



CHAPTER III EXPERIMENTAL

3.1 Materials

A branched t-octylphenolpolyethoxylate, OP(EO)₇ with an average of 7 moles of ethylene oxide per mole of octylphenol (trade name Triton X-114) from Dow Chemical Inc. (South Charleston, USA) was used as a nonionic surfactant in this study. Reagent grade toluene from BDH Laboratory Supplies with purity of 99.5% toluene was selected as a representative of pollutant in coacervate phase solution. Glass raschig ring with the length of 5 mm, width of 5 mm, wall thickness 3 mm was used as packing material in stripping column.

3.2 Experimental Apparatus

3.2.1 Design and Experimental Setup of Co-current Vacuum Stripper

A pilot flash vacuum stripper was fabricated for the study of vacuum stripping of toluene as shown in Figure 3.1 and Figure 3.2. The design parameters of the vacuum stripping equipment are given in Table 3.2. The vacuum stripper consists of a water jacketed packed column, a feed tank (stainless steel), a product tank (stainless steel), a vacuum pump, a master flex pump and a water circulation bath.

The fed solution was pumped to the top of the column by masterflex pump. The feed's temperature was controlled at 30°C. The inner glass column was packed with glass raschig rings. The column and the product tank were maintained under vacuum by a rotary vacuum pump. The liquid flows down the column due to gravity and is collected in the product tank. The vacuum suction line was at the bottom of the column to operate the column in a co-current mode. The vacuum suction line end within the column was covered with a cap to prevent liquid from directly entering the line. The vapor and foam generated in the column flow into the foam trap. The foam was disappeared as it passed through the suction line. The vapor was drawn out of the foam trap by a vacuum pump, and the liquid resulting

