CHAPTER IV
RESULTS AND DISCUSSION

4.1 Microemulsion Formation

For microemulsion formation with motor oil, a mixture of 1.5wt% ADPODS, 5wt% AOT and 5wt% Span 80 was used as a base condition in this study. When the system was scanned for any ADPODS, AOT or Span 80, the other two surfactant concentrations were kept constant. Each of surfactant concentration was varied to find a proper formulation that formed microemulsions at a lowest salinity. The solubilization is defined as the volume of either oil or water dissolved per unit mass of surfactants. The solubilization parameters were plotted as a function of salinity, the interception of SPw and SPo can be used to indicate the optimum salinity and the solubilization parameter at this point is called the optimum solubilization parameter (S^*).

From the previous work, when an amount of ADPODS was reduced, NaCl concentration decreased in microemulsion formation because the HLB value of ADPODS is very high. So varying ADPODS concentration was a starting point in this work.

4.1.1 <u>Effect of ADPODS Concentration</u>

In order to observe the effect of ADPODS concentration on microemulsion formation with motor oil, both AOT and Span 80 concentrations were fixed at 5wt%. Then the ADPODS concentration was varied from 0.5wt% to 1 wt%. From Figure 4.1, as the ADPODS concentration decreases, the formation of Winsor type III microemulsion appears at a slightly lower salinity and the optimum salinity decreases markedly. The HLB value of the studied system was increased with increasing ADPODS concentration because of a very high HLB value of ADPODS. The system that formed the middle phase microemulsion at the lowest salinity of 0.19 wt% with the formulation of 0.5 % ADPODS, 5% AOT and 5% Span 80 was selected for further study.

