

## CHAPTER IV

### RESULT AND DISCUSSION

#### 4.1 Phase Behavior of Surfactant Systems

##### 4.1.1 Screening of Anionic Surfactant for Microemulsion Formation

Summary of single anionic surfactant phase behavior at 30°C is shown in Table 4.1.

**Table 4.1** Phase behavior result, single anionic surfactants at 30°C

Anionic surfactant	Phase behavior after 10 days / 30°C									
	1	2	3	4	5	6	7	8	9	10
SDBS	N/A	N/A	N/A	N/A	N/A	SP	SP	SP	SP	SP
SDS	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MES	SP	SP	SP	N/A	N/A	N/A	-	-	-	-
Lipal 835 I	N/A	N/A	N/A	N/A	SP	IIItn	IIItn	IIItn	IIItn	IIItn
Lipolan PB-800 CJ	SPtk	SPtk	SPtk	SPtk	SPtk	SPtk	SPtk	SPtk	SPtk	SPtk
NaCl (%wt)	1	2	3	4	5	6	7	8	9	10

Note :

I = Winsor type I

II = Winsor type II

III = Winsor type III

tn = thin layer

tk = thick layer

N/A = not change

SP = sponge at the middle

SDBS with low salinity concentration at 1wt%, 2wt%, 3wt%, 4wt% and 5wt% NaCl systems provided the Winsor type I phase behavior, since surfactant molecules prefer to dissolved in the water phase and remained the excess decane at the top. However, the SDBS with 6wt%, 7wt%, 8wt%, 9wt% or 10wt% NaCl could not provide phase transition from Winsor type I to Winsor type III, just a sponge layer was appeared at the middle between water phase and decane phase. This

phenomena could refer as ion precipitation between anionic ion of surfactant head group and cationic ion of Na from salt, because Na ion accumulated around the anionic surfactant micelles. However, adding of nonionic surfactant maybe solve ion precipitation phenomena, because nonionic surfactant molecules able to intermingle between the SDBS molecule and effect to reduce anionic charge concentration. Therefore, SDBS surfactant will keep to mix with nonionic surfactant at the next step.

However, SDS systems at any salinity concentration had no phase interaction, that mean SDS could not dissolve with decane. Because lipophilic tail of the surfactant ( $C_{12}$ ) is not significant longer than the decane molecule ( $C_{10}$ ) and the surfactant molecule is contains only straight chain. Therefore, the space between SDS molecules had not enough for dissolved decane. So, SDS surfactant were screened out to mix with nonionic surfactant at the next step.

MES systems were performed with 1wt%, 2wt%, 3wt%, 4wt%, 5wt% and 6wt% NaCl only, since surfactant solution was very viscous like a gel at above 6wt% NaCl. Some interesting phase behavior occurred at 1wt%, 2 wt% and 3wt% NaCl, sponge layer of the surfactant was occurred at the middle between water phase and decane phase as same as SDBS systems, this phenomena could refer as ion precipitation too. However, MES with 4wt%, 5wt% or 6 wt% NaCl systems had no phase interaction. This anionic surfactant were screened out and did not prepare for mixed surfactant step.

The another phase behavior results of Lipal 835I with 6wt%, 7wt%, 8wt%, 9wt% and 10wt% NaCl systems showed a very thin layer of colorless liquid at the middle. The first reason was the surfactant molecule contains both of sulfosuccinate and isopropyl alcohol with a long alkyl chain ( $C_{20}$ ), that mean the surfactant molecule contains with double polar parts and a large non-polar part. So, Lipal 835I molecule could not shows the outstanding property to dissolve in water phase or decane phase. The second reason was the surfactant molecule is very high molecular weight molecule when compared to water and decane. That cause the surfactant molecule preferred to accumulate together as the thin layer phase. Maybe nonionic surfactant can improve the phase behavior of this anionic surfactant by extend the

micelle spaces, so Lipal 835I surfactant will keep to mix with nonionic at the next step.

