

CHAPTER III

EXPERIMENTAL SECTION

3.1 Materials

3.1.1 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 was done by dissolving SDS in distilled water, then filtered through a fritted glass filter. The precipitated solution 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 overnight before use.

3.1.2 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 as received.

3.1.3 Calcium chloride dihydrate

Analytical grade calcium chloride dihydrate was obtained from J.T. Baker Chemicals B.V.-Deventer-Holland and was used as received.

3.1.4 Water

Distilled and deionized water with a conductivity of 2 μ mho/cm was used.

3.2 Experimental Methods

All experiments in this study were performed at a constant temperature of 30 °C and a 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. All the foaming tests were done by the Ross-Miles method (ASTM D1173-53). Foamability was taken as the foam height at time = 0 min. and foam stability was measured by the foam height at time = 5 min. The foam stability was also represented by the “stability index” defined as the ratio of foam height at time = 5 min. to foam height at time = 0 min. It has values ranging from 0 to 1. Whatman ashless filter papers (No. 40) were used to filter out precipitates in supernatant experiments.

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

The pipette (Figure 3.1) was constructed from glass having the following dimension: 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 200 ± 0.2 ml at 30°C

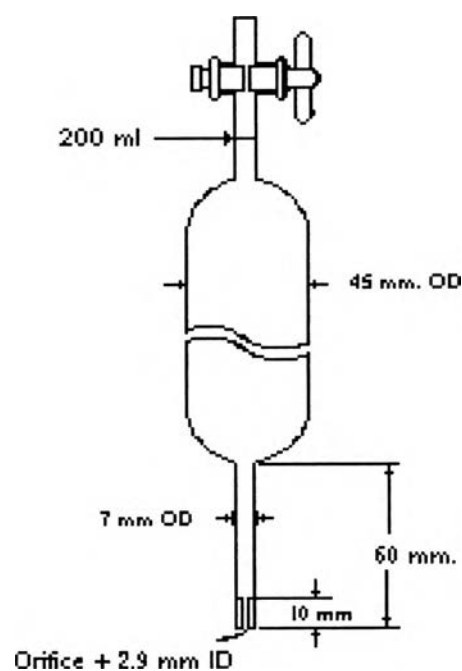


Figure 3.1 The Ross-Miles pipette

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.

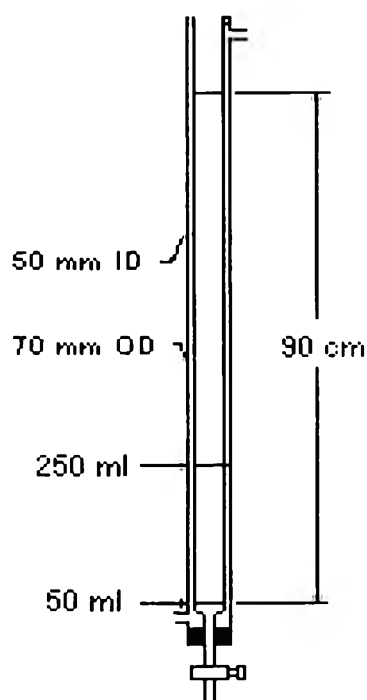


Figure 3.2 The Ross-Miles receiver

3.2.2 Experimental 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 opened 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.

3.2.3 Supernatant experiments

To prepare supernatant solutions, the surfactant mixture was allowed to equilibrate at 30 °C for 12 hours at pH 7. The precipitates were then filtered out and the foaming of the supernatant clear solution was measured by the Ross-Miles method.