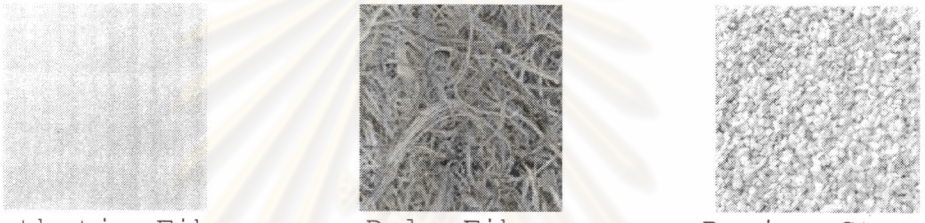
CHAPTER III

EXPERIMENTAL AND ANALYTICAL TECHNIQUES

3.1 Experimental Technique

The experiments were conducted in a vertical packed column, 10 mm. in diameter and 150 mm. in length. Three different coalescing media, natural palm fiber, synthetic fiber and pumice stone are used in this study. Each medium is packed in the column to the height of 100 mm with packing density of 0.29, 0.11 and 0.41 gm/cm^3 for palm fiber, synthetic fiber and pumice stone, respectively. Figure 3.1 shows photographs of each medium.



Synthetic Fiber Palm Fiber Pumice Stone

Figure 3.1 Coalescing media

Palm oil-in-water emulsion used in this study is prepared by homogenizing crude palm oil in deionized water. The concentration of emulsion is 1wt% palm oil. The emulsion is separated into two 500 ml portions and used for a coalescing and gravity separation studies.

Figure 3.2 shows schematic diagram of the experimental system. Palm oil-in-water emulsion is poured in a 500 ml container and is pumped into bottom part of packed column. Exit flow at the top part of the packed column is returned to the container. A 10 ml sample is taken from the container every 20 minutes during each experiment which has a total time of 100 minutes. Temperature of the system is controlled by placing the container on a temperature controlled oil bath. The experiments were conducted at temperatures of 60, 70 and 80°C and flow velocities of 0.12, 0.25, 0.40 and 0.50 mm/sec

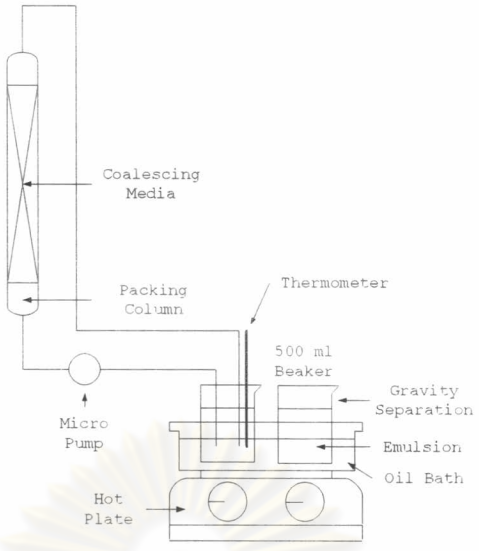


Figure 3.2 Schematic diagrams of the experimental apparatus

3.2 Analytical Technique

A modified version of Partition-Gravimetric for determination of oil content in the emulsion is used to determine amount of palm oil in water emulsion in this study.

Sample Preparation

1. Ten milliliters of sample is taken every 20 minutes during each experiment.
2. The sample is then acidified with 1 ml of aqueous solution of 1:1 HCl in a sample test tube. The amount of acid should be enough to obtain a pH of 2.
3. The sample is centrifuged for 5 minutes.

Analytical Procedures

1. The sample in the test tube is transferred to a separatory funnel.
2. The test tube is carefully rinsed with 10 ml hexane. The rinsed hexane is added to the solution in the separatory funnel.
3. The separatory funnel is shaken vigorously for 2 min.
4. The separatory 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 to room temperature in a desiccator before it is weighted.
11. Amount of oil in the sample can be calculated from the weight of oil in the beaker.

A schematic diagram of analytic apparatus is shown in Figure 3.3.

