CHAPTER III
EXPERIMENTAL SECTION

3.1 Materials

3.1.1 Amphoteric Surfactant

Miranol Ultra C32 or cocoamphoacetate obtained from Unilever Thai Holdings Ltd., was used as the amphoteric surfactant. It was used without further purification. Miranol Ultra C32 used in this study was a commercially available product with 39% concentration of cocoamphoacetate and 61% of sodium chloride plus water. It appeared as a slightly viscous amber liquid. The structure of Miranol Ultra C32 is shown in figure 3.1.

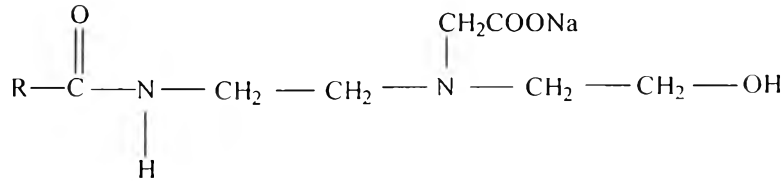


Figure 3.1 The chemical structure of Miranol Ultra C32.

3.1.2 Fatty Alcohol

The fatty alcohol used was the mixture of 15% cetyl ($C_{16}H_{33}OH$) which was the molecular weight of 258 g/mol and 85% stearyl alcohol ($C_{18}H_{35}OH$) which was the molecular weight of 286 g/mol respectively. It appeared as a waxy flak. Its melting point is equal to 48.5-53^{0}C. It was used as a component in the formation of lamellar phase and was obtained from Unilever Thai Holding Ltd., .

3.1.3 Solvent

Deionized distilled water was used as the pure solvent. It was used without further filtering or purification.

3.1.4 NH_4OH & HCl

0.01 M of ammonium hydroxide and 0.01 M of hydrochloric acid were used for adjusting pH.

3.2 Methodology

3.2.1 Sample Preparation of MUC32/FA emulsions

The fatty alcohol was heated to 75-85^{0}C and mixed with Miranol Ultra C32 and stirred with a glass rod for two to three minutes to obtain a homogeneous mixture. Then the mixture of fatty alcohol and Miranol Ultra C32 was poured into a main mixer beaker containing water at 75-85^{0}C. The whole mixture was then homogenized by a blade mixer at a slow speed of 110 rpm for 1 to 2 minutes before changing to a higher speed, which was 210 rpm for a period of 15 minutes.

Once the emulsion of Miranol Ultra C32 and fatty alcohol started to form, it was cooled down by turning off the heater and adding more water, whose temperature was controlled at 45^{0}C. The mixture was stirred at a slow speed of 110 rpm for a period of 10 minutes. Finally an emulsion Miranol Ultra C32 and fatty alcohol was obtained at room temperature.

Diagram 1 Flow chart diagram for the preparation of emulsion of Miranol Ultra C32 with fatty alcohol

