CHAPTER III
EXPERIMENTAL

3.1 Materials

3.1.1 Granular Activated Carbon (GAC)

The BPL 4x10 granular activated carbon (GAC) from Calgon Carbon Company, USA was used as an adsorbent. This activated carbon is for gas phase applications. Technical information of the GAC is shown in Table 3.1.

Table 3.1 Properties of BPL 4x10 GAC

Property	Value
Total surface area (N_2 BET method*)	1,100-1,200 m^2/g
Iodine number	1,050 (min)
CCl_4 number	60 (min)
Butane activity	23.3 (min)
Moisture (as packed)	2 % (max)
Hardness number	93 (min)

3.1.2 Polymeric Hydrophobic Adsorbent

Polymeric hydrophobic adsorbent used in this study was Amberite XAD-4 resin supplied by Rohm and Haas Company (Philadelphia, USA). Technical information of the adsorbent is shown in Table 3.2.

Table 3.2 Properties of XAD-4 resin

Property	Value
Density (dry)	1.08 g/mL
Surface area	725 m^2/g
Pore diameter (average)	9 nm
Pore volume (average)	0.98 mL/g
Mesh size (nominal)number	20-60
Porosity	0.5 mL/mL

3.1.3 Trichloroethylene (TCE)

Trichloroethylene (TCE) was used as volatile organic compound (VOC) to adsorb on the GAC. TCE was purchased from Lab-Scan Analytical Sciences with a reported purity of 99%. Relevant properties of TCE are shown in Table 3.2

Table 3.3 Properties of TCE

Property	Value
Molecular weight	131.39
Boiling point	87.2 °C
Vapor pressure (at 25 °C)	74.3 torr
Solubility (at 25 °C)	1,100 mg/l
Density (at 20 °C)	1.4642 g/l

3.1.3 Sodium Dodecyl Sulfate (SDS)

Sodium dodecyl sulfate was purchased from APS Ajax Finechem. Its purity is reported to be 96%. Properties of SDS are shown in Table 3.3.

Table 3.4 Properties of SDS

Property	Value
Molecular weight	288
Critical micelle concentration (in water)	8.1×10^{-3} M
Aggregation number (in water)	80

3.2 Methodology

3.2.1 Determination of the Breakthrough Adsorption Capacity of GAC

Breakthrough adsorption capacity of GAC before and after regeneration was determined in order to get the dynamic adsorption capacity of corresponding virgin and regenerated GAC. This part was carried out by feeding TCE containing air stream to the GAC bed and monitoring the concentration of TCE in effluent stream. The gas chromatography (GC) with a thermal conductivity detector (TCD) was used for on-line detection of TCE. The adsorption was terminated when TCE in effluent stream was equal to the feed stream.

3.2.2 Regeneration of the Spent GAC

Regeneration of spent GAC consisted of 3 steps

- Desorption of TCE: The spent GAC was contacted with SDS solution in order to determine desorption effectiveness of TCE adsorbed on GAC and to restore its adsorptive capacity by the desorption. In doing this, SDS solution at any particular concentration was fed to the GAC bed then the concentration effluent stream was analyzed. The gas chromatography (GC) equipped with head space facility was used to analyze the concentration of TCE in the effluent stream of SDS solution.

- Rinsing of the GAC bed: The regenerated GAC column was rinsed with water in order to determine if there was any further improvement of its regenerated adsorption capacity and the effectiveness of removal of SDS was also determined. In the rinsing step, water at any particular temperature was fed to wash

the removable SDS containing in the void of the GAC bed and adsorbed on the GAC surface out and the concentration of SDS in the effluent solution was measured. The total organic carbon analyzer was used to determine SDS concentrations in the effluent stream.

