CHAPTER III
EXPERIMENTAL SECTION

3.1 Materials

1) Surfactant

Sodium dodecyl sulfate or SDS obtained from Sigma Chemical Co., with minimum 99% purity, was used as the anionic surfactant without further purification.

2) Fatty acid

Lauric acid with 98% purity, stearic acid with 95% purity, and myristic acid with 95% purity were received from Aldrich Chemical Company, Inc. Behenic acid with 85% purity was obtained from Sigma Chemical Co.

These fatty acids were used for preparing calcium soaps in this study.

3) Calcium chloride dihydrate

Analytical reagent grade calcium chloride dihydrate was obtained from J.T. Baker Chemicals B.V.(Deventerr, Holland). Calcium chloride dihydrate has two important roles in this study. First, it was used in the precipitation of the calcium soaps. Second, calcium chloride was used to represent the hardness tolerance in the foam testing.

4) Methanol

Methanol with 100% high-performance liquid chromatography (HPLC)-grade from Merck was used as the solvent for fatty acids in calcium soap precipitation.

5) Sodium carbonate and sodium hydrogen carbonate

Sodium hydrogen carbonate with minimum 99.7% purity obtained from Riedel-dettaen was used to study the effect of pH on the foaming properties of SDS.

6) Water

Deionized water with pH around 5.5 was used in all experiments.

3.2 Experimental Equipment

3.2.1 Ross-Miles Method Equipment

The equipment for the Ross-Miles method was made in accordance with the ASTM standard D1173-53. The apparatus consists of two parts, the pipette and the receiver. The bulb of the pipette has 45±1.5 mm outside diameter and its ends are hemispherical. The upper part of the bulb is connected to a stem ending with a stopcock. The lower part of the bulb is connected to another stem of 7±0.5 mm outside diameter and length 60±2 mm. At its lower end is fitted an orifice of 2.9±0.02 mm inside diameter and a length of 10±0.05 mm constructed from precision bore tube with ends ground square. This orifice is sealed to the stem. The pipette is calibrated to contain 200±0.2 ml at 20°C. The pipette is shown in Figure 3.1.

The receiver as shown in Figure 3.2 is a jacketed tube of 50 mm internal diameter. The external diameter of the jacket is 70 mm. The lower end of the receiver has a stopcock to drain the liquid. There are three marks on the receiver, one at the 50 ml point measured with the stopcock closed and is at the cylindrical part of the tube.

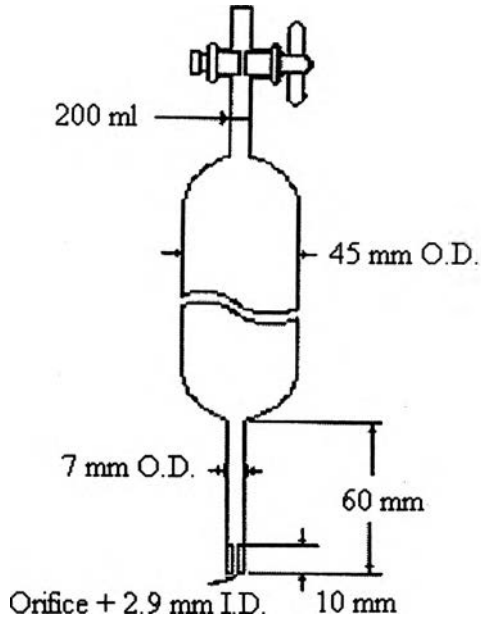

Figure 3.1 The Ross-Miles pipette.

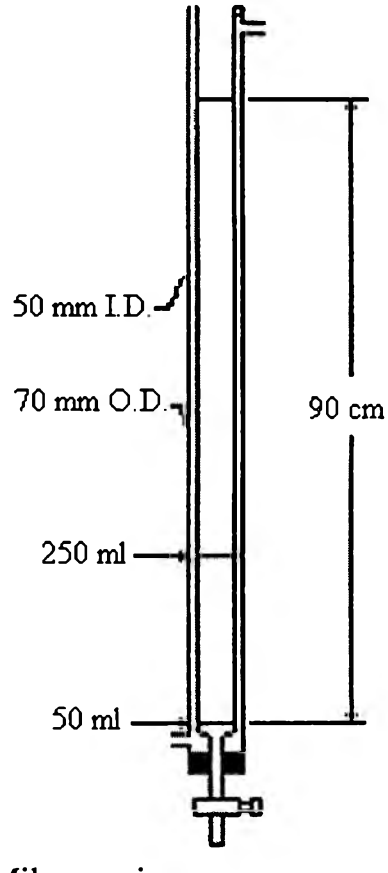

Figure 3.2 The Ross-Miles receiver.

The second mark is at the 250 ml point and the third is at 900 mm above the 50 ml mark. Figure 3.3 shows the mounting of the pipette on top of the receiver when in use.

