CHAPTER III
EXPERIMENTAL SECTION

3.1 Materials

1) Surfactant

Sodium dodecyl sulfate (SDS) obtained from Sigma Chemical Co., with 99+% (capillary GC) purity, was used as the anionic surfactant without further purification.

2) Fatty acid

Lauric acid, myristic acid and stearic acid from Unilever Thai Holding Limited were used to prepare calcium soaps in this study.

3) Calcium chloride dihydrate

Analytical grade calcium chloride dihydrate was obtained from J.T. Baker Chemicals B.V.-Deventer-Holland and was used to prepare calcium salt of fatty acid.

4) Water

Distilled water was used in this study. This distilled water had a conductivity of 2μmho/cm.

3.2 Experimental Methods

3.2.1 Preparation of Calcium Soap Precipitate

Calcium salts of C12, C14 and C18 fatty acid were prepared by dissolving the fatty acid in methanol HPLC grade and filtering through a fritted glass filter to remove the impurity. Calcium chloride solution was then added and the solution cooled down to 10^0C to precipitate out Ca-SO. Methanol and excess calcium chloride were removed by vacuum suction and washed with distilled water before drying in the oven at about 60^0C for 24 hours.

Calcium salts of C8 was prepared by dissolving sodium caprylate in distilled water and calcium salt was precipitated by adding calcium chloride solution.

3.2.2 Foam Measurements

All experiments in this study were performed at a constant temperature of 30^0C and constant pH of 7. Sodium hydroxide and hydrochloric acid were used to adjust the pH of the solution. A pH meter (Benchtop pH/ISE Meter, Model 420A with Triode pH electrode Model 91-578N) was used for pH measurements.

3.2.2.1 Ross-Miles Method (ASTMD 1173-53)

Apparatus

The pipette (Figure 3.1) was constructed from glass having the following dimensions: for the bulb, 45± 1.5 mm outside diameter: for the lower stem, 7 ± 0.5 mm outside diameter. The upper stem was constructed to contain a solid stopper, straight bore, No.2, standard taper stopcock having a 2 mm bore and stem with 8 mm outside diameter. The lower stem was 60± 2 mm in length from the point of attachment to the bulb and contained an orifice sealed to the lower end. The orifice was constructed from

precision bore tubing having an inside diameter of 2.9 ± 0.02 mm and a length of 10 ± 0.05 mm with both ends ground square. The pipette was calibrated to contained 200 ± 2 ml at 20°C.

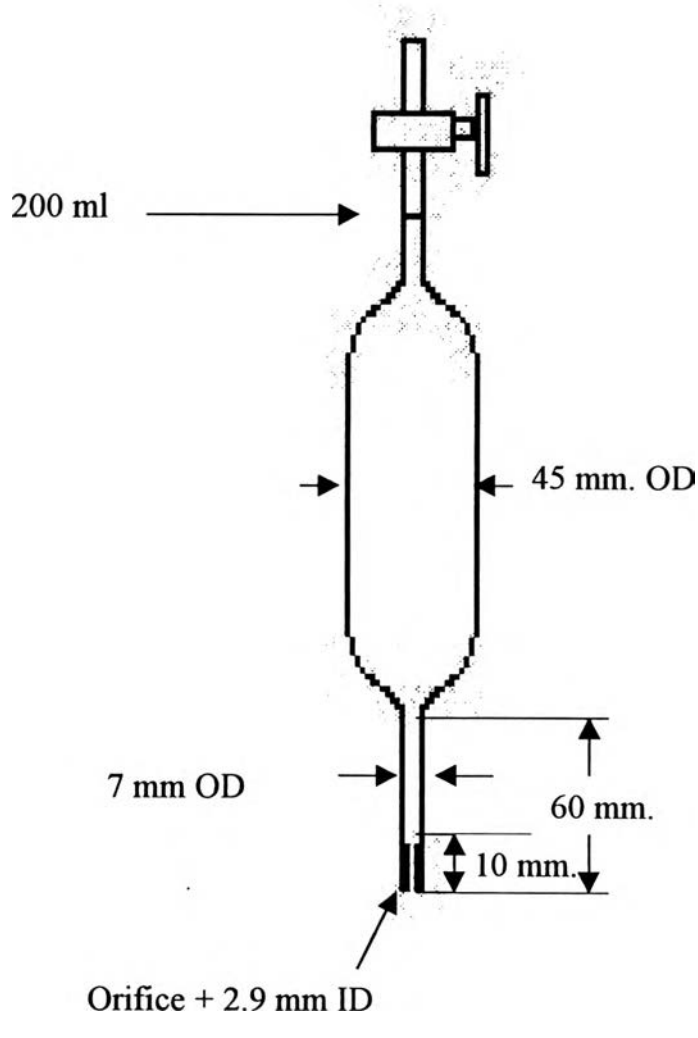

Figure 3.1 Foam pipette for the Ross-Miles test

The receiver (Figure 3.2) was constructed from glass having an internal diameter of 50± 0.8 mm. One end was constricted and sealed to a straight-bore, solid-plug, standard taper No.6, equipped with a stopcock having a 6 mm bore and 12 mm stems. The receiver tube was mounted in a standard-wall tubular water jacket, having an external diameter of not less than 70 mm, fitted with inlet and outlet connections.

