

## CHAPTER IV

### RESULTS AND DISCUSSION

#### 4.1 Microemulsion Phase Study

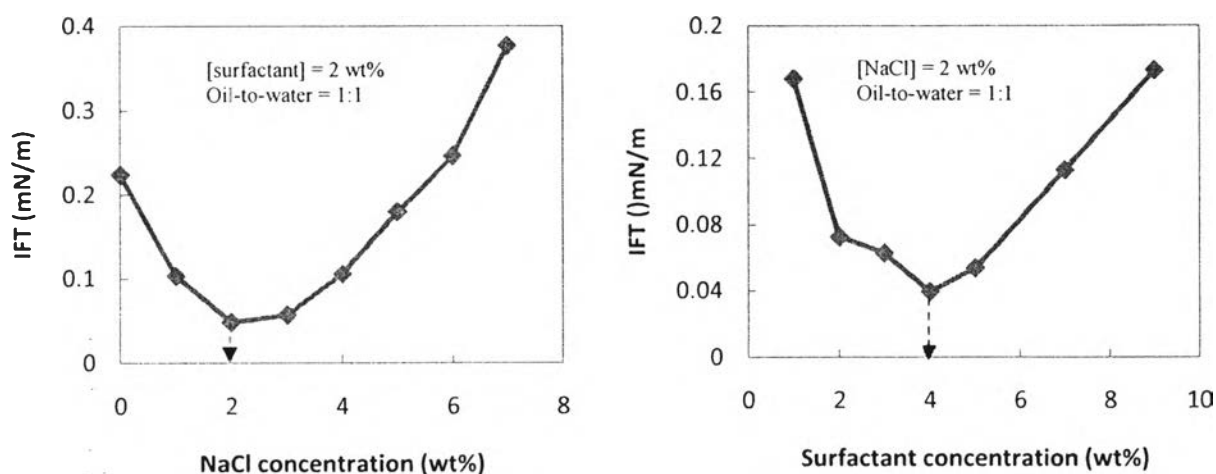
In this study,  $C_{14-15}(PO)_4SO_4Na$  was selected to form microemulsion with motor oil, which was found to exhibit low IFT. However, this studied system exhibited only two obvious phases, the water and oil excess phases. For the layer of middle phase, it could not be clearly observed visually. Consequently, the measurement of the phase transformation became difficult to identify whether the system had a middle phase or not. Hence, the phase diagram of motor oil with alfoterra is not shown here. The IFT of the system was measured by the spinning drop tensiometer to examine the existence of Winsor Type III microemulsions. The diagrams of IFT as a function of surfactant concentration and salinity are illustrated here.

##### 4.1.1 Effect of Surfactant Concentration on IFT

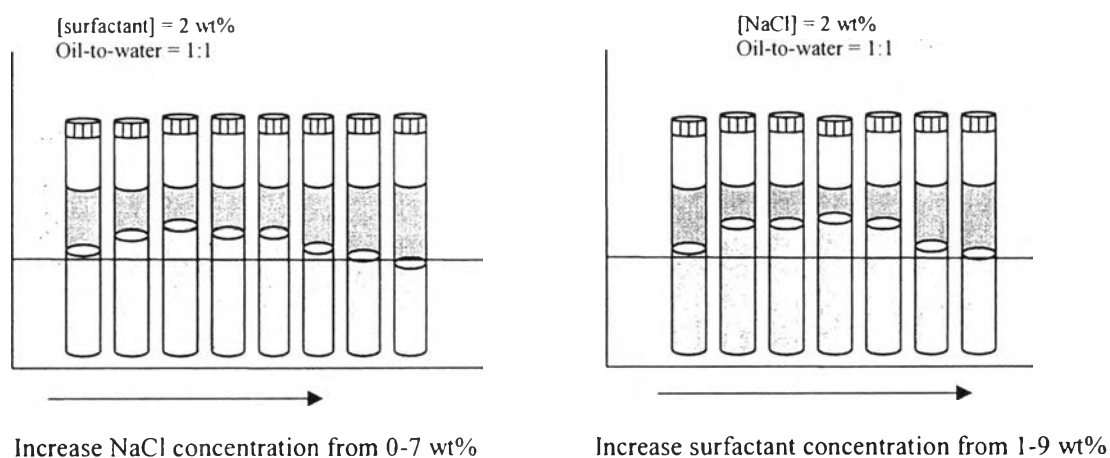
Figure 4.1 illustrates the effect of Surfactant Concentration on IFT at 2 wt% salinity and oil to water initial volumetric ratio of 1:1. The IFT of the system decreased rapidly when surfactant concentration increased from 1 to 2 wt% and reached minimum at 4 wt% surfactant concentration. Beyond the optimum surfactant concentration, the IFT increased with increasing surfactant concentration. This is because the repulsive force between the anionic head groups of Alfoterra increases with the increase in the Alfoterra concentration. Therefore, micelle is difficult to form leading to lower oil solubilization. The IFT also showed that microemulsion was not formed ( $>10^{-3}$  mN/m). The phase height was shown in figure 4.2