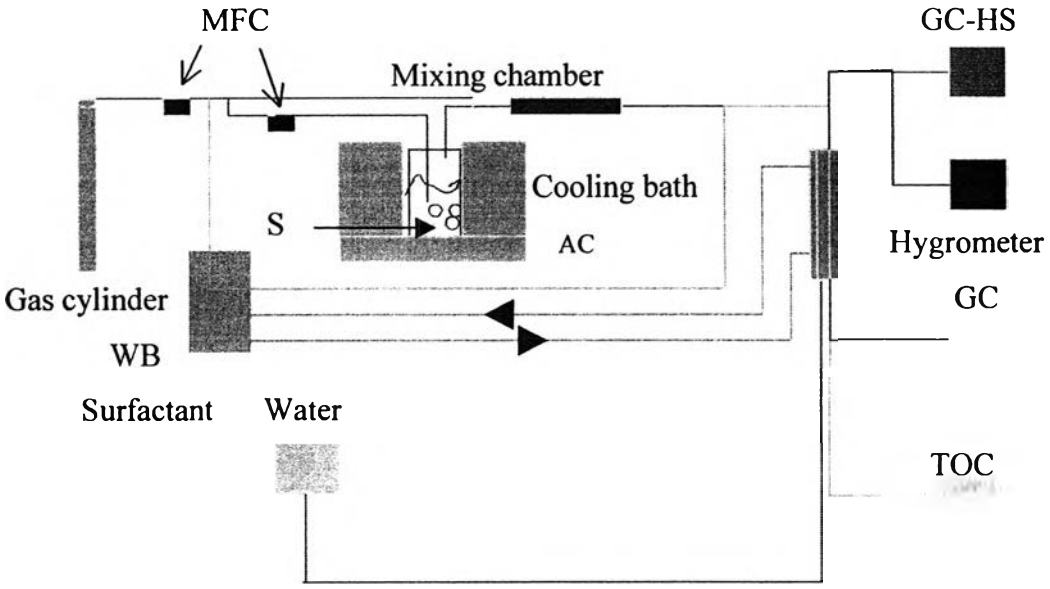
- Drying of the GAC bed: The rinsed and non-rinsed GAC were completely dried by passing a warm air stream to the bed in order to prepare the regenerated GAC ready to adsorb TCE in another adsorption cycle. The drying step was complete when the moisture content in the effluent air stream is less than 2%.

3.2.3 Adsorption-Desorption of SDS on/from GAC Surface

Adsorption-desorption isotherms of SDS on/from GAC surface at 30 to 70°C were carried out in order to determine ability of adsorption and desorption of SDS on and from GAC at any particular temperature. The total organic carbon analyzer was used to analyze SDS concentrations in the equilibrium solution.

3.3 Experimental Equipment

A schematic flow diagram of the system used in this study is shown in Figure 3.1.



AC = Adsorption column GC = Gas chromatograph
GC-HS = Gas chromatography head space MFC = Mass flow controller
TOC = Total organic carbon S = TCE saturator
WB = Water bath

Figure 3.1 Schematic diagram of adsorption and regeneration system.

3.4 Experimental Procedure

3.4.1 Regeneration of GAC and XAD-4

Firstly, the dynamic adsorption experiment of fresh GAC was performed in order to get a breakthrough curve. The contaminated air stream consisting of 1,030 ppmv of TCE, which was obtained from mixing of TCE saturated stream held at 20°C in a cooling bath and air zero grade stream, was continuously fed to the adsorption column at a flow rate of 450 ml/min. The saturated TCE stream was provided by passing air zero stream through the TCE liquid in the saturator. The adsorption column temperature was held at 30°C by circulating water from water bath and jacket around the column. The flow rate of air zero stream to the saturator and the main stream was controlled by a mass flow controller. The effluent stream from the column was detected using an online gas chromatograph. The adsorption

was terminated when the concentration of TCE in the effluent stream was equal to the feed stream. At this state, the GAC was saturated with TCE. The diagram of the dynamic adsorption step is shown in Figure 3.2.