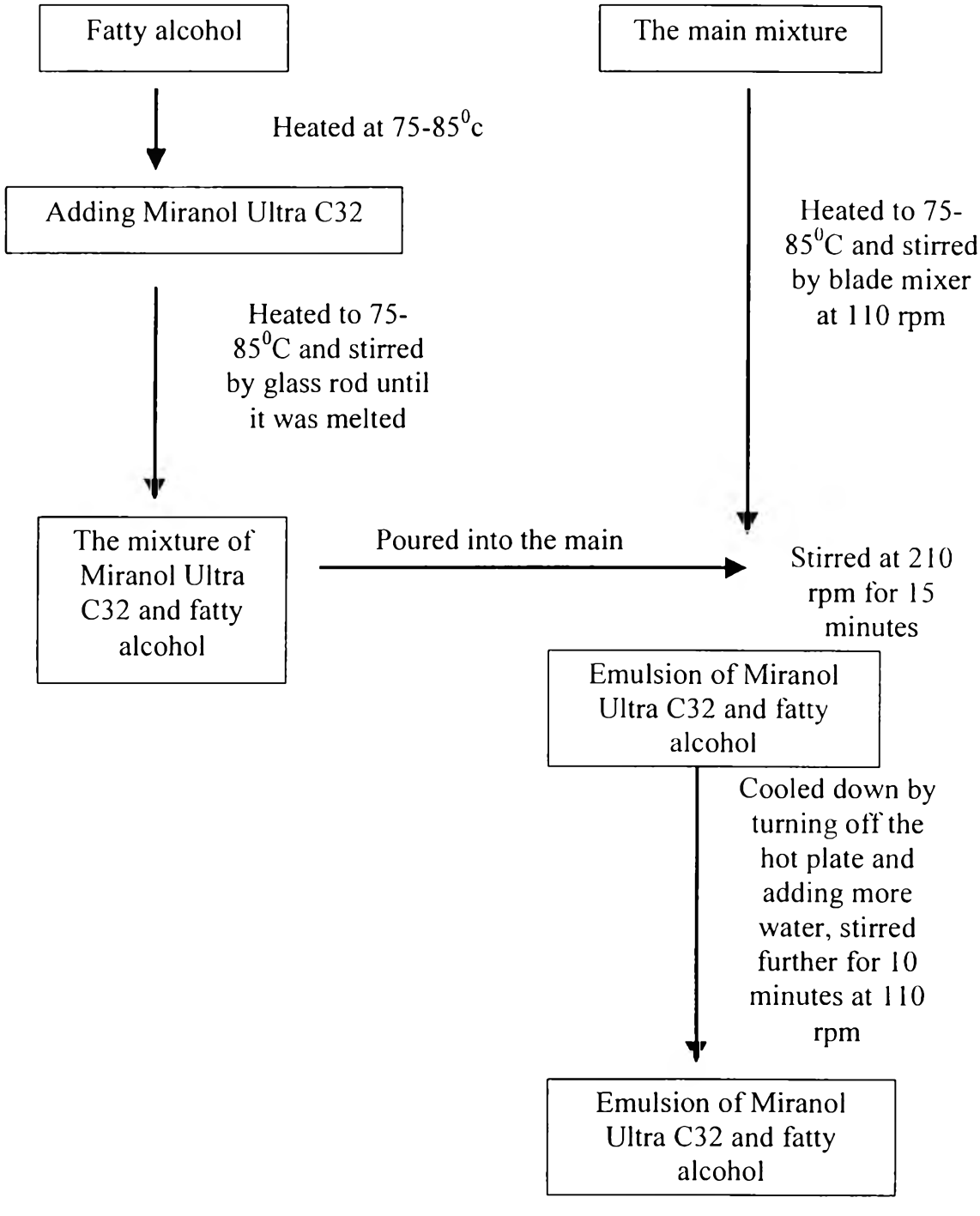
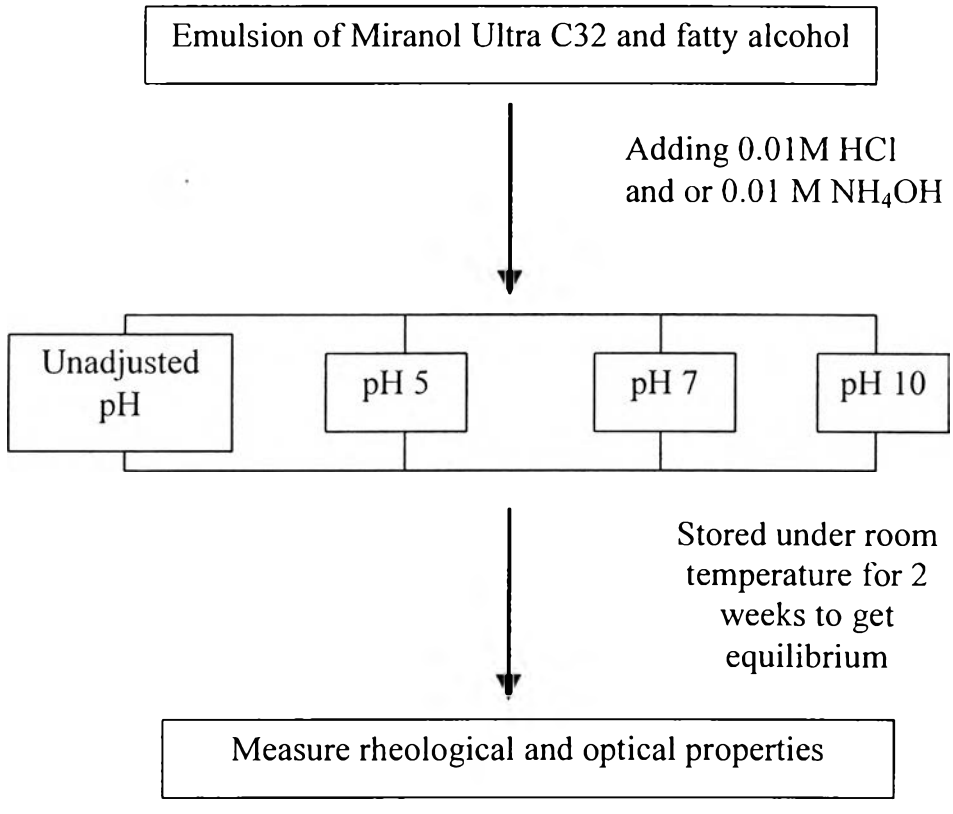