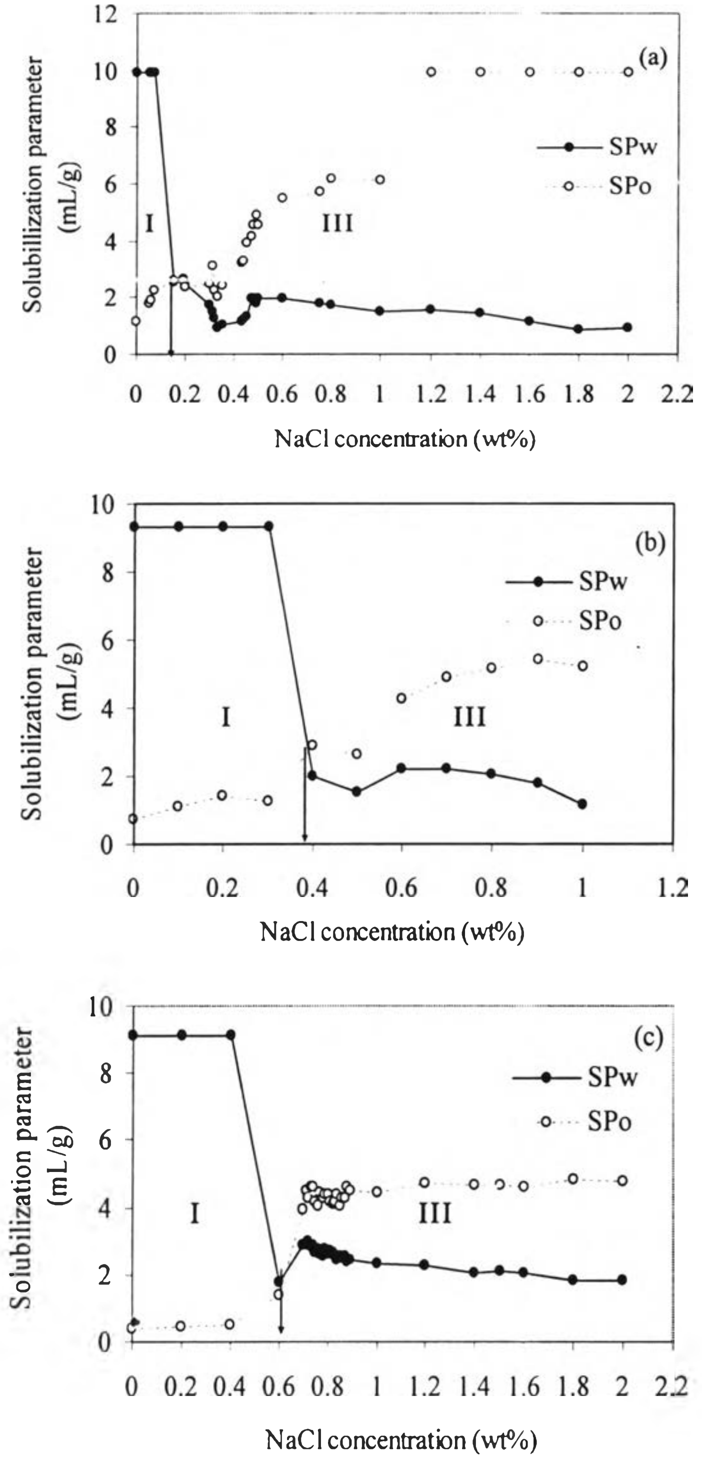

Figure 4.1 Solubilization parameter as a function of NaCl concentration at different surfactant formulations at the oil to water volumetric ratio of 1 to 1, (a) 0.5wt% ADPODS, 5wt% AOT, 5wt% Span 80, (b) 0.75wt% ADPODS, 5wt% AOT, 5wt% Span 80, (c) 1wt% ADPODS, 5wt% AOT, 5wt% Span 80.

4.1.2 Effect of AOT Concentration

The ADPODS concentration and the Span 80 concentration were fixed at 5wt% while the AOT concentration was varied from 3wt% to 5wt%. The effect of AOT concentration is illustrated in Figure 4.2. As the AOT concentration increases, the middle-phase microemulsion is formed at a lower salinity. Interestingly, an increase in AOT concentration from 4% to 5% resulted in shifting the phase transformation to Winsor type III since AOT has a moderate HLB value to enhance the formation of the middle phase.

4.1.3 Effect of Span 80 Concentration

To observe the effect of Span 80 concentration of mixed surfactant system on microemulsion formulation. Both of the ADPODS and AOT concentrations were fixed at 5 wt% while the Span 80 concentration was varied from 1 to 3 wt%. As seen in Figure 4.3, when the Span 80 concentration increases, a Winsor type III microemulsion appears at a lower salinity and the optimum salinity shifts to a higher value. It can be explained that according to all extremely low HLB value of Span 80, increasing Span 80 concentration leads to decreasing the HLB value of the system. A lower HLB value directly promotes phase transformation from Winsor type I to Winsor type III microemulsions as shown in the phase diagram (Figure 4.3). The system that formed the middle phase microemulsion at the lowest salinity of 0.5 wt% with the formulation of 0.5 wt% ADPODS, 5 wt% AOT and 3 wt% Span 80 which was selected for detergency experiment.

