

CHAPTER IV
MICROEMULSION FORMATION AND DETERGENCY WITH
OILY SOILS: III. MECHANISM OF OILY SOIL REMOVAL

ABSTRACT: This study aim to investigate the correlation of oil removal efficiency and low interfacial tension (IFT) generated by microemulsion formation. A mixture of sodium dioctyl sulfosuccinate (AOT) and alkyl diphenyl oxide disulfonate (ADPODS) and sorbitan monooleate (Span 80) was selected as a detergent formulation to determine detergency performance for highly hydrophobic oils: hexadecane and motor oil. The maximum detergency was found where supersolubilization (SPS) occurs in a type I microemulsion region close to type III region and in a type III region. It was found that the oil removal in the rinse step was almost as much as in the washing step for the SPS and the type III regions. Results of the IFT values between washing solution and the oils before and after the wash step and the detergency resulted from different rinsing methods indicated the effect of low IFT on spreading of the oil on fabric substrate and hence give the clue for the detergency mechanisms.

KEY WORDS: microemulsion, detergency, alkyl diphenyl oxide disulfonate, low IFT, spreading effect, detergency, oil removal mechanism

INTRODUCTION

Microemulsion is thermodynamically stable with remarkable properties of low interfacial tension and high solubilization. Microemulsion system usually consists of at least three components namely; oil, water and surfactant. In a system that one parameter is changed and results in a decrease in HLB of the system, which is known as a 'scan' system, the transition from Winsor type I to type III and to type II microemulsions will be observed. The transitions are correlated to IFT of the system associated to the microstructure of microemulsion in each type. Figure 1 is a typical phase diagram of the transitions of a scan system. The region on the left hand side of Figure 1 is known as type I where oil in water (o/w) microemulsion exists along with an excess oil phase. In the middle phase or type III where microemulsion becomes bicontinuous structure, excess oil and water phases exist along with the middle phase microemulsion. When HLB decreases further, the transition from type III to type II occurs as shown on the right hand side, the microstructure transforming into water in oil (w/o) microemulsion, which exists with an excess water phase. The point in the middle phase where interfacial tension between the oil phase and the middle phase ($IFT_{O/M}$) equals interfacial tension between the middle phase and the excess water phase ($IFT_{M/W}$) is known as the minimum IFT or the optimum condition. At the region where type I close to type III is known as supersolubilization (SPS) region, is believed to have high solubilization and low IFT. From literatures, several papers (1,2,3) reported that at SPS region, micelle are 'swollen' and its solubilization capacity can be enhanced up to 1 order of magnitude higher than that of a regular micelle system. Because of high solubilization and low IFT in this supersolubilization region as well as being in o/w microemulsion phase, it is interesting to apply for detergency application.

There have been several research works to prove the correlation of the phase behavior of microemulsion system and detergency performance since 1970s (4,5,6,7,8,9,10,11,12). It has been known that the maximum detergency corresponds to the optimum condition in the middle phase. In a nonionic system with temperature scan, the point at the optimum is known as phase inversion temperature (PIT). For system with anionic surfactant, salt is commonly used as a scan variable and the salt concentration at the optimum point in the middle phase is known as

optimum salinity (S^*). This is due to the fact that at the optimum condition, the system has the lowest IFT. Dillan and coworkers (4, 5) explained the mechanism of oily soil removal from substrate influenced by interfacial tension between oil and washing solution. Solans and his coworkers (6,7,11) reported their research works on detergency result from the microemulsion system at low temperatures. They illustrated detergency efficiency as a function of temperature and electrolyte concentration which maximum detergency was found respond to PIT (Phase Inversion Temperature). Mori *et. al* (8) revealed the relationship between dynamic contact and detergency behavior of a nonionic surfactant system with triolien. Their work showed the relation of phase behavior at different temperatures with detergency performance. Azemar and Goel (9,12) reported similar results of the maximum detergency corresponding to the minimum interfacial tension. However, this concept was challenged by Thompson (10) who argued that maximum detergency was not always corresponded to minimum interfacial tensions. Instead, he proposed the concept of contact angle as well as cohesive and adhesive works between oil and fabric substrate to respond for the maximum detergency. Rybinsky (13) reviewed researcher works related to detergency and illustrate the surface chemistry in detergency. He proposed the interface processes involving in detergency.