##### 4.1.2 Effect of NaCl Concentration on IFT

From the result, The IFT decreased with increasing NaCl concentration from 1 – 2 wt% and it reached minimum at 2 wt% NaCl concentration which is the optimum condition for experiment. After that, IFT increased with increasing NaCl concentration. This is because when NaCl is added into the system, it reduces the repulsive force between anionic head groups resulting in increasing aggregation



**Figure 4.1** The effect of surfactant and NaCl concentration on interfacial tension (IFT).

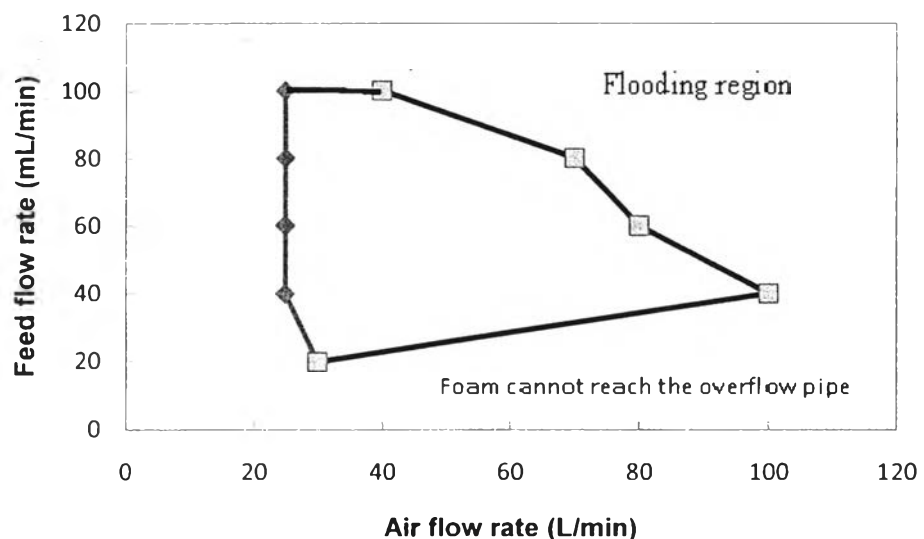


**Figure 4.2** The effect of surfactant and NaCl concentration on phase height.

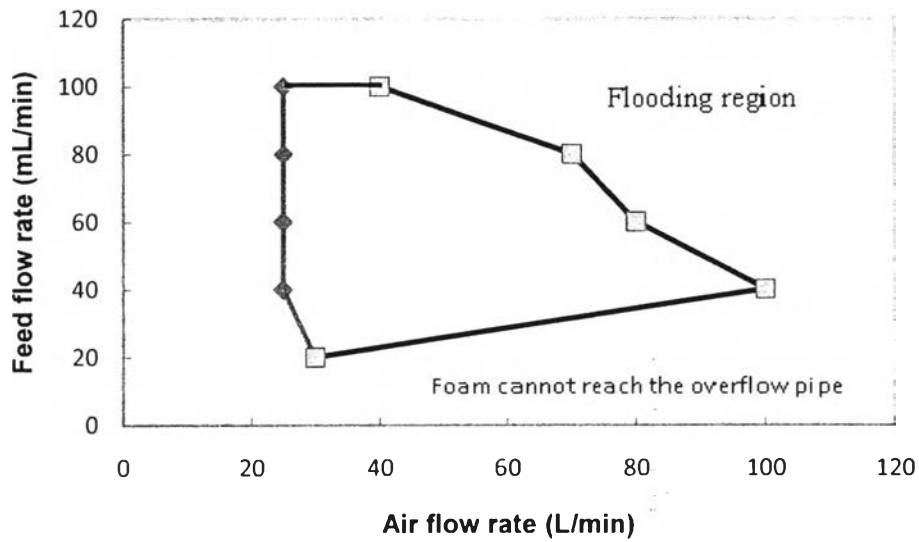
numbers, so the amount of solubilized oil into the inner core micelles increases leading to the reduction of IFT. At very high NaCl concentrations, the charge at the head group of surfactant is neutralized, so the effect of hydrophobic group seems to be predominant. The Winsor type was change from type I to type II as an increase in IFT.