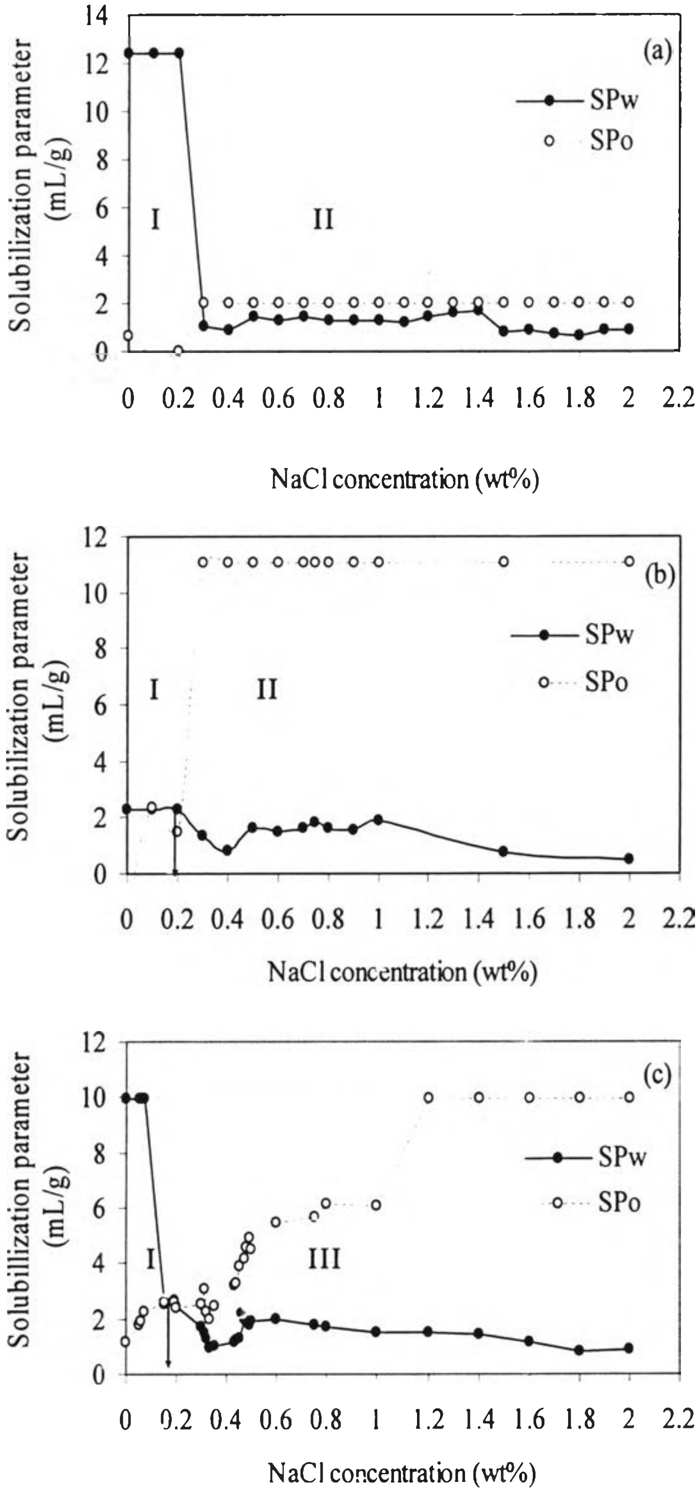

Figure 4.2 Solubilization parameter as a function of NaCl concentration at different surfactant formulations at the oil to water volumetric ratio of 1 to 1, (a) 0.5wt% ADPODS, 3wt% AOT, 5wt% Span 80, (b) 0.5wt% ADPODS, 4wt% AOT, 5wt% Span 80, (c) 0.5wt% ADPODS, 5wt% AOT, 5wt% Span 80.

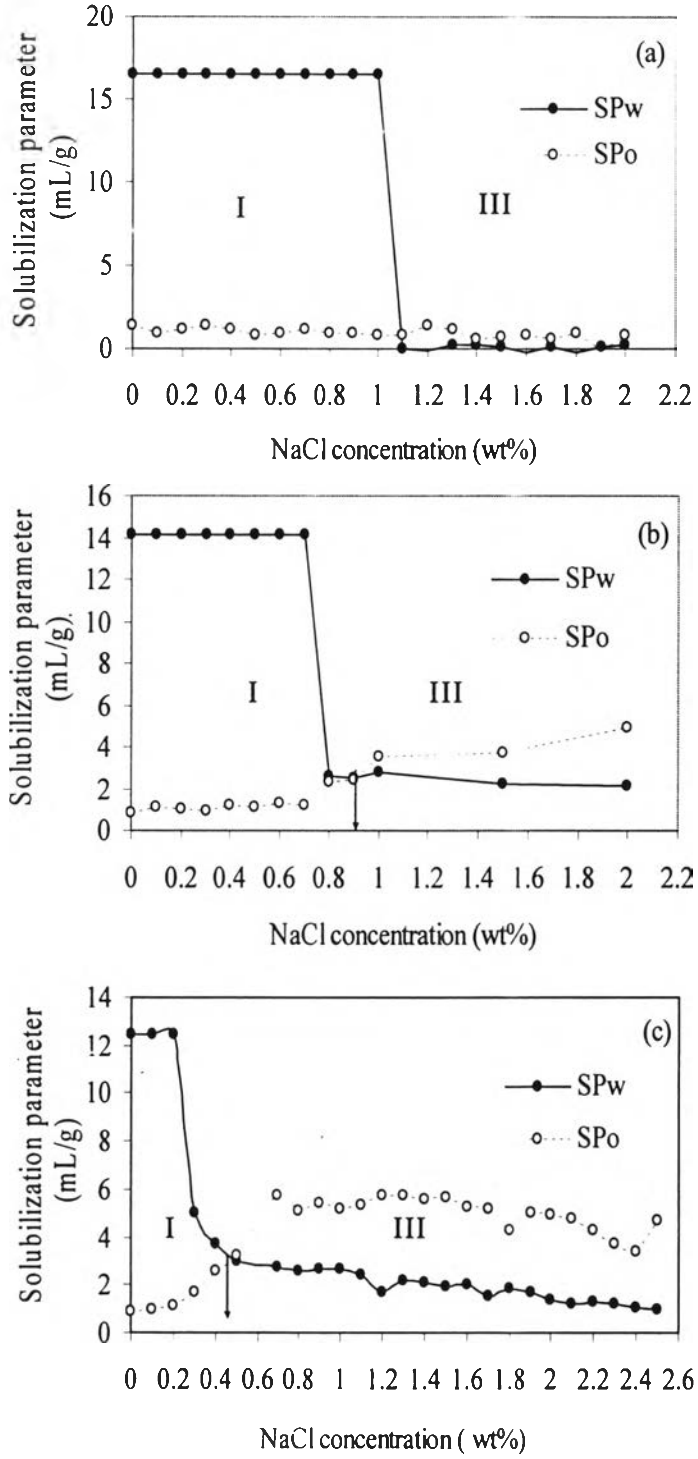

Figure 4.3 Solubilization parameter as a function of NaCl concentration at different surfactant formulations at the oil to water volumetric ratio of 1 to 1, (a) 0.5wt% ADPODS, 5wt% AOT, 1wt% Span 80, (b) 0.5wt% ADPODS, 5wt% AOT, 2wt% Span 80, (c) 0.5wt% ADPODS, 5wt% AOT, 3wt% Span 80.

