

CHAPTER III

EXPERIMENTAL PROCEDURES

3.1 Materials

All chemicals were obtained from suppliers and used as received. Precipitated silica, Hi-Sil 255 was purchased from Sakdarongreungkij Co., Ltd. (Bangkok, Thailand). The BET surface area was 170 m²/g. Cetyltrimethylammonium bromide (CTAB), a cationic surfactant, at a purity of > 98%, acetophenone (purity > 98%) and magnesium chloride anhydrous (MgCl₂) were purchased from Sigma-Aldrich Co. LLC. (Missouri, USA). Sodium chloride (NaCl), sodium hydroxide and hydrochloric acid were purchased from RCI Labscan Limited (Bangkok, Thailand). Sodium carbonate anhydrous (Na₂CO₃), with a purity of > 99%, was purchased from Ajax Finechem Pty Ltd (Taren Point NSW, Australia). Magnesium sulfate anhydrous (MgSO₄), with a purity of > 99%, was purchased from QRëC (New Zealand). Deionized water was obtained from Nontri Co., Ltd. (Bangkok, Thailand).

3.2 Equipment

- Total Organic Carbon (TOC) analyzer, Shimadzu TOC 5000
- UV-VIS Spectrophotometer, Perkin Elmer Lamda 10
- METTLER TOLEDO, pH meter
- METTLER TOLEDO, Balance
- Syringe filter
- Vial (size 20 ml)
- Glass bottle (size 20 ml)

3.3 Experimental Procedures

3.3.1 Adsorption of Surfactant on Precipitated Silica

Batch adsorption experiments were carried out using aqueous solutions of cetyltrimethylammonium bromide (CTAB) at a constant pH. CTAB stock solutions were mixed with varying amounts of deionized water. Then, the solutions were added with 0.5 g of silica in 20 ml vials. The temperature of the mixture was kept at 30 °C and constant pH 8 was maintained by the addition of small amounts of dilute NaOH or HCl. The vials were agitated for 4 days until equilibrium was achieved. After equilibrium, the bulk liquid phase was filtered using a syringe filter and the bulk phase CTAB concentration was measured using a total organic carbon (TOC) analyzer (Shimadzu, TOC 5000). A simple mass balance determined the amount of adsorbed CTAB. The experiments were repeated with four different electrolytes (NaCl, MgCl₂, MgSO₄ and Na₂CO₃) and at five different ionic strength values (0.1, 0.5, 1, 5 and 10 mM).

3.3.2 Adsolubilization of Organic Solute

The adsorption of acetophenone on the precipitated silica in the absence of surfactant was not significant. Batch studies of adsolubilization were carried out using a moderately polar organic solute (acetophenone). The organic solute was dissolved in a mixture of equilibrium CTAB solution and 0.5 g of precipitated silica in a 20 ml glass bottle, which had equilibrated at constant pH 8 for 4 days, as described above. The mixture was kept at 30 °C and shaken occasionally for 4 days until equilibrium was achieved. After that the bulk phase concentration of the solute was sampled and analyzed. The bulk phase concentration of acetophenone was measured by UV-VIS spectrophotometer (Perkin Elmer, Lambda 10) at the wavelength 245 nm. A simple mass balance was performed by subtracting the initial concentration of solute with the bulk concentration of solute at final or equilibrium to obtain the amount of adsolubilized solute. The experiments were repeated with four different electrolytes (NaCl, MgCl₂, MgSO₄ and Na₂CO₃) and at five different ionic strength values (0.1, 0.5, 1, 5 and 10 mM). It is very important to note that upon changing electrolyte and 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.