



## CHAPTER III EXPERIMENTAL

### 3.1 Materials

In this study, a Branched Secondary Alcohol Ethoxylates 2-(2,6,8-trimethylnonan-4-yloxy) ethanol, (Tergitol TMN-6) was use as the nonionic surfactant. Reagents grade benzene with 99% purity, toluene with 98% purity, ethyl benzene with 99% purity, dichloroethylene with 98% purity, trichloroethylene with 98% purity, and perchloroethylene with 98% purity. All chemical were purchased from Sigma-Aldrich (Singapore). Deionized water was use for preparing all solutions.

### 3.2 Apparatus

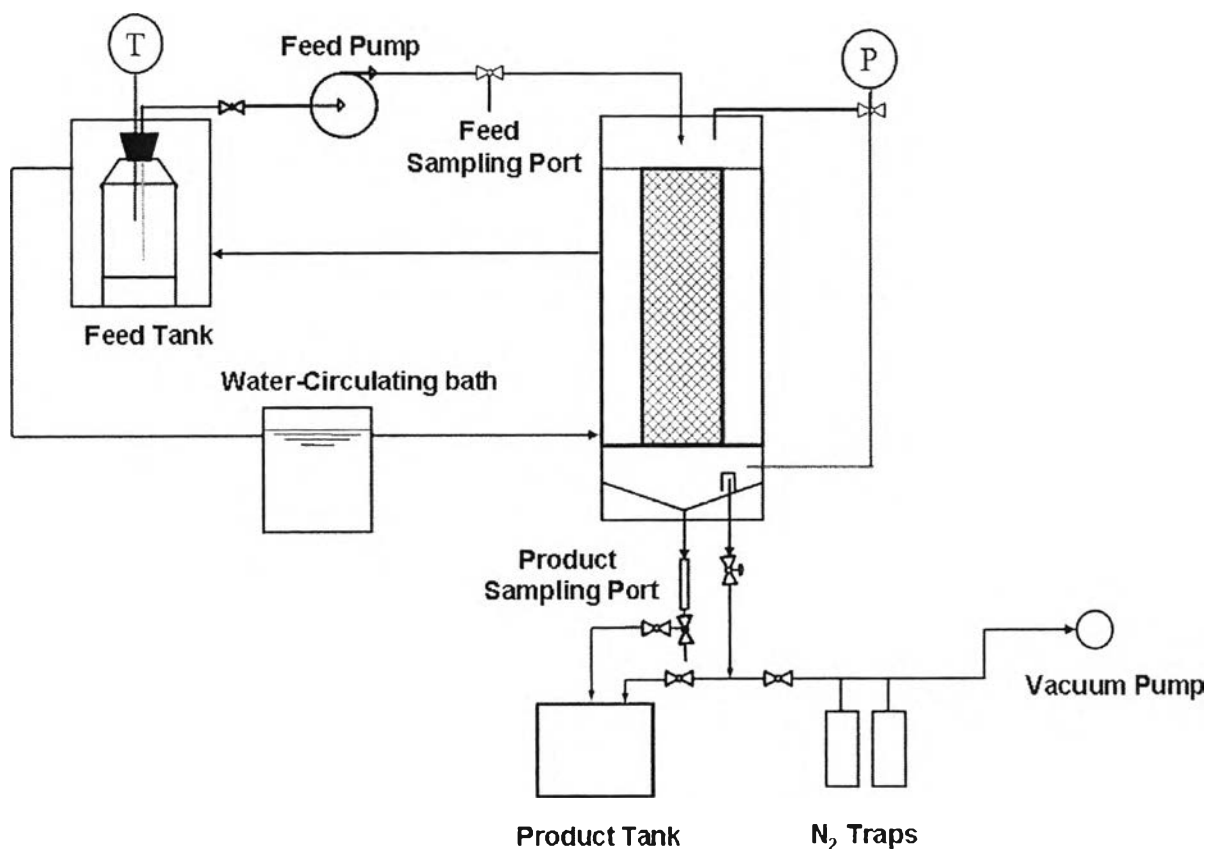
#### 3.2.1 Batch Operation

For vapor-liquid equilibrium study, headspace auto-sampler (G1888, Agilent Technology, USA) with gas chromatograph (6890N, Agilent Technology, USA) was used to analyze the quantity of the HVOCs in aqueous and surfactant solution. 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.

