

## CHAPTER III EXPERIMENTAL



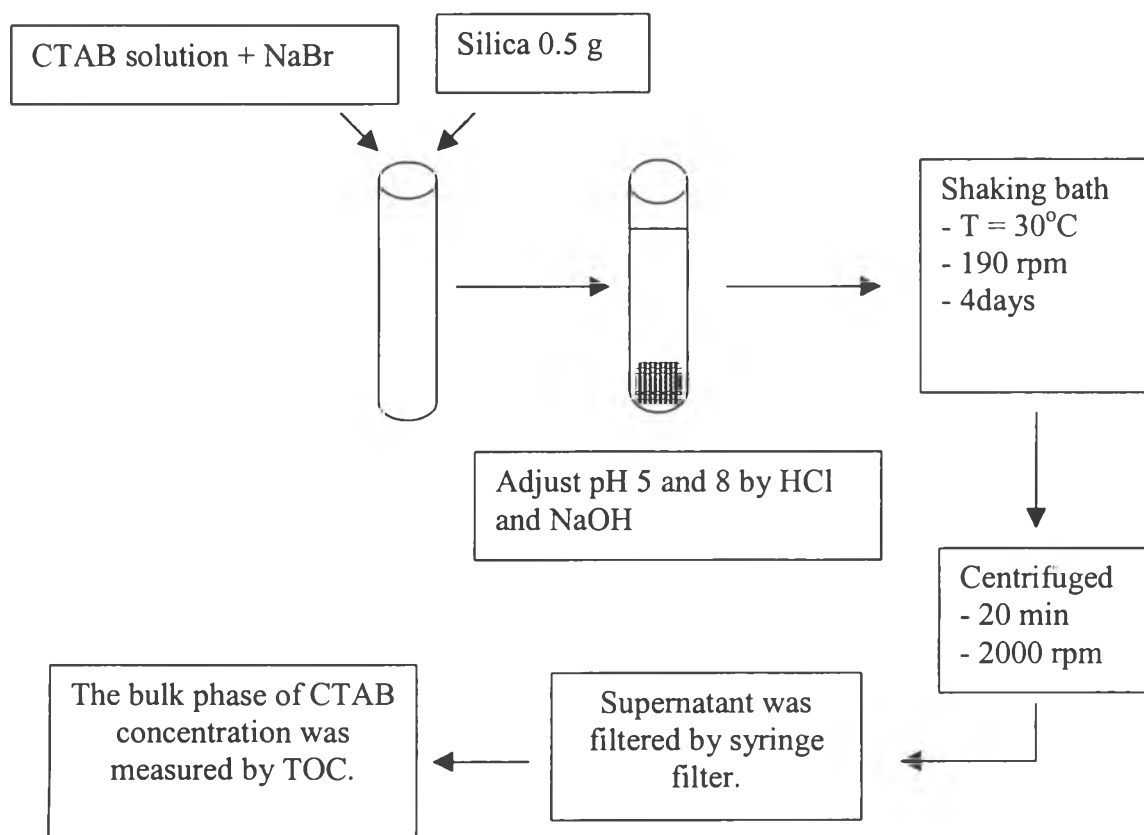
### 3.1 Materials

All chemicals were obtained from commercial sources and used as received. Cetyltrimethyl ammonium bromide (CTAB), at a purity of 99% was purchased from Aldrich (Milwaukee WI, USA). Hi-Sil<sup>®</sup> 255, precipitated silica was received from PPG-Siam silica Co., Ltd (Rayong, Thailand) with the reported surface area of 170 m<sup>2</sup>/g. Sodium bromide (NaBr) was purchased from Sant Chemical Co., Ltd. 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. Deionized water was obtained from Nontri Co., Ltd.

### 3.2 Methods

#### 3.2.1 Adsorption of Surfactant on Precipitated Silica.

Batch adsorption experiments were carried out using aqueous cationic surfactant (cetyltrimethyl ammonium bromide, CTAB) at a constant pH. The CTAB aqueous solution was mixed with the silica particle in a 20 ml vial. The temperature of the mixture was kept at 30 °C with agitation until equilibrium was reached (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 experiment was repeated at different pH values (5 and 8) and different ionic strength values (1, 10, and 100 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.