## 4.2 Operational Zones

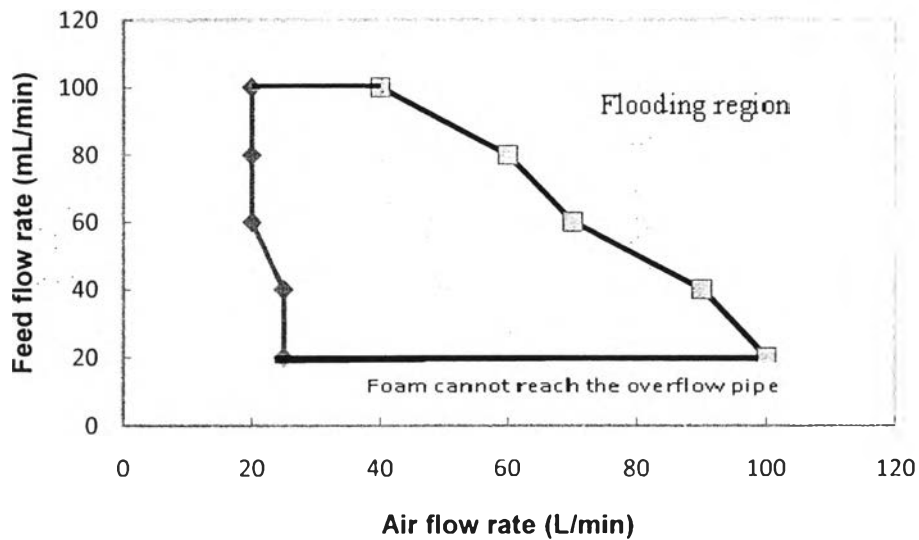
To operate a multistage froth flotation successfully, one has to consider two important process constraints: foamability (foam formation) and flooding. To achieve surfactant separation, a sufficient air flow rate is needed to produce foam outlet of the top stage. Meanwhile, the flooding of the solution in the column may interrupt or reduce the separation efficiency if the system is operated under a very high flow rate of air and/or a very high feed flow rate. Figure 4.2 – 4.4 show the operational regions at different feed concentration. They also show the boundary of the no-foam regions and the flooding region. As shown in figure 4.2-4.4, the liquid flooding in a stage depends on both the liquid flow rate and the air flow rate. The operational zone of each feed concentration is quite similar, but both the boundary lines for flooding and foam forming are slightly different among these three conditions, indicating that the presence of any surfactant concentration can affect the operation of a multistage froth flotation.



**Figure 4.3** Flooding points and operating zones of the multistage froth flotation at a surfactant concentration = 0.1 wt%, foam height = 60 cm and number of stages = 4.



**Figure 4.4** Flooding points and operating zones of the multistage froth flotation at a surfactant concentration = 0.2 wt%, foam height = 60 cm and number of stages = 4.



**Figure 4.5** Flooding points and operating zones of the multistage froth flotation at a surfactant concentration = 0.5 wt%, foam height = 60 cm and number of stages = 4.