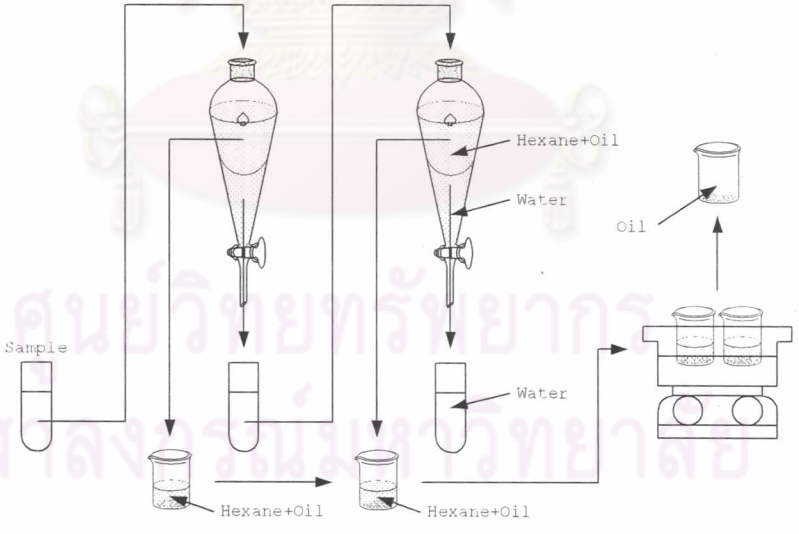


Figure 3.3 Schematic diagrams of analytic apparatus

3.3 Experimental and Analytical Error

Experimental Error

This section is conducted to verify repeatability of the experiments. The results are calculated for their average values, percentage of maximum and percentage of minimum errors. Equations 3.1 to 3.3 are used for calculation of average value, percentage of maximum and percentage of minimum errors of the experiment. Four experiments are conducted using palm fiber bed packed and are operated at a temperature of 80°C and a flow velocity of 0.5 mm/sec.

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

$$\%\text{Maximum error} = \frac{\text{maximum conc.} - \text{average conc.} \times 100}{\text{average conc.}} \quad (3.2)$$

$$\%\text{Minimum error} = \frac{\text{average conc.} - \text{minimum conc.} \times 100}{\text{average conc.}} \quad (3.3)$$

The results are summarized in Table 3.1 and illustrated in Figure 3.4.

Table 3.1 %wt oil content in study on experimental Error of flow through the palm fiber at 80°C and 0.5 mm/sec

Time (min)	Experiment (%wt Oil Content)				Average (%wt)	Max Error (%)	Min Error (%)
	01	02	03	04			
0	0.83	0.81	0.90	0.83	0.85	6.74	3.90
20	0.73	0.72	0.67	0.76	0.72	5.73	7.05
40	0.61	0.64	0.64	0.62	0.63	2.35	2.27
60	0.55	0.53	0.59	0.55	0.56	5.71	4.00
80	0.47	0.45	0.42	0.50	0.46	9.45	9.66
100	0.46	0.42	0.40	0.47	0.44	6.64	8.70
				Average percent error		6.10	5.93

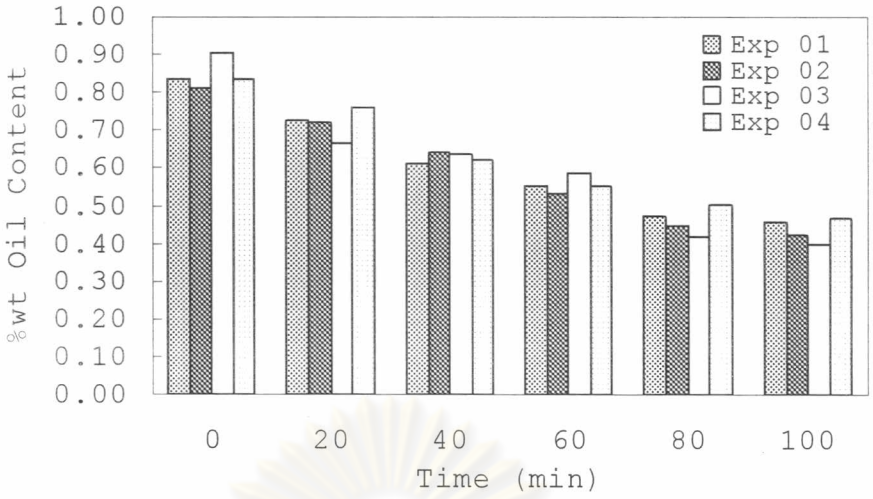


Figure 3.4 %wt oil content in study on experimental Error of floe through the palm fiber at 80°C and 0.5 mm/sec

The result shows that the average experimental error is approximately 6%.

Analytical Error

Four samples are taken from an experiment at same time. The samples should represent the same amount of oil. Error in each sample analysis would be error in transferring the oil from an emulsion into hexane. The results are summarized in Table 3.2.

Table 3.2 %wt oil content in study on analytical Error.

Experiment (%wt Oil Content)					Average (%wt)	Max Error (%)	Min Error (%)
01	02	03	04	05			
0.82	0.83	0.82	0.80	0.81	0.82	2.02	2.11

The result shows that the average analytical error is approximately 2%.