CHAPTER III
EXPERIMENTAL

3.1 Materials

3.1.1 Surfactant

Dehydol LS3, LS5, LS7, and LS9 linear alcohol ethoxylates (Nonionic surfactants) or $C_{12-14}EO_3$, $C_{12-14}EO_5$, $C_{12-14}EO_7$, and $C_{12-14}EO_9$ subsequently with purity 99% were used in this work. They were supplied by Cognis (Thailand) Co., Ltd. NPE-6 and NPE-9 (Nonylphenol ethoxylates) were also used in this study, which were purchased from Dow Chemical Co., Ltd. with a purity of 99%. The general properties of the studied surfactant are summarized in Table 3.1.

Table 3.1 General properties of studied surfactants

Surfactant	Molecular weight	Chemical formula
Dehydol LS 3	318.75	$C_{12-14}(CH_2CH_2O)_3OH$
Dehydol LS 5	403.60	$C_{12-14}(CH_2CH_2O)_5OH$
Dehydol LS 7	496.46	$C_{12-14}(CH_2CH_2O)_7OH$
Dehydol LS 9	578.35	$C_{12-14}(CH_2CH_2O)_9OH$
NPE-6	487	$C_9H_{19}C_6H_4(OCH_2CH_2)_6OH$
NPE-9	~616	$C_9H_{19}C_6H_4(OCH_2CH_2)_9OH$

3.2 Experimental Procedures

In this work, the properties of fatty alcohol ethoxylates—$C_{12-14}EO_3$, $C_{12-14}EO_5$, $C_{12-14}EO_7$, and $C_{12-14}EO_9$—were studied and the results were compared to those from nonylphenol ethoxylates—NPE-6 and NPE-9. The CMC, cloud point, contact angle, and gel range, were studied. The foam characteristics were determined in terms of foamability and foam stability by using the simple shaking test, the Ross–Miles foam test, and pneumatic foam test.

3.2.1 Critical Micelle Concentration (CMC)

The aqueous surfactant solutions were diluted by distilled water at different concentrations. Surface tension versus concentration was measured by using the tensiometer (Krüss Easy Dyne). During the measurement, the chamber was kept at 25°C. The surface tension as a function of the log of surfactant concentration was constructed for each aqueous surfactant solution. The best linear fit to the descending and horizontal lines of the plot was obtained, and their intersection is used to calculate the CMC.

3.2.2 Cloud Point Temperature

The cloud point measurement was modified from the standard test method D 2024-65. The aqueous surfactant solution prepared at concentration of 1.0 %. The test solution was poured into test tube and agitated slowly with the thermometer and then the test tube was heated in the water bath until the test solution becomes definitely cloudy. After that the test tube solution was allowed to cool slowly until it becomes clear, and then the temperature was recorded.

3.2.3 Contact Angle Measurement

Surfactants are often used as wetting agent and, in this case two aspects are important: the spreading rate of solution on the solid surface and the equilibrium contact angle. However, this test was concerned only with the equilibrium aspect. The contact angle was measured by the sessile drop technique with the drop shape analysis instrument (Krüss, DSA10). A 10 μL drop of test solution was then placed onto the studied surfaces which consist of nonpolar parafilm, HDPE (High Density Polyethylene), and nylon6, 6. The contact angle was measured after 1 minute in order to allow equilibrium to occur. During the measurement, the chamber was kept at 30°C and saturated with water vapour to prevent drop evaporation effect.

3.2.4 Foam Characteristics Experiments

3.2.4.1 Simple Shaking Foam Test

The aqueous surfactant solutions were prepared at studied concentration. The simple shaking test was modified from the method suggested by Piispanen et al. A quantity of 40 mL of test solution containing different surfactant concentrations was poured into a 100-mL graduated cylinder. The cylinder was turned up–side down for 10 times at a rate of 1 turn per 2 seconds. The foam heights were measured immediately for 60 minutes for indicating the foamability and foam stability, respectively.