### 4.3 Froth Flotation Performance

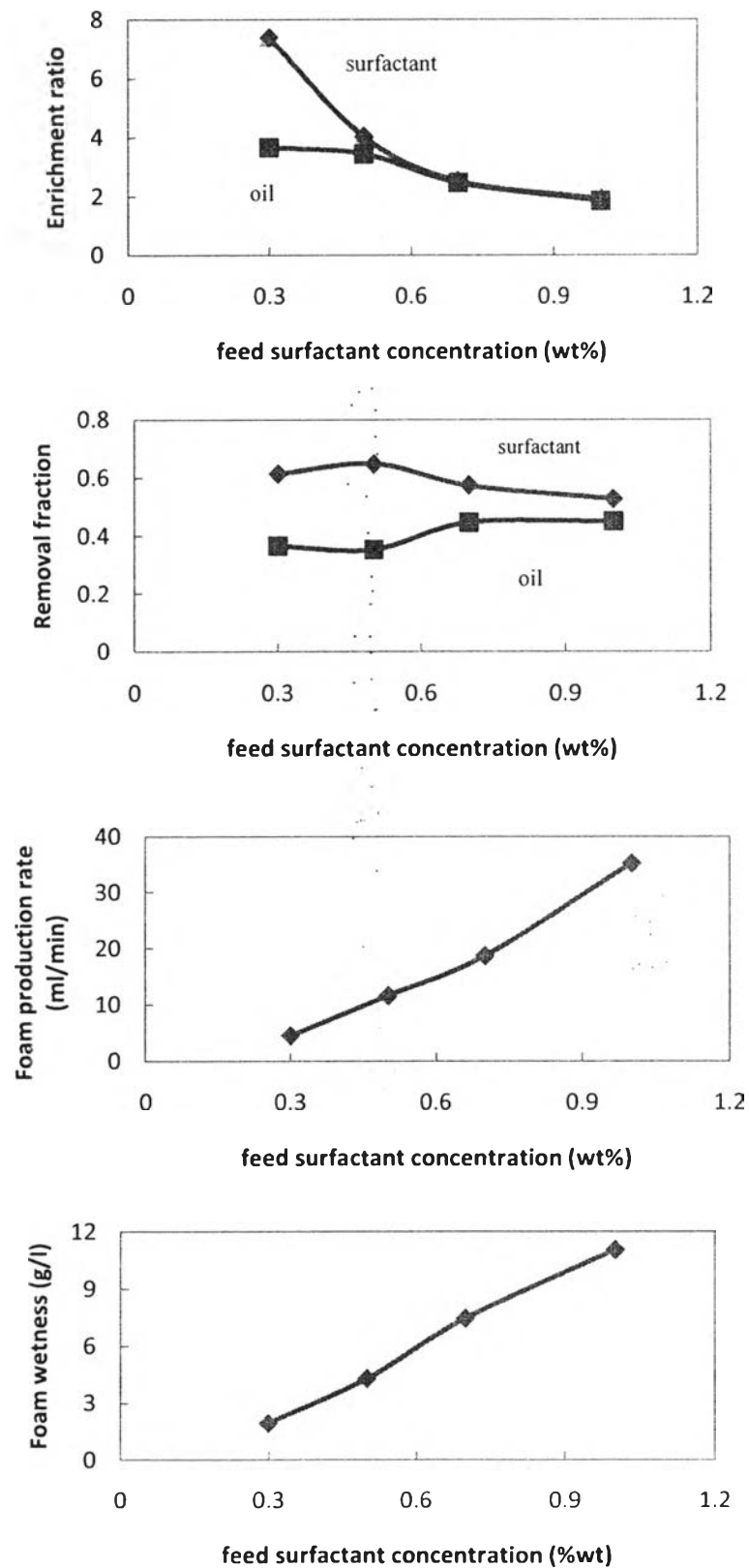
Oil removal and enrichment ratio are significant parameters to indicate the performance of froth flotation process. In addition, the surfactant removal, foam

wetness and foam flow rate should be determined and used for froth flotation performance evaluation.

Generally, high oil removal efficiency is a vital requirement for an effective froth flotation process but it is not the sole factor. The enrichment ratio also indicated the separation efficiency. It is defined as the ratio of concentration of oil in the overhead froth to that in the feed. In order to achieve the separation, the enrichment ratio must be greater than one. Moreover, the higher the enrichment ratio, the better the separation is.