In addition to the works on correlation of phase behavior and detergency performance, extensive studies on detergency mechanism have been conducted in last few decades. Roll-up or rollback mechanism is believed to play a major role in oily soil removal (14). Emulsification-solubilization (sometimes called snap-off) is also responsible for detergency process (15). In the roll-up mechanism, reduction of interfacial tensions between the oil and the bath (water) and the substrate (solid) and the bath result in an increase in the contact angle of the attached oil droplet with the substrate until it reaches 180° ideally and as a result the oil droplet detaches from the substrate. In the emulsification or snap-off mechanism, incomplete detachment of the elongated droplet occurs and the oil detached is emulsified. Molecular solubilization of the oil into micelles can also be a removal. Figure 2 illustrates comparatively the roll-up and snap-off mechanisms. Since attainment of ultralow oil-water interfacial tensions and high oil solubilization capacities are integrally related to microemulsion formation (16), current studies of oily soil detergency

mechanisms have been focused on the relation between microemulsion formation and detergency.

In this present study, in addition to confirm the correlation of low interfacial tension with detergency result, we further explored that the supersolubilization region is also able to yield sufficient efficiency in cleaning. From our previous study (17), it was found that the mixed surfactants of 3% AOT, 2% ADPODS and 2% Span80 was able to form microemulsion with motor oil. In addition, our studied system showed high solubilization and low IFT at SPS region. As a consequence, the objectives of the present study were to compare the detergency results at SPS and at the optimum regions. In addition, the correlations of IFT and oil removal as well as the mechanism of detergency were investigated.

MATERIALS AND EXPERIMENTAL PROCEDURE

Materials. The alkyl diphenyl oxide disulfonate (ADPODS) used in this research was a commercial grade anionic surfactant supplied by Dow Chemical Co. known as Dowfax 8390 (Midland, MI, USA). Dioctyl sodium sulfosuccinate (AOT) was purchased from Fisher (Fair Lawn, NJ, USA) in 100% solid. Sorbitan monooleate (Span 80) was obtained from ICI Uniquema Co. (Wilmington, DE, USA). Hexadecane (99 % purity) and Oil red O (Solvent Red 27, CI. No. 26125) were purchased from Aldrich (Milwaukee, WI, USA). The motor oil used in the study was a commercial motor oil for gasoline engine, type SAE 10W-30 (Castrol GTX). All chemicals were used as received without any further purification. Fabric for detergency test, a standard unsoiled blend polyester/cotton (65/35) was purchased from Test Fabrics Co. (Middlesex, NJ, USA.)

Phase behavior study. Phase studies of both model oils were carried out by adding an aqueous surfactant solution and oil at a volumetric ratio of unity in flat bottom screw cap tubes. The solutions were well shaken and left in a water bath for equilibration at 25°C. IFT between phases at equilibrium was measured by a spinning drop tensiometer (model 500 Texas University).

Detergency experiment. Soiled swatches were prepared by soaking in dyed oil and were left to dry over night at room temperature. Before soiling, the oil was dyed by an oil-soluble Oil red O following the method purposed by Goel (18). The

washing solution used in the detergency experiment was prepared to have a total active surfactant concentration of 0.12% by diluting the selected formulation which contained 3%AOT, 2%ADPODS and 2% Span 80. The washing experiment was performed by a Terg-O-Tometer, in 1,000 mL of washing solution, 20 min wash, 3 min first rinse and 2 min second rinse with D.I. water. Four swatches were washed in each bucket for one cycle as replication. In order to examine the correlation between phase behavior and detergency performance, salt was added to washing solution to make up the solution to have the same salinity as the phase studied formulation. Detergency performance was determined by percentage of oil removal. The oil contents of pre-wash and post-wash swatches were extracted by 2-propanol and measured the absorbance at 520 nm by a UV/VIS Spectrophotometer (Hewlett Packard 8452 A). A commercial liquid detergent product under trade name "Tide" of Procter & Gamble Co., USA, was also selected for the detergency experiment for comparison. It was purchased from a supermarket in Norman, OK in April, 2002.

