CHAPTER III
EXPERIMENTAL

3.1 Materials

Sodium dodecyl sulfate (SDS) and sodium octanoate, anionic surfactants, at a purity of 99% were obtained from Sigma Chemical Co., Ltd. (St. Louis, USA). Sodium chloride, with a purity of 99% was obtained from Ajax Chemical Co., Ltd. (Auburn, NSW, Australia). BPL 4×10 bituminous coal-based granular activated carbon (GAC) was supplied by Calgon Carbon Co., Ltd. (Pittsburgh, USA). GAC was reported to have the BET surface area of 1,100-1,200 m^2/g. Polymeric resin, Amberlite XAD-4 was purchased from Acros Organics Co., Ltd. (New Jersey, USA). XAD-4 was reported to have the BET surface area of 750 m^2/g. Distilled water was obtained from Nonsri Co., Ltd. (Bangkok, Thailand).

3.2 Methods

3.2.1 Materials Preparation

Granular activated carbon (GAC) and Amberlite XAD-4 resin were used as adsorbents in this research. Each experiment, GAC was purified by washing with distilled water several times to remove inorganic impurities. After a final rinse with water, the adsorbent was dried at 100°C for at least 48 hours in the oven. The treated carbon was stored in a dessicator. For XAD-4 resin, it was used as received in a touch-dry form.

3.2.2 Adsorption Experiments

All adsorption experiments were carried out in a batch liquid mode of adsorption. The procedure could be described as shown in Figure 3.1. A fixed amount of dry GAC (0.1 g) or XAD-4 resin (0.2 g) was mixed with 10 ml of aqueous solution of surfactant in a 24 ml glass vial. The aqueous solutions of surfactant were prepared by mixing a stock solution of surfactant with distilled water. The vials were

shaken at 110 rpm on the water bath shaker (Memmert WBU45) with a temperature control.

To determine an equilibrium time for the adsorption, samples were taken from the vials at specific time interval during the adsorption. The solid-liquid separation was achieved by centrifugation (ALC, 4256) and then the supernatant was filtered by a syringe filter to remove any particulate present in the solution. The remaining concentration of surfactant was determined by using TOC analyzer (Shimadzu, TOC 5000). When the remaining concentration of surfactant is relatively constant, it is considered to be an equilibrium time for the adsorption.

For the adsorption isotherms, the experiments were carried out using various initial concentrations of SDS in the range of 100-25,000 μM and 100 μM-450 mM for sodium octanoate. After mixing the solid adsorbent and surfactant solution in the vials, the vials were placed on the shaker as previously described. Upon reaching equilibrium, solutions were sampled with a micropipette and analyzed by using TOC analyzer. All experiments were carried out in triplicate.

The amount of surfactant adsorbed on the solid adsorbent was calculated using an appropriate mass balance. Adsorption isotherms were then constructed by plotting the amount of surfactant adsorbed on the solid adsorbent with corresponding equilibrium concentration. For ionic strength effect, experiments were carried out with the addition of 10, 50 and 100 mM of sodium chloride in the SDS solution. Then, the experiments were conducted as mentioned above.

3.2.3 Desorption Experiments

The experiments were first carried out to determine the time required for the desorption to reach equilibrium. After equilibrium was achieved, 8 ml of solution was withdrawn and replaced with 8 ml of distilled water as shown in Figure 3.2. This would give a dilution with a factor of five. The vials were shaken and the samples were taken using similar procedure as stated in the adsorption part. When the remaining concentration of SDS is relatively constant, it is considered to be an equilibrium time for the desorption. The desorption experiments were carried out for three different concentrations, 500, 12000, and 25000 micromolar of SDS, representing low, intermediate and high ranges of concentrations, respectively.