#### 4.3.1 Effect of Feed Concentration

To observe the effect of feed concentration, a feed flow rate was fixed at a constant of 50 mL/min, which is located in the operation zone. An air flow rate was also fixed at 50 L/min and NaCl concentration was fixed at 1 wt%. The effect of feed concentration on the oil and surfactant separation parameter is shown comparatively in fig 4.5. For any given feed concentration, as surfactant concentration increases, increasing foamate production rate and foam wetness explains the lower enrichment ratio of both oil and surfactant. An increase in surfactant concentration results in an increase in the excess surface concentration of the surfactant, leading to a decrease in the surface tension. Moreover, the surfactant can enhance both foamate production rate and foam wetness. The foam becomes more stable and hold more water, thus leading to an increase in foam wetness confirmed experiment. Interestingly, a higher enrichment ratio in multistage froth flotation column occurred at lower surfactant concentration; but this improvement was limited by a minimum surfactant concentration for enough foaming to generate the overhead froth. For the oil removal fraction, it slightly increased and reached maximum at 0.7 wt% of surfactant concentration because of an increase in foamate production rate and foamability. Then, it remained almost unchanged around unity. But the surfactant removal fraction slightly

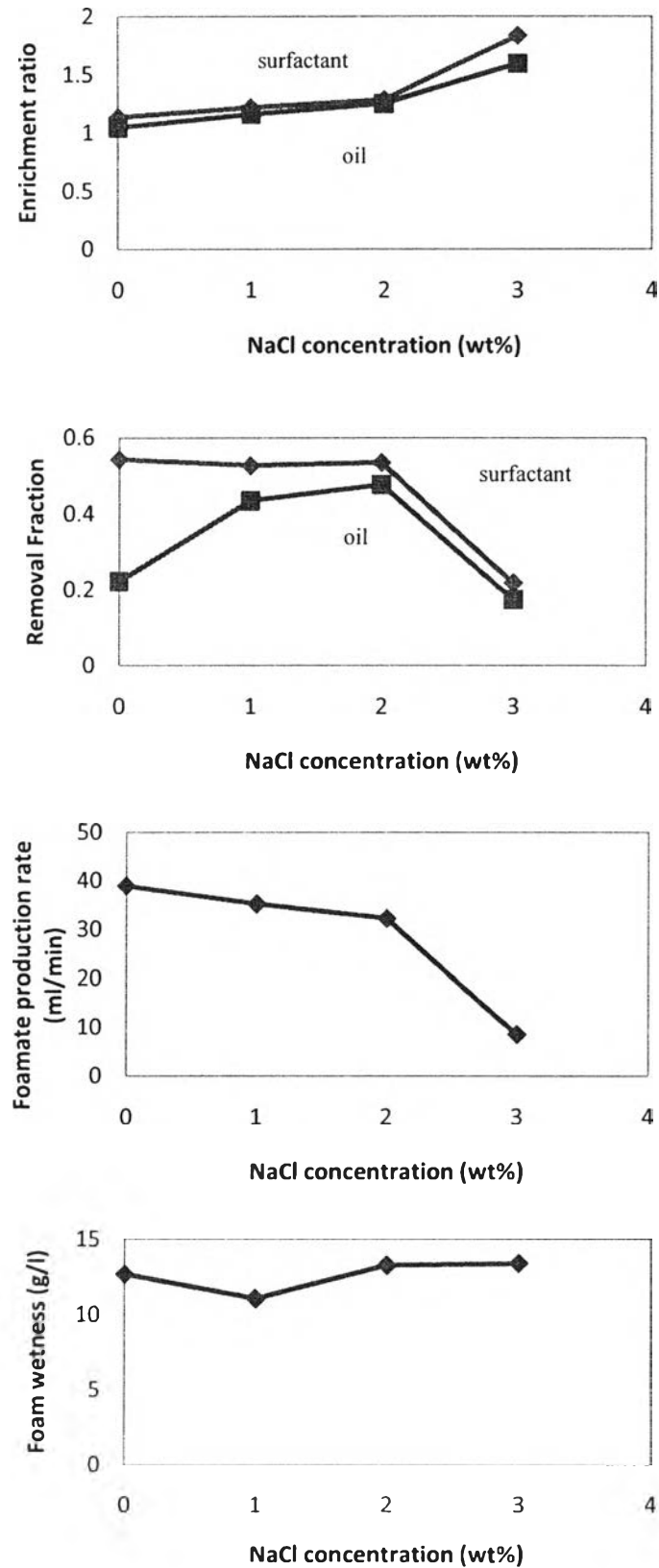


**Figure 4.6** Effect of feed concentration on separation efficiency (air flow rate = 50 L/min; feed flow rate = 50 mL/min and NaCl concentration = 1 wt%.)

decreased and reached maximum at 0.5 wt% of surfactant concentration. Then, it slightly decreased with increasing feed concentration.

