CHAPTER III

EXPERIMENT

3.1 Materials

3.1.1 Phenol was used as organic contaminant model and was used as received from Univar as analytical grade. Its solubility is about 8.2×10^{15} per 100 parts of water and in needles form.

3.1.2 Sodium dodecyl sulfate (SDS) or sodium lauryl sulfate was used as anionic surfactant as received from Kao Industrial (Thailand) Co., Ltd. as commercial grade. The trade name is Emal 10 powder.

3.1.3 Activated carbon was Filtrasorb-300, a granular liquid-phase activated carbon, manufactured by Calgon Corporation and received from White Group Co. Its surface area is about 950-1,050 m^2/gram and pore volume is 0.85 mL/gram

3.1.4 Methanol was used as mobile phase of HPLC and manufactured from Unichrome. Its purity is 99.7%.

3.2 Equipment

3.2.1 The column that used for adsorption studying is solvent-resistance with 2.5 cm. inside diameter and 120 cm length. It was manufactured from Rainin. This column was jacketed and circulated with water to maintain the temperature at 25°C. An adjustable plunger with filter at

both ends was used to restrain the carbon packed bed at the fixed level and to minimize the void volume existed in the bed.

3.2.2 Pump used in this work is Masterflex Microprocessor pump drive model 600 rpm/7524-05 (230 VAC) was manufactured by Cole-Palmer Instrument Co.. Its speed control range is 10-600 rpm.

3.2.3 Pump head model is 7518-12 that was manufactured by Cole-Palmer Instrument Co.. The tubing used is model 7518-24, and made from Tygon also made from the same company. This pump head permits the maximum flowrate of 1680 mL/min. at 600 rpm.

3.2.3 Sampling collector is used to collect sample in every hour. It was manufactured from ISCO, Inc. The model is Foxy Jr. Fraction Collector used with 12-13 mm outside diameter tubes. In collecting sample, its maximum drop speed is 5.5 drops/second.

3.2.4 Temperature controller that was used to maintain adsorption column at 25 °C was manufactured from Poly Science with model 9601 Digital Temperature Controller.

The schematic diagram of experimental apparatus that used in this study is illustrated in figure 3.1.

