then started to keep the emulsions for next sample collection. The process was continued until steady state. The steady state condition was established when at least three samples consecutively had the same concentration value by approximate. The experiment were operated at flow rate 3, 5.4, 11, 16 and 20 ml/min and voltage at 0, 2, 4, 6, 8 and 10 V/cm.

3.2 Analytical Techniques

A modified version of ASTM number D4281-93 for determination of oil content in the emulsion is used to determine the amount of palm oil in water emulsion in this study.

Sample Preparation

1. During the demulsification of emulsions by electric fields, the sample was taken 10 ml by syringe at the specific time.
2. The sample was acidified with 1 ml of aqueous solution of 1:1 HCl in a sample test tube. The amount of acid should be enough to obtained pH 2.
3. The sample was centrifuged about 10 minutes.

Analytical Procedures

1. The sample in the test tube is transfered to a seperatory funnel.
2. The test tube is carefully rinsed with 10 ml of hexane. The rinsed hexane is added to the solution in the seperatory funnel.
3. The seperatory funnel is shaken vigorously for 2 min.
4. The seperatory funnel is placed on the ring stand to allow separation of phases.

5. Aqueous phase and small amount of organic phase are drained into original sample test tube.
6. Solvent phase is drained through a funnel containing a filter paper and 10 gram of Na_2SO_4 , both of which have been hexane – rinsed, into a pre – weighted beaker.
7. Aqueous phase and small amount of organic phase are extracted again following step 1 to 6. The solvent phase is added to previous solvent.
8. Hexane is evaporated from solvent phase by placing the beaker in an oil bath controlled at 85°C .
9. After the evaporation is completed, the beaker is dried in an oven at a temperature of 103°C for 15 min.
10. The beaker is allowed to cool down to room temperature in a desiccator before it is weighed.
11. The amount of oil in the sample can be calculated from the weight of oil in the beaker.

A schematic diagrams of analytical apparatus is shown in Figure 3.2.

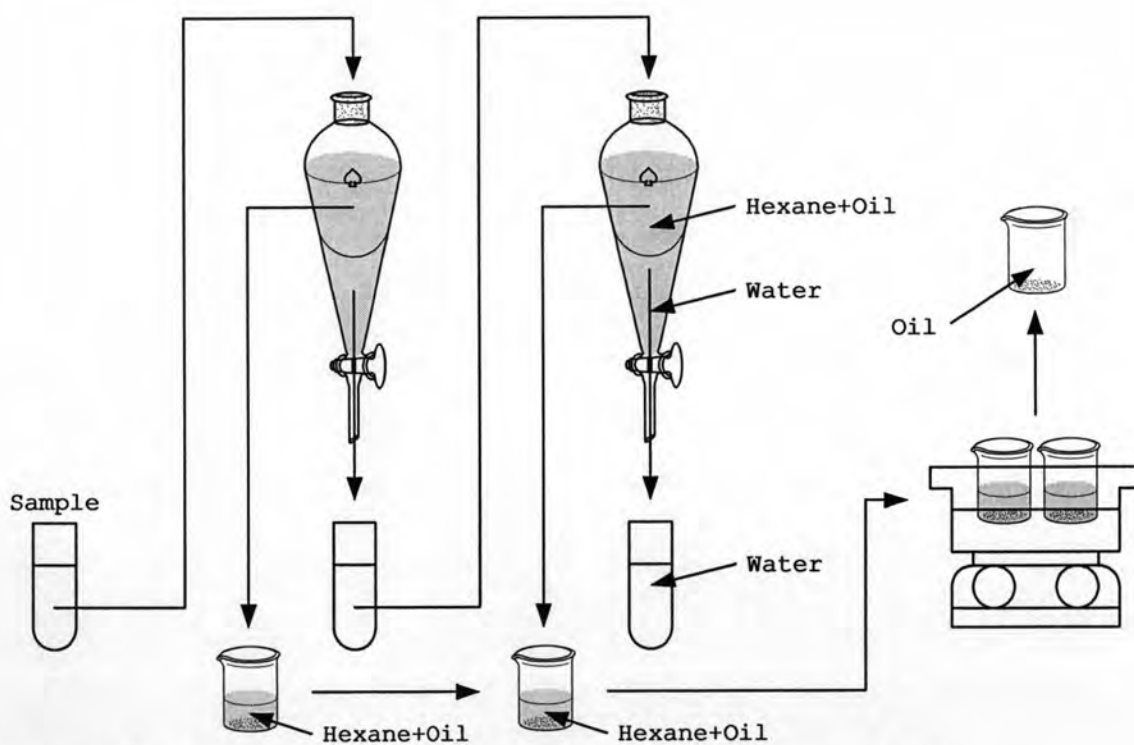


Figure 3.2 Schematic diagrams of analytical apparatus (Amaralikit, 2004).

3.3 Experimental and Analysis Error

3.3.1 Experimental Error

This section is conducted to verify repeatability of the experiments. The results are calculated for their average and standard deviation value by Equations 3.1 to 3.2

$$\text{Average value, } \bar{X} = \frac{\sum x}{n} \quad (3.1)$$

$$\text{Percent deviation from average value} = \frac{\sqrt{(X - \bar{X})^2}}{\bar{X}} \times 100 \quad (3.2)$$

3.3.2 Analytical Error

Three samples of emulsion feed stock were taken from the experiment in the same time. The samples should represent the same oil content. Error in each sample analysis would be error in transferring the oil from an emulsion into hexane.