#### 4.3.2 Effect of NaCl Concentration

Figure 4.6 shows the effect of NaCl concentration on the oil and surfactant separation parameter. The enrichment ratio of oil and surfactant increased slightly with increasing NaCl concentration in the study range because increasing NaCl concentration decreased the repulsive force between the negative-charged heads of the surfactants. Consequently, the hydrophobic characteristics of the foam surface increase leading to higher surfactant and oil adsorption at the surface of the foam lamellae. The combined effect between this effect and IFT lead to explanation of the increasing in oil removal with increasing NaCl concentration from 0 wt% to 2 wt% . At 3 wt% NaCl concentration, the surfactant removal and oil removal dramatically decreased because the system possessed poor foamability, foam stability and foamate production rate which probably due to dramatically decreasing electrostatic repulsion between the charged surfactant monolayer at the two surfaces of the lamellae. From this effect, foam cannot be generated to reach the froth when the NaCl concentration exceeded 3 wt%. For the foamate production rate, it slightly decreased with increasing NaCl concentration from 0 wt% to 2 wt%. At the NaCl concentration of 3 wt%, the foamate production rate dramatically decreased. This is because further decreasing the repulsive force decreased the thickness of the foam lamella. Hence, the foam lamella can easily collapse leading to the decreasing of the foamate production rate. For the foam wetness, NaCl concentration didn't affect too much