For Lipolan PB-800 CJ systems, the result showed a very thick layer of sponge phase at the middle with all NaCl concentration similar to the SDSB systems. But, Lipolan PB-800 CJ is mix of C<sub>14</sub> and C<sub>16</sub> alkyl chain and also differently contained of double bond structure. So, the Lipolan PB-800 CJ micelle has the tail space more than SDBS micelle, that cause more decane was able to dissolve into the Lipolan PB-800 CJ micelle than the SDBS micelle. However, an ion precipitation phenomenon was occurred by effect of salinity concentration as same as another anionic surfactant. Therefore, Lipolan PB-800 CJ will mix with nonionic surfactant to improve phase behavior at the next step.

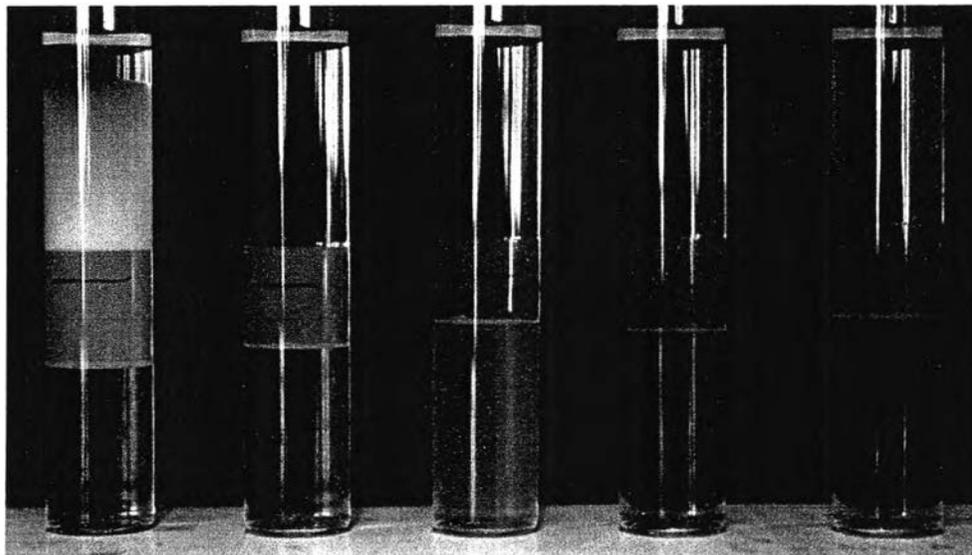
At 50°C, had no effects to phase behavior results of the anionic surfactant systems when compared with 30°C. Since temperature did not mainly effect to phase transformation of anionic surfactant.

#### 4.1.2 Screening of Mixed Anionic-Nonionic Surfactant for Microemulsion Formation

Nonionic surfactant, Tergitol<sup>®</sup> TMN6 or Triton<sup>®</sup> X-100, was mixed with anionic surfactant to improve the phase behavior of single anionic surfactant to form a microemulsion or Winsor type III phase behavior.