Diagram 2 Flow chart diagram for the preparation of emulsion of Miranol Ultra C32 with fatty alcohol



3.3 Apparatus

3.3.1 du Nouy Pt Ring Tensiometer

Air - water surface tensions were measured at $26\pm1^{0}C$ using a du Nouy ring tensiometer (KRUSS, model Digital-tensiometer K10T). The apparatus was first calibrated against a set of standard liquids, an excellent agreement with the literature values was found (e.g. $\gamma_{air\text{-}water} = 72.3\ mNm^{-1}$).

3.3.2 Zeta-Potential Meter System 3.0+

Zeta potential measurements were performed with a zeta meter (Zeta-meter, model zeta-meter system 3.0+) using a rectangular quartz capillary cell with a Pt anode and code. It measured the mobility of particles under an electric field.

3.3.3 Optical Measurement

3.3.3.1 Optical Microscope

An optical microscope (Leica, model DMRXE) was used to investigate the structures for the effect of temperature in this experiment. In optical microscopy, information is obtained by light transmission through or reflection from matter. The optical microscope consists of a light source, a condenser, two lens systems and other accessories. The system is capable of producing magnified images from 10X through 1400X, thereby premitting observation of structures too small for unaided visual observation. The resolution is approximately 0.2 μm. Camera attachments permit photographic recording of the image by a Sony video camera. Studies can be conducted using

transmitted and reflected light, polarized light, bright field, dark field, differential interference contrast, and phase contrast illumination.

Thermal microscopy can be conducted at temperature ranging from 20^0C to 70^0C with the aid of thermal stage (Leitz Wetzlar). This allows phase transitions of various material to be directly observed and transition temperatures measured.

3.3.3.2 Laser Scanning Microscope

Laser scanning microscope (Zeiss, LSM410 inverse) was used to investigate the morphology of emulsions for the effect of aging time, fatty alcohol concentration and pH. The system is based on the Axiovert 100, 135 or 135M microscope family. This microscope allows conventional microscope using usual contrast methods such as bright field, dark field, differential interference contrast, phase contrast, fluorescence and polarization. The basic LSM system is based on a beam scan system, a detector unit and a control computer. The over view and schematic drawing of LSM410 are given in figures 3.2 and 3.3 respectively .

A laser is scanned horizontally over the sample and the beam is redirected by a beam splitter and is based on the sample via the scanner and the objective. By using the scanning technique, the lateral resolution can be improved by a factor of 2 to 3 compared with the classical light microscope. The depth resolution depends on the wavelength of the light source, as well as the pinhole size (Ribbe, 1997).

The laser is equipped with a Helium-neon laser with wavelength of 543nm (green), 633nm (red) which can all be used for reflection as well as fluorescent studies. In order to improve the speed of the scanning

process, the pinhole can be replaced by a rotating disk containing hundreds of pinholes through which the light is collected.