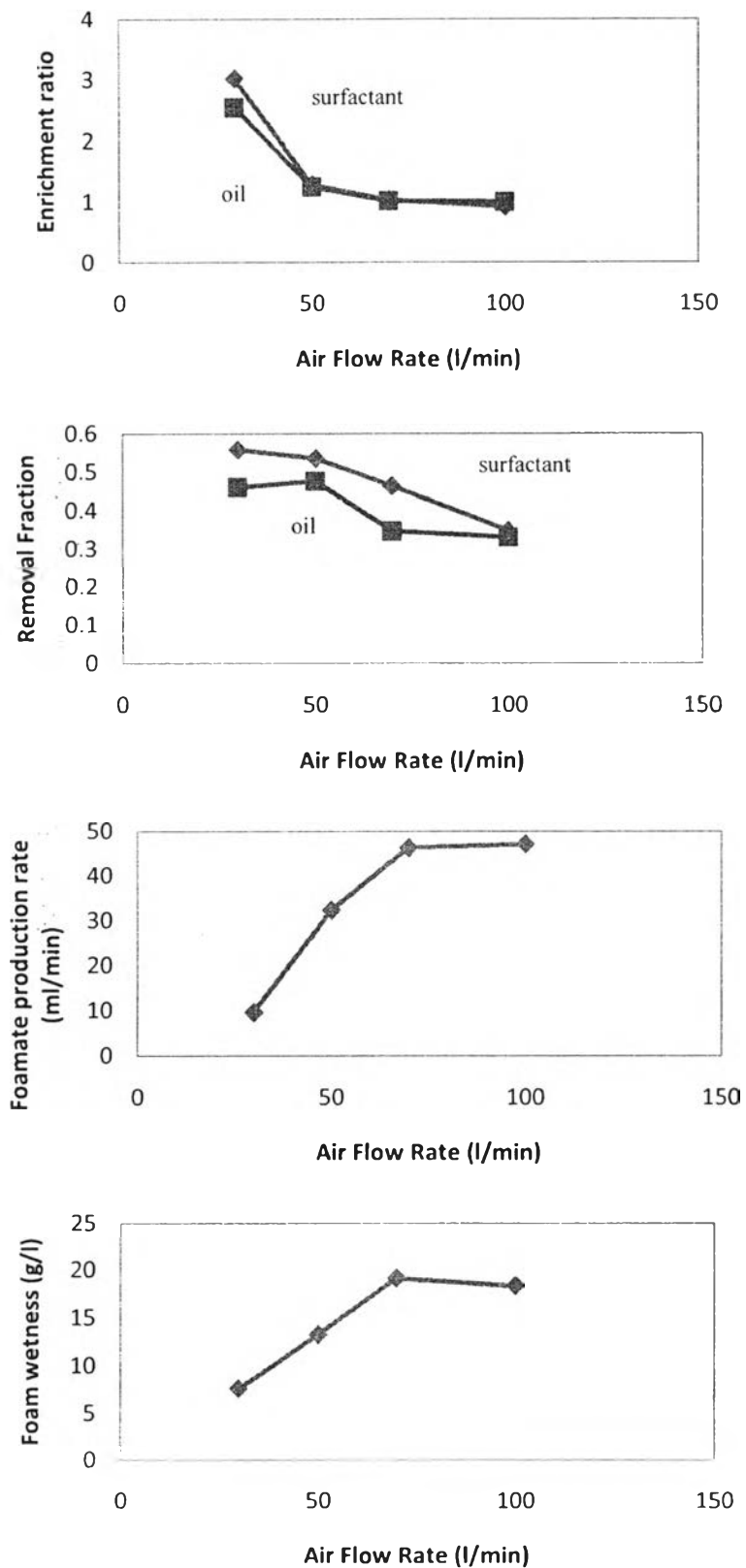


**Figure 4.7** Effect of NaCl concentration on separation efficiency (air flow rate = 50 L/min; feed flow rate = 50 mL/min and surfactant concentration = 0.1 wt%).



#### 4.3.3 Effect of Air Flow Rate

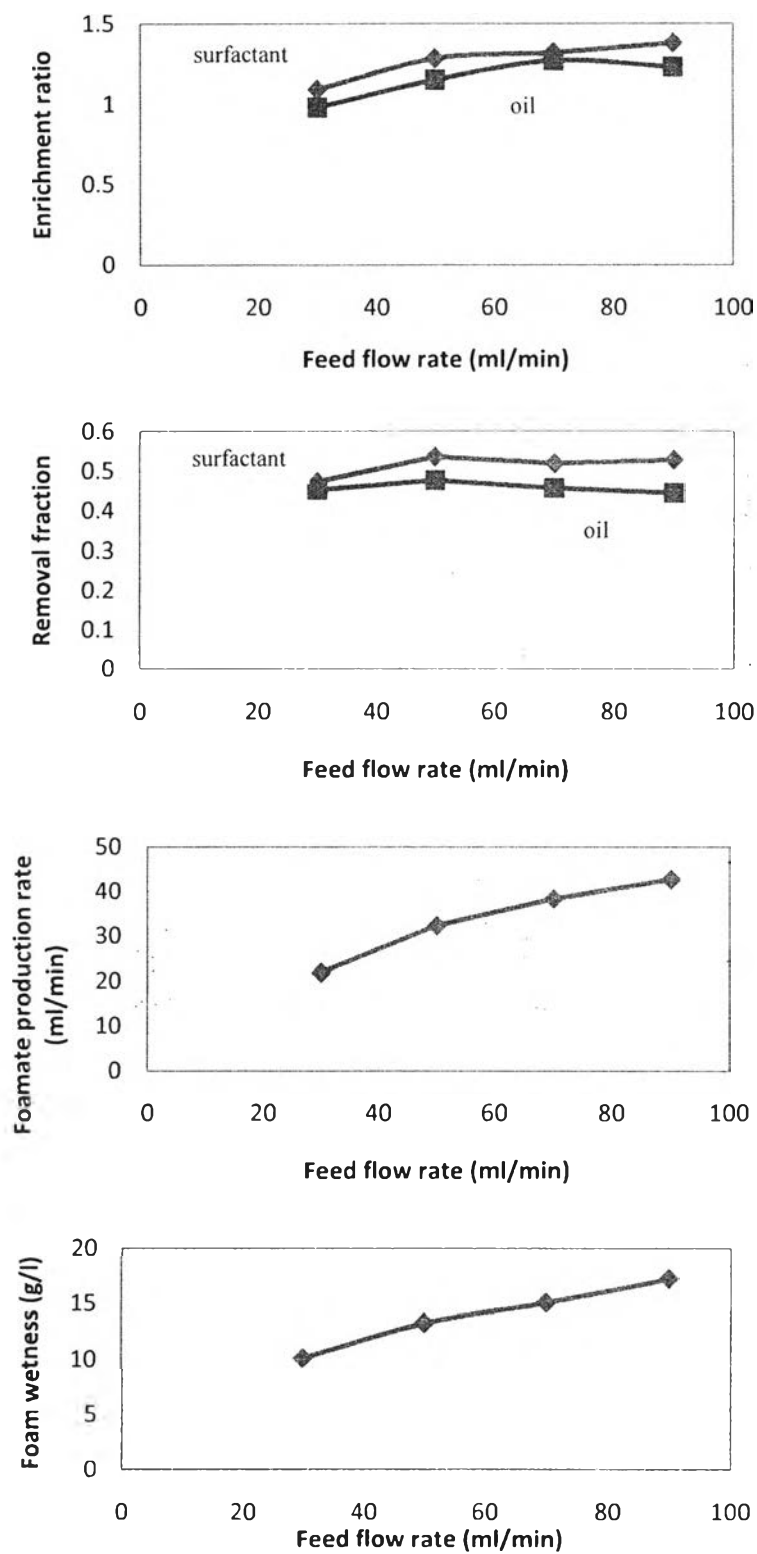
An air flow rate lower than the minimum limit (30 mL/min) caused a low production of foam which collapsed before reaching the overhead outlet at the top of the column. In contrast, an air flow rate greater than the maximum limit (100 mL/min) could not be used because of flooding effect. From figure 4.7, the result shown that the higher air flow rate shown the lower enrichment ratio of surfactant and motor oil. This can be explained in that a higher air flow rate simply produce more bubble passing through the solution, result in less time for water drainage. Hence, a larger amount of water can be carried with the produced froth to the top of the column, leading to an increase in the foam wetness confirmed experimentally. An increase in air flow rate also tended to break the foam as well as to produce wetter foam. However at an air flow rate higher than 50 L/min, the enrichment ratio of oil and surfactant remained unchanged. The removal fraction of motor oil slightly increased with increasing the air flow rate in range of 30 to 50 L/min. Then, it slightly decreased with increasing the air flow rate in range of 50 to 100 L/min because increasing air flow rate lead to have more bubble swarm passing though the solution. Not only the number of bubbles in the column but also the flow pattern in the column that was affected by a high air flow rate. The circulation velocity induced by the bubble swarm rising through the column enhanced the turbulence at the froth/collection zone interface, so some amount of adsorbed motor oil in the froth was entrained back into the solution. For the surfactant removal fraction, it slightly decreased with increasing in air flow rate. This can be explained by the effect of the air flow rate on the flow platen as described before. An increase in the air flow rate directly increased the foamate production rate, resulting in the increase in the water fraction of produced foam. Consequently, the foam produced became wetter or contained more water.



