

CHAPTER III EXPERIMENTAL

3.1 Materials

All chemicals were obtained from commercial sources and used as received. Cetyltrimethyl ammonium bromide (CTAB), a cationic surfactant, at a purity of 99% was purchased from Aldrich Chemical Company (Milwaukee WI, USA). Precipitated silica, Hi-Sil®255 was obtained from PPG-Siam silica Co., Ltd. (Rayong Thailand). Hi-Sil 255 was reported to have the BET surface area of 170 m²/g with an average particle size of 64 μm. Acetophenone was purchased from Fluka Co., Ltd. with a purity of >98%. Toluene was purchased from Lab-Scan Co., Ltd. with a purity of > 99.5%. Sodium hydroxide and hydrochloric acid were purchased from Lab-Scan Co., Ltd. Sodium bromide (NaBr) was ordered from Sant Chemical Co., Ltd. Deionized water was obtained from Nontri Co., Ltd.

3.2 Methods

3.2.1 Adsorption of Surfactant on Precipitated silica (Adsorption Isotherms).

Batch adsorption experiments were carried out using aqueous cationic surfactant (cetyltrimethyl ammonium bromide, CTAB). CTAB stock solutions previously made were diluted with deionized water to form a series of 20 ml solutions with varying surfactant concentrations. These CTAB solutions were added to vials containing 0.5 g of silica. The temperature of the mixture was kept at 30°C with agitation until equilibrium was reached which took approximate 4 days (Favoriti *et al.*, 1996). After equilibrium, the bulk liquid phase was filtered using a syringe filter and the bulk phase concentration of CTAB was measured using a total organic carbon analyzer (TOC) (Shimadzu, TOC 5000) (Behrends *et al.*, 1999). A simple mass balance was performed to give the amount of adsorbed CTAB on silica. The experiments were carried out at two different pH values (5 and 8) and an ionic strength of 10 mM. The pH of the solution was adjusted by using NaOH or HCl and

ionic strength was adjusted by using NaBr. Schematic diagram of the adsorption of CTAB on silica is shown in Figure 3.1. From the CTAB adsorption experiments, the adsorption isotherms at two pH values could be constructed. After that, for each pH, precipitated silica with different surfactant adsorption was selected from 3 different regions corresponding to 3 different equilibrium surfactant concentrations in the adsorption isotherm. In each region, surface adsorption for both pH were carefully chosen to be very similar which equilibrium concentration and adsorbed amount are reproducible as determined by using elemental analyzer (EA) and total organic carbon (TOC).