3.2.4.2 Ross-Miles Foam Test

The Ross-Miles test was modified from the standard test method D1173-53. A quantity of 200 mL of test solution was poured into a pipette and 50 mL of test solution was also transferred into the bottom of glass column. The pipette was placed on the top of the glass column with 90 cm in height from the surface level of the test solution in the glass column. The foam height was measured immediately after the test solution ran out of the pipette and measured again at 5 minutes. The rate of foam collapse indicate that the stability of foam (foam stability) while, the initial foam height indicate that the power of foam formation (foamability).

3.2.4.3 Pneumatic Foam Test

A quantity of 100 mL of a test solution was transferred to a glass cylindrical column, having inside diameter of 4 cm and a height of 150 cm. Filtered air was introduced through the sintered glass disk which has a pore size of diameter of 16-40 μm. The flow rate of air was kept constant at 20 ml/min by using a mass flow controller. The solution in the column was aerated continuously until the foam height in the column was constant and then the maximum foam height was recorded. After that, the flow of the filtered air was stopped. The time required for the foam volume to collapse by half was recorded. Foamability is defined as the ratio of maximum foam height to initial solution height; whereas foam stability ($t_{1/2}$) is the time required for the foam volume to collapse by half.

3.2.5 Dynamic Surface Tension

The aqueous surfactant solutions were prepared at studied concentrations. The dynamic surface tension of each test solution was measured by using the maximum bubble pressure method. The dynamic surface tension, corresponding to the surface of the air bubbles is found an important property for the foamability. An air was fed into capillary tube to generate the air bubble. During the process, the pressure inside the pressure inside the air bubble was monitored and recorded with time until the burst of the air bubble. The maximum rate of the decrease surface tension was calculated using the dynamic surface tension data. The change of curve in the dynamic surface tension with surface bubble lifetime can be divided to 4 regions that consist of induction, rapid fall, meso–equilibrium, and equilibrium. The first three regions are important in high speed dynamic process such as foaming (Tamura, 1995). In this test, dynamic surface tension measurements were carried out using a bubble pressure tensiometer (Krüss, BP2) and the temperature was maintained at 30°C.

3.2.6 Gel Range Experiments

The aqueous surfactant solutions were prepared at studied concentrations in the range of 5 to 100 wt.%. The concentration of each surfactant was observed by rare eyes and measured the viscosity by Brookfield Viscometer Model DV–III. The procedure was started with choosing the spindle and adjusted properly with the equipment. Setting speed of agitation was also important for each type of surfactants. The Brookfield Viscometer determines viscosity by measuring the force to turn the spindle in the solution at a given rate. The measuring value is given as the viscosity in the centipoises unit. During the measurement, the temperature was maintained at 25°C.