**Figure 4.8** Effect of air flow rate on separation efficiency (feed flow rate = 50 ml/min; NaCl concentration = 2 wt% and surfactant concentration = 0.1 wt%).

#### 4.3.4 Effect of Feed Flow Rate

An increase in feed flow rate slightly decreased the enrichment ratio of surfactant. This can be explained in that an increase in feed flow rate simply increased the quantity of surfactant available in the froth column. Hence, it directly improved the foamate production rate to confirm the increasing in surfactant enrichment ratio. For the enrichment ratio of oil, it slightly increased with increasing the feed flow rate in range of 30 to 70 ml/min because of increasing in foamate production rate. It slightly decreased with increasing the feed flow rate in range of 70 to 100 ml/min because of the increasing of the foam wetness. For the effect of removal fraction, the oil removal fraction slightly increased with increasing the feed flow rate in range of 30 to 50 ml/min due to an increase in foamate production rate. Then, it slightly decreased with increasing the feed flow rate in range of 50 to 100 ml/min because an increase in feed flow rate reduced the residence time of the liquid remaining in the column so the mass transfer between solution phase and foam was less than at the lower feed flow rate. For the surfactant removal, it slightly increased with increasing the feed flow rate in range of 30 to 50 ml/min because of the effect of feed flow rate on foamate production rate. Then, it remained unchanged. The effect of feed flow rate was found to be much lower than the other effects. The effect of feed flow rate on separation efficiency was shown in figure 4.8. These improvements in performance with increasing liquid feed flow rate were limited by minimum flow rate required to reach the flooding condition.



**Figure 4.9** Effect of feed flow rate on separation efficiency (air flow rate = 50 L/min; NaCl concentration = 2 wt% and surfactant concentration = 0.1 wt%).