

## CHAPTER III EXPERIMENTAL

### 3.1 Materials

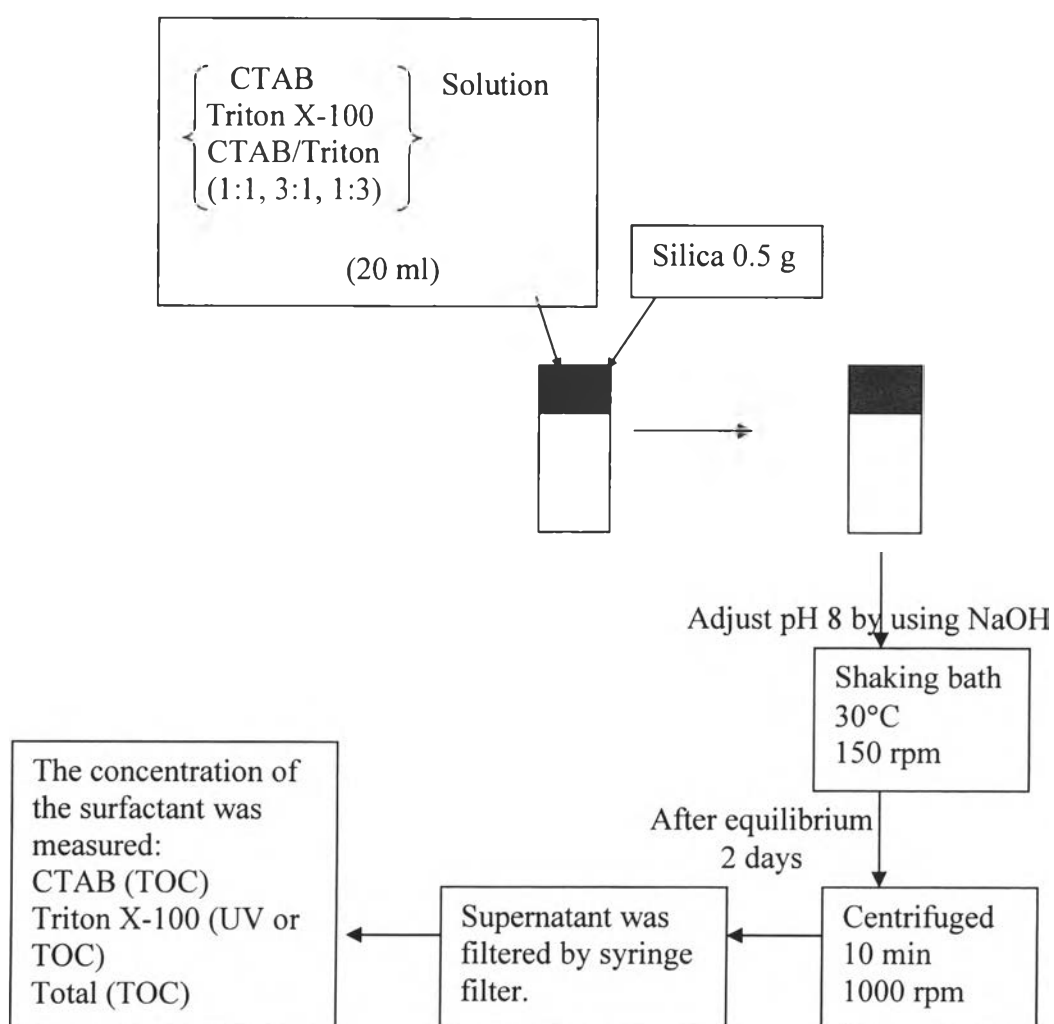
All chemicals were obtained from commercial sources and were used as received. Precipitated silica, Hi-Sil<sup>®</sup>255 was kindly provided by PPG-Siam Silica Co.,Ltd. (Rayong, Thailand) with BET surface area and average particle size of 170 m<sup>2</sup>/g and 64 μm, respectively. Acetophenone was purchased from Fluka Co., Ltd. with a purity of >99%. Sodium hydroxide and toluene with a purity of > 99.5% were purchased from Carlo Erba (Milan, Italy). Cetyltrimethylammonium bromide (CTAB), a cationic surfactant, at a purity of 96%, and Triton X-100, a nonionic surfactant, were purchased from Fluka Co., Ltd (Steinheim, Switzerland). Deionized water was obtained from Nontri Co., Ltd (Bangkok, Thailand).

