

CHAPTER III

EXPERIMENTAL SECTION

3.1 Materials

1) Surfactant

Sodium dodecyl sulfate (SDS) obtained from Henkel Company with a purity greater than 96.5 % was used as the anionic surfactant. It is an anionic surfactant with a negatively charged sulfate head group and an alkyl chain length of twelve carbon atoms. It was purified by recrystallization. The recrystallization process (Perrin, 1988) was done by dissolving SDS in distilled water, then filtered through a fritted glass filter. The filtrate was left overnight in a refrigerator to allow crystals to fall out. The precipitated solution was then filtered through a fritted glass filter. The second step was to reprecipitate SDS by using methanol. The crystals were dissolved in 100% HPLC grade methanol. The methanol was warmed up slightly to enhance the solubility of SDS. This solution was left overnight in the freezer of the refrigerator to introduce crystallization. The SDS crystals were filtered and then dried under vacuum overnight before use.

2) Soap

The sodium salt of caprylic acid(n-octanoic acid) - Sodium caprylate - with the 99+ % (capillary GC) purity, manufactured by Sigma Chemical Co., was used as the soap in this study. It has an alkyl chain length of eight carbon atoms. It was used without further purification.

3) Calcium Chloride Dihydrate

Analytical grade calcium chloride dihydrate was obtained from J.T. Baker Chemicals B.V.-Deventer-Holland and was used without purification.

4) Water

Distilled and deionized water was used throughout this study. This distilled and deionized water had a conductivity of 2 $\mu\text{mho/cm}$.

3.2 Experimental Methods

All experiments in this study were performed at constant temperature of 30°C and constant pH of 7. Sodium hydroxide and hydrochloric acid were used to adjust the pH of the solutions. A pH meter (Benchtop pH/ISE Meter, Model 420A with Triode pH electrode Model 91-578N) was used for pH measurements. This study was separated into 2 parts. The first part was done by the Ross-Miles method (ASTM D1173-53). The second part was done by a new Mixing method.

3.2.1 Ross-Miles Method (ASTM D 1173-53)

Apparatus

Pipette

The pipette (Fig 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 ± 0.2 ml at 20°C .

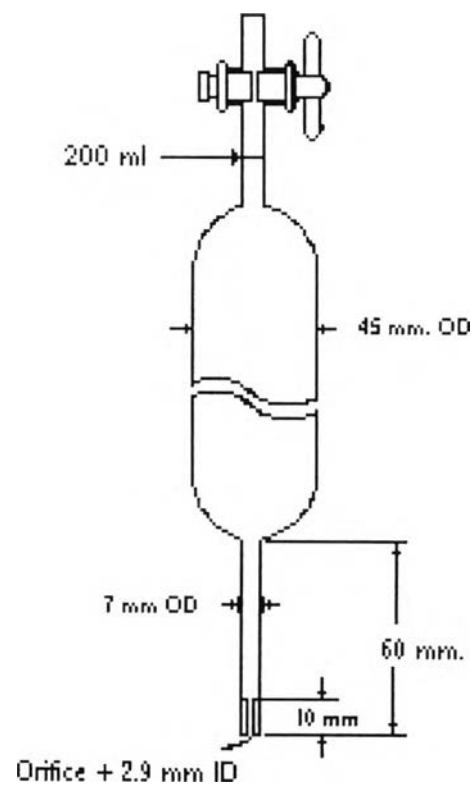


Figure 3.1 Foam Pipette for the Ross-Miles Test.

Receiver

The receiver(Fig.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.