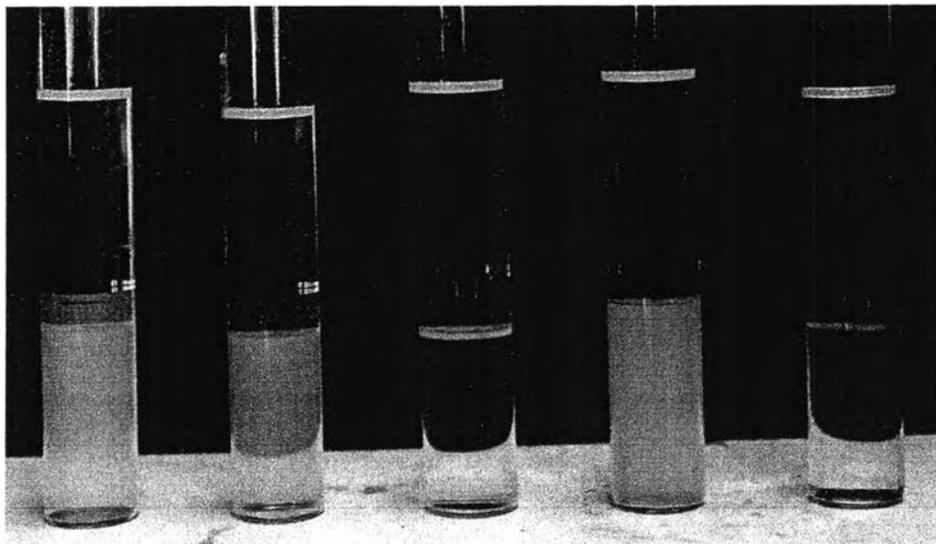
Table 4.2 shows phase behaviors of mixed anionic-nonionic surfactant systems. Only SDBS could provide the perfect middle phase which was considered as Winsor type III or microemulsion behavior when mixed with nonionic surfactant, Tergitol<sup>®</sup> TMN6 or Triton<sup>®</sup> X-100.

Mixed SDBS/Tergitol<sup>®</sup> TMN6 surfactant systems could provide Winsor type III phase behavior at 7wt% to 10wt% NaCl concentration. The perfect middle phase which is the most symmetry distribution middle phase was obtained at about 8.5wt% NaCl system as shown in Figure 4.1.



**Figure 4.1** Microemulsion phase behavior of SDBS/Tergitol<sup>®</sup> TMN6 with 7wt%, 7.5wt%, 8wt%, 8.5wt% and 9wt% NaCl (left to right).

Figure 4.2 is shows microemulsion phase behavior result and phase height distribution of SDBS/Triton<sup>®</sup> X-100 mixed surfactant systems with 10wt%, 11wt%, 12wt%, 13wt% and 14wt% NaCl. Mixed SDBS/Triton<sup>®</sup> X-100 also provided a Winsor type III phase behavior at the middle with wide range from 8wt% to 14wt% of NaCl concentration (Appendix B). But the middle phase layer of SDBS/Triton<sup>®</sup> X-100 was thinner than the middle phase layer of SDBS/Tergitol<sup>®</sup> TMN6. Since Tergitol<sup>®</sup> TMN6 molecule is contain a branch of alkyl group, that caused micelle spaces were extented more than the Triton<sup>®</sup> X-100 micelle spaces. However, an avantage of SDBS/Triton<sup>®</sup> X-100 systems is suitable for using at high salinity concentration when compared with SDBS/Tergitol<sup>®</sup> TMN6.



**Figure 4.2** Microemulsion phase behavior of SDBS/ Triton<sup>®</sup> X-100 with 10wt%, 11wt%, 12wt%, 13wt% and 14wt% NaCl (left to right).

While Lipal 835I mixed with Tergitol<sup>®</sup> TMN6 or Triton<sup>®</sup> X-100 could not provide the middle phase at 2wt% to 10wt% NaCl concentration (formed only 2wt%, 4wt%, 6wt%, 8wt% and 10wt% NaCl). Because compatibility in the term of non-polar charge between anionic and nonionic surfactants is too low, since lipophilic chain length is too different ( $C_{20}$  and  $C_{14}$ ).