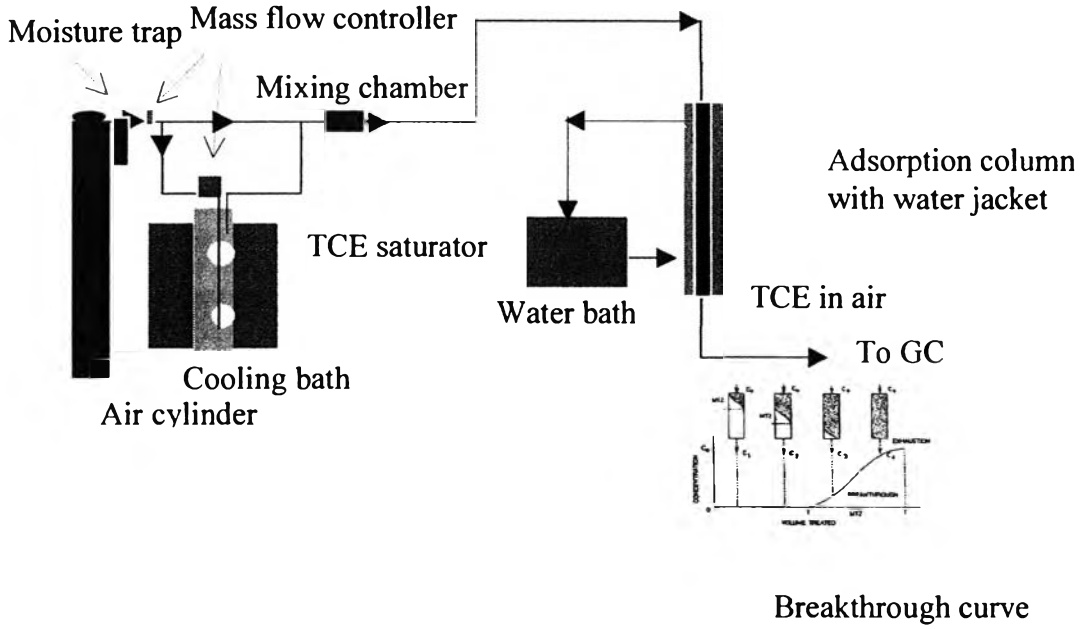


Figure 3.2 Set-up of adsorption step.

Secondly, the desorption of TCE using SDS solution was carried out: The TCE saturated activated carbon was flooded a surfactant solution containing different concentrations of SDS. The flow rate of SDS solution was kept constant at a desired value throughout the experiment by using a rotameter. The temperature of the GAC column was maintained at 30°C by using the circulating water from the water bath and the water jacket around the column. The effluent stream of TCE-containing SDS solution was periodically collected. The collected effluent solution were analyzed the amount of TCE by using gas chromatography equipped with headspace. The desorption was terminated when the TCE in the effluent was not detectable. The diagram of desorption step is shown in Figure 3.3.

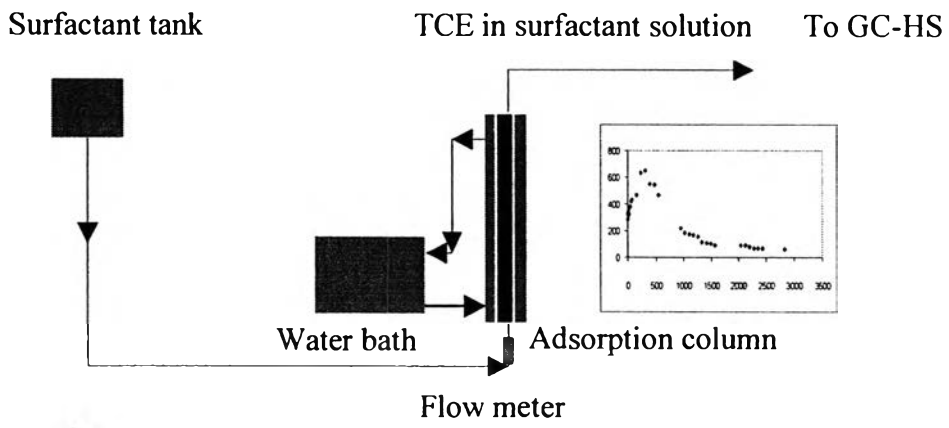


Figure 3.3 Set up of desorption step.

Thirdly, the desorbed GAC was rinsed by distilled water to remove excess SDS in the packed bed. The bed was flood by distilled water at any desired flow rate. The effluent was periodically collected and analyzed for surfactant concentration by using a total organic carbon analyzer (TOC). The rinsing was ended when the SDS was not detectable. The diagram of flushing step is shown in Figure 3.4.

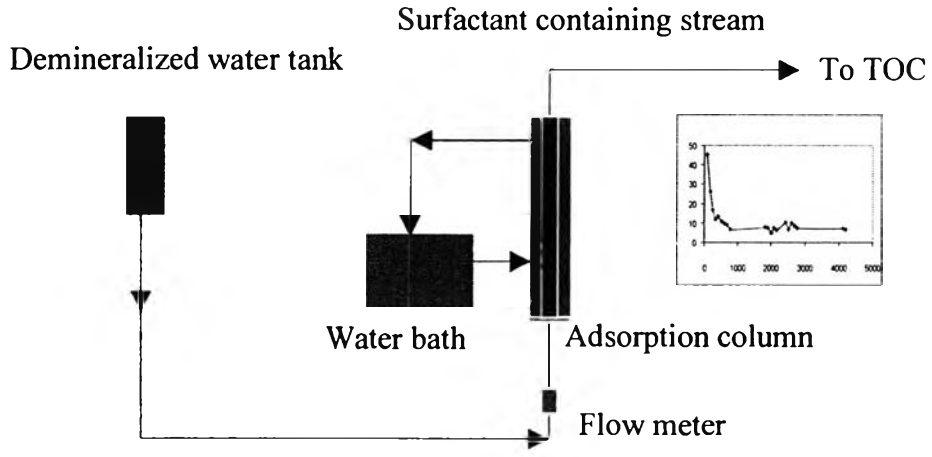


Figure 3.4 Set up of flushing step.