For the desorption isotherm, after dilution, the experiments were carried out until reaching equilibrium and the samples were taken and analyzed in the same manner as described in the adsorption part. The concentration of surfactant in the solution at equilibrium was analyzed by using TOC analyzer. The amount of surfactant desorbed from adsorbent at corresponding equilibrium concentration was plotted to generate the desorption isotherms.
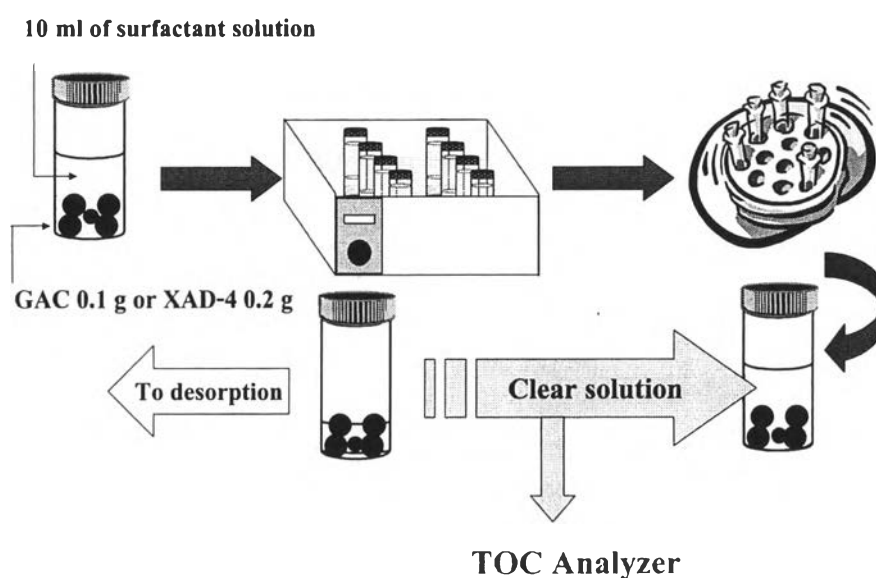

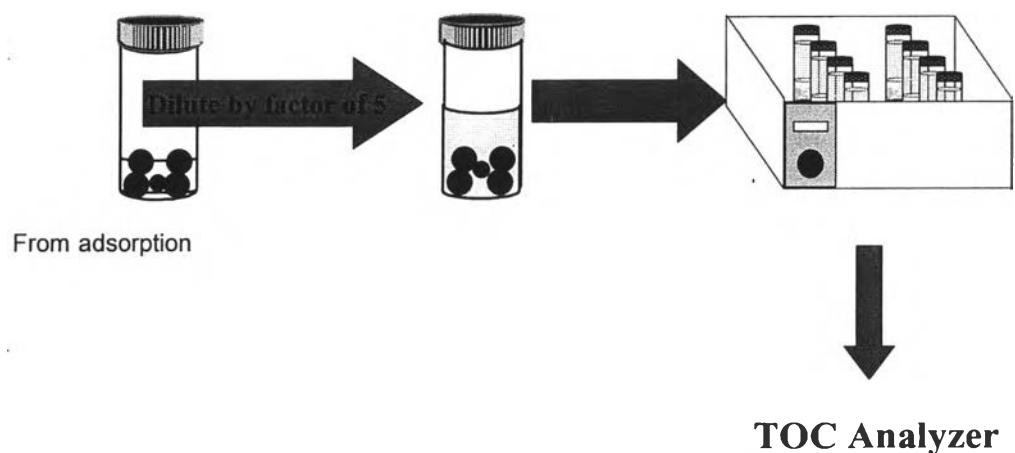**Figure 3.1** Flow chart of adsorption isotherm experiment.

Figure 3.2 Flow chart of desorption isotherm experiment.

3.3 Data analysis

TOC is a main equipment used in this study for the measurement of the concentration of SDS in aqueous solution in the adsorption and the desorption studies. By performing single mass balance, adsorption and desorption of the surfactant in the unit of μmole/gram of adsorbent can be determined by the following equation;

$$Q = \frac{(C_0 - C_e).V}{m}$$

C_0 = initial concentration of surfactant (μmole/L)

C_e = concentration of surfactant at equilibrium (μmole/L)

m = weight of adsorbent (g)

Q = amount adsorbed or desorbed surfactant (μmole/g)

V = volume of solution (L)