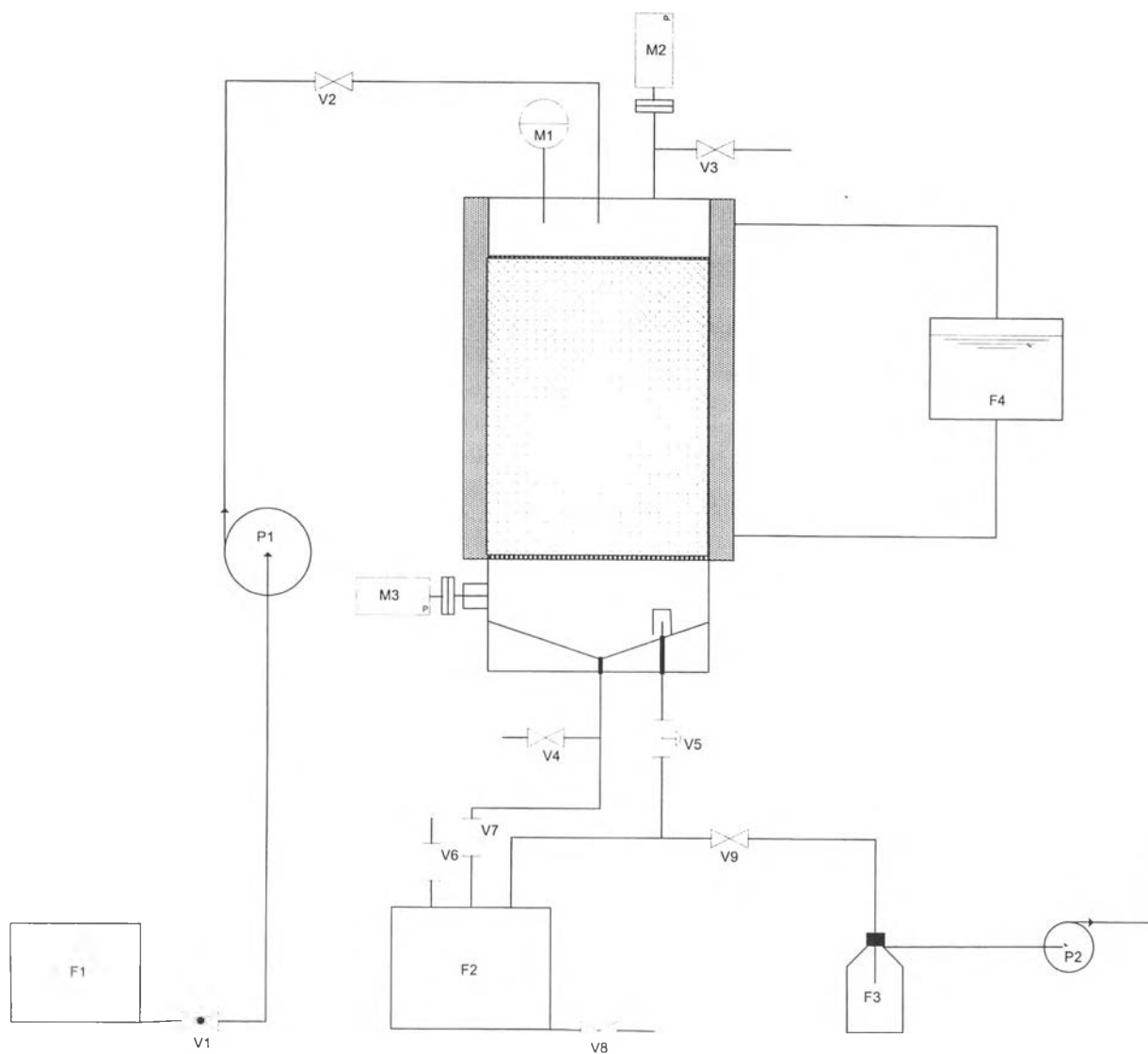


Figure 3.1 Profile of continuous co-current vacuum stripper.

Table 3.1 Label of number equipment

F1	Feed tank (stainless steel)
F2	Product tank (stainless steel)
F3	Foam and volatile solute trap
F4	Circulating bath
M1	Temperature gauge
M2-M3	Pressure gauge
P1	Feed pump (peristaltic pump)
P2	Vacuum pump (rotary vane pump)
V1	Ball valve for ¼ in stainless steel tube
V2-V9	Needle valve for ¼ in stainless steel tube

Table 3.2 Design parameters of the flash vacuum stripping column

Packed column inside diameter	3 in
Packing Height	9 in
Type	Glass raschig rings
Size	5×5 mm
Void fraction	0.584
Liquid line diameter	¼ in
Vapor line diameter	¼ in
Sample line diameter	¼ in
Operating temperature	30°C