4.1.4 Effect of NaCl

The effect of salt on microemulsion formation was studied by adding salt in the system as known as salinity scan. Salinity scan results for the motor oil system with different formulations are presented in Figures. 4.1-4.3. For any given composition of the mixed surfactants with low salinity, the system exhibited an oil in water microemulsion or Winsor type I microemulsion. With increasing salinity, the system tranformed from Winsor type I micremulsion to Winsor type III and then to Winsor type II microemulsion as a typically anionic surfactant based system since increasing salinity causes the system become more lipophilic or more surfactants moving out from the water phase to the oil phase.

As shown in Figure 4.1, the optimum salinity increases with increasing ADPODS concentration because adding ADPODS increases a HLB value of the system, the higher salinity required to reduce the system's HLB to form middle phase microemulsion. As can be seen in Figure 4.2, due to the low ADPODS concentration and the high concentration of Span 80 therefore HLB of this system is very low. When adding AOT increases HLB of this system leading to shift the system from Winsor type II to Winsor type III microemulsions. Interestingly, the optimum salinity was not affected by changing AOT concentration since AOT has a relatively HLB value, From Figure 4.3, the optimum salinity decreases with increasing Span 80 concentration since adding Span 80 reduces a HLB of this system leading to lowering salinity required to shift the system from Winsor type I to Winsor type III microemulsions.

4.2 Detergency Performance

For this part, the formulation was selected for detergency experiment consisted of 0.5wt% ADPODS, 5wt% AOT and 3 wt% Span 80. The standard polyester/cotton blend (65/35) was used as a testing fabric.

4.2.1 Effect of Surfactant System

Figure 4.4 shows the oil removal by the selected formulation at different salinities as compared to three single surfactant formulation and the commercial detergent. In this washing experiment, the active surfactant concentration was fixed at 0.119% according to the previous study (Parichat, 2005). All of pure Span 80 and AOT systems as well as the commercial detergent gave relatively low efficiency in oil removal for all range of studied salinities. For the case of pure ADPODS system, the oil removal was much higher than those of both AOT and Span 80 systems and the commercial detergent. Moreover, for the pure ADPODS system, the oil removal increased with increasing salinity. Interestingly, the selected formulation of 0.5 wt% ADPODS, 5wt% AOT and 3wt% Span 80 gave the highest oil removal for any given salinity. This is because the selected formulation consists of three surfactants having very high, moderate and very low value of HLB. As a result, this mixed surfactant system can provide high solubilization capacity for both oil and water leading to having a very low or ultralow IFT in the system. As known, a decrease in IFT will promote the oil removal as described in the previous work (Parichat, 2005).

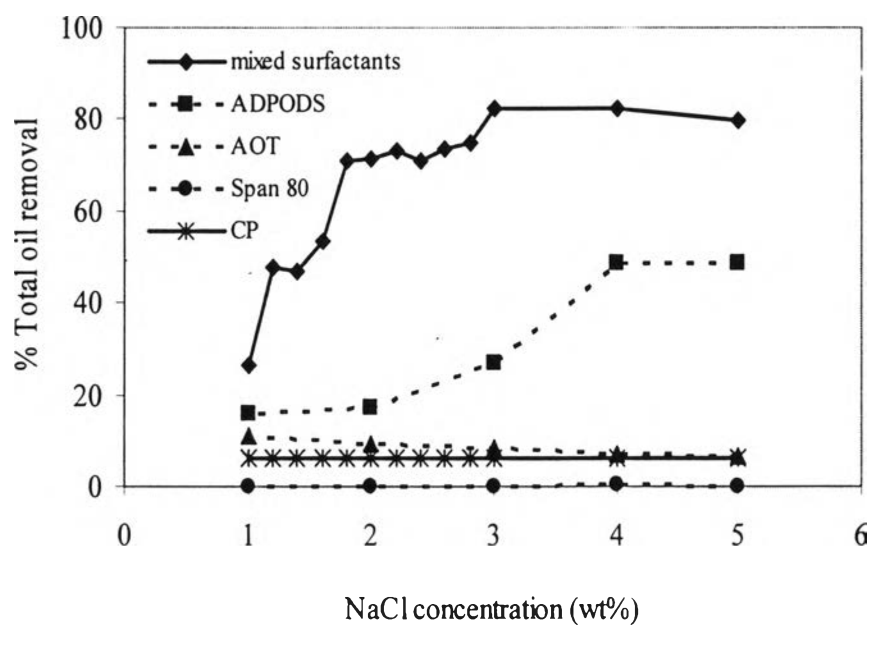

Figure 4.4 The effect of surfactant system (single and mixed surfactant system) on oil removal on polyester/cotton blend (65/35) at various salinities with the selected formulation at a total surfactant concentration of 0.119%.