For Lipolan PB-800 CJ as anionic surfactant systems, Winsor type II phase behavior occurred at 2wt% and 4 wt% NaCl when mixed with Tergitol<sup>®</sup> TMN6 or Triton<sup>®</sup> X-100 non-ionic surfactant systems. That mean the non-ionic surfactant had able to extend the space of Lipolan PB-800 CJ tails and dissolved decane more than used single Lipolan PB-800 CJ. However, at high salinity concecntration (6wt% and 8wt%) surfactant phase was formed as gel/sponge layer at the middle. That means the salinity stilled as main effect to this mixed surfactant phase behavior, therefore ion precipitation by cationic ion from Na was occurred. Phase behavior of mixed Lipal 835I/Tergitol<sup>®</sup> TMN6, Lipal 835I/Triton<sup>®</sup> X-100, Lipolan PB-800 CJ/Tergitol<sup>®</sup> TMN6 and Lipolan PB-800 CJ/Triton<sup>®</sup> X-100 systems are shown in appendix B.

Mixed anionic-nonionic surfactant systems at 50°C also showed phase behavior similar to phase behavior at 30°C, only slight changed of phase height but

microemulsion (Winsor type III) still occurred at the same NaCl concentration (shown in appendix C). That means the temperature increased from 30°C to 50°C could not affect the phase behavior transition of the studied mixed surfactant, since the nonionic surfactant ratio in mixed surfactant was less than the ratio of anionic surfactant (1wt% : 2wt% , respectively). Therefore, salinity concentration still acts as the majority effect to the phase behavior. It could be implied that, at 50°C the mixed SDBS/Tergitol<sup>®</sup> TMN6 and mixed SDBS/Triton<sup>®</sup> X-100 surfactants still had the capacity to perform as well as the capacity at 30°C. Therefore, determination of the optimum surfactant formulas and spontaneous imbibition tests will mainly focus at 30°C only.

**Table 4.2** Phase behavior result, mixed anionic-nonionic surfactants at 30 °c (same as 50 °c)

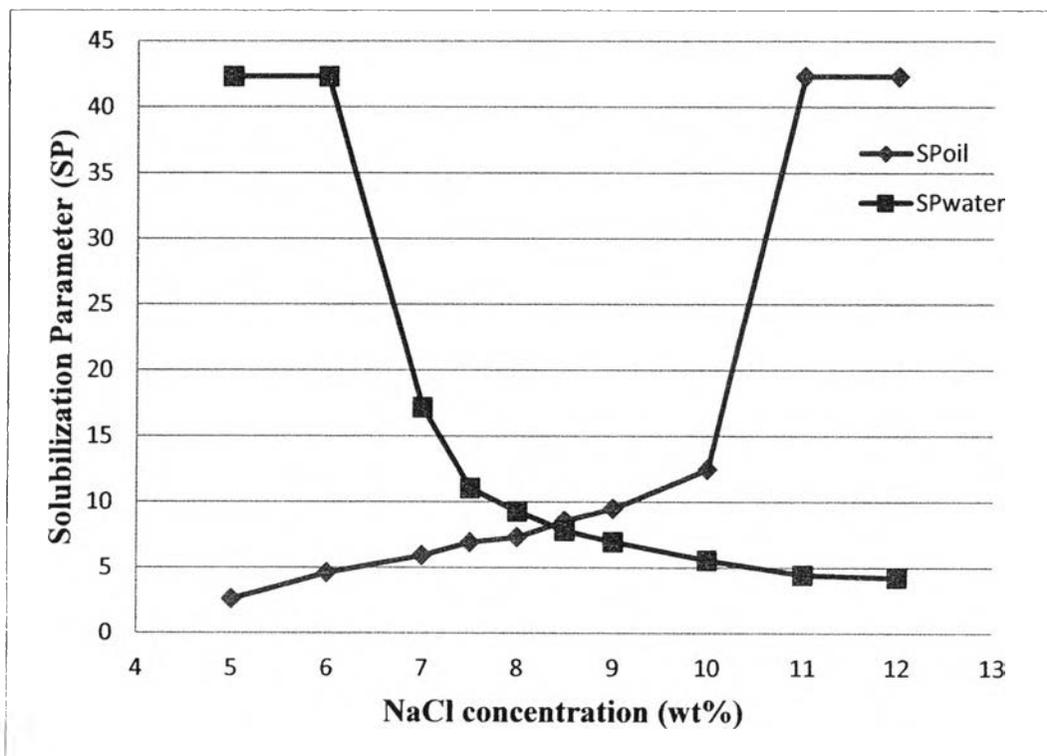
Surfactant	Phase behavior after 10 days / 30 °c					
	Anionic	SDBS	SDBS	Lipal 835 I	Lipal 835 I	Lipolan PB-800 CJ
Non-ionic	Tergitol® TMN6	Triton® X-100	Tergitol® TMN6	Triton® X-100	Tergitol® TMN6	Triton® X-100
NaCl (wt%)						
2	-	-	No phase interaction	No phase interaction	Winsor type II	Winsor type II
3	-	-	-	-	-	-
4	-	-	No phase interaction	No phase interaction	Winsor type II	Winsor type II
5	Winsor type I	Winsor type I	-	-	-	-
6	Winsor type I	Winsor type I	No phase interaction	No phase interaction	Gel/sponge	Gel/sponge
7	Winsor type III	Winsor type I	-	-	-	-
8	Winsor type III	Winsor type III	No phase interaction	No phase interaction	Gel/sponge	Gel/sponge
9	Winsor type III	Winsor type III	-	-	-	-
10	Winsor type III	Winsor type III	No phase interaction	No phase interaction	-	-
11	Winsor type II	Winsor type III	-	-	-	-
12	Winsor type II	Winsor type III	-	-	-	-
13	-	Winsor type III	-	-	-	-
14	-	Winsor type III	-	-	-	-