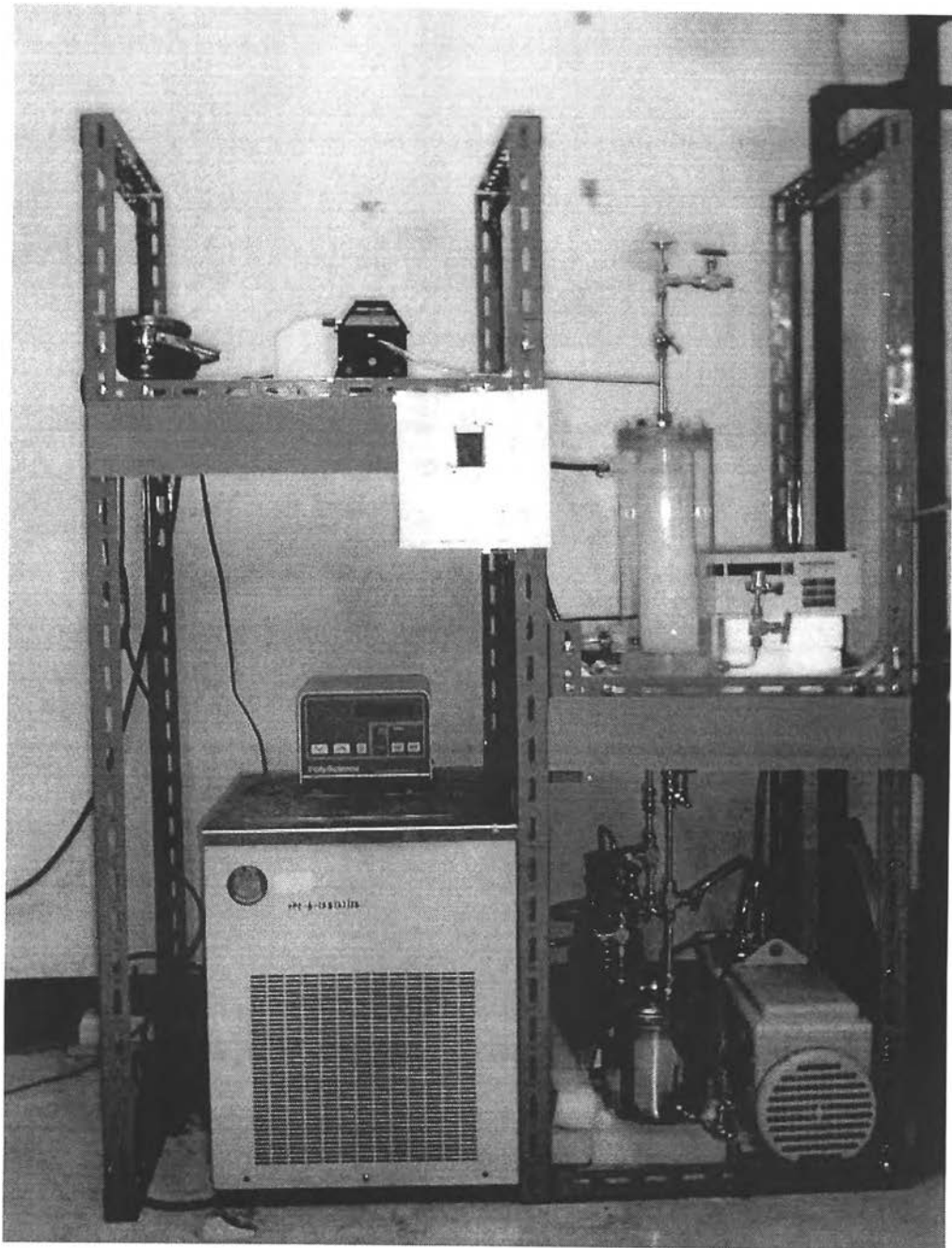


Figure 3.2 A continuous co-current vacuum stripper.

from the foam was collected in the foam trap. An acrylic pipe was used as a water jacket to enclose the acrylic packed column. Water was preheated to the desired temperature and continuously circulated in the annulus to keep the glass column isothermal.

The concentration of OP(EO)₇ was measured by using a CE 2000 series UV-spectrophotometer (Cecil Instrument Limited, Cambridge, England) at wavelengths 224 nm. The toluene concentration was measured by a gas chromatograph with a flame ionization detector (Perkin Elmer, Inc., Shelton, USA). Because of the high volatility of toluene, static headspace sampling was used as the sample injection technique with no interference of the high molecular weight nonionic surfactant. The conditions used for determination of toluene concentration were as follows: column, Supelcowax 10; gas carrier, ultra-pure nitrogen with the flow rate of 20 ml/min; oven temperature, 100°C isothermal; injector temperature, 150°C; and detector temperature, 250°C. The external standard quantitative calibrations were obtained for the analysis of surfactant and toluene.

3.2.2 Design and Experimental Setup for Equilibrium Data Determination

The 22-ml glass vials with Teflon-coated septa were used to determine the equilibrium data. The temperature was controlled by a water bath.