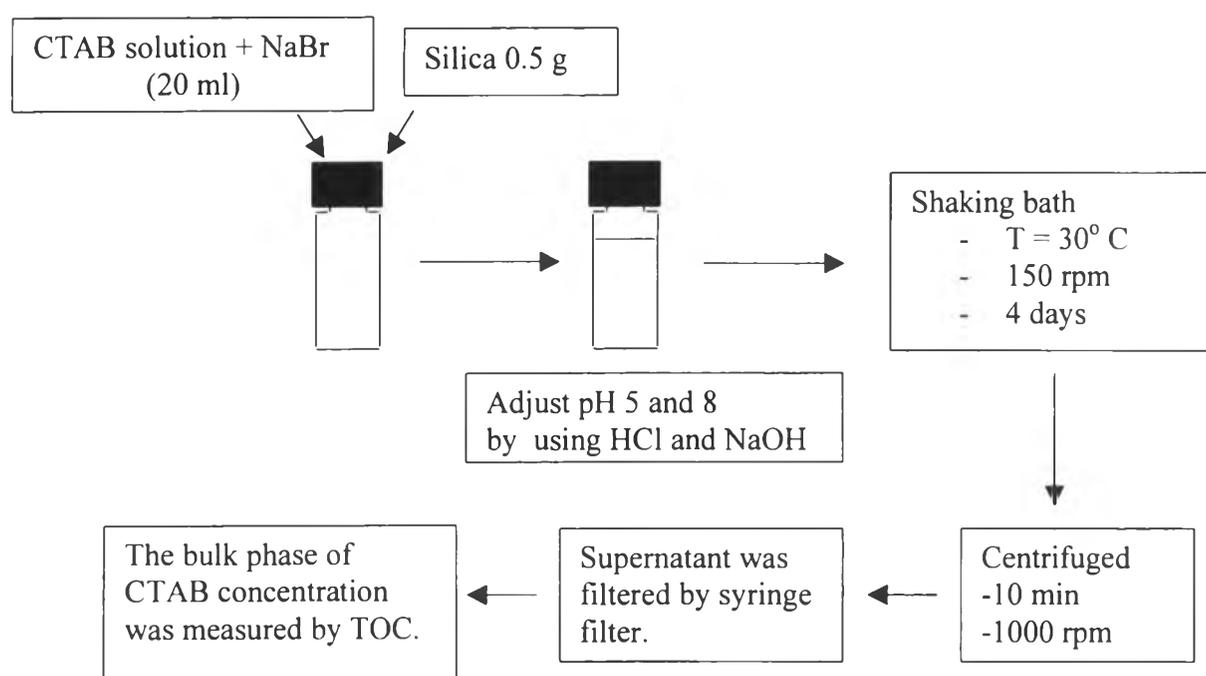


Figure 3.1 Schematic diagram of the adsorption of CTAB on silica.

3.2.2 Adsolubilization of Organic Solute into surfactant Adsorbed on Precipitated silica.

For silica in the 3 regions described above, batch studies of adsolubilization of nonpolar and moderately polar organics solutes were then carried out using toluene and acetophenone, respectively. The experiments were divided into 2 parts: single solute and mixed solute systems. For the adsolubilization

adsorption in the 600 ml glass bottle. The temperature of the mixture was kept at 30° C. The mixture was shaken once per day until equilibrium was achieved. After that the bulk phase concentration of the solute was sampled and analyzed. The concentration of toluene was measured by headspace gas chromatography (Nguyen *et al.*, 1988). The bulk phase concentration of acetophenone was measured by a UV-VIS spectrophotometer (Perkin Elmer, Lambda 10) at 245 nm (Thakulsukanant *et al.*, 1997). A simple mass balance was performed by subtracting initial concentration of solute with the bulk concentration of solute at equilibrium to obtain the amount of solute adsorbed in the admicelle. For mixed-solute systems, the solute of interest (e.g., toluene) is added into the CTAB equilibrium system in the presence of the other solute (e.g acetophenone). The experimental conditions and analytical methods were similar to those of the single solute system. The experiments were repeated at various surface adsorption in three regions selected previously at ionic strength of 10 mM and pH of 5 and 8. Schematic diagram of the adsolubilization of toluene and acetophenone is shown in Figure 3.2.

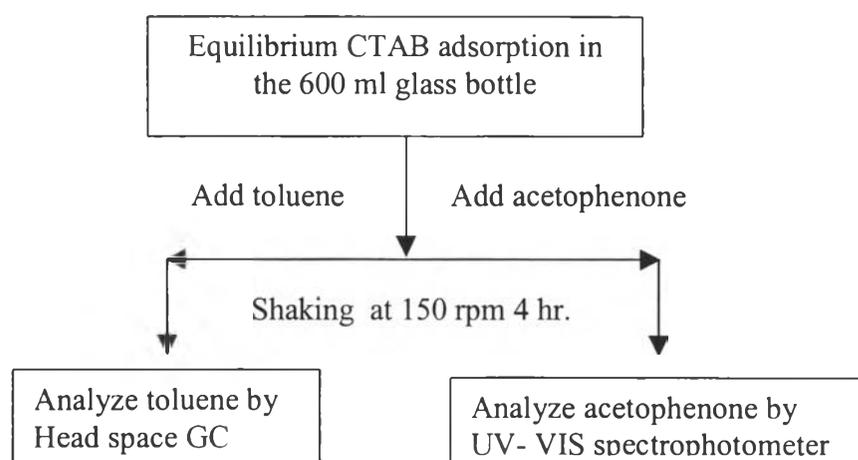


Figure 3.2 Schematic diagram of the adsolubilization of toluene and acetophenone.

3.3 Data analysis

3.3.1 Surfactant adsorption isotherm

An adsorption isotherm for CTAB was constructed by plotting the amount of CTAB adsorbed per gram of silica versus equilibrium concentration of CTAB.

3.3.2 Partition coefficient (K)

The adsorbed surfactant on the solid can be treated as a pseudophase and the partition coefficient (K) of solute solubilized in admicelles used to describe the distribution of substance between the aqueous phases and the surfactant coverage. It can be calculated as follow (Behrends and Hermann, 1988 and Holzheu S. *et al.*, 2000)

$$K = \frac{X_{admicelle}}{X_{bulk}}$$

Where $X_{admicelle}$ = mole fraction of solute in the surfactant coverage

X_{bulk} = mole fraction of solute in the bulk

Partition coefficient plots (K) can then be generated by plotting the partition coefficient (K) vs mole fraction of surfactant coverage ($X_{admicelle}$).