## 4.2 Microemulsion Phase Behavior and Determination of Optimum Formula

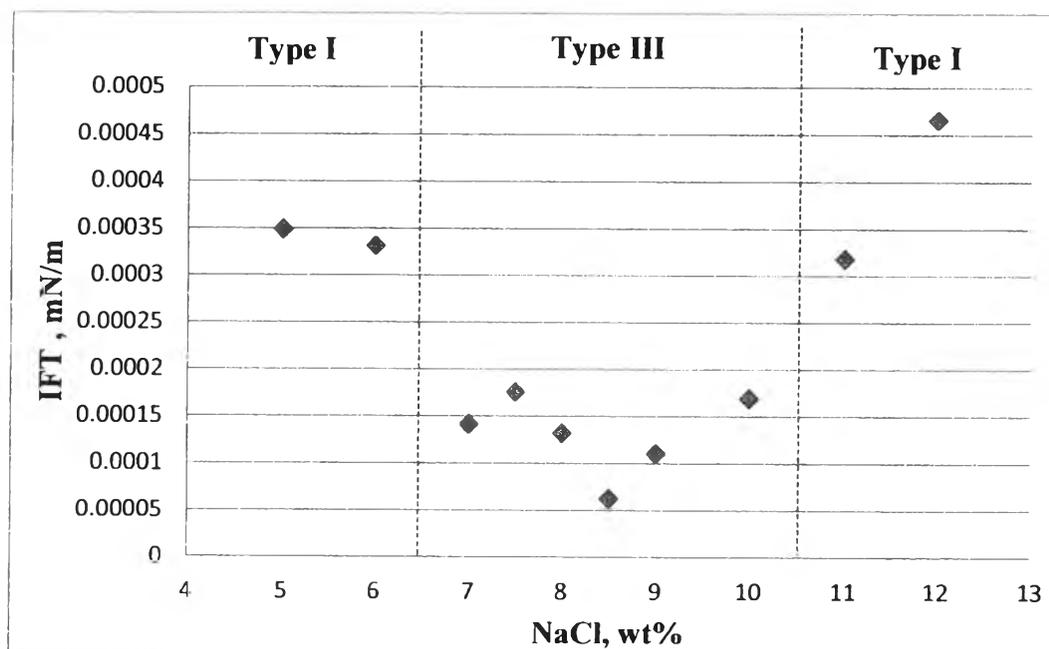
From phase behavior of mixed anionic-nonionic surfactants results, the Winsor type III or microemulsion phase behavior were obtained from mixed of SDBS/Tergitol<sup>®</sup> TMN6 and SDBS/Triton<sup>®</sup> X-100 systems. In this step, solubilization parameter and interfacial tension value were used to determine the optimum salinity of these mixed surfactants.