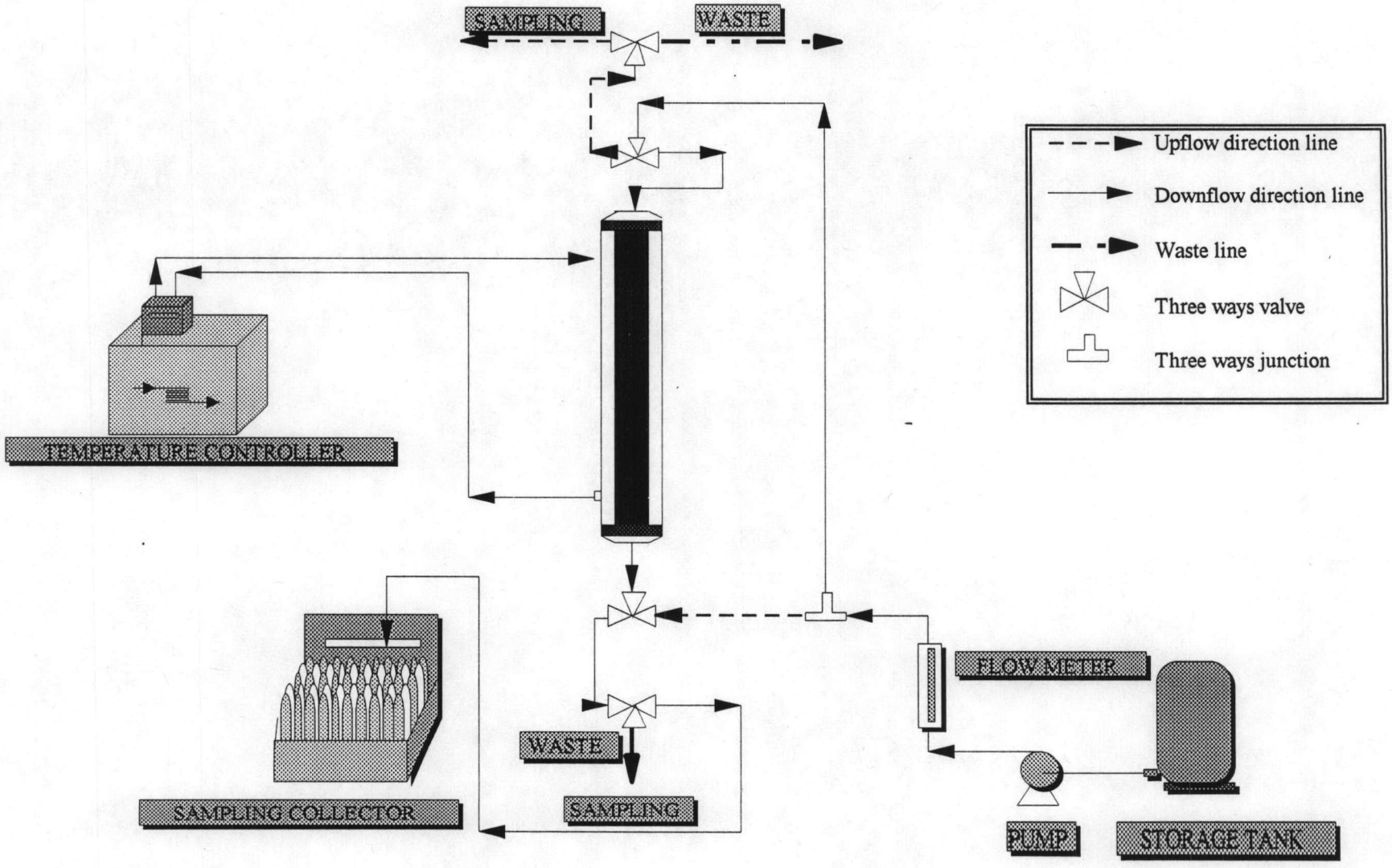

Figure 3.1 Schematic diagram of experimental equipments

3.2.5 Analytical equipment used to measure phenol concentration in the effluent include the High-Performance Liquid Chromatography (HPLC) system, which all of them were manufactured by Perkin-Elmer Corporation, and the adsorbance detector and its accessories.

- Pump is Series 200 LC Pump. Its flowrate range is 0.01-10.0 mL/min. with the maximum pressure of 420 bar (6200 Psi.).

- Degasser model is 200 Vacuum Degasser. It can operate with maximum flowrate of 10 mL/min. and there are 4 independent channels. It used gas permeation through a fluoropolymer membrane as the degassing process.

- Perkin Elmer 900 Series Interface and 600 Series Link are used to analog to digital interfaces that acquired data and store it in a local buffer memory and transmit the readings to the host computer.

- Absorbance detector or Ultra-violet or UV detector was used with wavelength of 269 nm. It was manufactured by Applied Biosystems Division of Perkin-Elmer Corporation. The model is 759A that provided the wavelength among 190-700 nm. The light source is deuterium.

- HPLC Column that was used to detect phenol was Supelcosil LC-18. This column had specification 15.0 cm. length, 4.6 mm. inside diameter, and packed with 5 μm particles.

- Filtered paper used for filtered sampling and mobile phase solvent was 0.45 micron with diameter of 13 mm. for sample and 47 mm. for mobile phase solvent. Both of two sizes are manufactured by Gelman Science.

Nylaflo was used for sample and methanol whereas cellulose nitrate was used for distilled water.

3.2.6 The concentration of SDS was analyzed by conductivity detector in the flushing step.

3.3 Methodology

3.3.1 Carbon preparation

Fresh activated carbon received from company may contain some salts so desalting process is important to do before using it. The first, carbon was boiled in distilled water at a temperature of about 100 °C for 5 hours. It was decanted when it was cooled down and washed with distilled water several times. The conductance of the decanted water was checked by conductivity meter until it nearly reached the conductance of distilled water, at this point, there is a small amount of salt left on the carbon. The carbon was dried at a temperature of 100°C for 2 days and kept away from air to avoid carbon degradation by oxidizing with oxygen.

3.3.2 Surfactant-Enhanced Carbon Regeneration process

Treated carbon weighing 58 g was put into the column, and the plunger was used to confine the fixed bed height at approximate 21.5 cm. The column was temperature controlled by a temperature controller at 25 °C. The packed carbon bed was flushed with distilled water for 3 hours in the upflow direction to ensure that no excess air bubbles, which can affect organic adsorption were in the carbon pores. After that, phenol which was used as the organic contaminant in this study, was loaded in the upward direction with a flowrate

of 100 mL/min. The phenol concentration used was 10, 20, and 30 mg/L. Before switching the valve to load phenol into the carbon bed, the waste line was always used to avoid the error of mixing of the left over water in the column and phenol solution used. This procedure was done every time that there is changing of adsorption conditions. The outlet solution was collected by sample collector every hour and the phenol concentration in gathered samples was measured by HPLC using an absorbance detector at a wavelength of 269 nm.

When the bed was saturated with loaded phenol solution as shown in breakthrough curve. Then SDS solution was introduced to the packed bed to desorb the phenol from carbon by solubilization. This step was called as regeneration step and time used in this step was 50 hours. The effluent was also collected every hour and phenol concentration was analyzed by HPLC using an absorbance detector.

The flushing step followed. Water was flowed through the bed again to eliminate the left SDS adsorbed on carbon bed for 50 hours. In this step, the conductivity detector was used to analyze SDS concentration in the effluent. Eventually, the conductance of outlet measured after 50 hours operating was in the range of 3-6 μs/cm. which is close to that of distilled water.

Finally, the phenol solution was reloaded on the bed in readsorption step for 20 hours. The HPLC with absorbance detector was used to measure the phenol concentration in the effluent again in this step.

3.3.3 Analytical method

High-Performance Liquid Chromatography (HPLC) was used for measuring phenol concentration in the collected effluent in the adsorption step, the regeneration step, and the readsorption step. The mobile phase used was 65% water and 35% methanol. Both of methanol and water were filtered with membrane of 45 μm before using. The mobile phase passed through the Supelcosil LC-18 with a flowrate of 0.8 mL/min. and the retention time. It is noteworthy that all of samples should be filtered with 45 μm. membrane before injection into Supelcosil LC-18 column.