3.3 Methodology

3.3.1 Experimental Procedures

3.3.1.1 *Determination of equilibrium time for toluene in aqueous and coacervate phase solution*

In order to measure the equilibrium time of toluene in aqueous and coacervate phase solution, several identical 22-ml glass with Teflon-coated septa containing solution of toluene were placed in an isothermal water bath. Then, samples were collected every 30 minutes to determine the toluene concentration in vapor phase by gas chromatography with headspace autosampler. Then, the equilibrium time of toluene in aqueous and coacervate phase solution was observed.

3.3.1.2 *Determination the correlation of toluene partial pressure and linear responses of the GC detector at equilibrium condition*

Two of 22-ml glass vials with Teflon-coated septa containing toluene in water solution were placed in an water bath to control the temperature. After reaching equilibrium, one sample was used to determine the gas chromatographic responses in vapor phase, and another one was used to detect the toluene concentration in liquid phase by gas chromatography with headspace autosampler. From this experiment, the correlation between toluene partial pressure and gas chromatographic responses of vapor phase can be determined by using the Henry's law constant for toluene in water solution to transform the toluene concentration in liquid phase to the partial pressure of toluene in vapor phase.

3.3.1.3 *Determination the apparent Henry's law constant for toluene in coacervate phase solution*

Two of 22-ml glass vials with Teflon-coated septa containing the coacervate phase solution at various toluene concentrations were placed in an isothermal water bath until equilibrium was reached. Then, one sample was used to detect the toluene concentration in liquid phase, while another one was used to determine the gas chromatographic response in vapor phase by gas chromatography with headspace autosampler.

3.3.1.4 *Procedure for co-current vacuum stripping operation*

a. Coacervate phase solution preparation

After coacervate phase solution was prepared. The solution was added into feed tank, which is connected to valve V1.

b. Starting the circulating water system

Water was allowed to circulate in jacket around the packed column until the temperature in column was 30°C by using the circulating water bath.

c. Starting the vacuum stripper

The feed tank was heated until the temperature of feed solution was 30°C. Then closing V2, V3, V4, V6, and V8 and opening V5, V7, and

V9, the vacuum pump was started. Wait for a period of time until the system is vacuumed to the desired pressure.

Once the desired vacuum is reached, the feed pump was started and valve V1 was fully opened. Then valve V2 was opened slowly to such an extent that there is no foaming in the feed line. The flow rate was adjusted by using controller at the feed pump. Simultaneously, the stop clock is started. The system would be run until steady state was attained. Then, the product samples were taken from V4 by using a gas tight syringe.

Packed column studies were performed to obtain the overall volumetric mass transfer coefficient ($K_x a$), number of transfer unit (NTU), and height of transfer unit (HTU). The liquid feed surfactant (OP(EO)₇) concentration was prepared at 300 mM to prevent the cloud point effect. The temperature was kept constant at 30°C, and pressure variation was between 40 and 130 torr. The liquid flow rate was varied from 8 to 30 mL/min.

3.3.2 Data Calculation and Analysis

a. Equilibrium time for toluene-water and coacervate phase solution system was observed by the consistency of the toluene concentration in vapor phase of each initial toluene concentration in liquid phase. For convenience, the longest equilibrium time was selected to use as a standard equilibrium time for each system experiment.

b. By applying the true Henry's law constant for toluene at 30°C, the relation between gas chromatographic responses of vapor phase and partial pressure of toluene can be resolved.

c. From the correlation between gas chromatographic responses of vapor phase and partial pressure of toluene together with the total toluene concentration in liquid phase which measured from coacervate phase solution system, the apparent Henry's law constant for toluene in coacervate phase solution can be calculated.

d. By using equation 2.1 or 2.2, the solubilization constant can be found by using correlation of toluene partial pressure and apparent Henry's law constant for toluene in coacervate phase solution.

e. The vapor-liquid equilibrium partition coefficient (K) can be calculated from equation 2.15 for toluene and equation 2.21 for water.

f. The overall volumetric mass transfer coefficient ($K_x a$), number of transfer unit (NTU), and height of transfer unit (HTU) can be calculated by using the rate equation (equation 2.28) for co-current stripping, the partition coefficients, and flash calculation.