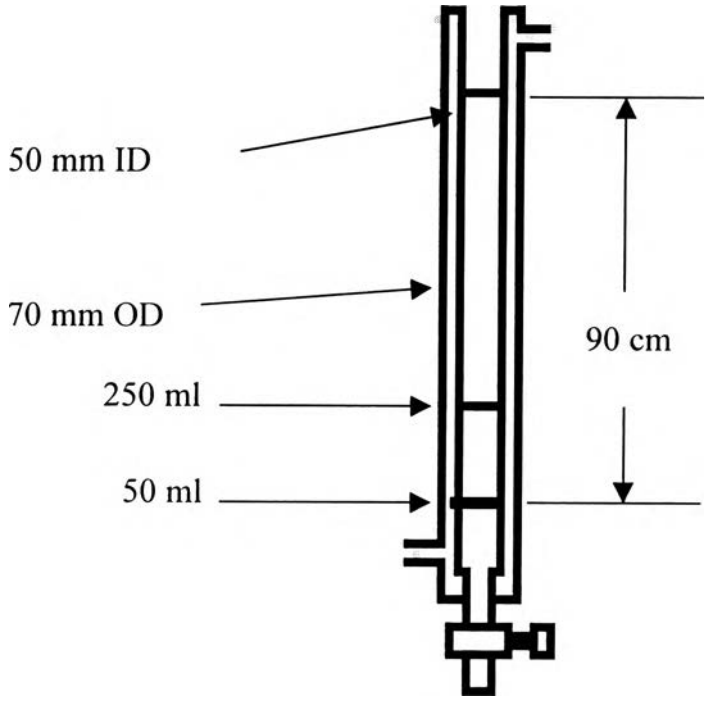

Figure 3.2 Foam receiver for the Ross-Miles test.

Procedure

Water, controlled at 30^{0}C, was circulated through the water jacket of the receiver so as to bring it to the proper temperature. About 50 mL of the solution was run into the receiver using the pipette and the level of the solution in the receiver was adjusted to be exactly at the 50 mL mark. The pipette was the filled with solution to the 200 mL mark, using a slight suction for the purpose. It was placed immediately on top of the receiver and the stopcock was open to run the solution into the receiver. When all the solution was run out of the pipette, a reading of foam height was taken immediately to measure the foamability of the solution. Foam stability was determined by measuring the foam decay after a certain time.

3.2.2.2 Shake Method

25 ml of the test solution was run into a 38x200 mm test tube with a screw cap by using a pipette. The solution was placed in a water bath at 30^{0}C for 1 hour. It was then taken out and shaken by hand in a consistent way 10 times. Initial foam height was measured after 5 second. Foam stability was determined by measuring foam height after a certain time.

3.2.3 Contact Angle Measurement

Ca-SO pellets were prepared by grinding the Ca-SO particles by mortar until fine powder was obtained. It was transferred into stainless steel punch and die (13 mm diameter) and pressed at 10,000 kg force using a hydraulic press (BIO-RAD, P/N 15011) with 2 minutes dwell time. The pellets were stored in 30% relative humidity chamber at ambient temperature prior to use. The contact angle measurement was done by placing Ca-SO pellet inside a glass chamber controlled at 30^{0}C. A drop of 20 μl solution was placed on the solid surface by using micro syringe. The picture of contact angle was captured after 15 sec and 10 minutes by digital camera. The Photo Shop program was used to measure the angle.

3.2.4 CMC Measurement

The CMC was determined by measuring the surface tension as a function of bulk concentration of surfactant. The surface tension was measured by digital tensiometer (KRÜSS Instruments). The ring was obtained from Du Nouy with the following specifications: platinum-iridium type wetting length 119.95 mm, ring-radius 9.6545 mm, and wire radius 0.185 mm. The point at which the surface tension became constant was taken as the CMC of the solution.

3.2.5 Preparation of Supernatant Solution

Supernatant solutions of SDS with Ca-SO were prepared by mixing the SDS solution with Ca-SO by using the magnetic stirrer for 12 hours at 30^0C and removing the Ca-SO particles from the solution by filtering through a fritted glass filter.