4.2.2 Effect of Salinity

The effect of NaCl concentration on detergency performance was carried out by varying NaCl concentration at 0.119 % active concentration of the selected formulation; 0.5 wt% ADPODS, 5wt% AOT and 3 wt% Span 80.

In detergency experiments, the washing solution to oil ratio was much higher than the 1:1 ratio used in the phase studies. In addition, the washing solution in this study contained only 0.119 % active concentration of mixed surfactants. As a result, the equilibrium phase behavior in the washing bath may be different from the results of the phase studies conducted at an oil/water ratio of 1 to 1. Also, since phase behavior equilibrium time can be weeks or months as compared to a 20 min wash cycle in the detergency experiment as confirmed by the measurement of the high IFT values during the wash step as compared to those obtained from the phase studies (see Figure 4.5). Therefore to obtain an accurate salinity that forms middle phase microemulsion during the washing process, NaCl concentration was varied. Figure 4.5 shows the oil removal and the IFT between the washing solution and the oil as a function of salinity of the selected formulation. With increasing salinity of the system, the oil removal increased and beyond 3 wt% NaCl, the oil removal remained almost constant. It is very interesting to point out that the optimum salinity for the maximum oil removal in the washing process was much higher than that of the phase studies (3wt% versus 0.5wt%). This can be explained that in the washing process, there are two steps of washing and rinsing. During the rinse step, the spent washing solution is drained out and it is refilled with water. As a result, great reduction of both surfactant concentration and salinity occurs because of the dilution effect. Hence, an excessive amount of NaCi is needed to ensure the system to have a very low or ultralow IFT during the rinse step. As known from the previous work (Chantra, 2003 and Parichat, 2005), the oil removal in the rinse step is almost as high as that in the wash step for the system operated under the Winsor type III microemulsion condition.

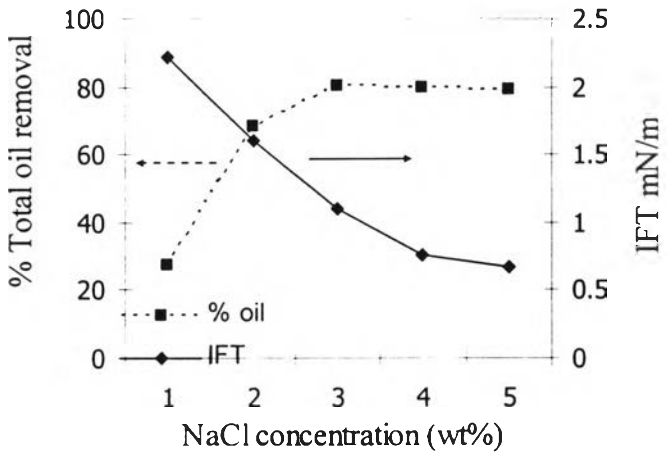

Figure 4.5 The total oil removal and IFT between the rinsing water and the oil as function of salinity using the selected formulation at 0.119% active surfactant.

4.2.3 Correlation between Surfactant Concentration and Oil Removal

In this study, only the ADPODS concentration was determined due to analytical instrument available. However, it is believed that for the concentration of ADPODS could be the same trend to the total concentration of the three surfactants. As expected, in Figure 4.6, an increase in salinity in the system decreases both of oil removal and the ADPODS concentration in the washing solution after the wash step since the IFT system decreases with increasing NaCl concentration resulting in increasing the spreading effect. As compare to the initial surfactant concentration of 0.119 wt% (≈1,190 ppm), most of the surfactants adsorb onto the fabric surface. The adsorption of the surfactants increased with increasing the salinity.

As being mentioned before, the wash step still has large amount of both of the oil and surfactants adsorbed onto the fabric surface. A rinse step is needed to further remove both of the oil and surfactants. Figure 4.7 and 4.8 illustrates the total oil removal and the ADPODS concentration in the rinse water at different salinity. The results indicated that not only most of the oil to be removed but also the surfactant desorption appear in the rinse step.