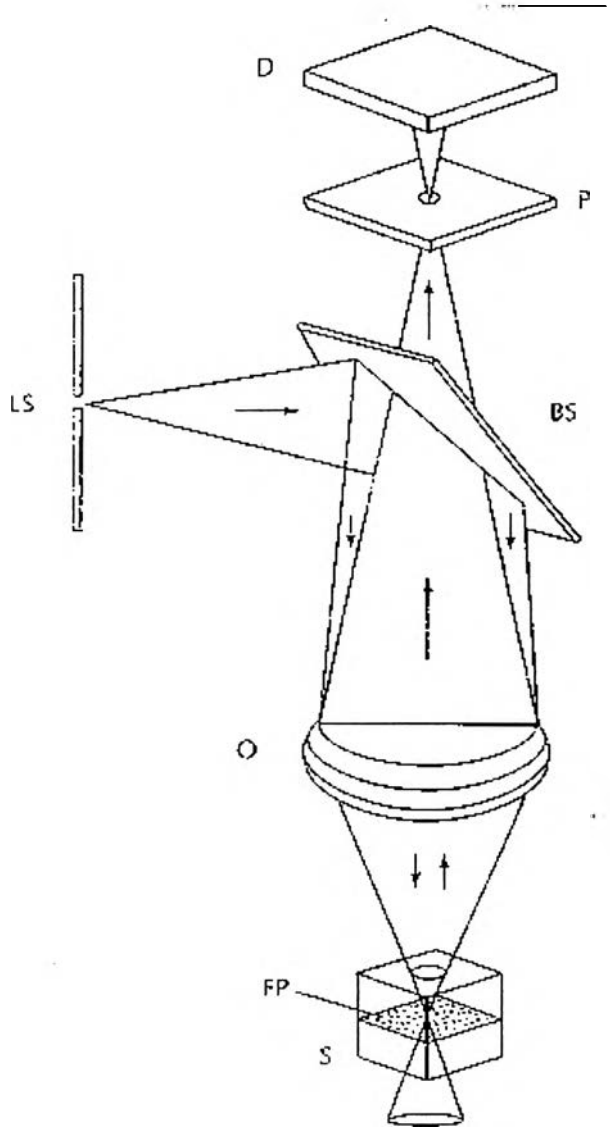


Figure 3.2 Set-up of a laser scanning confocal microscope; light source (LS), detector (D), pinhole (P), beam splitter (BS), objective (O), focal plane (FP), specimen (s).

