



CHAPTER III EXPERIMENTAL

3.1 Materials

A branched *t*-octylphenolpolyethoxylate, OP(EO)₇ having an average of 7 moles of ethylene oxide per mole of octylphenol (Triton X-114) purchased from Dow Chemical Inc. (South Charleston, WV, USA) was used as the nonionic surfactant. Reagent grade benzene with 99.8% purity, toluene, ethylbenzene, trichloroethylene, and tetrachloroethylene with 99.5% purity were purchased from J.T. Baker (Phillipsburg, NJ, USA). Reagent grade 1,2 dichloroethane with 99.8% purity was purchased from Labscan Co, Ltd. (Bangkok, Thailand). All chemicals were used as received. Deionized (DI) water was used for the solution preparation. The properties of the surfactant and the VOCs are detailed in appendix A.

3.2 Apparatus: Vacuum Stripping Packed Column

A schematic of the vacuum stripping unit is shown in Figure. 3.1. The cylindrical stripping column was 6.4-cm inside diameter 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 5×5 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.

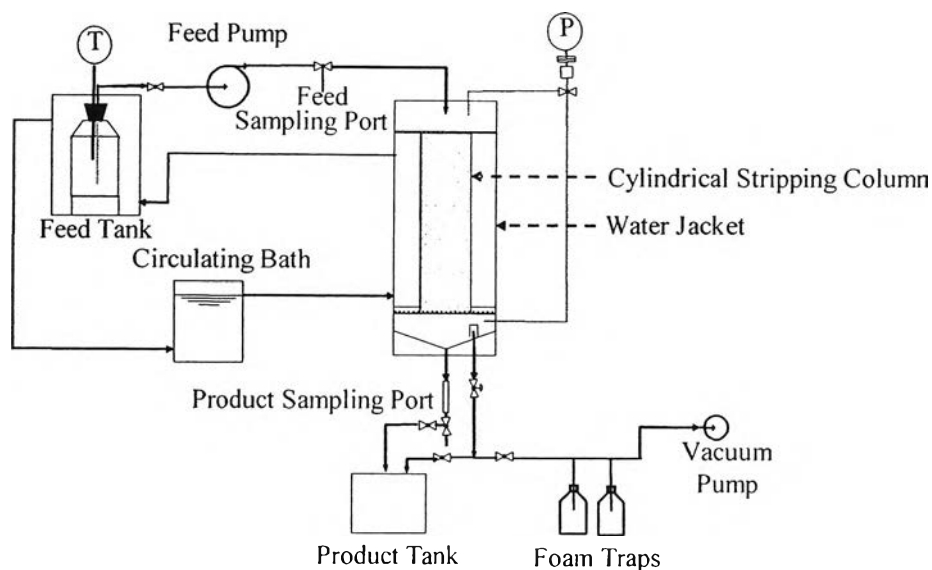


Figure 3.1 Schematic of the vacuum stripping unit.

3.3 Methodology

3.3.1 Viscosity Determination

The viscosity of the surfactant solution containing the organic solute was measured by a Brookfield digital rheometer (DV-III, Brookfield Engineering Laboratories Inc.). Spindle (RVDV-III) numbers 21 and 27 were used to determine the viscosity of the surfactant solution. The spindle's rotating speed was set at 250 rpm. The viscosity of each liquid sample was measured at least three times. Since VOC loss can occur during viscosity measurement, the VOC concentration was measured directly by taking 100 μL of the liquid sample from the viscosity meter and then analyzing by a gas chromatograph connected with a headspace autosampler (HSGC).

3.3.2 Analytical Techniques

The concentration of OP(OE)_7 was measured by using a CE 2000 series UV-spectrophotometer (Cecil Instrument Limited, Cambridge, England) at a

wavelength of 224 nm. The VOC concentration was measured by a gas chromatograph with a flame ionization detector (Agilent technology, USA). Due to high volatility of the VOC, a static headspace autosampler was used as sample injection technique with no intervention of heavy nonionic surfactant molecule. A gas-tight syringe was used to collect product sample solution from the vials in batch experiment and from the vacuum stripping unit in continuous operation. Then, 100 μ L of liquid sample was transferred into the 22-mL glass vials with Teflon-coated septa and aluminum holed caps. The conditions of gas chromatograph for VOC concentration determination were as following; capillary column, HP-5 (5% Phenyl Methyl Siloxane), Agilent 19091J-413; 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, 175°C isothermal; injector temperature, 200°C; and detector temperature, 300°C. The standard quantitative calibrations were made to obtain VOC concentration in liquid phase.