Fourthly, the desorbed GAC was drained and then dried at 50°C by passing dry warm air. The dried and warm air was obtained from passing air zero-grade through the coil submerged in 50°C water bath. The temperature of the GAC

column was maintained at 50°C. The moisture content in the effluent air was detected online by using a hygrometer. The bed was presumably dried when the moisture content in the effluent air stream was less than 2%. The diagram of drying step is shown in Figure 3.5.

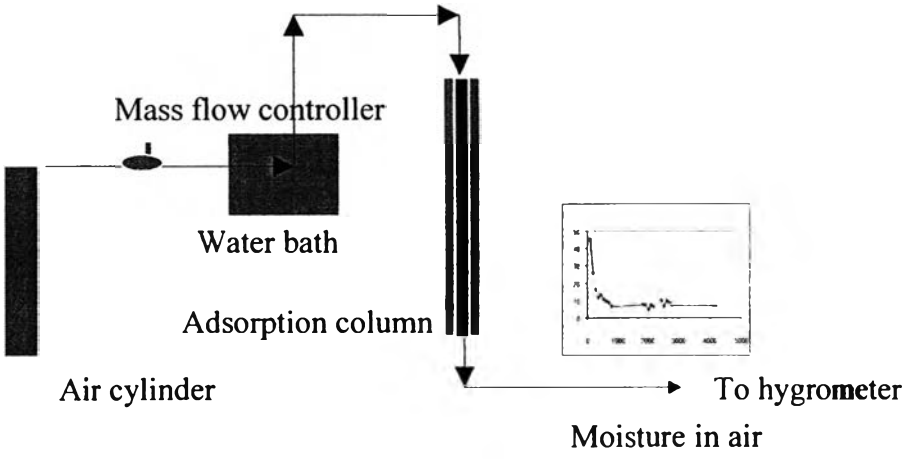


Figure 3.5 Set up of drying step.

After completion of those 4 steps, the adsorption of the regenerated, dried GAC was carried out as mentioned above. The corresponding breakthrough curves of adsorption of the fresh and the regenerated GAC were then compared. In the case of investigation of the rinsing effect, the rinsing step was skipped. For the investigation of the effect of bed height, the GAC was packed in the column at desired heights.

3.4.2 Adsorption-Desorption Isotherms

In the study of surfactant adsorption isotherms, about 0.1 g GAC was placed in 25 mL vials. Then, 10 ml of SDS solutions at various concentrations from 100 to 200,000 μM were added to the vials. The vials were shaken at a speed of 110 rpm in a water bath at any desired temperature for 96 h to reach equilibrium. The solution was separated from GAC and centrifuged at 4,000 rpm for 5 min. The clear solution was filtered. The concentration of SDS in the equilibrium solution was analyzed by the total organic carbon analyzer. Amounts of SDS adsorbed on GAC at

corresponding equilibrium concentration were plotted to construct adsorption isotherms.

In the study of desorption, equilibrium solution in the adsorption experiment were diluted with the factor of 5. The vials were shaken and the procedure stated in the adsorption experiment was carried out. The amounts of SDS desorbed into the solution and corresponding equilibrium concentration were plotted to construct desorption isotherms.

3.5 Investigated Parameters

Intevestigated parameters in this study were the regeneration condition namely, concentration of SDS solution, flow rate of SDS solution, flow rate of rinsing water, and temperature of rinsing water. In addition, the effect of bed height was also investigated. The investigated parameters in this study are shown in Table 3.4.

Table 3.5 Investigated parameters

Parameters	Value
Bed height	1.1-3.6 cm
Rinsing	With rinsing and without rinsing
Concentration of surfactant solution	0.074-0.2 M.
Flow rate of surfactant solution	5-15 ml/min
Temperature of rinsing water	30-50 °C
Flow rate of rinsing water	5-15 ml/min