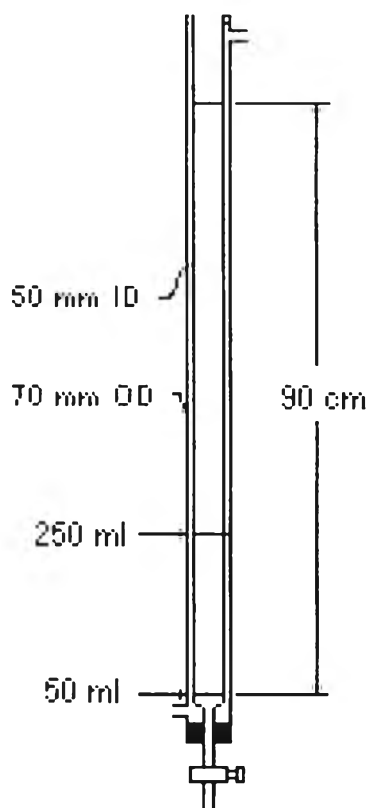


Figure 3.2 Foam Receiver for the Ross-Miles Test.

Procedure

Water, controlled at 30°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 then filled with the 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 and again after 5 minutes.

3.2.2 Mixing Method

Apparatus

The equipment used in this part is shown in Fig. 3.3. The jacket cylindrical foam column consists of two 60 cm long acrylic columns with diameter of 75 mm and 100 mm, and a thickness of 3 mm.

A mechanical stirrer (ALC, CENE-LEC HD 365) with 6 cm three pitched blade was used to stir the solution at 1200 rpm.

Procedure

Water, controlled at 30°C, was circulated in the water jacket of the foam column to bring the temperature inside the column to 30°C. About 350 mL of the test solution preheated to 30°C, was placed inside the column and the level of the solution was adjusted to the 350 mL mark on the foam column. The mechanical stirrer was started to stir the solution for 2.5 minutes. The stirrer was then stopped and the foam height was read off the column immediately and again after 5 minutes.

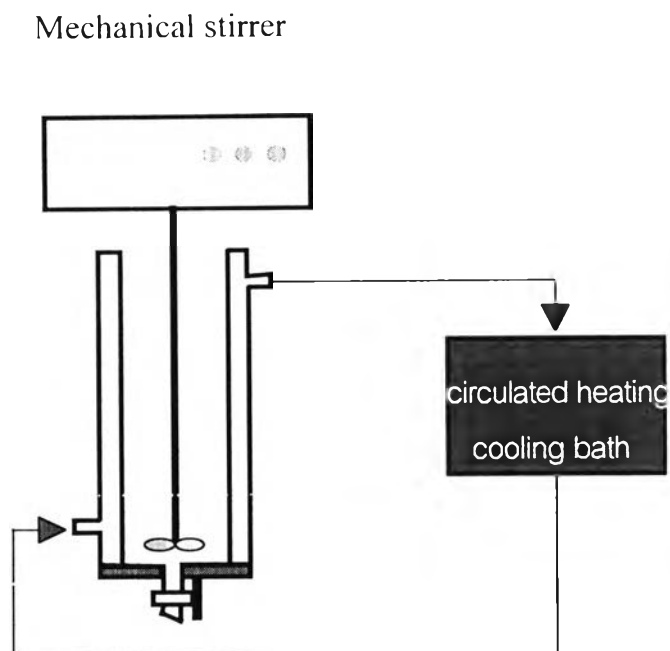


Figure 3.3 Schematic flow diagram of the Mixing method.

3.2.3 Determination of CMC

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 K 10 ST (KRÜSS Instruments). The ring was obtained from Lecompte Du Nouy with the following specifications: platinum-iridium type, wetting length 119.95 mm, ring-radius 9.545 mm, and wire radius 0.185 mm. Before measurement, the digital display and balance beam of tensiometer were adjusted to zero. The accuracy of tensiometer was checked by triple-distilled water before use.

The vessel cell containing the liquid sample was rinsed three times for each experiment. The sample was loaded into vessel and the ring was dipped into sample. The pointer of balance beam was moved to negative value. Then, the instrument was switched on. When the pointer of balance beam

returned to zero again, the value of surface tension was shown on the instrument. Each experiment was repeated five times with the same sample.

Finally, the surface tension (mN/m) was plotted as a function of bulk concentration and the point at which the slope suddenly changed was taken as the CMC.