### 4.2.1 Solubilization Parameter of SDBS/Tergitol<sup>®</sup> TMN6

Figure 4.3 shows solubilization parameter of SDBS/Tergitol<sup>®</sup> TMN6 systems at 5wt%, 6wt%, 7wt%, 7.5wt%, 8wt%, 8.5wt%, 9wt%, 10wt%, 11wt% and 12wt% NaCl. Intersection point of  $SP_{oil}$  and  $SP_{water}$  lines was showed at 8.5wt% NaCl, which is refer as the optimum salinity ( $SP^* = SP_o = SP_w$ ) of SDBS/Tergitol<sup>®</sup> TMN6 mixed surfactant. The lowest interfacial tension between mixed SDBS/Tergitol<sup>®</sup> TMN6 and decane is  $6.25 \times 10^{-5}$  mN/m was occurred at 8.5wt% NaCl concentration. However, ultralow IFT value between SDBS/Tergitol<sup>®</sup> TMN6 and decane also occurred at Winsor type III phase behaviors (7wt%, 7.5wt%, 8wt%, 9wt% and 10wt% NaCl concentration) as shown in Figure 4.4.



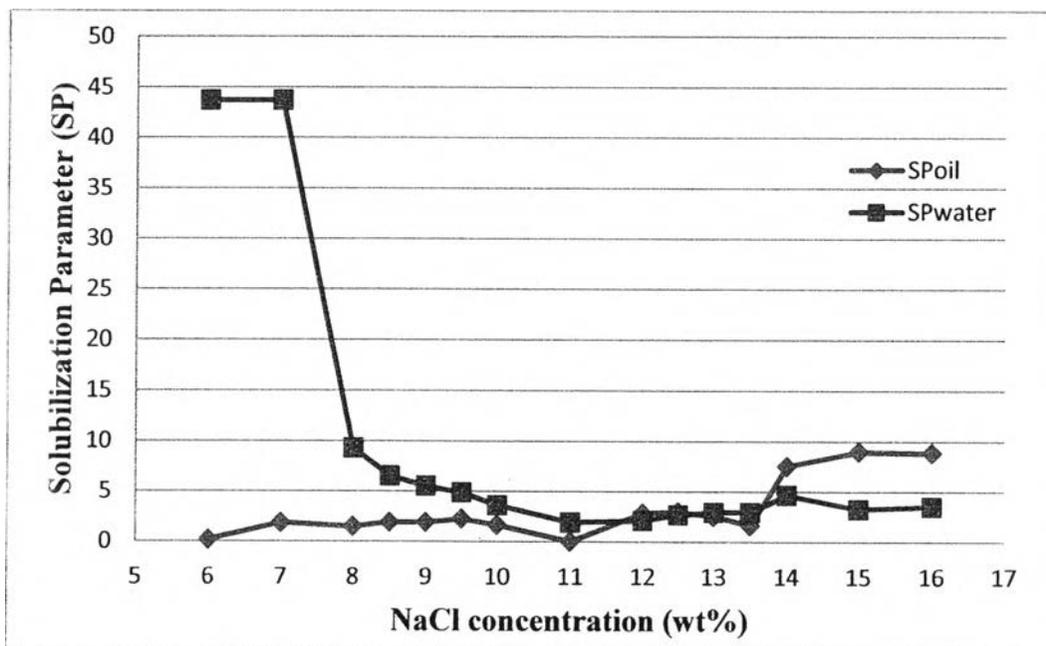
**Figure 4.3** Solubilization parameter of SDBS/Tergitol<sup>®</sup> TMN6 as a function of salt salinity.