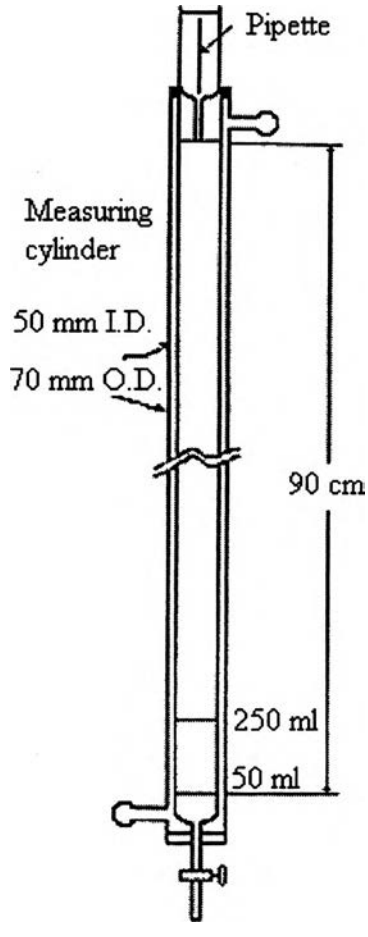

Figure 3.3 Schematic of equipment for Ross-Miles foam test.

3.3 Experimental Methods

3.3.1 Preparation of Calcium Soap Precipitate

Calcium salts of C_{12}, C_{14}, C_{18}, and C_{22} fatty acids were prepared by dissolving each type of these fatty acids in hot methanol until clear solution was obtained. Then, calcium chloride solution with 20% excess was added and

the solution was cooled down to 10°C to precipitate out the calcium soap. Methanol and the rest of calcium chloride were removed by vacuum suction and washed with distilled water. Next, calcium soaps of fatty acids C_{12}, C_{14}, C_{18}, and C_{22} were dried in the oven at 45, 50, 55, and 60°C respectively for 24 hours. Before use, all calcium soaps were stored in the desiccator to get rid of the humidity.

3.3.2 Contact Angle Measurement

Calcium soap pellets were prepared by grinding the soap particles by mortar until fine particle was obtained. 0.3 g of calcium soap was placed in stainless steel pinch and die (13 mm diameter) and pressed at 8,000 kg using a hydraulic press (BIO-RAD, P/N 15011) with 3 minutes dwell time.

Sessile-drop contact angle technique (Garrett et al., 1993) was used in this study. In this experiment, the temperature was kept constant at 30°C. The SDS solution with varying concentration was dropped on the calcium soap pellet and the picture was captured at 15 seconds by a camera. The contact angle at the air-liquid-solid interface was determined by using the Photoshop program. The example of contact angle measurement in this study was shown in Figure 3.4.

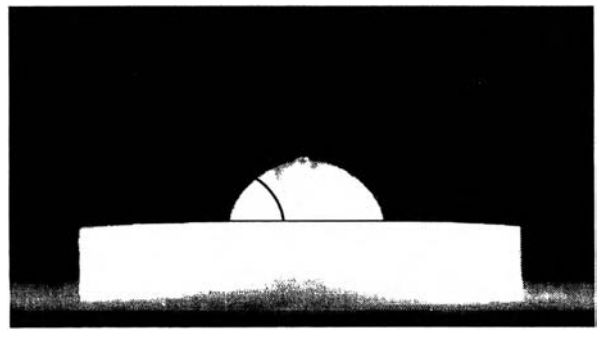

Figure 3.4 The contact angle of water on the calcium soap C_{12} pellet.

3.3.3 Foam Measurement by Ross-Miles Method

All experiments were performed at a constant temperature of 30°C. The solutions for these experiments were prepared by mixing the SDS solution with calcium soap by using magnetic stirrer for 12 hours and the solution was then kept in a water bath at 30°C for 1 hours to reach equilibrium before testing.

Ross-Miles method involved filling a pipette with a 200 ml of the test solution; the solution was allowed to fall a specified distance into a 50 ml of the same solution which was contained in a column. When all the solution was run out of the pipette, a reading of foam height taken immediately was used to represent the foamability. On the other hand, foam stability was determined by measuring the foam height after a certain time.

In this experiment, it can be divided into 5 parts.

- Foaming properties of SDS solution in the presence of calcium soaps

In this part, it was used to verify the antifoaming mechanism of calcium soap precipitate in the SDS solution. The concentration of SDS was 10 mM which is above its CMC. Calcium soap concentration used was 0.5 mM which is above the solubility of soap as evidenced by the presence of soap precipitate in the solution. The pH of test solution was constant at 7.

- The effect of particle size of calcium soap on the foaming properties of SDS solution

In this part, the particle size of calcium soap used was in the range of 212-425 μm and less than 212 μm. The concentrations of SDS and calcium soap were constant at 10 mM and 0.5 mM, respectively. The pH of test solution was controlled at 7.

- The effect of concentration of calcium soap on the foaming properties of SDS solution

In this case, CaC_{14} and CaC_{22} were used to study the effect of their concentrations on the foaming properties of SDS. The concentration of SDS was 10 mM. The concentrations of calcium soap used were 0.5 and 2 mM. The pH of the test solution was not controlled in this experiment.

- The effect of hardness tolerance on the foaming properties of SDS solution in the presence of calcium soaps

In this experiment, the effect of anionic precipitate on the foaming properties of SDS was studied. The concentrations of SDS and calcium soap used were 10 and 0.5 mM, respectively. In this case, $CaCl_2$ (calcium ion) was used to represent the hardness and its concentration was varied at 2 and 5 mM.

- The effect of $NaHCO_3$ on the foaming properties of SDS solution in the presence of calcium soaps

In this experiment, the concentrations of SDS and calcium soap used were constant at 10 and 0.5 mM. At the same time, the concentration of $NaHCO_3$ was varied at 50 and 100 ppm.