

CHAPTER III EXPERIMENTAL

3.1 Chemicals and Materials



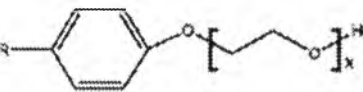
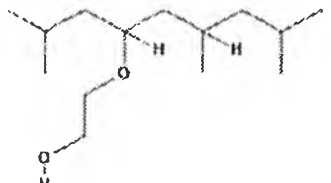
Five anionic surfactants used in this research were alpha olefin sulfonic acid sodium salt or Lipolan PB-800CJ (96.8 wt% active ingredients), di-2-ethylhexyl sodium sulfosuccinate/isopropyl alcohol or Lipal 835I (33.5 wt% solution in water) obtained from Lion Corporation, sodium dodecyl sulfate (SDS 99 wt% active ingredients), fatty acid methyl ester sulfonic acid (MES, 98 wt% active ingredients) and sodium dodecylbenzene sulfonic acid (SDBS, 88 wt% active ingredients) obtained from ACROS organics. Chemical structure and molecular weight of the anionic surfactants are shown in Table 3.1.

Non-ionic surfactants utilized in this thesis were polyethylene glycol tert-octylphenyl ether (Triton[®] X-100, 100% activity) obtained from Fluka chemical and trimethylnonylpolyethylene glycol (Tergitol[®] TMN6, 90% activity) obtained from Sigma life science. Chemical structure and molecular weight of the non-ionic surfactants are also shown in Table 3.1.

Decane, colorless liquid with 99+% activity, from ACROS organics was used as a simulated light crude oil. Sodium chloride (NaCl) with 99.5% minimum assay from CARLO ERBA was used to determine the salinity effect. Oil Red O (dye content 99%), which is a dye for oil was obtained from sigma-aldrich.

Flat-bottom-screw cap tubes were used as a container for microemulsion forming. Cathetometer from Titan tool supply was used to measure phase height. Sandstone core sample is used as rock for spontaneous imbibition test.

Table 3.1 Chemical structure and molecular weight of the studied surfactants

Surfactant	Type	Chemical structure	C (alkyl)	MW
Lipolan PB-800CJ	Anionic	$\left. \begin{array}{l} RCH_2CH(OH)(CH_2)_mSO_3Na \\ RCH=CH(CH_2)_nSO_3Na \end{array} \right\} \text{Mixing}$	14-16	-
Lipal 835I	Anionic	ROCOCH ₂ -ROCOCH-SO ₃ Na	20	444.56
SDS	Anionic		12	288.372
MES	Anionic	RCH(SO ₃ Na)COOCH ₃	16-18	-
SDBS	Anionic		18	348.48
Triton® X-100	Non-ionic		14	647
Tergitol® TMN6	Non-ionic		14	230.3868

3.2 Microemulsion Formation

Phase behavior of microemulsion system is studied in order to determine the suitable surfactants to form microemulsion with decane. The middle phase or Winsor type III is targeted due to the ultralow interfacial tension of the surfactant systems. Symmetry distribution of the middle phase is also referred as the best surfactant system.

This experimental was divided for two steps. The first step is anionic surfactant screening, only single anionic surfactant was provided for surfactant systems. Phase behavior results were observed by visual only. The second step, non-ionic surfactant was mixed with anionic surfactant that is shown the interesting phase behavior results from the first step.

3.2.1 Screening of Anionic Surfactant for Microemulsion Formation

Single anionic surfactant systems with 2wt% were prepared with varies salt concentration at 1wt% , 2wt%, 3wt%, 4wt%, 5wt%, 6wt%, 7wt%, 8wt%, 9wt% and 10wt% of NaCl. Equal volume of surfactant solution (5 ml) and decane (5 ml) were mixed in the Flat-bottom-crew cap tube. The system was left to reach phase equilibrium, until phase level stable, up to 10 days at 30°C and 50°C.

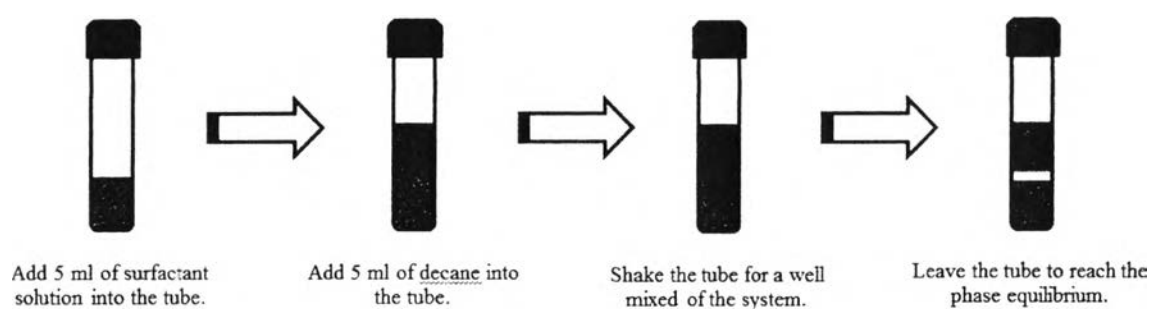


Figure 3.1 Schematic experiment of microemulsion preparation.