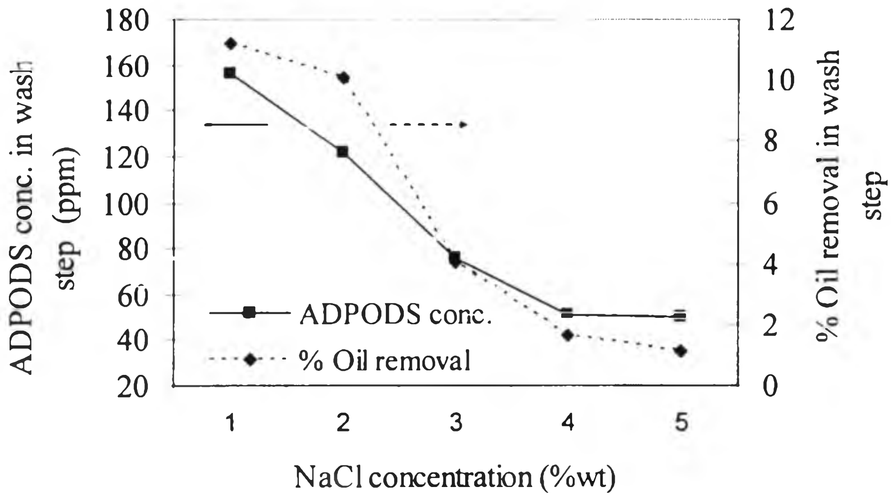

Figure 4.6 The relationship between total concentration of ADPODS in the washing solution after wash step and % oil removal in wash step.

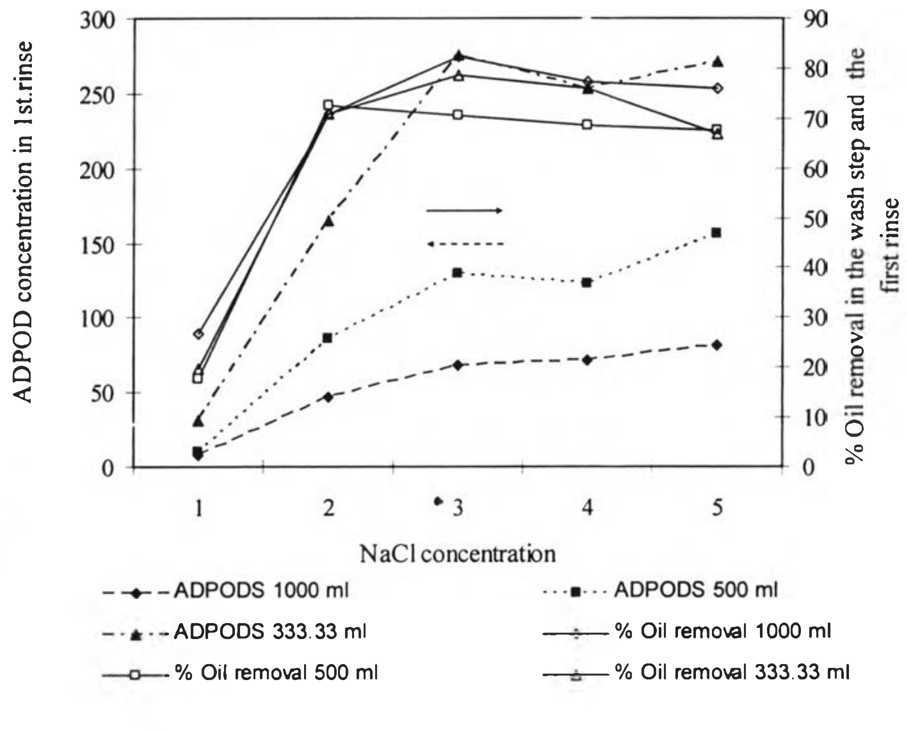

Figure 4.7 Total concentration of ADPODS after the first rinsing step and % oil removal in the wash step and the first rinse.

