

CHAPTER III

EXPERIMENTAL SECTION

3.1 Materials

3.1.1 Preparation of Quartz

Natural high purity quartz (SiO_2 , 99.8%) purchased from Cernic International Co., Ltd. was used in the study. The specification of the ground quartz particles size were less than 300 mesh fraction. The ground quartz particles were leached with a hot concentrated hydrochloric acid (HCl) solution (37.5 % by wt.) to remove iron contaminant until no discoloration of the acid by dissolved iron was observed. Finally, the ground quartz particles were washed with triply-distilled water to remove chloride ions until the pH remained constant, was the pH of distilled water, about 6.2 ± 0.05 and with specific conductance of $3.0 \times 10^{-6} \text{ ohm}^{-1} \text{ cm}^{-1}$ was detected. The distilled water used was purchased from the Thai Pharmaceutical Organization. The purified ground quartz was stored in a glass container under triply-distilled water until used in the experiments.

The suspension of ground quartz/water system was filtered and dried at 80°C about 72 hours for all experiments. The average diameter of the quartz particles was measured by a Mastersizer X Ver. 2.15 (Malvern Instruments Ltd.). The average mean diameter value of the ground quartz was $28.29 \mu\text{m}$ and was calculated from the five measurement. The average specific surface area of the studied quartz was determined by the BET technique using a Quantasorb system with nitrogen adsorption. Six samples of

quartz were taken for measurement; the average specific surface area was 4.21 m²/g.

3.1.2 Surfactant

Reagent grade (98% purity) dodecyltrimethylammonium bromide (DTAB, C₁₂H₂₅N(CH₃)₃Br) obtained from Fluka Co., Ltd. Switzerland was used as received the collector in this flotation study.

3.1.3 Quartz Suspension Solution

All quartz suspensions used in this study were prepared to have a constant quartz loading of 50 ± 0.0050 g/l. The pH of the suspension solution was kept constant at pH 6.2 ± 0.05.

Initially the ground quartz was mixed with 50 ml of triply-distilled water and stirred with a magnetic stirrer at 500 rpm for 10 minutes. 50 ml of DTAB solution, with different concentrations, was then added. In this system, final DTAB concentrations were varied from 0.1 to 100,000 µmol/l. The pH was then adjusted to 6.2 ± 0.05 with sodium hydroxide (NaOH) or HCl solution at room temperature (26 °C). Reagent grade NaOH and HCl purchased from J.T. Backer Inc. were used. The aqueous solution of NaOH and HCl were prepared at concentration 0.012 mol/l.

After adjusting the pH, the suspension solutions were stirred with a magnetic stirrer 20 minutes and then wait for 20 minutes to surfactant adsorption equilibrium. These suspension solutions were ready the experimental studies.

3.2 Experimental Procedures

3.2.1 CMC Determination

Surface tension measurement was employed to determine the CMC of DTAB in aqueous solution at the pH of 6.2 ± 0.05 at room temperature ($26\text{ }^{\circ}\text{C}$). In this study, the surface tension was measured by using a tensiometer with the Du Noüy method (Digital Tensiometer K 10 ST, KRÜSS Instrument). The measuring ring was the platinum-iridium type having wetting length of 119.95 mm, ring-radius of 9.545 mm, and wire radius of 0.185 mm. Before measurement, the digital display and balance beam of tensiometer were adjusted to zero every time. The accuracy of the tensiometer was checked by triply-distilled water before use.

The DTAB solutions with concentrations ranging from 1.0×10^{-4} to 0.1 mol/l were adjusted to 6.2 ± 0.05 under room temperature ($26\text{ }^{\circ}\text{C}$) by adding NaOH or HCl solution. Each concentration was measured three times.

3.2.2 Adsorption Isotherms Experiment

This experiment was carried out in order to find the amount of surfactant that was adsorbed on the solid surface as a function of surfactant concentration. This is known as the adsorption isotherm. The amount of surfactant adsorbed was calculated by performing a mass balance using the initial and final concentrations of the surfactant. 100 ml of the suspension solution containing 50 g quartz and different concentrations of surfactant in the range of 0.1 to 100,000 $\mu\text{mol/l}$ was prepared. Then prepared, followed 3.1.3. The supernatant portion above the ground quartz was clarified by using a centrifuge at 2500 rpm for 10 minutes. The centrate for was filtered by using a

0.45 μm nylon66 membrane to remove all the suspended solids. This filtered concentrate was kept in a sealed 25 ml vial for further analysis.