Measurement of dynamic interfacial tension. To gain a better understanding about oil removal mechanism during washing process, dynamic IFT between the washing solution and the dyed oil were measured. The washing solution prepared same concentrations as used for laundry experiments was a dense phase while the dyed oil was a light phase in the measurement. The diameter of the oil drop was measured versus time to be used to calculate dynamic IFT..

RESULTS AND DISCUSSION

Phase Behavior with Salinity Scan. Phase studies of the mixed surfactants of 3% AOT, 2% ADPODS and 2% Span 80 with hexadecane and motor oil were scanned by salt concentration. The transitions from type I to type III and then to type II of Winsor's were observed in the range from 2.5 to 22.5% NaCl, for hexadecane where only transition from type I to type III was observed for the system with motor oil in the same range of salinity scan (10). The equilibrium IFT at SPS and at the optimum for both oils is shown in Table 1. For a surfactant solution to oil ratio at unity, the salinities at SPS and at the optimum for hexadecane were around 5% and 8% NaCl, respectively. For the system with motor oil, the salinities of 8% at SPS region and

16% NaCl at optimum region are higher than those of the hexadecane system since motor oil is more hydrophobic than hexadecane.

Dynamic IFT during Washing Process. From the results of dynamic IFT of our studied system as shown in Figure 3, for any given salinity, it shows spontaneous reduction of the dynamic IFT with time. When salinity was increased, it took longer time for the system to reach steady state. Raney and coworkers (19,20) studied the dynamic IFT value of hexadecane both with nonionic surfactants and a blend nonionic and ionic surfactants. Their studied system tended to take longer time to reach equilibrium as compared to the present system. In our studied system with hexadecane, the IFT value is significantly reduced in a great magnitude at 8.0% NaCl as illustrated in Figure 3. It indicates that at this salinity, the studied system already reaches the middle phase or Winsor's type III microemulsion in comparison to the phase behavior as shown in Table 1 as also described in our previous work (17).

Similar result was found in the system with motor oil; however, the largest magnitude of reduction occurred at 16% NaCl (Figure 4). For both studied oils, the difference between the dynamic IFT value and the minimum IFT at the optimum salinity indicates a shift of phase transition. This shift of the phase transition is resulted from the spontaneous formation of the middle phase or Winsor's type III microemulsion at the interface. As compared to the IFT values obtained from the phase behavior results and during the wash step, the supersolubilization region is believed to be occurred at 12 % NaCl and the middle phase appears at 16% NaCl for motor oil (see Figure 4).

Detergency Performance. The oil removal and IFT are plotted as a function of salinity for hexadecane and motor oil as illustrated in Figure 5 and Figure 6 respectively. The washing experiment with the commercial liquid detergent product (CP) at the same active surfactant concentration of 0.12% was also carried for comparison. The graphs show a good correlation between IFT and detergency for both oils. When the IFT value was lowered, the higher detergency performance was found as expected. From the result, it can be concluded that the detergency efficiency is strongly dependent on salinity in the system. In addition, for both oils, the oil removals at the SPS region and at the middle phase were found insignificantly

different. The reason for the indifferent washing results in both regions may be explained by the fact that the solubilization capacity in the SPS region is almost as high as that in the middle phase, even though the IFT from these two regions may be substantially different in one order of magnitude. It is also interesting to note that the studied system of both oils almost any salinity showed a better performance than the commercial liquid detergent product (CP).