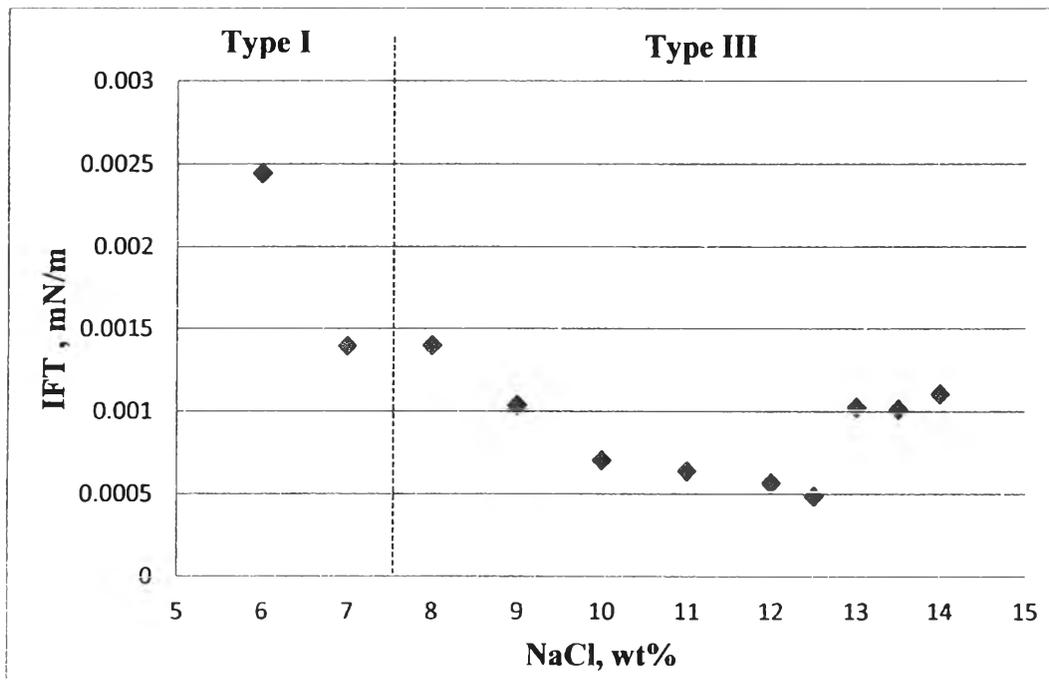
**Figure 4.4** The interfacial tension between SDBS/Tergitol<sup>®</sup> TMN6 and decane as a function of salt salinity.

#### 4.2.2 Solubilization Parameter of SDBS/Triton<sup>®</sup> X-100

Solubilization parameter value of mixed SDBS/Triton<sup>®</sup> X-100 systems is shown in Figure 4.5. Intersection point between  $SP_{oil}$  and  $SP_{water}$  was showed at 12.5wt% NaCl which is refer as the optimum solubilization parameter. Interfacial tension between mixed SDBS/Tergitol<sup>®</sup> TMN6 and decane results (Figure 4.6) show  $4.88 \times 10^{-4}$  mN/m is the lowest IFT value was also occurred at 12.5wt% NaCl concentration. However, the condition with ultralow interfacial tension also obtained from 10wt% to 12.5wt% NaCl range which is the middle region of Winsor type III phase behavior.



