



CHAPTER III EXPERIMENTAL

3.1 Materials

In this study, a secondary AE surfactant having an average of 11 to 15 carbon atoms and 7.3 moles of ethylene oxide, Tergitol 15-S-7, was purchased from Union Carbide. A linear AE surfactant having similar number of carbon atoms with Tergitol 15-S-7 and 5 moles of ethylene oxide, Dehydol LS5, was kindly contributed by PTT Chemical Public Company Limited (Thailand). Reagent grade benzene (BEN) with 99.8% purity, toluene (TOL), ethylbenzene (ETB), trichloroethylene (TCE), and tetrachloroethylene (PCE) with 99.5% purity were purchased from J.T. Baker (Phillipsburg, NJ, USA). Reagent grade 1,1-dichloroethylene (1,1-DCE) with 99.8% purity was purchased from S.M. Chemical Co., Ltd. (Bangkok, Thailand). Deionized (DI) water was used for preparing all the solutions.

3.2 Equipment

Batch Experiment

For vapor-liquid equilibrium study, headspace autosampler (G1888, Agilent Technology, USA) with gas chromatograph (6890N, Agilent Technology, USA) was used to analyze the quantity of the VOCs. The obtained data are utilized for calculating the apparent Henry's law constant and solubilization constant. Total Organic Compounds (TOC 5000A, Shimadzu) analyzer is used for measuring the surfactant concentration.

Continuous Operation

A picture of the co-current vacuum stripping using a packed column is shown in Figure 3.1. The cylindrical stripping glass column had a 6.5-cm inside diameter and a 34-cm height, surrounded by a 16-cm diameter acrylic water jacket. The stripping column was packed with 5 mm×5 mm glass Raschig rings to a height of 30 cm, and was connected with a 3-hole distributor at the top of the column. A 1-L feed tank was made of glass and was placed inside a 16-cm diameter acrylic

water jacket. A product tank was made of stainless steel. The system was maintained under vacuum by a rotary vane pump. The feed solution was sent to the stripping column using a peristaltic pump. The operating temperature was controlled via a water circulating bath.

