CHAPTER III
EXPERIMENTAL SECTION

3.1 Materials

Precipitated silica, Hi-Sil® 255 was obtained from PPG-Siam silica Co., Ltd. (Rayong Thailand). The BET surface area was 170 m^2/g with an average particle size of 64 μm. The point of zero charge (ZPC) of precipitated silica is $2 < pH < 3$ (Iler, 1979). Cetyltrimethyl ammonium bromide (CTAB), a cationic surfactant, was obtained from Fluka Co., Ltd. with a manufacturer-report purity of > 98%. Toluene was purchased from Lab-Scan Co., Ltd. with a purity of > 99.5%. Acetophenone was purchased from Fluka Co., Ltd. with a purity of > 98%. Sodium hydroxide and hydrochloric acid were purchased from Lab-Scan Co., Ltd. All materials were used as received. Deionized water was obtained from Nontri Co., Ltd.

3.2 Method

The experimental method can be divided into 3 parts: adsorption isotherm development, adsolubilization, and analysis.

3.2.1 Adsorption of Surfactant on Solid Surface

Batch adsorption experiments were carried out using aqueous solutions of cetyltrimethyl ammonium bromide, (CTAB) at a constant pH. This was done by mixing and varying amounts of a CTAB stock solutions with deionized water. The solution was mixed with the silica in 20 ml vials. The temperature of the mixture was kept at 30 °C and constant pH was maintained by the addition of NaOH or HCl. The vials were agitated for four days until

equilibrium was achieved. (Favoriti *et al.*, 1996). Then the bulk phase was filtered using a syringe filter and the supernatant CTAB concentration was measured using a total organic carbon analyzer (TOC)(Shimadzu TOC 5000) (Behrends *et al.*, 1999). A simple mass balance determined the amount of adsorbed CTAB. The experiment was performed at pH's of 5 and 8. .

3.2.2 Adsolubilization of Organic Solutes into Adsorbed Aurfactant on Solid Surface

The adsorption of toluene and acetophenone on the precipitated silica in the absence of surfactant was not significant. Batch studies of adsolubilization were carried out using nonpolar and moderately polar organic solutes (toluene and acetophenone). For the adsolubilization of a single solute, the organic solute was dissolved in a mixture of 600 ml CTAB solution and precipitated silica, which had equilibrated at constant pH for four days, as described above. The mixture was kept at 30 °C and shaken occasionally for four days . The bulk phase concentration of the solute was then analyzed. by (1) placing 1 ml of the supernatant into a 20 ml vial for Head-space Gas Chromatography analysis and (2) UV-VIS spectrophotometer at the wave length 245 nm analysis. The equilibrium concentration of toluene was obtained by the head-space gas chromatography analysis and the equilibrium of acetophenone was obtained by UV-VIS spectrophotometer. A simple mass balance was performed by subtracting the final or equilibrium solute concentration from the initial concentration to obtain the amount of adsolubilized solute. For mixed-solute systems, the solute of interest (e.g., toluene) was added into the CTAB equilibrium system in the presence of known quantities of the other solute (e.g., acetophenone). The experimental conditions and analytical methods were similar to those of the single solute system. The bulk phase concentration of toluene was measured by a head-

space gas chromatography (Nguyen *et al.*, 1988). The bulk phase concentration of acetophenone was measured by a UV-VIS spectrophotometer (Perkin Elmer Lamda 10) (Thakulsukanant *et al.*, 1997). The experiment was repeated at pH's of 5 and 8.

3.2.3 Analysis

3.2.3.1 Surfactant Adsorption Isotherms

CTAB adsorption isotherms were constructed by plotting the amount of CTAB adsorbed per gram of silica versus the equilibrium CTAB concentration

3.2.3.2 Adsolubilization Isotherms

Adsolubilization isotherms were generated by plotting the amount of toluene and acetophenone adsolubilized per gram of silica versus the equilibrium solute concentration.

3.2.3.3 Adsolubilization Equilibirum Constant (K)

The adsolubilization equilibrium constant or partition coefficient (K) can be used to describe the distribution of solutes between the aqueous phases and the admicelle pseudophase (Kitiyanan *et al.*, 1995; and Holzheu *et al.*, 2000):

$$K = \frac{X_0}{C_u} \tag{3.1}$$

where X_0 is mole fraction of adsolubilizate in admicelle

C_u is the concentration of solute in the bulk aqueous phase

Adsolubilization equilibrium constant plots (K) were generated by plotting the adsolubilization equilibrium constant (K) versus the mole fraction of solute in the admicelle (X_0).