CHAPTER III
EXPERIMENTAL

3.1 Materials

All Chemicals were obtained from commercial sources and used as received. Cetyl trimethyl ammonium bromide (CTAB), a cationic surfactant, with a purity of 96% and Triton X-165 (70% in water), nonionic surfactant were purchased from Fluka Co., Ltd. (Milwaukee, WI, USA.). Triton X-305 (70% in water), nonionic surfactant was kindly provided by Dow Chemical Thailand Ltd. Acetophenone with a purity of > 98% was purchased from Fluka Co., Ltd. Toluene with a purity of > 99.5% and Sodium Hydroxide (NaOH) with a purity of > 99.5% were purchased from Lab-Scan Co., Ltd. (Bangkok, Thailand). Precipitated silica, Hi-Sil®255 was supplied by PPG-Siam silica Co., Ltd. (Rayong, Thailand). The BET surface area was reported at 170 m^2/g with an average particle size of 64 μm. Deionized water was obtained from Nonsri Co., Ltd. (Bangkok, Thailand).
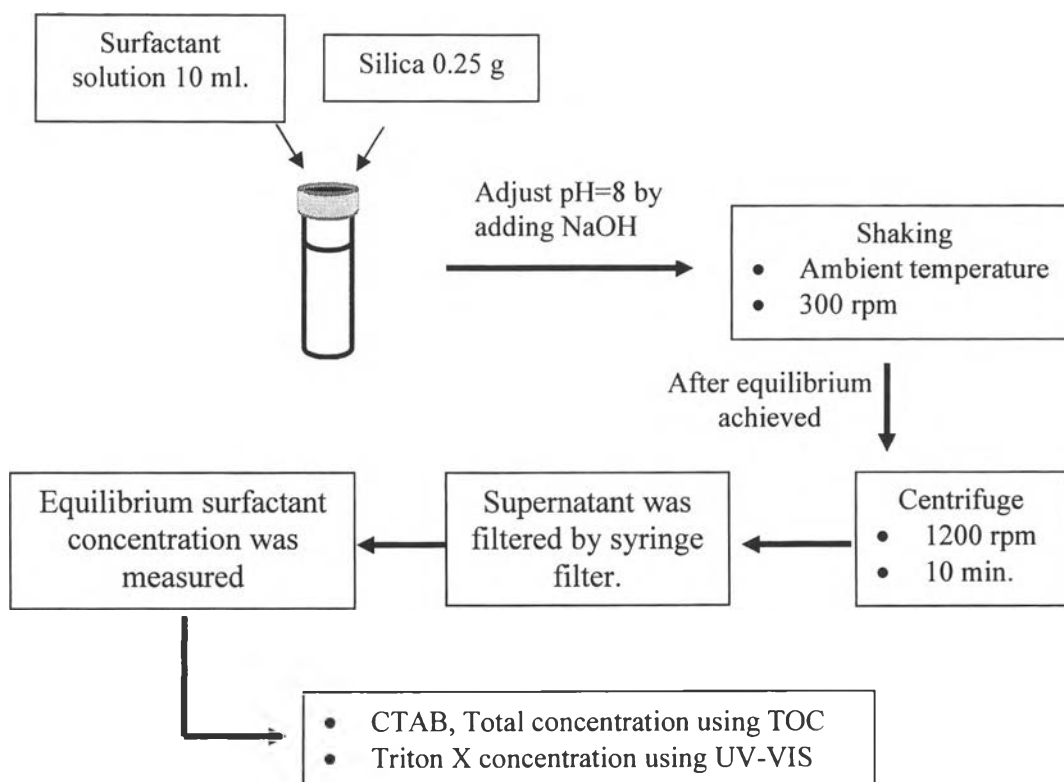
3.2 Methods

3.2.1 Adsorption of Surfactant on Precipitated Silica

3.2.1.1. Adsorption of a Single Surfactant

Batch adsorption experiments were carried out using aqueous solutions of surfactant at constant pH = 8 and at ambient temperature. Surfactant stock solutions were diluted with distilled water to form a series of 20-ml solutions with varying surfactant concentrations. These solutions were then mixed with 0.25 g. of silica in 20-ml glass vials. The temperature of the mixture was kept at ambient temperature (27°C) and constant pH=8 was maintained by the addition of small amounts of dilute NaOH. The vials were agitated on an orbital shaker for 2 days until equilibrium was achieved (Pradubmook *et al.*, 2003). The bulk phase was then removed through a syringe filter and the equilibrium surfactant concentration in the supernatant will be determined. CTAB concentration was analyzed by using Total Organic Carbon analyzer (TOC) (Shimadzu TOC 500) (Behrends *et al.*, 1999). For

Triton X-165 and Triton X-305, the equilibrium surfactant concentration was analyzed by using UV-VIS spectrophotometer (Perkin Elmer, Lamda 10) at 275 nm. A simple mass balance was performed to amount of adsorbed surfactant on precipitated silica. Surface coverage in region III from adsorption isotherm was selected for adsolubilization of organic solute.

Schematic diagram of the adsorption of surfactant on precipitated silica is shown in Figure 3.1.

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

### 3.2.1.2. Adsorption of Mixed Cationic: Nonionic Surfactants

Similar procedure to the adsorption of single surfactant was used in this part. Batch adsorption experiments were carried out using aqueous solution of CTAB and Triton X (165 or 305) with different molar ratios (1:1, 1:3, 3:1) at constant pH = 8 and at ambient temperature. After equilibrium was reached, the bulk phase was filtered by using syringe filter. In the binary system, TOC and UV-VIS sprctrophotometer were systematically used to analyze the total surfactant

concentration and Triton X-(165 or 305) concentrations. Then CTAB concentration was calculated by subtracting Triton X-(165 or 305) concentrations from total surfactant concentration.