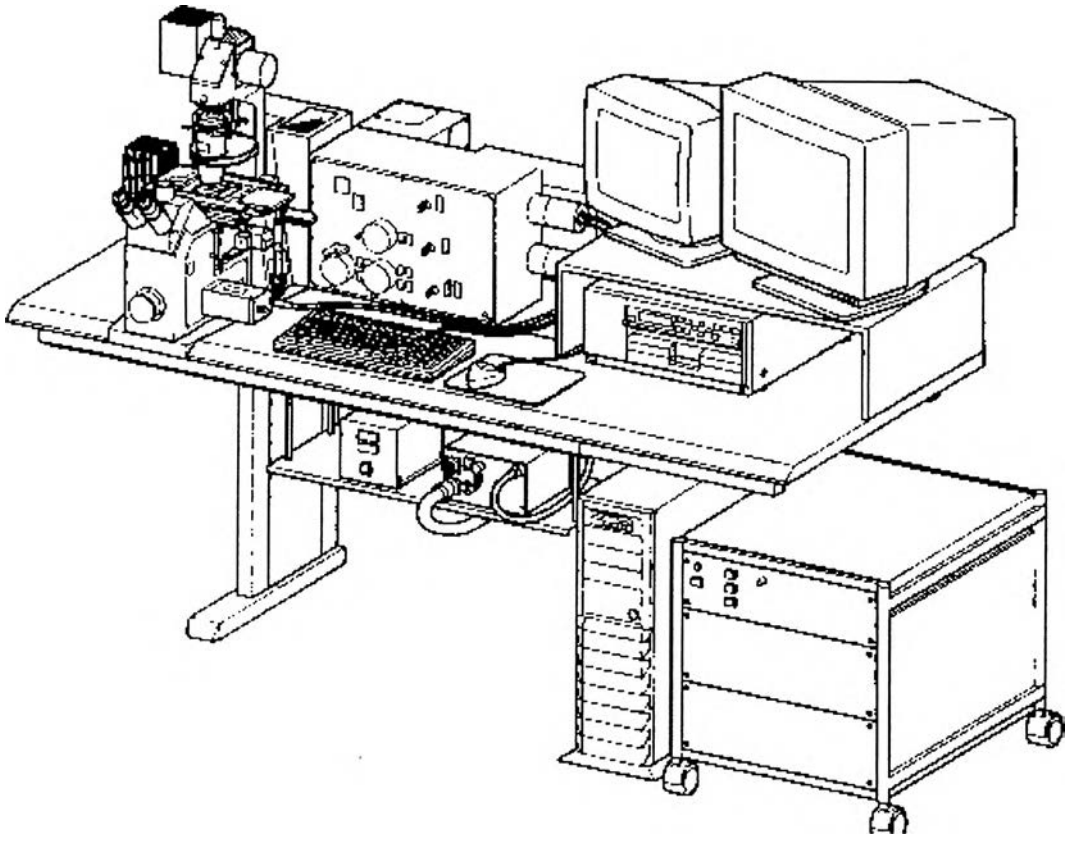


Figure 3.3 General overview of the laser scanning microscope.

3.3.4 Rheometer

Strain controlled fluid rheometer (Rheometric Scientific Inc., model ARES) was used to measure the rheological properties of emulsion in both oscillatory and steady state modes. The system consists of test station, power chassis, temperature sensing control system. In this experiment, cone-and-plate geometry with cone angle 0.04 rad , a diameter of 50 mm and a gap size 0.051± 0.001 mm was used.

The geometrical characteristic of a cone and plate rheometer is shown in figure 3.4. A flat circular plate and linearly concentric cone are rotated relative to each other. The major advantage of a cone and plate rheometer is the constant shear rate throughout all the sample.

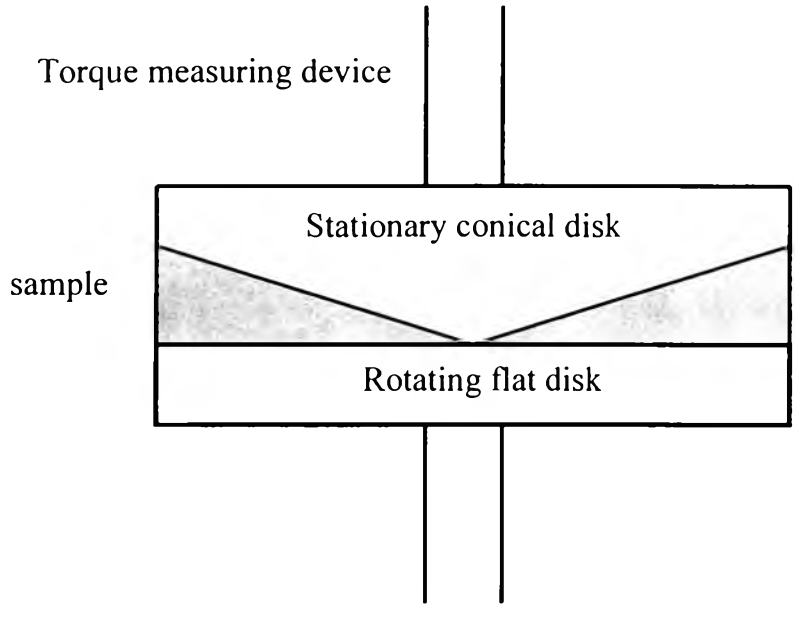


Figure 3.4 Principal features of cone-and-plate.

3.3.4.1 Oscillatory test

Rheological properties in oscillatory experiments were measured as a function of strain amplitude and at a fixed frequency (1Hz) with temperature 26±1^{0}C. Initial strain and final strain were 0.1 and 200% respectively. In these measurements, the level of strain was determined in order to ensure that all measurements were made within the linear viscoelastic regime.

After the fluid's linear viscoelastic regime has been established by a strain sweep, its structure can be further characterized using a frequency sweep at a strain below the critical strain. The measurements were by varying the frequency (0.03 to 100 Hz) at a fixed strain. The value of strain used was chosen to be within the linear viscoelastic regime. In these measurements, G', G"and tanδ were determined as a function of frequency.

3.3.4.2 Steady State

Steady flow and dynamic properties were measured with a rheometer equipped with a cone and plate (diameter 50 mm and cone angle 0.04 rad). The temperature of the samples was maintained constant at 26 ± 1^{0}C. Shear rates were varied from 0.1 to 1000 s^{-1}.