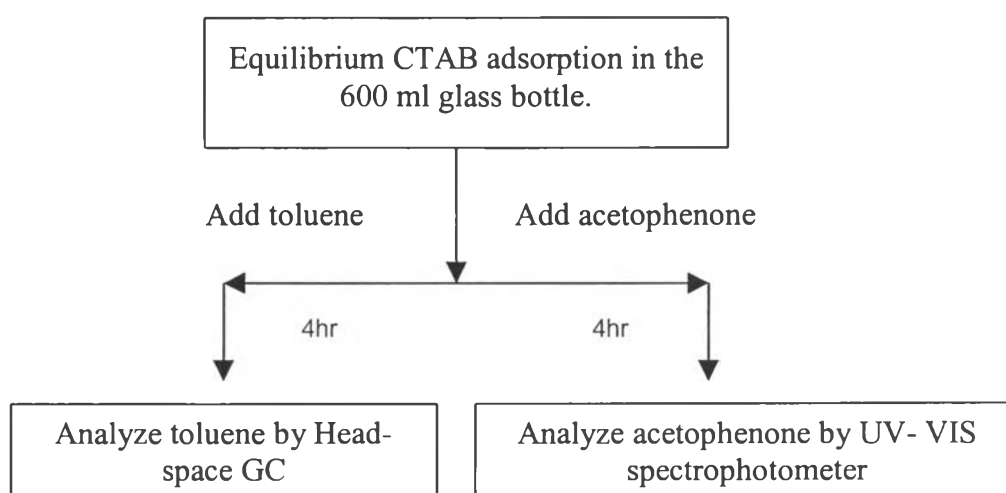


**Figure 3.1** Schematic diagram of the adsorption of CTAB on silica.

### 3.2.2 Adsolubilization of Organic Solute into Surfactant Adsorbed on Precipitated Silica.

Batch studies of adsolubilization were then be carried out using nonpolar and moderately polar organic solutes (toluene and acetophenone). For the adsolubilization of a single solute system, the organic solute was mixed with equilibrium CTAB adsorption in the 600 ml glass bottle. The temperature of the mixture was kept at 30 °C. The mixture was occasionally agitated until equilibrium was achieved. After that the bulk phase concentration of the solute was sampled and analyzed. The amount of bulk phase was used 1 ml into 20 ml of septum sealed vial for head-space gas chromatography analysis and 5 ml into cuvette for UV-VIS spectrophotometer, respectively. 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 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, Lambda 10) at the wave length 245 nm. (Thakulsukanant *et al.*, 1997). The experiments were repeated at different pH values (5 and 8) and ionic strengths ranging from 1 to 100 mM for pH 5 and ionic strengths ranging from 1 to 10 for pH 8. The pH value was adjusted by using NaOH or HCl. Different ionic strengths were achieved by varying NaBr concentration of the aqueous solution. It is very important to note that upon changing ionic strength of the system, the critical micelle concentration (CMC) of the CTAB adsorption on silica was also changed. The lower CMC was obtained at higher ionic strength. Therefore, in this part of the study, the experiments were carefully designed and carried out at a predetermined CTAB concentration below CMC. 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 Isotherms

CTAB adsorption isotherms 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 surfactant coverage on solid phases could be treated as a pseudophase and the partition coefficient (K) could be used to describe the distribution of substance between the aqueous phases and the surfactant coverage to calculate as follow (Behrends and Herrmann, 1998; Holzheu *et al.*, 2000):

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

Where  $X_{\text{admic}} =$  mole fraction of solute in the surfactant coverage (admicelle).

$X_{\text{bulk}} =$  mole fraction of solute in the bulk.

Partition coefficient plots (K) was generated by plotting the partition coefficient (K) versus mole fraction of surfactant coverage ( $X_{\text{admic}}$ ).