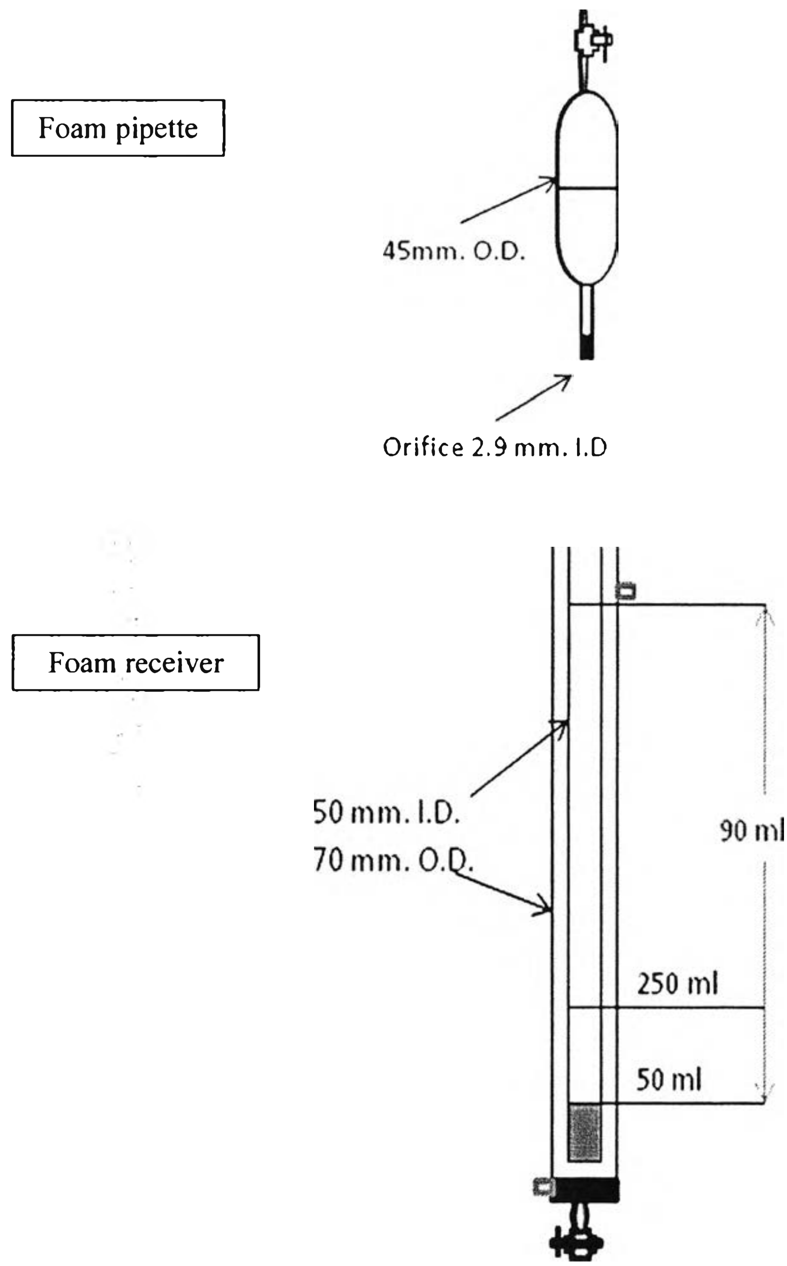

Figure 3.1 Schematic of foam pipette and foam receiver of the Ross–Miles foam apparatus.

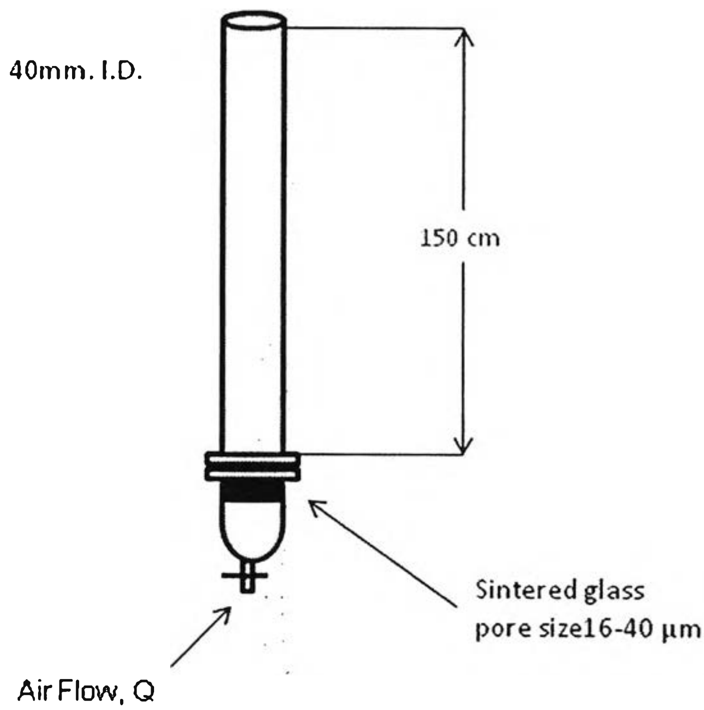

Figure 3.2 Schematic of foam column of the Pneumatic foam test apparatus.