3.2.2 Screening of Mixed Anionic-Nonionic Surfactant for Microemulsion Formation

Single non-ionic surfactant was mixed with single anionic surfactant in the concentration of 1wt% and 2wt% respectively. Only salt concentration of the microemulsion systems which the middle phase appeared (include of sponge, gel or precipitate) at the first step were prepared to performed in this step. The systems was left to reach phase equilibrium, until phase level stable, up to 10 days at 30°C and 50°C. The surfactant formula was selected base on Winsor type III showing the symmetry distribution middle phase as shown in the Figure 3.2.

Equivalent height of upper level and lower level from baseline indicates that the surfactant system has capacity to dissolve an equal quantity of water and decane into the middle phases. This condition was determined as the most suitable surfactant formula with ultralow interfacial tension of the middle phase.

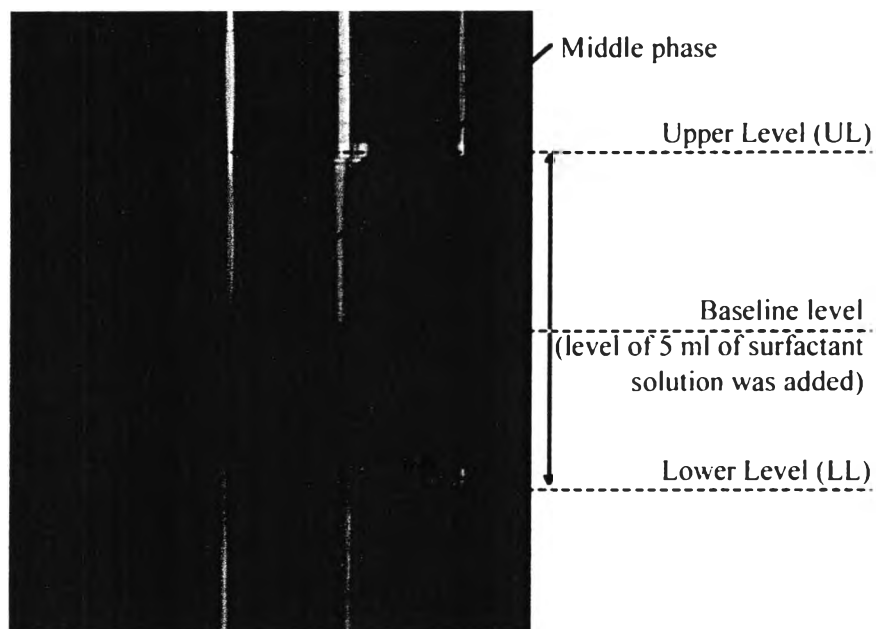


Figure 3.2 Symmetry distribution of middle phase, when height from baseline level to upper level is equal with height from baseline level to lower level.

3.3 Measurement of Microemulsion Interfacial Tension

Spinning drop tensiometer (SVT 15; Dataphysics) was utilized to confirm the ultralow interfacial tension (IFT) of microemulsion phase and excess decane and/or excess water at 30°C.

3.4 Spontaneous Imbibition Test

Spontaneous imbibition test was performed to determine the surfactant ability to recover decane from the core sample. Only the suitable surfactant formulas those were determined from the mixed anionic-nonionic surfactant screening step were used in this step. The same core sample was used for limiting the variation possibly come from using different core samples.

3.4.1 Core sample preparation

Cylinder sandstone sample was cut to 2 inches diameter and 3 inches length as shown in Figure 3.3. It was cleaned by soak in tetrahydrofuran (THF) for 2 days, then in chloroform for another 2 days and displaced the chloroform by soaking the core in methanol for 1 day. Finally, the core sample was soaked in water and dry until weight constant.

Subsequently, the cleaned core sample was submerged into decane at 30°C in the close system until weight constant to confirm the core was saturated with decane.

However, surfactant solution and decane are colorless liquid. In this step, Oil Red O dye color was added into decane to dye the decane turned to red color for better visual and easily decane distinguish out from surfactant solution.

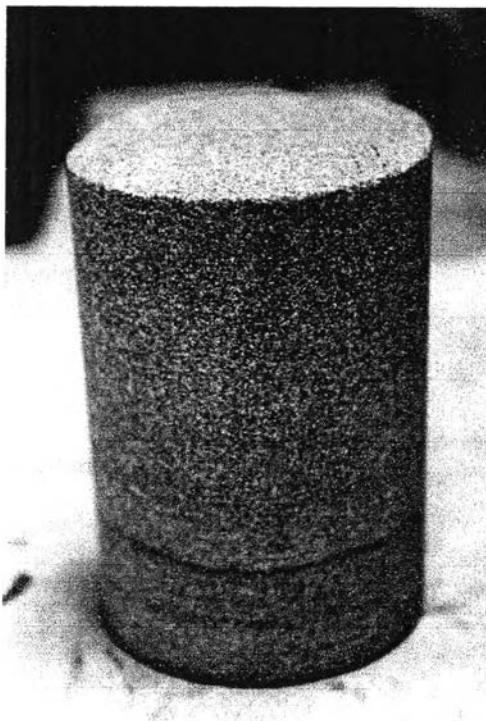


Figure 3.3 Sandstone core sample was used for spontaneous imbibitions test.

3.4.2 Decane recovery

After the saturated core sample had been submerged in the suitable surfactant solution in the close system at 30°C for 10 days, The decane was removed from the core were float to the top of surfactant solution. The amount of removed decane was determined by weight of decane which was removed by surfactant/decane mixture extraction.