Figures 7 and 8 show the IFT profiles with different salinities in the washing solution before and after wash step for hexadecane and motor oil, respectively. As mentioned before, for both studied oils, IFT values were found to decrease remarkably with increasing salinity. As shown in Figure 7, after wash step for hexadecane removal, an IFT value increases significantly at the SPS and Winsor's type III (middle phase) regions. Similar result was found for the system with motor oil (Figure 8). The result gives a clue that the spent washing solution at both SPS and a middle phase regions have a lower surfactant concentration left in the solution resulted from the surfactant adsorption on the fabric surface. The decrease in surfactant concentrations directly attributes to increasing IFT after wash step. Further experiment on determination of surfactant concentrations in the washing solution before and after the wash step was carried out to verify this hypothesis. In this study, only the ionic surfactants (AOT and ADPODS) were determined due to analytical instrument available. However, it is believed that the concentration of these two surfactants could be the same trend of the total concentration of the three surfactants. As expected, the concentrations of the total surfactants (AOT and ADPODS) at the SPS and at the middle phase were found substantially lower than those at low salinities (see Figure 9.). This result can be explained that at the SPS and the middle phase regions (5% and 8% NaCl for the system with hexadecane and 12% and 16% NaCl for the motor oil), significant amounts of surfactants from the washing solution adsorb on the fabric surface as monolayer because of the counter ion effect on ionic surfactant adsorption.

As mentioned earlier, salinity has a strong effect on detergency performance. Hence, another experiment was conducted for the hexadecane system with allowing the salinity of the rinsing water to be the same as that of the washing solution. Interestingly, for the rinse step with brine for both at the SPS and the middle phase

regions, the hexadecane removal fails dramatically as compared to D.I. water to be used in the rinse step (see Figure 10). The result obtained from the present study will be used to support over proposed mechanisms later.

In order to understand how oil is removed during the cleaning process, the oil removals by both wash and rinse steps were determined for both model oils with the studied formulation at the SPS and the middle phase regions in comparison to the commercial detergent (CP). As can be seen from Table 2, for both model oils with our studied formulation, the oil removal efficiency by the rinse step is as high as that by the wash step while for the commercial detergent, the oil removal mostly occurs in the wash step.

Proposed Mechanisms of Detergency. Since oily soil removal from fabric in the washing process directly involves interfacial energy and contact angle between fabric or substrate and the oil, wetting and spreading must be taken into consideration in order to obtain better understanding about the detergency mechanisms of oily soil removal.

In general, when a soiled fabric is placed in surfactant solution, the contact angle increases with decreasing IFT. The contact angle (θ) can be described by a well known Young's equation:

$$\cos \theta = (\gamma_{sw} - \gamma_{os}) / \gamma_{ow} \quad (1)$$

where γ , s, w, and o represent interfacial tension, solid, water, and oil, respectively.

In the wash step, some amount of oil is simply removed by the mechanical force from agitation. When a soiled swatch is placed in the bath and agitation starts, the surfactant molecules move around and coalesce with each other to form micelles as well as contact to the fabric surface and the surface of oil. However, in this work the contact angle in detergency process cannot be measured. Therefore, the proposed mechanisms for detergency will be explained on the basis of interfacial tension or interfacial energy change.

Under the low IFT regions (e.g. SPS and middle phase), the oil removal is believed to be occurred by emulsification-solubilization mechanism relating to an increasing of contact angle between oil and fabric as well as the spreading of the oil on the substrate. Figure 11 illustrates the microscopic feature of proposed

mechanism in each step of the washing process. At the time is less than zero before a swatch is placed in the washing bath, oil drops cover the surface of swatch (see Figure 11a). At the SPS and the middle phase regions, an IFT value between the oil and the water is lowered because some surfactant monomers associated with counter ions from the added salt are coadsorbed on the fabric surface to form the close pack monolayer with head out and tail in as shown in Figure 11b. The contact angle is increased since both values of γ_{sw} and γ_{ow} are lowered by the presence of surfactant as shown in Eq.(1). As described before and as shown in Figure 3 and Figure 4, the wash step is considered as dynamic process with respect to a progressive decrease in IFT. As a result, the oil droplet can be initially removed from the fabric as known the snap-off mechanism since the contact angle initially increases. However, the IFT value of the system will further decrease to a vary low or ultralow level in an order magnitude of 10^{-1} to 10^{-2} mN/m leading to another important phenomena of spreading. The tendency of the oil droplet to spread on the fabric surface depends on the condition of interfacial energy as described in Equation (2):