The equilibrium concentration of surfactant was determined by Hewlett Packard series 1050 High Performance Liquid Chromatography (HPLC) with a conductivity detector (model 550 Alltech Associates, Inc.) The conductivity detector was set up at condition of positive signal and temperature of 40 $^{\circ}\text{C}$. The sensitivity of the detector for high surfactant concentrations was adjusted to about 500 μS and for low surfactant concentrations was about 100 μS . The HPLC column was a Nova-Pak phenyl reverse-phase column (Waters Division of Millipore, Millipore Corporation, Milford, MA). The mobile phase solvent was a mixture of 80 % HPLC grade methanol (Lab-Scan Asia, Ltd.) and 20 % triply-distilled water. The mobile phase solvent was filtered by using a 0.45 μm nylon 66 membrane to remove all foreign suspended solids. The flowrate of the mobile phase was controlled at 1 ml/min. Each concentrate sample was analyzed 4 times.

3.2.3 Electrophoretic Mobility Measurement

The electrophoretic mobility of charged quartz particles in aqueous solution containing DTAB at different concentrations were determined by using a Zeta Potential Meter (Zeta Meter System 3.0). The electrophoretic mobility is the moving speed of a colloidal particle under an electric field and has a unit of microns-cm/volt-seconds. The electrophoretic mobility was measured by observing the movement of the particle quartz in horizontal direction under applied voltage. Under microscope observation, the time required for the particle moving for a fixed distance was measured. The operating unit has a microprocessor for calculating the electrophoretic mobility based on the timing and distance over which the particle was being tracked and at the applied voltage.

The specific conductance of quartz particle that didn't have the movement of particle was also measured by the zeta potential meter. This measured value of the specific conductance was used to set a proper voltage for applying to measure value of electrophoretic mobility or zeta potential. For solutions with low specific conductance, a high voltage was required. In contrast, when the value of specific conductance increased, an applied voltage was lowered. Distance was set at full scale divisions of the micrometer. If a particle moved very slowly, the half or fourth scale divisions of the micrometer was set for measuring distance.

The GT-2 electrophoresis cell had molybdenum and platinum anode and a platinum cathode. The molybdenum anode was used when the system had high specific conductance (more than 1,000 micro-ohms) and the platinum anode was used for low specific conductance (less than 1,000 micro-ohms). The electrodes were cleaned with triply-distilled and dried before and after measurement. Before measurement the electrophoresis cell was cleaned with triply-distilled water three times and rinsed with each time about five milliliters of the solution flushed through the cell two times. The well mixed suspension was then poured into the electrophoresis sample cell. For each sample, the zeta potential was measured about 10 values, in order to take an average arithmetic mean of the zeta potential with small standard deviation.

3.2.4 Flotation Experiments

This experiment was carried out to determine the effect of DTAB concentration on flotation efficiency. The flotation efficiency was calculated from the percent ratio of weight of quartz floated and weight of initial quartz. 100 ml of the suspension solution having 50 g quartz/l and different concentrations of surfactant from 0.1 to 100,000 $\mu\text{mol/l}$, was loaded into the flotation cell. A schematic of the cell is shown in Figure 3.1. The flotation cell

used in this experiment looks quite similar to Hallimonnd tube (Fuertenau *et al.*, 1957).

