CHAPTER III
EXPERIMENTAL SECTION

3.1 Materials

Nonylphenoxypoly (ethyleneoxy) ethanol with an average of 8, 9, and 10 moles of ethylene oxide per mole of nonylphenol, $NP(EO)_8$, $NP(EO)_9$, and $NP(EO)_{10}$ from Rhone-Poulenc (Thailand) Co., Ltd. (Igepal CO-610, Igepal CO-630 and Igepal CO-660) were the nonionic surfactants used in this study. The water was deionized and distilled.

3.2 Experimental Equipment

3.2.1 Ross-Miles Method Equipment

The equipment for the Ross-Miles method was made in accordance with the ASTM standard D1173-53. The apparatus consists of two parts, the pipette and the receiver. The bulb of the pipette has 45 $\pm$ 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 $\pm$ 0.5 mm outside diameter and length 60 $\pm$ 2 mm. At its lower end is fitted an orifice of 2.9 $\pm$ 0.02 mm inside diameter and a length of 10 $\pm$ 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 $\pm$ 0.2 mL at 20^{o}C. The pipette is shown in Figure 3.1.

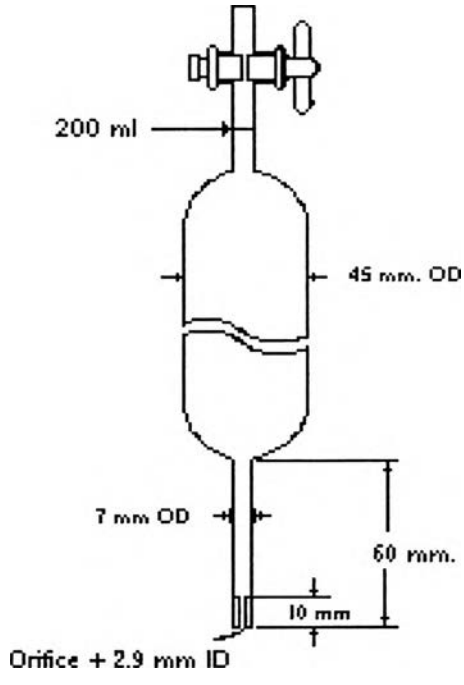

Figure 3.1 The Ross-Miles pipette.

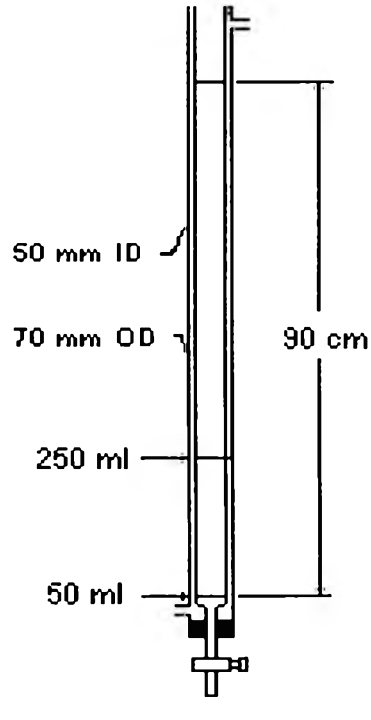

Figure 3.2 The Ross-Miles receiver.

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 250mL 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.

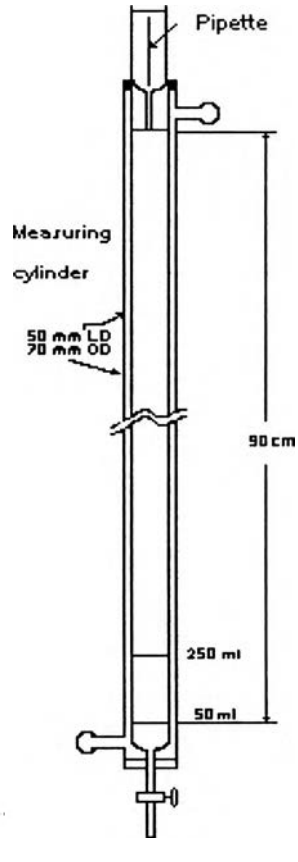

Figure 3.3 Schematic of equipment for Ross-Miles foam test.