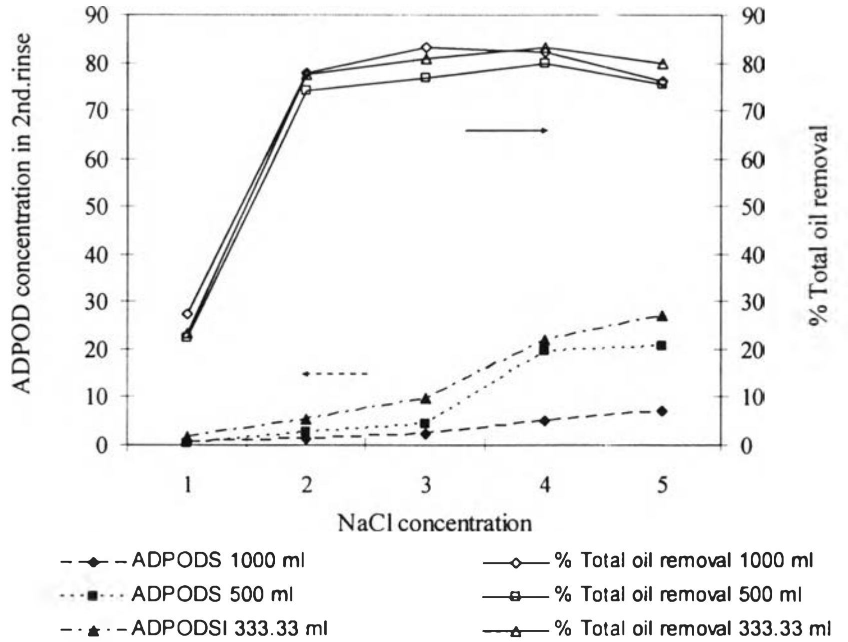

Figure 4.8 Total concentration of ADPODS after the second rinsing step.

4.2.4 Effect of Amount of Rinsing Water on Oil Removal

The oil removal of each step in detergency process is illustrated in Figure 4.9. From this result, it was found that the oil removal in the first rinse step was as high as that in the wash step. For considering the oil removal in first and second rinsing step at different volumes of rinsing water, the difference of the oil removal was obtained. These results show that the different amount of rinsing water can affect the detergency performance in the first and the second rinsing steps, From the Figure 4.9, it can be concluded that 333.33 ml of distilled water of rinsing step are sufficient for detergency process.

In addition, the total oil removal different volume of rinsing water was illustrated in Figure 4.10. From the result, the oil removal by the wash step was considerably lower than that by the rinse step for all three different ways of the rinse step. For first rinsing step with different amount of rinsing water, the total oil removal is the same. For considering the total oil removal with two rinse steps at different amount of rinsing water, it indicated that an amount of rinsing water was not affect significantly the total oil removal. From the results, two times of rinsing step with the lowest volume of rinsing water was recommended for operating any washing machine units.