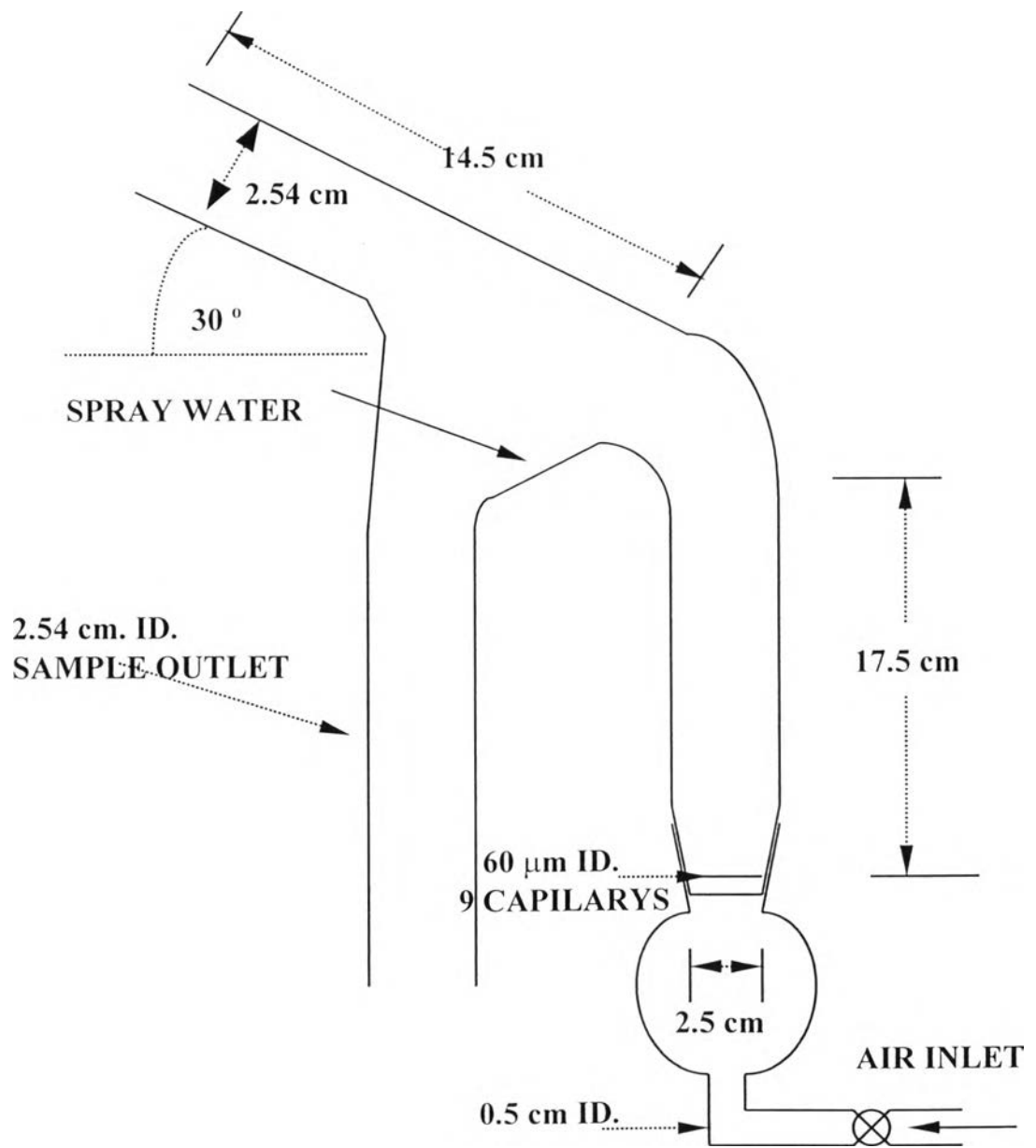


Figure 3.1 Flotation cell used in this experimental study.

It was necessary to shake well to ensure the suspension solution sample would have uniform concentration before pouring into the flotation cell. The suspension solution was aerated with air (zero grade supplied by Praxair Co., Ltd.) at different flowrates of 5.7, 6.5, and 8.4 ml/s. The air flowrate was calibrated by a soap film flowmeter. The flotation experiment were performed for each sample 90 seconds for each sample. During foam overflow, triply-distilled water was sprayed to wash out the flowing foam at the sample outlet of the flotation cell, since the foam tended to break and then retend in the column as a result of the quartz particles. The washed overflow foam was collected in a beaker. The foam collected was mixed with 50 ml of triply-distilled water about 5 times and then filtered with filter paper (number 2, Whatman International, Ltd.). The filtered quartz was washed with distilled water about 5 times to ensure no surfactant adsorbed on the quartz particles. The filtered quartz was then dried at 80 °C for 72 hours. The weight of the dried quartz was then measured.

3.2.5 Measurement of Aggregate Size

The sizes of quartz aggregates formed at different DTAB concentrations were measured by a LSM 410 Inverse Laser-Scan Microscope System (ZEISS instrument Co., Ltd.). 20 ml of the sample was prepared as describe in 3.1.3. The sample without surfactant at pH 6.2 ± 0.05 under room temperature was prepared into measure for background. At each concentration, the sample was poured into 25 ml vial. It was shacken by hand 20 times to create a homogeneous suspension. Then, a sample dropped on a clear glass plate. The size of clear glass plate was 25.4 x 76.2 mm, 1-1.2 mm thickness. The sample was then closed with cover glass 25.4 x 25.4 mm, 0.5 mm thickness. The specimen was inverted and placed on the microscope stage. Lens 20 x 5 were selected for using. The position area for measurement was selected

by watching at the eyepiece of microscope. The specimen was put into position for transmitted light. Then, the sample was measured and results shown in a monitor. The figure on the monitor was then printed. For each size of quartz aggregated, the area of the quartz aggregates was found by measured distance, as compared with a reference scale, around its boundary. The area was calculated and converted to a diameter as if the area was equal to the circle area.