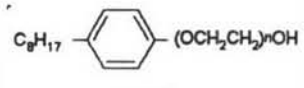
# CHAPTER III
# EXPERIMENTAL

## 3.1 Materials

In this study, two different surfactants was used: Cetylpyridinium chloride (CPC) and Polyethylene glycol tert-octylphenyl ether ($OPEO_{10}$). Cetylpyridinium chloride (CPC) as a cationic surfactant was supplied from Zealand Chemical (Steinheim, Germany) with above 99% purity. $OPEO_{10}$ from Fluka (Switzerland) of higher than 98% in purity represented a nonionic surfactant. Distilled water was used in all experiments. Chemical properties of the surfactants provided by the suppliers are listed in Table 3.1.

**Table 3.1** Chemical properties of the studied surfactants

| Surfactant | Chemical formula | Molecular weight | Purity |
|---|---|---|---|
| cetylpyridinium chloride (CPC) | $n\text{-}C_{16}H_{33}N(CH{=}CH)_2CHCl$ | 358.01 | > 99% pure |
| polyethylene glycol tert-octylphenyl ether ($OPEO_{10}$) |   | 625 | >98% pure |

## 3.2 Apparatus

The multi-stage foam fractionation apparatus used in this study is shown in Figure 3.1. The column did not have water jacket because previous work showed that temperature did not have much effect for the separation. The column was built by using acrylic with tray spacing equal to 15 cm. The inner and outer diameters of the column are 18 and 18.03 cm, respectively. The unit has multiple stages and could accompany up to 5 stages. Bubble-caps were made from stainless steel with 2 cm of

diameter and 6 cm of height and it has 22 bubble caps per tray. Figure 3.2 illustrates the model of bubble-cap tray. The dimensions of the multi-stages foam fractionator are given in Table 3.2.