### 3.2 Experimental

#### 3.2.1 Adsorption of Surfactant on Precipitated Silica

Batch adsorption experiments were carried out using aqueous solutions of cetyltrimethylammonium bromide (CTAB) at pH 8. CTAB stock solutions were mixed with varying amount of deionized water. The mixture was added to vials containing 0.5 g of silica. The temperature of the mixture was kept at 30°C and pH was adjusted to 8 with NaOH. The vials were agitated for 2 days until equilibrium was achieved. The bulk phase was then removed through 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 mass balance was performed to determine the amount of adsorbed CTAB. Similar adsorption experiments were carried out for the systems using nonionic surfactant, Triton X-100, and mixtures of CTAB and Triton X-100 at various molar ratios (1:1, 3:1, 1:3). For Triton X-100, the concentration of the surfactant in the supernatant was measured by UV-VIS spectrophotometer (Perkin Elmer, Lamda 10) at wavelength

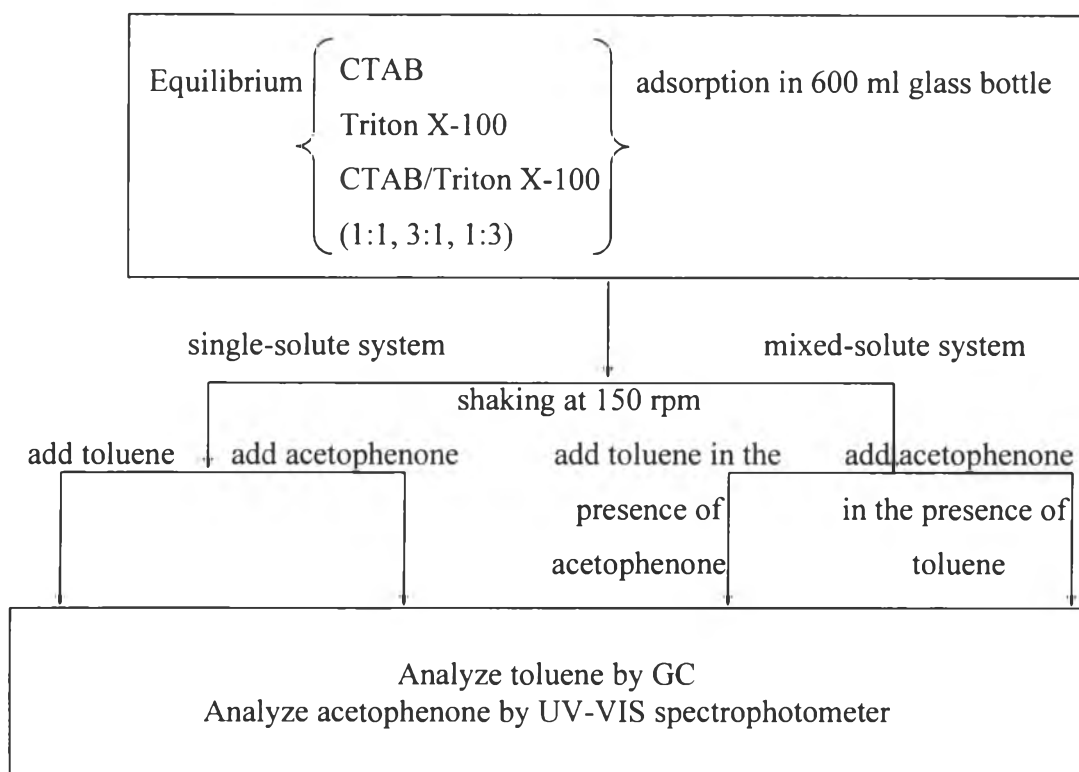
275 nm (Huang and Gu, 1987). For mixed surfactants, both TOC and spectrophotometer were systematically used to determine the concentration of total surfactants and Triton X-100, and thus, CTAB concentration could be subsequently determined. Schematic diagram of the adsorption experiments is shown in Figure 3.1.



**Figure 3.1** Schematic diagram of the adsorbed surfactant on silica.

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

Batch studies of adsolubilization were carried out using a nonpolar and moderately polar organic solute (toluene and acetophenone). For the adsolubilization of a single-solute system, the organic solute was dissolved in a mixture of 600 ml CTAB solution and precipitated silica, which had equilibrated at pH 8 for 2 days. The mixture was kept at 30°C and agitated on magnetic stirrer. After the sedimentation, the bulk phase 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, Lamda 10) at wavelength 245 nm (Thakulsukanant *et al.*, 1997). 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.

A mass balance was performed by subtracting initial concentration of solute with equilibrium concentration of solute to obtain the amount of adsolubilized solute. For mixed-solute systems, the solute of interest (e.g., toluene) was added into equilibrated CTAB-silica samples in the presence of a known quantity of added acetophenone. The experimental conditions and analytical methods were the same as in the single-solute system. Similar experiments were also performed for other systems (Triton; CTAB/Triton 1:1, 3:1, 1:3).

### 3.3 Data Analysis

#### 3.3.1 Surfactant Adsorption Isotherm

Adsorption isotherms of CTAB and Triton X-100 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 of toluene and acetophenone were generated by plotting the amount of adsolubilized solute versus equilibrium concentration of solute.

#### 3.3.3 Partition Coefficient (K)

The surfactant adsorption on solid phases can be treated as a pseudophase and the partition coefficient (K) of solute solubilized in admicelles could be used to describe the distribution of substance between the aqueous phases and the surfactant coverage. It can be calculated as follow (Holzheu *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 ( $X_{admicelle}$ ).