#### 3.2.2 Continuous Operation

A schematic of the vacuum stripping unit is shown in Figure 3.1. The inside diameter of cylindrical stripping column was 6.4-cm and 30.5-cm in height and was made from acrylic fiber; there was also a 12.7-cm diameter acrylic water jacket. The stripping column was packed with 25.5-cm in height of 5 mm × 5 mm glass Raschig rings. A 1 L glass feed tank was placed inside a 16-cm diameter acrylic water jacket. A product tank was made of stainless steel. The system was maintained under vacuum conditions by a rotary vane pump. The feed solution was

sent to the stripping column using a peristaltic pump. The operating temperature was controlled using a water circulating bath.



**Figure 3.1** Schematic of the vacuum stripping unit. (Kungsanant *et al.*, 2008).

### 3.3 Methodology

#### 3.3.1 Analytical Techniques

The concentration of surfactant was measured by using a Total Organic Compounds (TOC 5000A, Shimadzu). The HVOC concentration was measured by a gas chromatograph with a flame ionization detector (Agilent technology, USA). Due to high volatility of the HVOC, a static headspace auto-sampler was used as sampler injection technique with no intervention of heavy nonionic surfactant molecule. A gas-tight syringe was used to collect product sampler solution from the vials in batch experiment and from the vacuum stripping

unit in continuous operation. Then, 100  $\mu\text{l}$ , of liquid sampler was transferred into the 20-ml glass vials with Teflon-coated septa and aluminum holed caps. The conditions of gas chromatograph for HVOC concentration determination were as following; stabile wax column, Agilent; carrier, helium with the flow rate of 15 mL/min; make up gas, ultra-pure nitrogen with the flow rate of 30 mL/min; oven temperature, 60°C isothermal; injector temperature, 125°C; and detector temperature, 300°C. The standard quantitative calibrations were made to obtain HVOC concentration in liquid phase.

### 3.3.2 Vapor-Liquid Equilibrium

#### 3.3.2.1 *The Partition Behavior of the HVOCs in Aqueous and Coacervate Phase Solution.*

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

The partitioning of HVOCs in aqueous phase solution is measure by adding 4 mL of the water containing the HVOCs in several identical 20-mL glass vials with Teflon-coated septa and aluminum holed caps in order to prevent the leakage of HVOCs. 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 minutes. The equilibrium time of the HVOCs is found via the correlation between the concentration of HVOCs and time. For the surfactant-containing system, the partitioning of HVOCs 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 HVOCs 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.

At low solute concentration, the value of  $K_s$  can be estimated as its infinite dilution value and is independent of solute concentration (Choori *et al.*, 1998).

### 3.3.2.3 Determination of the Apparent Henry's law Constant ( $H_{app}$ ) of HVOCs in Coacervate Phase Solution.

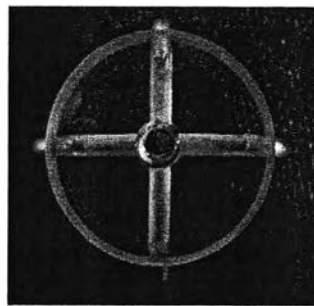
Several identical 20-mL glass vials containing 4 mL of an aqueous solution containing the HVOC were prepared. The HVOC was protected from leakage by capping the vials with Teflon-coated septa and aluminum holed caps. The HVOC concentration was varied but was kept below its water solubility limit. After reaching equilibrium, the HVOC concentration in the headspace and liquid phases was analyzed by the gas chromatograph connected with the headspace auto-sampler. Finally, the equilibrium correlation between HVOC concentration in vapor and in liquid phases was obtained. For the surfactant-containing system, due to higher solubility in the surfactant solutions, the HVOC concentrations were varied from 100 to 2000 ppm. The experiments were conducted in a similar manner as those for the HVOC-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 pass through the liquid distributor as shown in Figure 3.2. 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 HVOC concentration analysis.

In this work, the feed solution contained 2000 ppm of HVOC and 450 mM of surfactant concentration. The feed flow rate was fixed at 1.0 mL/min, the absolute column pressure is varied from 53 torr. The efficiency of stripping process was observed and reported in term of the overall liquid phase volumetric mass transfer coefficient ( $K_L a$ ) and the percentage of HVOCs removal



**Figure 3.2** Liquid Distributor (Kungsanant *et al.*, 2008).