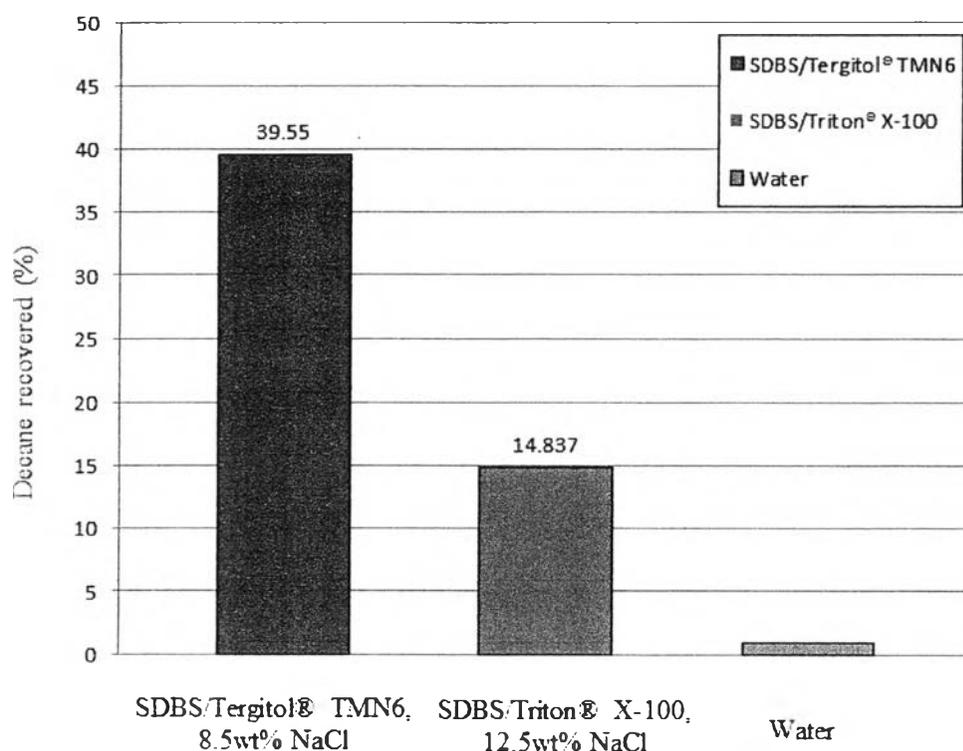
**Figure 4.5** Solubilization parameter of SDBS/Triton<sup>®</sup> X-100 as a function of salt salinity.



**Figure 4.6** The interfacial tension between SDBS/ Triton<sup>®</sup> X-100 and decane as a function of salt salinity.

Finally, 8.5wt% and 12.5wt% NaCl concentration are the optimum salinity for SDBS/Tergitol<sup>®</sup> TMN6 and SDBS/Triton<sup>®</sup> X-100 respectively. These formulas had preparing for recover decane from sandstone core sample in imbibition test.

#### 4.3 Determination the Suitable Surfactants Performance for Recover Decane from the Sandstone Core by Spontaneous Imbibition.



**Figure 4.7** Decane recovered from sandstone core by the suitable surfactant formulas at 30°C.

Decane recovery result by the suitable surfactant formulas are shown in Figure 4.7. The highest amount of recovered decane is 39.55%, was recovered by mixed SDBS/Tergitol® TMN6 surfactant at 8.5wt% NaCl concentration. While mixed SDBS/Triton® X-100 surfactant at 12.5wt% NaCl concentration could recover decane from the same sandstone core only 14.837%. The result confirm that SDBS/Tergitol® TMN6 has better performance to recover decane from sandstone core sample at 30°C than SDBS/Triton® X-100, according to the IFT value and microemulsion phase behavior from previous result. Size and weight of nonionic surfactant are also effect to decane recovery performance, Tergitol® TMN6 molecule is smaller and lighter than Triton® X-100 molecule that caused

mixed SDBS/Tergitol<sup>®</sup> TMN6 surfactant could entry into porous of the sandstone core easier than mixed SDBS/Triton<sup>®</sup> X-100 surfactant.

While water system could not recover decane out from the sandstone core sample, although some amount of decane appeared in water phase, but decane recovered was very little for determining the recovered amount (appendix F). So, the percentage of decane recovered by water was not reported.