3.2.2 Spray Method Equipment

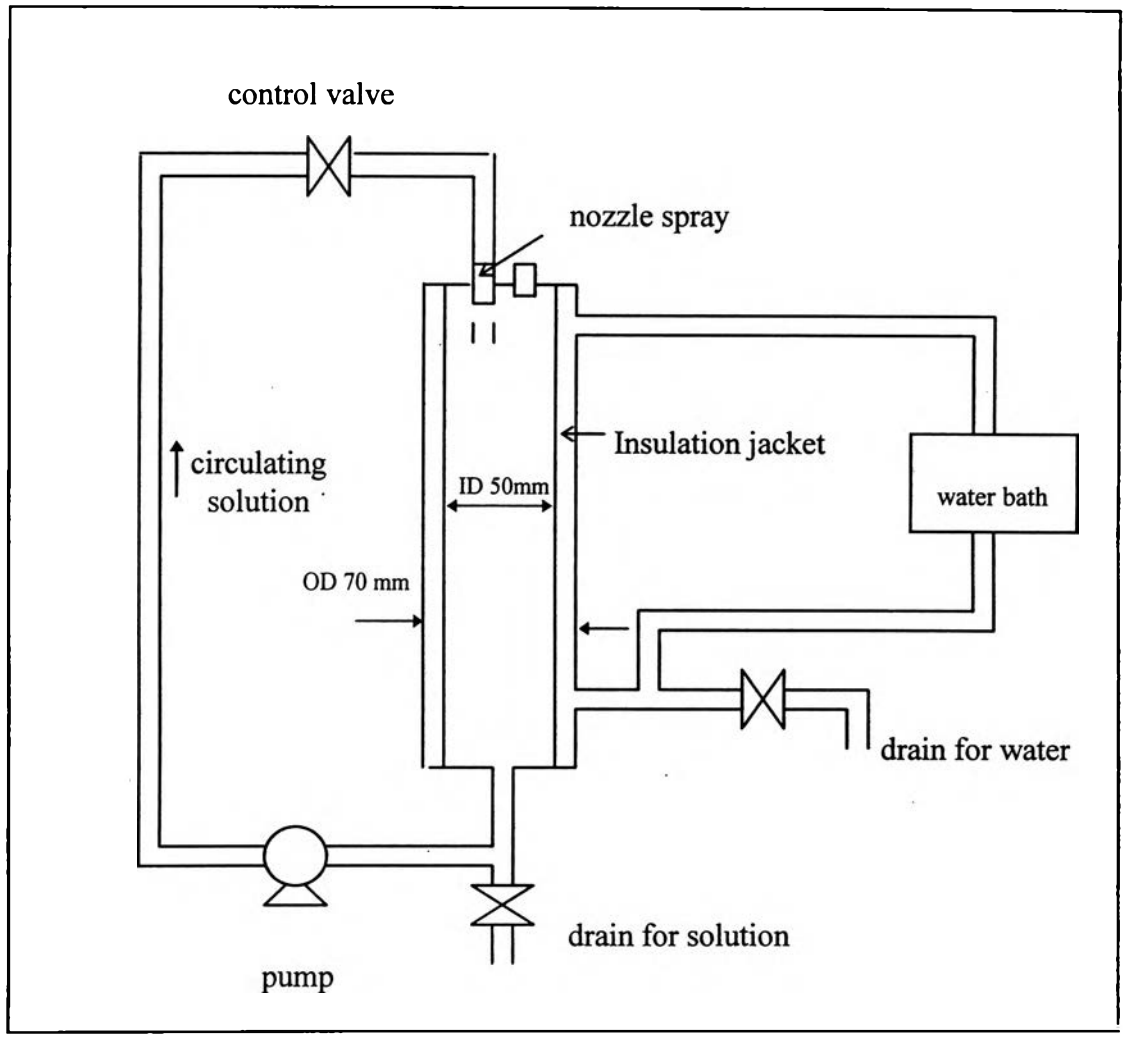

Figure 3.4 Schematic diagram of the Spray method.

A schematic flow diagram of the spray foam apparatus used in this study is shown in Figure 3.4. The column is a jacketed tube of 50 mm internal diameter. The external diameter of the jacket is 70 mm. The lower end of the measuring cylinder has a stopcock to drain the liquid. It has a spray nozzle and centrifugal pump for withdrawing the solution from the bottom of the column, circulating it through a nozzle and spraying onto the surface of the same surfactant solution.

3.3 Experimental Methods

3.3.1 Cloud Point Test

To determine the cloud point temperature, surfactant solutions were prepared at the desired concentration and 50 mL of the solution was transfered to a test tube, 25 mm in internal diameter. The solution was then warmed in a hot-water bath until cloudy by stirring occasionally with a thermometer. The solution was then removed from heat, placed on a stand, and stirring was given occasionally until the solution was again clear. The temperature at which the solution became clear was recorded to the nearest 0.1°C. This temperature was taken as the cloud point of the solution (Schmitt T.M., 1992).

3.3.2 Ross-Miles Method

This method was carried out by 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 to the desired temperature. 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 slight 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 at 5 minutes after the flow had been stopped.

3.3.3 Spray Method

This method was carried out by 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 column to the desired temperature. A volume of 500 mL of the test solution was placed in the cylinder and was left in there until the test solution temperature reached the desired temperature. A volume of 250 mL of the test solution was then withdrawn from the column to circulate in the line connecting the column and the spraying device. The initial height of the solution in the column was read and the feed pump was set at the desired rate. The solution was pumped at a flow rate of 325 mL/min by centrifugal pump before going through the line and entering at the top of the column through a nozzle, spraying the solution onto the surface of the solution in the column. The foam height was monitored every minute for one hour. The pump was then closed and the foam height was read every 5 minutes for another 30 minutes to follow the decay in foam height after the spray was stopped.