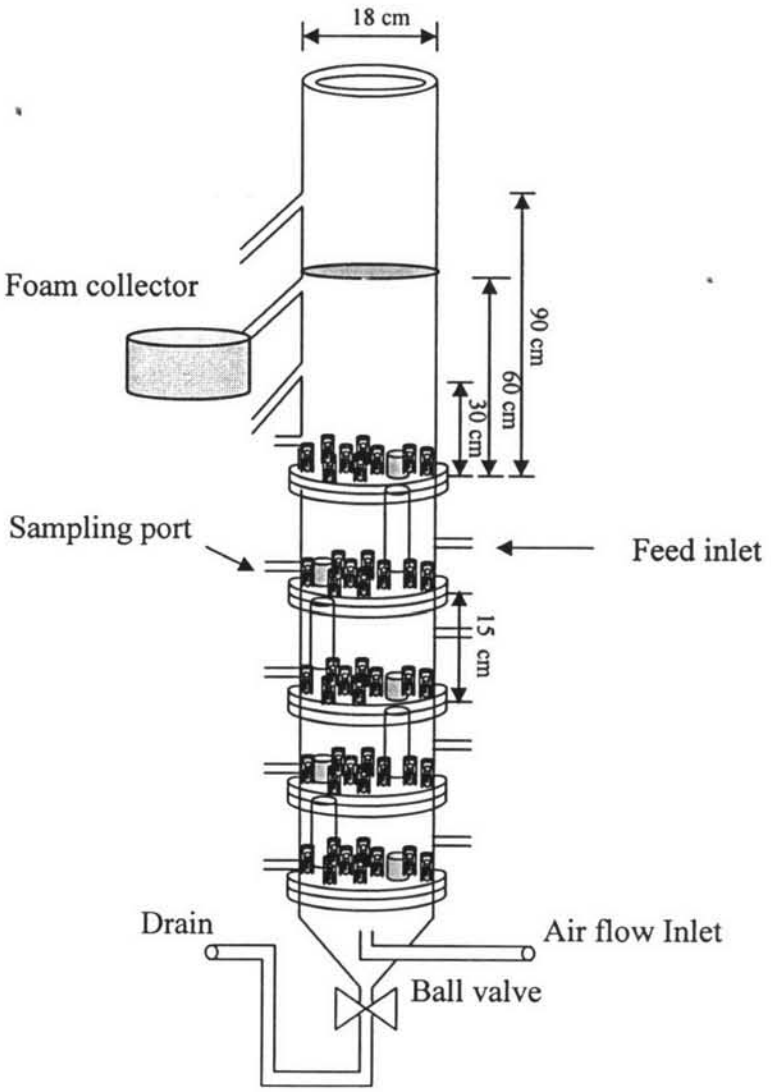



**Figure 3.1** Schematic of multistage foam fractionation column.

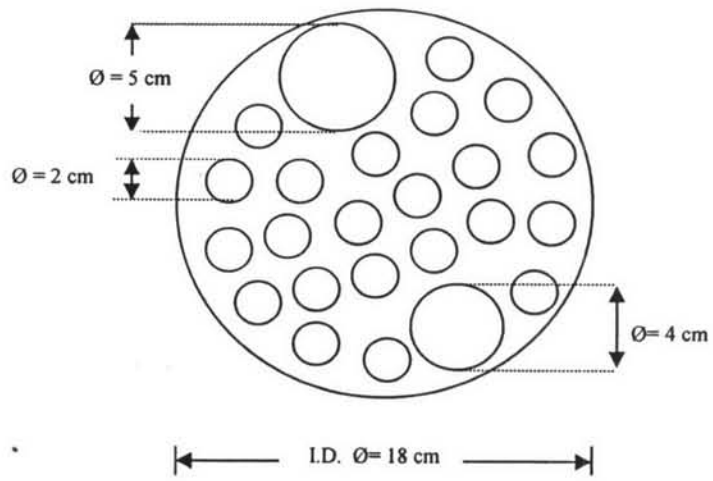



**Figure 3.2** Schematic of tray (Top view).

**Table 3.2** Dimensions of the multi-stage foam fractionation column

| | |
|---|---|
| Tray spacing | 15 cm |
| Column diameter<br>- Inner<br>- Outer | <br>18 cm<br>18.03 cm |
| Weir of bubble cap<br>- Diameter<br>- Height | <br>2 cm<br>6 cm |
| Number of bubble caps per tray | 22 |

### 3.3 Methodology

The foam fractionation was operated in continuous mode with aqueous solution containing surfactant. The surfactant solution was continuously fed a constant flow by using a peristaltic pump. The compressed air was introduced to the bottom tray of the column. The foam was collected at 60 cm from the liquid surface of the top tray. It was received by beaker for measured time. Then, it was frozen, thawed and then weighed to get the collapsed foamate volume.

The foam fractionation system was studied under steady state conditions. Steady state was insured when all measured parameters were invariant with time. The surfactant concentration in the feed solution was kept constant at 0.225 mM. The range of the operating parameters is summarized in Table 3.3. In each experiment, foam wetness (grams of foam solution/liter of foam), the surfactant concentration (mM) in the collapsed foam solution, and the surfactant concentration (mM) in the inlet and outlet stream were measured. The concentrations of CPC and $OPEO_{10}$ were measured by an UV-visible spectrophotometer (Shimadzu,2550) at wavelength of 260 nm and 275 nm, respectively.

The critical micelle concentration (CMC) of each surfactant and the mixtures were calculated as the concentration where the specific surface tension versus surfactant concentration showed an abrupt change in slope. The measurement of surface tension of surfactant solutions was carried out by using Contact Angle Instrument (DSA 10, Kruss).

In this research, glass column having an internal diameter of 5 cm and a height of 100 cm was used to investigate the foam ability and foam stability of all surfactant solutions. The steps of this method started with pouring a certain amount of 250 ml of solution into the column and it was consequently sparged by a constant flow rate of air 0.1 L/min. The foam height was measured as a function of time was to indicate the foam ability of the system. When the foam reached the highest level at which it was stable, the air flow was suppressed and then the time that it took for collapse to the half of maximum point was measured as foam stability.

**Table 3.3** Operating parameters

| | |
|---|---|
| Feed inlet<br>- Type of surfactant<br>- Total concentration<br>- Flow rate | <br>CPC, CPEO10 and Mixed<br>0.225 mM<br>50 ml/min |
| Air inlet<br>- Flow rate | <br>80 l/min |
| Foam height | 60 cm |
| Feed position at tray number | 1-5 |
| Bottom reflux at position tray number | 1-5 |
| Bottom reflux ratio | 0.25-1 |

### 3.4 Data Analysis

After the steady state was established, the effects of several parameters on the multi-stage foam fractionation performance were investigated in a continuous mode of operation. Efficiency of the surfactant recovery process was evaluated in terms of %surfactant recovery and enrichment ratio as given below:

$$\% \text{ Surfactant recovery} = \frac{(C_i F_i - C_e F_e)}{C_i F_i} * 100 \qquad (3.1)$$

$$\text{Enrichment ratio} = \frac{C_f}{C_i} \qquad (3.2)$$

where $C_i$ is the surfactant concentration in the influent stream (mM)

$C_e$ is the surfactant concentration in the effluent stream (mM)

$C_f$ is the surfactant concentration in the foam concentrated stream (mM)

$F_i$ is the feed flow rate (ml/min)

$F_e$ is the effluent flow rate (ml/min)