CHAPTER III
EXPERIMENTAL

3.1 Material

Talcum powder (meets analytical specification of Ph. Eur., BP) was obtained from Sigma-Aldrich Co.

Three types of surfactants were used in this study. The nonionic surfactant was alcohol ethoxylate (AE7, 99% purity) suppied by Thai Ethoxylate Company Limited. The cationic surfactant was cetyl trimethyl ammonium bromide (CTAB), and the anionic surfactant is sodium dodecyl sulfate (SDS).

For other chemicals, Sodium hydroxide (NaOH, analytical purity grade), Hydrobromic acid (HBr, analytical purity grade) and Nitric Acid (HNO_3, analytical purity grade) were supplied by Lab-Scan Analytical Sciences (BKK, Thailand). Hydrofluoric acid was purchased from Sigma-Aldrich.

3.2 Equipments

1. Quanta Chrome, Autosorb-1MP, BET Instrument
2. Malvern/Mastersizer X, Particle Size Analyzer
3. JEOL 5200-2AE, Scanning Electron Microscope (SEM)
4. Shimadzu, TOC 5000, Total Organic Analyzer (TOC)
5. Zeta-Meter System 3.0
6. DENVER Instrument, pH meter
7. Kruss, DSA 10, Contact Angle Instrument
8. AXIOS PW4400, PANalytical, X-ray Fluorence (XRF)
9. UV-VIS spectrophotometer (Shimadzu, UV-1800)

3.3 Experimental Procedures

3.3.1 Determination of Specific Surface Area

1 g of talcum was degassed at 250°C overnight, their specific surface areas were determined by nitrogen adsorption BET measurement using a surface area analyzer (Quanta Chrome, Autosorb-1MP).

3.3.2 Particle Size Determination

Particle size and particle size distribution of talcum powder was determined by laser technique using particle Size Analyzer (Malvern/Mastersizer X).

3.3.3 X-ray Diffraction Spectroscopy (XRD)

X-ray diffraction (XRD) patterns were obtained by using a Rigaku, Rint X-Ray diffractometer system (RINT 2200) with Cu tube for generating CuKα radiation (1.5406 Å) and nickel filter. XRD was employed to obtain the structure of talcum powder. A talcum powder was packed in glass specimen holder which is placed in the goniometer using CuKα small radiation and operated at 40 KV and 30 mA. This prepared catalyst samples were scanned from 10 degrees to 80 degrees (2θ) with the scanning speed of 0.02 degrees/min. The data from XRD was analyzed and recorded by an on-line computer.

3.3.4 Chemical Analysis

Chemical analysis of talcum powder was measured by X-Ray Fluarescence Spectrometer (XRF).

3.3.5 Surface Morphology

Surface morphology of talcum powder was measured by scanning electron microscope (SEM) (JEOL, LS002) and transmission electron microscope (TEM).

3.3.6 PZC Measurement

A very small amount of talcum powder was added into deionized water having different solution pH by using a 0.1 M HBr or 0.1 M NaOH solution. After that, the solution is then stirred at room temperature for 24 h. The solution was then transferred to an electrophoretic cell of a zeta meter (Zeta Meter 3.0+ unit) equipped with a microscope module. Then apply a suitable voltage according to the solution conductivity. Finally, the average zeta potential value of talcum powder was obtained.

3.3.7 Adsorption Isotherm Experiments

The experiments of surfactant adsorption were carried out in order to find the amount of surfactant adsorbed on talcum powder as a function of surfactant concentration and pH solution.

A surfactant stock solution of alcohol ethoxylate (AE7), cetyl trimethyl ammonium bromide (CTAB), and sodium dodecyl sulfate (SDS) was diluted with distilled water to obtain various surfactant concentrations and added to screw cap vials containing 0.3 g of talcum powders and initial pH was adjusted at different values by adding 1 M NaOH or 1 M HBr solution. The filled vials were allowed to equilibrate at 25°C for 5 days to achieve steady state. After equilibrated, the supernatants were filtered using a Nylon syringe filter with 0.45 μm pore size. The supernatants were analyzed for bulk phase concentrations of surfactant by using a total organic analyzer (TOC) (Shimadzu, TOC 5000). All experiments were conducted with three replicates at 25°C and the obtained data are averaged to calculate the amount of surfactant adsorbed by using the concentration difference method.

3.3.8 Zeta Potential Measurements

An amount of 3 mg of talcum powder was added into a surfactant solution with various surfactant concentrations at 0.2CMC, CMC and 2CMC which have different pH values at 5, 7 and 9. After that, the solution is then stirred at room temperature for 24 h. The solution was then transferred to an electrophoretic cell of a zeta meter (Zeta Meter 3.0+ unit) equipped with a microscope module. Then apply a

suitable voltage according to the solution conductivity. Finally, the average zeta potential value of talcum powder was obtained.

3.3.9 Dispersion Stability Measurements

The dispersion stability of talcum powder in the presence and absence of studied surfactant was investigated using a UV-VIS spectrophotometer (Shimadzu, UV-1800). First, the talcum powder in surfactant solution was scanned wavelength, which light wavelength was found to not significantly affect the light absorbance for talcum powder and surfactants. In this study, the wavelength was fixed at 550 nm (visible light region). The dispersed talcum powder systems were prepared at 0.1 g of talcum powder in the presence and absence of studied surfactant with the pH of 5, 7 and 9. The solution pH was adjusted by adding either a 1 M NaOH or 1 M HBr solution. The studied surfactants with various surfactant concentrations at 0.2CMC, CMC and 2CMC are alcohol ethoxylate (AE7), cetyl trimethyl ammonium bromide (CTAB), and sodium dodecyl sulfate (SDS). After being well mixed, the prepared solution was transferred to a cuvette of the spectrophotometer and the solution without talcum powder for each system was transferred to the other cuvette as a blank. Then the absorbance (at the position of ¾ from the top or ¼ of the way from the bottom of the cuvette) was measured as a function of time at room temperature until 12,000 sec. A high absorbance indicates high dispersion stability and a low absorbance indicates low dispersion stability.

3.3.10 Contact Angle Measurement

The contact angle measurement was carried out using the sessile drop technique by a contact angle measuring instrument (Kruss, DSA 10). The talcum powder was first compressed into smooth sheet by using a compression molding. A 10 μL of a surfactant solution containing different surfactant concentrations was placed onto the talcum powder surface and then the contact angle was measured after 20 seconds to ensure a maximum surfactant adsorption onto the solution/surface interface.