

CHAPTER III

EXPERIMENTAL

3.1 Materials

All chemicals were obtained from commercial sources and used as received. Precipitated silica, Hi-Sil[®]255 was provided by PPG-Siam Silica Co., Ltd. (Rayong, Thailand) with BET surface area and average particle size of 170 m²/g and 64 μm, respectively. Benzene was purchased from BDH Co., Ltd. (Poole, England) with a purity of >99.7%. Toluene was purchased from Carlo Erba (Milan, Italy) with a purity of > 99.5%. Ethylbenzene was purchased from Fluka Co.,Ltd. (Steinheim, Switzerland) with a purity of >98.0%. 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). Sodium hydroxide and hydrochloric acid were purchased from Lab-Scan Co., Ltd. (Samutsakorn, Thailand). Deionized water was obtained from Nontri Co., Ltd (Bangkok, Thailand).

3.2 Experimental

3.2.1 Adsorption of Surfactant on Precipitated Silica

Batch adsorption of both single and mixed surfactant systems were carried out as follows. CTAB stock solutions were diluted with deionized water to form a series of 20 ml solutions with varying surfactant concentrations. The mixture was added to vials containing 0.5 g of silica and adjusted to different pH values (3, 5, and 8) by adding NaOH or HCl. The vials were then placed on a shaker and kept at a constant temperature of 30 °C. After equilibrium was reached, the bulk phase was filtered using syringe filter and CTAB concentration in the supernatant was measured using total organic carbon analyzer (TOC) (Shimadzu TOC 5000) (Behrends *et al.*, 1999). Simple mass balance was performed to give the amount of adsorbed CTAB per gram of silica. Similar adsorption experiments were carried out for the systems using nonionic surfactant, Triton X-100, and mixtures of CTAB and Triton X-100 at equimolar ratio. For Triton X-100, 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.

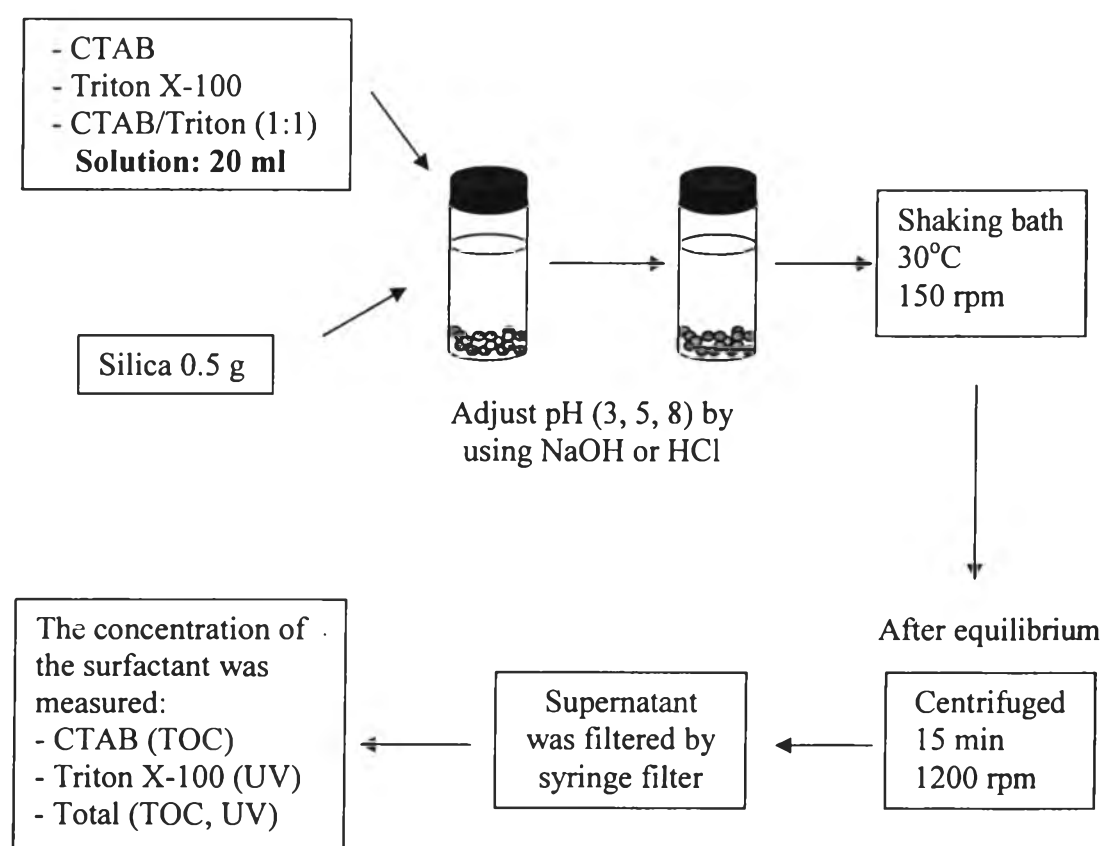


Figure 3.1 Schematic diagram of the adsorbed surfactant on silica.