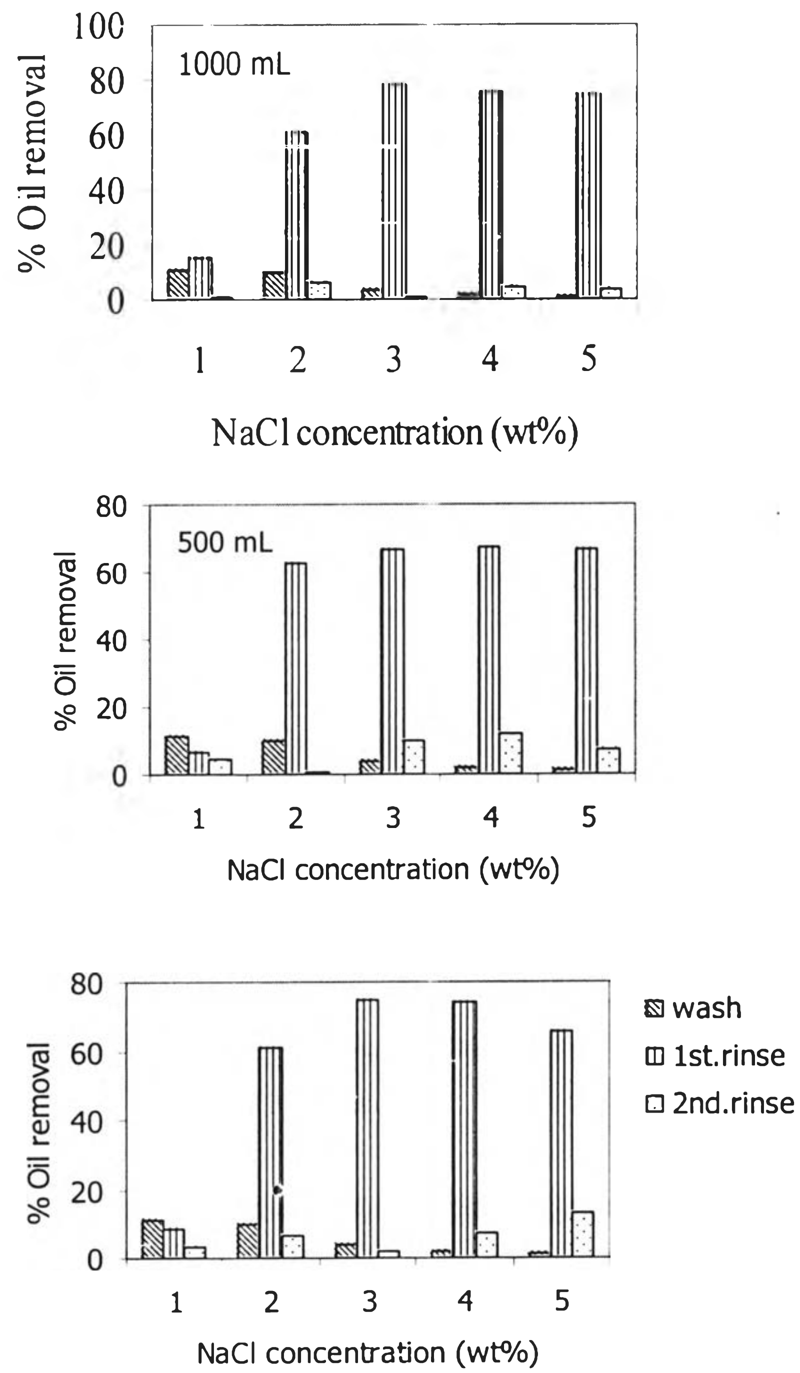

Figure 4.9 The relationship between NaCl concentration and oil removal on polyester/cotton blend (65/35) in each step at different amount of rinsing water with the selected formulation at 0.119% active surfactant concentration.

4.2.5 Correlation of Interfacial Tension and % Oil Removal

In this study, the selected formulation was measure the interfacial tension as varying salinity by using a spinning drop tensiometer, the measurement of interfacial tension was done in the washing step by measuring the interfacial tension between oil and washing solution.

Figure 4.10 shows the relationship between interfacial tension, NaCl concentration and % oil removal with the formulation 0.5 wt % ADPODS, 5 wt % AOT and 3 wt % Span 80. It was found that with increasing salinity, the interfacial tension between oil and washing solution slightly decreased but % oil removal extremely decreased in washing step. However, in Figure 4.11 shows a clear inverse relationship between the interfacial tension and accumulation oil removal.

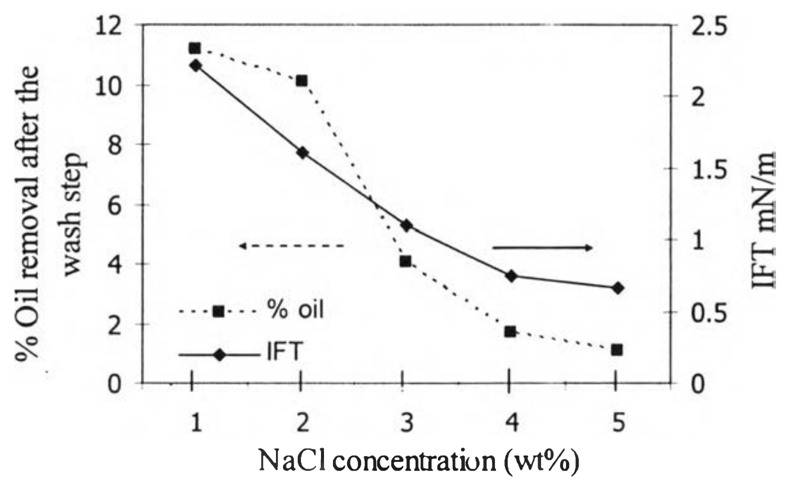

Figure 4.10 % oil removal after the wash step and IFT between the washing solution and the oil as a function of salinity in wash step using the studied formulation with 0.119% active surfactant concentration.

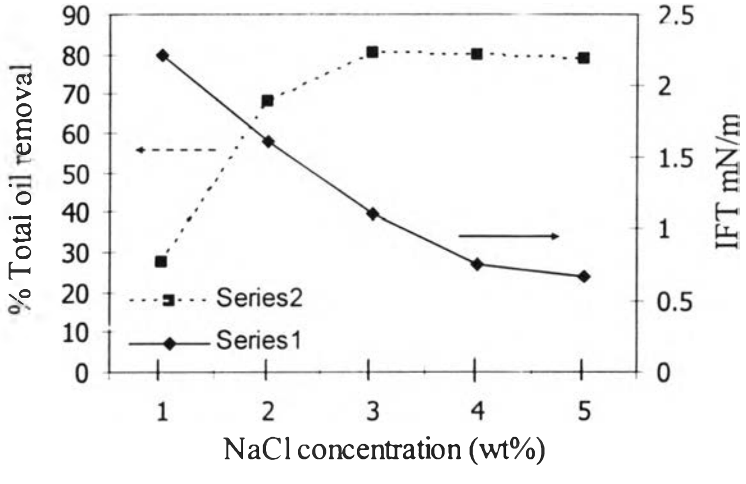

Figure 4.11 % Total oil removal and IFT between the washing solution and the oil as a function of salinity with two rinse steps(1000 mL of rinsing water for each rinse) using the studied formulation with 0.119% active surfactant concentration.