

## CHAPTER III

### EXPERIMENTAL

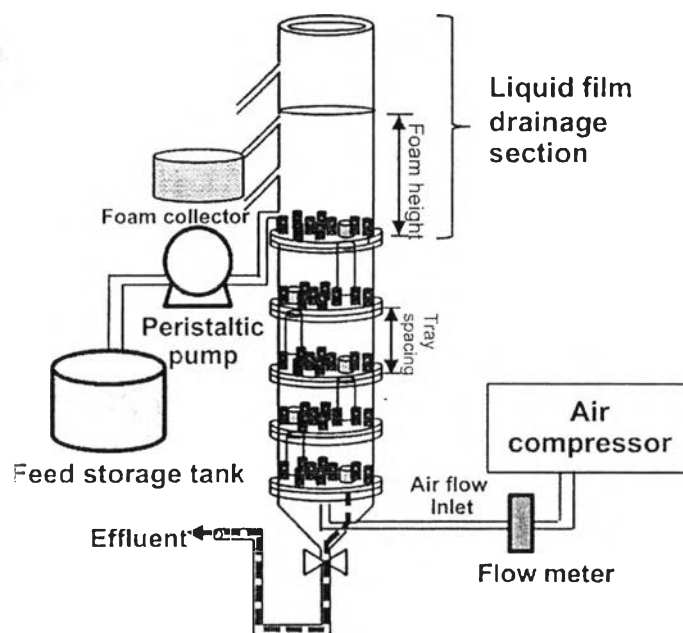
#### 3.1 Materials

Sodium dodecyl sulfate (SDS) , cadmium nitrate ( $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ ), copper nitrate ( $\text{Cu}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ ) and nickel nitrate ( $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ) with a purity of 97 % , > 99 % , > 99 % and > 99 % , respectively, were used as received without further purification. Deionized water was used in all experiments.

#### 3.2 Equipment

##### 3.2.1 The Foam Fractionation Unit

The multistage ion foam fractionation apparatus used in this study and the arrangement and the dimensions of the bubble-caps in each tray are shown in Figure 3.1. The multistage ion foam fractionation column was made from an acrylic cylinder with a 17.4 cm inner diameter and was assembled to have different trays of up to 5 stages. Each tray had 22 bubble caps with a weir height of 6 cm and a cap diameter of 2 cm. The tray spacing was 15 cm. Each bubble cap tray, except the lowest tray, had a downcomer with a diameter of 4 cm to allow liquid overflow to a lower tray. The liquid level in each tray was fixed at a weir height of 3 cm. On the top tray, there was a foam column having the same inner diameter (17.4 cm) and different foam outlet heights at 30, 60, and 90 cm.



**Figure 3.1** The multistage foam fractionation unit in this research.

### 3.3 Methodology

#### 3.3.1 Individual Heavy Metal

The ion foam fractionation system was operated in continuous mode in this study. A feed solution containing different Cd, Cu, Ni and SDS concentrations was prepared by dissolving  $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ ,  $\text{Cu}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ ,  $\text{Ni}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$  and SDS in deionized water. The feed solution was continuously fed into the top of the column at different feed flow rates in the range of 25–100 mL/min, corresponding to the feed flux flow rate range of 1–4 L/m<sup>2</sup> min, by using a peristaltic pump. Compressed air from an air compressor was introduced under the bottom tray and was regulated by rotameter to have flow rates in the range of 30–100 L/min. The experiments were carried out at room temperature (25–27 °C) because of the insignificant influence of temperature on foam fractionation performance. The foam was collected at the top of the column at different

foam heights (30, 60, and 90 cm). The collected foam was left to collapse by itself to obtain the foamate. Samples of the feed solution, the collapsed foam, and the effluent were collected after steady state conditions (around five hours). The steady state condition was justified when all measured parameters were invariant with time. The volumetric flow rates of both foamate and effluent were also measured. The data obtained from at least three runs were averaged and used to assess the process performance of the multistage ion foam fractionation system. By performing mass balances of heavy metal and SDS, the average error was found to be less than 10 %. Various evaluating parameters were used to assess the process performance of the studied multistage ion foam fractionation system, including the enrichment ratios of SDS and individual heavy metal, SDS recovery, individual heavy (Cd, Cu, and Ni) removal, and the separation factors of Cd, Cu, Ni, and SDS, as shown in Equations 4.1-4.3:

$$\text{Enrichment ratio} = C_f/C_i, \quad (4.1)$$

$$\% \text{ SDS recovery or } \% \text{ Cd removal} = (V_i C_i - V_e C_e)(100)/(V_i C_i), \quad (4.2)$$

$$\text{Separation factor} = C_f/C_e, \quad (4.3)$$

where  $C_f$  and  $C_i$  are the SDS or individual heavy metal concentration in the foamate (collapsed foam solution) and in the influent (feed), respectively.  $C_e$  is the SDS or individual heavy metal ion concentration in the effluent stream.  $V_i$ ,  $V_f$ , and  $V_e$  are the volumetric flow rate of feed, foamate, and effluent, respectively.

### 3.3.2 Mixed Heavy Metals

The ion foam fractionation system was operated in continuous mode in this study. A feed solution containing different mixed heavy metals and SDS concentrations was prepared by dissolving  $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ ,  $\text{Cu}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ ,  $\text{Ni}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$  and SDS in deionized water. The feed solution was continuously fed into the top of the column at different feed flow rates in the range of 25–100 mL/min,

corresponding to the feed flux flow rate range of 1–4 L/m<sup>2</sup> min, by using a peristaltic pump. Compressed air from an air compressor was introduced under the bottom tray and was regulated by rotameter to have flow rates in the range of 30–100 L/min. The experiments were carried out at room temperature (25–27 °C) because of the insignificant influence of temperature on foam fractionation performance. The foam was collected at the top of the column at different foam heights (30, 60, and 90 cm). The collected foam was left to collapse by itself to obtain the foamate. Samples of the feed solution, the collapsed foam, and the effluent were collected after steady state conditions (around five hours). The steady state condition was justified when all measured parameters were invariant with time. The volumetric flow rates of both foamate and effluent were also measured. The data obtained from at least three runs were averaged and used to assess the process performance of the multistage ion foam fractionation system. By performing mass balances of heavy metal and SDS, the average error was found to be less than 10 %. Various evaluating parameters were used to assess the process performance of the studied multistage ion foam fractionation system, including the enrichment ratios of SDS and individual heavy metal, SDS recovery, mixed heavy metals removal, and the separation factors of SDS and mixed heavy metals, as shown in Equations 4.1-4.3.

### 3.4 Measurement and Analytical Method

The concentration of SDS was measured by using a total organic carbon analyzer (Shimadzu, TOC-VCSH). The concentration of heavy metal ions was determined by an atomic adsorption spectrophotometer (AAS, Varian, SpectrAA 300). The procedure for determining heavy metal concentration was carried out with consideration of the effect of SDS interference. Heavy metals standard solution containing SDS at the same concentration as in the diluted sample was used for obtaining a calibration curve. The measurement range of cadmium with high accuracy is 0.02–4 mg/L, according to the AAS specification.