The adsorption of each system was repeated at least three times and through a simple mass balance, the amount of surfactant adsorbed per gram of silica was calculated and plotted as a function of the equilibrium concentration on a log-log plot.

3.2.2 Adsolubilization of Organic Solute on Adsorbed Precipitated Silica

Batch adsolubilization studies were carried out using nonpolar and moderately polar organic solutes (toluene and acetophenone). The organic solute was dissolved in a mixture of 600-ml surfactant solution and precipitated silica, which had equilibrated at constant pH = 8 for 2 days, as described above. The mixture was kept at ambient temperature and shaken occasionally for 2 days. The bulk liquid phase was analyzed for the concentration of solute. The concentration of toluene was measured by headspace gas chromatography (Nguyen *et al.*, 1988) and the bulk phase concentration of acetophenone was measured by UV-VIS spectrophotometer 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 adsolubilized in the admicelles. This procedure was used for both single surfactant system (CTAB, Triton X-165 and Triton X-305) and mixed surfactant system (CTAB with Triton X-165 and CTAB with Triton X-305) with different molar ratios. Schematic diagram of the adsolubilization of toluene and acetophenone is shown in the Figure 3.2.
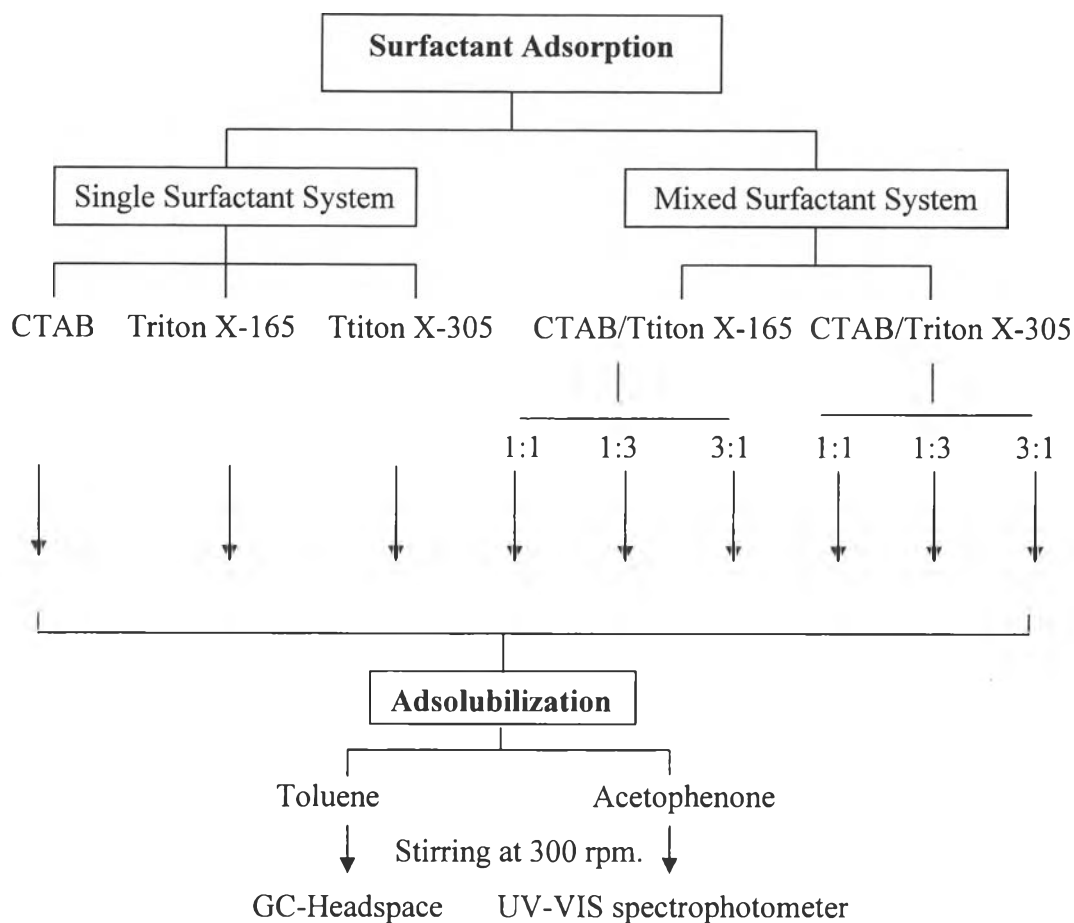
Figure 3.2 Schematic diagram of the adsolubilization of toluene and acetophenone.

3.3 Data Analysis

3.3.1 Adsorption Isotherm

Adsorption isotherms of CTAB, Triton X-165 and Triton X-305 in single- and mixed- surfactant systems were constructed by plotting the amount of surfactant adsorbed per gram of silica versus equilibrium concentration of surfactant.

3.3.2 Adsolubilization Isotherm

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

3.3.3 Adsolubilization Equilibrium Constant or Partition Coefficient (K)

The adsolubilization equilibrium constant or partition coefficient (K) was used to describe the distribution of solutes between the aqueous phases and the admicelle pseudophase (Holzheu *et al.*, 2000). K can be determined from the following equation.

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

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

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

Partition coefficient (K) plots were generated by plotting the partition coefficient (K) versus the mole fraction of solute in the admicelle ($X_{admicelle}$)