3.2.2 Adsolubilization of Organic Compounds into Surfactant Adsorbed on Precipitated Silica

These experiments were divided into 2 parts: single-surfactant and mixed-surfactant systems. For the adsolubilization of single surfactant system (CTAB and Triton X-100), the organic compounds were added into the equilibrium surfactant adsorbed on precipitated silica in the 600 ml glass bottle at different pH values (3, 5, and 8). The glass bottle was agitated at ambient temperature until

equilibrium for 2 days. After that the solids were separated and the supernatant was analyzed for organic compounds concentration by a headspace gas chromatography (Nguyen *et al.*, 1988). A simple mass balance was used to determine adsolubilization. For mixed-surfactants system (equimolar CTAB-Triton X), the organic compound (benzene, toluene, and ethylbenzene) was added into mixed surfactants equilibrium system. The experimental conditions and analytical methods were the same as in the single-surfactant system. Schematic diagram of the adsolubilization of organic compounds is shown in Figure 3.2.

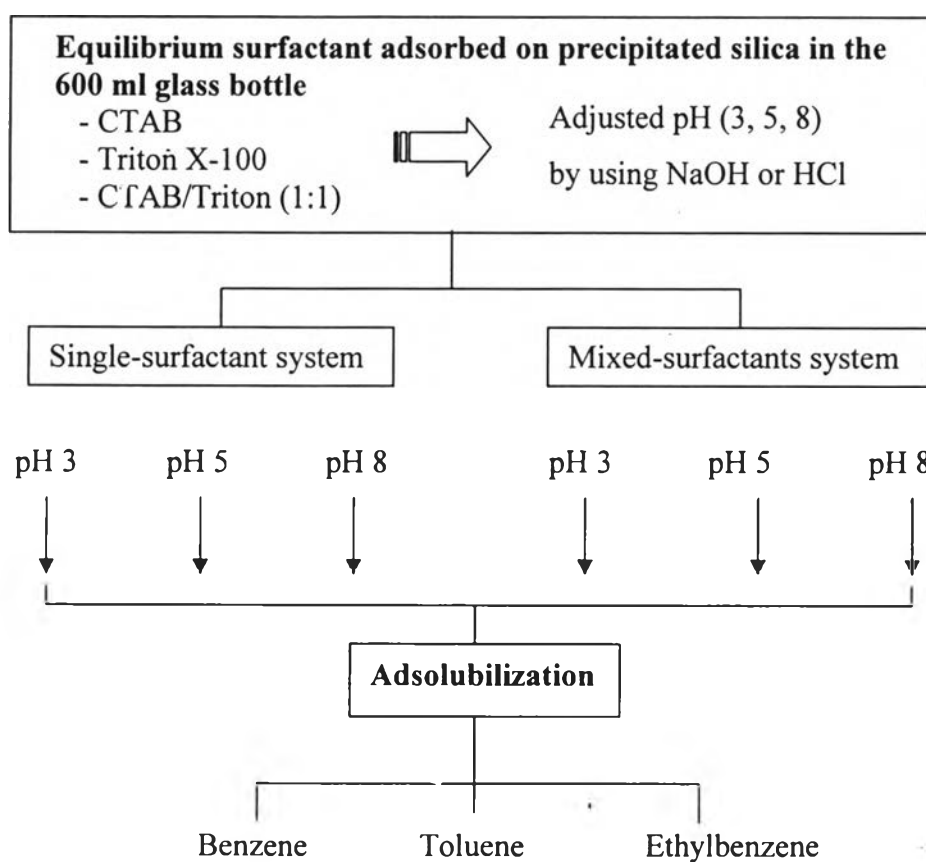


Figure 3.2 Schematic diagram of the adsolubilization of organic compounds.

3.3 Data Analysis

3.3.1 Surfactant Adsorption Isotherm

Adsorption isotherm of CTAB, Triton X-100, and equimolar mixed surfactants were constructed by plotting the amount of surfactant adsorbed per gram of silica versus equilibrium concentration of surfactant.

3.3.2 Adsolubilization Isotherm

Adsolubilization isotherm of organic compound (benzene, toluene, and ethylbenzene) was generated by plotting the amount of adsolubilized solute versus equilibrium concentration of solute.

3.3.3 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 surfactant coverage

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

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