CHAPTER III
EXPERIMENTAL

3.1 Materials

3.1.1 Nonionic Surfactants

Nonyl phenoxypoly(ethyleneoxy)ethanol with an average of 8, 9, and 10 moles of ethylene oxide per mole of nonyl phenol, $NP(EO)_8$, $NP(EO)_9$, and $NP(EO)_{10}$ from Rhodia (Thailand) Ltd. (Igepal CO-610, Igepal CO-630, and Igepal CO-660).

3.1.2 Anionic Surfactant

Sodium dodecyl sulfate (SDS) obtained from Sigma Chemical Co., with a purity greater than 99%. It is an anionic surfactant with a negatively charge sulfate head group and an alkyl chain length of twelve carbon atoms.

3.1.3 Sodium Chloride

Analytical grade sodium chloride was obtained from AJAX Chemicals and was used without purification.

3.1.4 Water

Distilled water was used throughout this study.

3.2 Experimental Equipment

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

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. 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.

3.3 Experimental Methods

3.3.1 Cloud Point Determination (ASTM D 2024-65)

To determine the cloud point temperature, nonionic surfactant solutions and mixed nonionic/anionic surfactant solutions with salt were prepared at the desired concentration. The solution was agitated until the surfactant was completely dissolved and 50±5 mL of the solution was

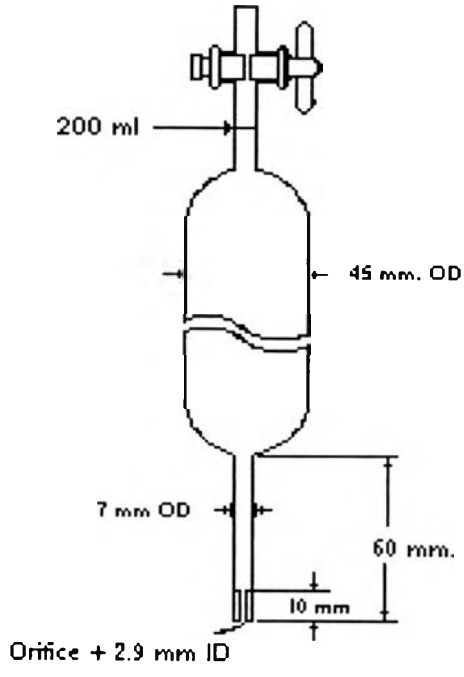

Figure 3.1 The Ross-Miles pipette.

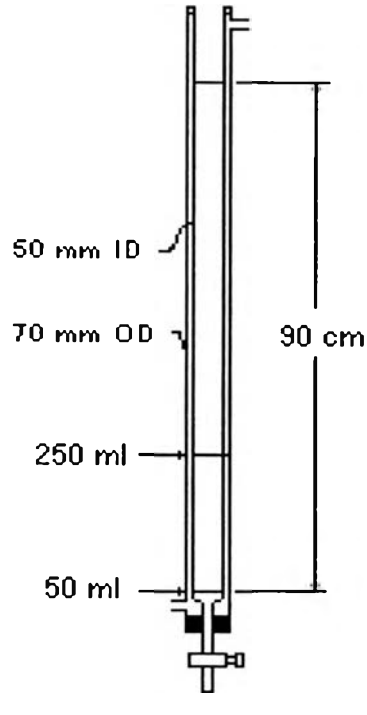

Figure 3.2 The Ross-Miles receiver.

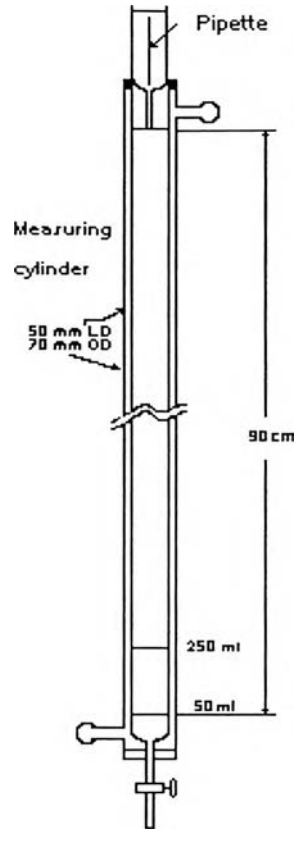

Figure 3.3 Schematic of equipment for Ross-Miles foam test.

transferred to a 25x200 mm test tube. The solution was then heated in a water bath until the solution became cloudy by stirring occasionally with a stirring rod. The solution was then removed from heat and stirring was given occasionally until the solution was again clear. The temperature at which the solution became clear was recorded to the nearest 1°C. This temperature was taken as the cloud point of the solution.

3.3.2 Ross-Miles Method

Preheating the water to be used to the desired temperature and adjusting the thermostat of the circulation bath so as to bring the water jacket of the receiver carried out this method. Then the pipette was mounted by using a clamp to ensure that the axes of the measuring receiver and the pipette must be in line with the upper calibration mark on the measuring receiver. A meter ruler was fastened behind the measuring receiver ensuring that its zero point coincided with the 250 mL calibration mark on the measuring receiver. The walls of the receiver was then rinsed with the test solution using approximately 50 mL solution. When the liquid had drained to the bottom of the receiver, the stopcock was adjusted so that the level of the solution was exactly at the 50 mL mark in the receiver. The pipette was then filled with the test solution up to the 200 mL calibration mark by applying a sight suction. It was immediately placed in position on top of the receiver and the solution was allowed to flow into the receiver until the solution was run out. The foam height was immediately measured at this moment and it was measured again after the flow had been stopped at 5, 10, 15, and 20 minutes respectively.

3.3.3 Shake Test Method

This method was carried out by shaking 25 mL of sample in a 38x200 mm test tube with screw cap. The temperature of the samples was adjusted in a temperature-controlled water bath from which the samples were removed only during the time of shaking. The test tube was gently shakenby hand 10 times as uniformly as possible, and the foam height was immediately measured at this moment and it was measured again at 5, 10, and 15 minutes respectively. All experiments were performed at least five times, and the average of the measurements was used for evaluations.

3.3.4 Surface Tension Determination (ASTM D 1331-89)

The ring of Digital Tensiometer K 10 ST (KRUSS Instrument) was obtained from Lecompte Du Nouy. The ring specifications are as follows: platinum-irridium type, wetting length 119.95 mm, ring-radius 9545 mm, and wire radius 0.185 mm. Before measurement, using cleaning solution, followed by a thorough rinsing with distilled water cleaned all glass materials. The platinum ring was rinsed by distilled water and then heated to white heat in the oxidizing portion of a gas flame of an alcohol burner. The accuracy of tensiometer was checked by triple-distilled water before use.

The digital display and balance beam of tensiometer were adjusted to zero. The vessel cell and the liquid sample were preheated at the desired temperature. The cleaned vessel cell containing the liquid sample was rinsed three times for each experiment. The sample was loaded into the vessel and the ring was dipped into sample. The pointer of balance beam was moved to a negative values. The temperature of the liquid sample was checked again. If the sample temperature equaled the desired temperature, then the instrument was switch on. When the pointer of the balance beam returned to zero again, the value of surface tension was shown on the instrument. Each experiment was repeated six times with the same sample, and the surface tension value was accepted when it reach steady state.