3.3.3 Equilibrium Determination

The partitioning behavior of VOC in water and the surfactant system was observed by modification of the Equilibrium Partitioning in Closed Systems (EPICS) method (Jiang *et al.*, 1997 and Valsaraj *et al.*, 1988). To determine the equilibrium time, several identical 22-mL glass vials with Teflon-coated septa and aluminum holed caps containing 4 mL of VOC in DI water were placed in an isothermal water bath controlled at a set temperature. One mL liquid samples were collected every 30 min by a gas-tight syringe. The liquid sample was transferred into the vial and immediately sealed with a Teflon-coated septum and an aluminum holed cap. VOC concentration in liquid phase was measured by HSGC. Then, the time required to reach equilibrium was determined.

Subsequently, to find the Henry's law constant for the surfactant-free system, two sets of 12 identical 22-mL glass vials with Teflon-coated septa and aluminum holed caps containing 4 mL of VOC in aqueous solution were prepared. The VOC concentrations ranged from 50 to 300 ppm. The first set was placed in the isothermal water bath, and the second set was incubated in the oven of the headspace

autosampler. After reaching equilibrium, the VOC concentration in the liquid phase of the first set was measured. The VOC concentration in the vapor phase of the second set was analyzed automatically by HSGC. Three samples were repeated for each VOC concentration. The equilibrium correlation between VOC concentration in vapor and in water phase was then obtained. For the surfactant containing system, the VOC concentrations were varied from 100 to 2 000 ppm in OP(EO)₇ solution. The experiments were conducted using the same procedure as those for the surfactant-free system, except that 100 μ L of the liquid sample was collected after equilibrium, and four samples were repeated for each VOC concentration.

3.3.4 Continuous Operation

The vacuum in the stripping column, product tank, and foam traps was created by the rotary vane pump. Water at constant temperature was circulated through the water jacket of the feed tank and stripping column to control the system temperature. The temperature of the feed solution and the packed column was maintained at desired temperature. After the column pressure was stable, the feed solution was pumped at the desired flow rate into the top of the column by the peristaltic pump. The liquid flowed down the column and was kept in the product tank. The stripping packed column was operated in co-current mode with a vapor suction line attached at the bottom of the column. The foam trap was installed at the vapor exit before entering the pump in order to prevent the rotary vane pump from liquid-induced damage. The vacuum stripper was operated continuously, and liquid samples were collected by a gas-tight syringe every 20 min. The liquid sample in the feed stream was collected at the feed sampling port, and that in the product stream was collected at the product sampling port. The vacuum stripping unit reached steady state within 90 min as indicated by constant VOC concentrations. The solution from the inlet and the outlet streams after steady state were taken and analyzed for VOC concentration.

The operating conditions in the vacuum stripping packed column were varied as follows:

- operating temperature: 30–50°C
- OP(EO)₇ concentration: 300–630 mM

- VOC concentration: 300–2 000 ppm
- absolute column pressure: 38–125 Torr
- feed flow rate: 1.1–9.8 mL/min
- number of distributor holes: 1–5 holes
- VOCs: Aromatic VOCs
 - benzene (BEN)
 - toluene (TOL)
 - ethylbenzene (ETB)
- VOCs: Chlorinated VOCs
 - 1,2 dichloroethane (DCE)
 - trichloroethylene (TCE)
 - tetrachloroethylene (PCE)

Vacuum stripping performance could be evaluated by calculation of the percentage of VOC removal and the overall liquid phase volumetric mass transfer coefficient of the VOCs. The sample of calculation and experimental results are described in appendices B and C, respectively.