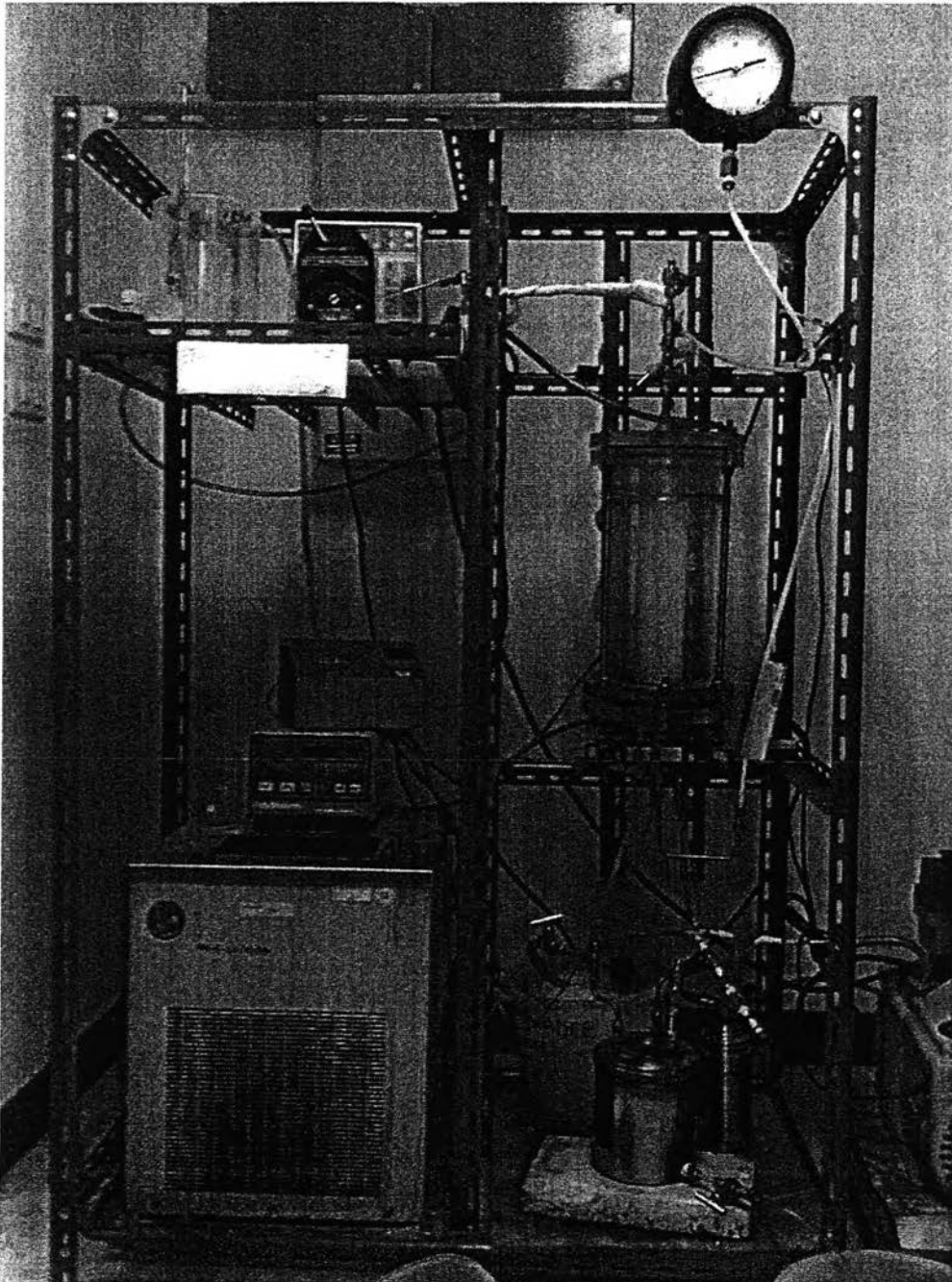


Figure 3.1 The co-current vacuum stripping using a packed column (Kungsanant *et al.*, 2008).

3.3 Methodology

3.3.1 Properties of Studied Surfactant

3.3.1.1 Cloud Point Temperature

The nonionic surfactant solution was prepared and agitated until homogeneously mixed. Then it was poured into a test tube. After that, it was placed into water bath and agitated slowly with the thermometer until the test solution becomes definitely cloudy and then, slowly cool down again. The CPT of nonionic solution was determined by visual observation of the temperature at which the clear solution suddenly turbid upon heating or cooling. Each of the results came from the averages of at least triplicate measurements.

3.3.1.2 Concentration of Surfactant in Coacervate Phase

The nonionic surfactant solution was placed in an isothermal bath controlled at 40°C. After phase separation, the sample in coacervate phase portion was collected and then, analyzed for finding the surfactant concentration by TOC analyzer.

3.3.2 Vapor-liquid Equilibrium

3.3.2.1 The Partition Behavior of the VOCs in Aqueous and Coacervate Phase Solution

The partitioning behavior of VOCs in water and coacervate phase solution was observed via modified equilibrium partitioning in closed systems (EPICS) method (Jiang *et al.*, 1997, Kungsanant *et al.*, 2008).

The partitioning of VOCs in aqueous phase solution is measured by adding 4 mL of the water containing the VOCs in several identical 22-mL glass vials with Teflon-coated septa and aluminum holed caps in order to prevent the leakage of VOCs. After that, the vials are placed in an isothermal chamber of the headspace autosampler controlled at 40°C. Then, the vapor sample in the headspace was automatically collected and then analyzed by a gas chromatograph every 10 minute. The equilibrium time of the VOCs is found via the correlation between the concentration of VOCs and time. For the surfactant-containing system,

the partitioning of VOCs in coacervate phase solution is observed by the same method.

3.3.2.2 Determination the Solubilization Constant, K_s

The value of K_s can be obtained by using correlation of VOCs partial pressure and apparent Henry's law constant in coacervate phase solution described as followings:

$$K_s = \frac{C_{sol}}{C_u C_m} \quad (2.2)$$

Where:

$$C_{sol} = C_{Total\ solute} - C_u$$

$$C_m = C_{Total\ surfactant} - CMC$$

$C_{Total\ solute}$ is total organic solute concentration, and $C_{Total\ surfactant}$ is total surfactant concentration.

3.3.2.3 Determination of the Apparent Henry's Law Constant (H_{app}) of VOCs in Coacervate Phase Solution

Several identical 22-mL glass vials containing 4 mL of an aqueous solution containing the VOC were prepared. The VOC was protected from leakage by capping the vials with Teflon-coated septa and aluminum holed caps. The VOC concentration was varied but was kept below its water solubility limit. After reaching equilibrium, the VOC concentration in the headspace and liquid phases was analyzed by the gas chromatograph connected with the headspace autosampler. Finally, the equilibrium correlation between VOC concentration in vapor and in liquid phases was obtained. For the surfactant-containing system, due to higher solubility in the surfactant solutions, the VOC concentrations were varied from 100 to 2,000 ppm. The experiments were conducted in a similar manner as those for the VOC-water system.

3.3.3 Continuous Operation

According to Figure 3.1, the pressure of the stripping unit was maintained under vacuum using the rotary vane pump. The operating temperature was controlled at 40°C using the water circulating bath. After the column pressure was stable, 1.0 mL/min of the feed solution was pumped to the top of the column by the peristaltic pump. The viscous surfactant-containing liquid flew down along the

packed column and was stored in the product tank. A vapor suction line was attached at the bottom of the stripping column to operate the column in co-current mode. At the end of line, a cold trap was installed to prevent the rotary vane pump from liquid-induced damage. After reaching the steady state, the liquid sample was collected by a gas-tight syringe in the feed and product streams for VOC concentration analysis.

In this work, the feed solution contained 2000 ppm of VOC and 450 mM of surfactant concentration. The feed flow rate was fixed at 1.0 mL/min, the absolute column pressure is varied from 38 to 94 torr, and the number of distributor holes was three. The efficiency of stripping process was observed and reported in term of the overall liquid phase volumetric mass transfer coefficient ($K_x a$) and the percentage of VOCs removal.