$$S = \gamma_{sw} - \gamma_{ow} - \gamma_{os} \quad (2)$$

where S is the spreading coefficient. The oil will spread spontaneously when S is \geq zero. However, the measurement of IFT value between liquid (oil) and solid (fabric) is impractical and inaccurate. In most cases, contact angle measurement is preferable. Therefore, the spreading coefficient has to be derived in terms of contact angle by the combination of Eq. (1) and Eq. (2), which will obtain:

$$S = \gamma_{ow} (\cos \theta - 1) \quad (3)$$

For our studied system at high salinities where γ_{ow} becomes ultralow, it can make the spreading coefficient approach zero even though the value in the blanket of Eq. (3) is not equal to zero. Hence, spreading of the oil tends to occur spontaneously and it is likely that the oil may further penetrate into fiber bundle when the system is operated at both the SPS and the middle phase regions. Marmur (21) proposed the term 'wicking' to define this type of spreading that liquid spreads into a porous

substrate driven by capillarity force. This results in more difficulty for the oil to be detached due to continuity penetration of the oil in the fiber bundle.

In the rinse step with D.I water (without salt), some counter ions adsorbed between surfactant monomers both in micelles and in the close pack monolayer at the interface will be simply dissolved into the D.I. rinse water. This results in some surfactant monomers of the close pack monolayer at the interface to be desorbed due to the increasingly repulsive force between the ionic heads causing an increasing interfacial energy (IFT). As a consequence, the spreading effect is reduced leading to the oil to be detached from the fabric surface and become droplets. Then, the oil droplets are easily detached from the fabric by agitation force (Figure 11c). Carroll (22,23) clearly explained that this type of oily soil removal mechanism relates to the formation of soluble complexes between the detergent and the soil and may result in the gradual dissolution of the soil even though it still adheres to the substrate. In addition, he mentioned further that in case the process involves solubilization of the soil by micelles, the success of this mechanism is dependent on the complexes viscosity. Compared to our case, the complex may refer to microemulsion. This may be an additional reason to explain why the detergency result of the studied system in the middle phase was insignificant different from the SPS region even though it provided a much lower IFT. This is because the Winsor's type III microemulsion of our studied system is obviously more viscous than that of the SPS region.

NOMENCLATURE

AOT	dioctyl sodium sulfosuccinate
ADPODS	alkyl diphenyl oxide disulfonate
IFT	interfacial tension (mN/m)
SPS	supersolubilization
Om	water in oil microemulsion
Wm	oil in water microemulsion
S	spreading coefficient

D.I. deionized water

Subscripts

O excess oil phase

W excess water phase

M middle phase

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Table 1

Salinity and IFT at Supersolubilization and at Optimum Regions of Both Oils with the Studied Formulation

<i>Regions</i>	<i>Oils</i>	
	<i>Hexadecane</i>	<i>Motor oil</i>
At supersolubilization		
Salinity (% NaCl)	5.0	8.0
IFT (mN/m)	0.009	0.022
At optimum		
Salinity (% NaCl)	8.0	16.0
IFT (mN/m)	0.004	0.014

Table 2

Oil Removal (%) in Each Step of the Washing Process at Two Low IFT Regions Compared to the Commercial Liquid Detergent (CP)

<i>Formulations</i>	<i>Oil removal (%)*</i>	
	<i>Wash step</i>	<i>Rinse step</i>
Hexadecane		
At SPS	89	11
At middle phase	55	45
CP	96	4
Motor oil		
At SPS	47	53
At middle phase	32	68
CP	82	18

Note *% removal in each step is based on total oil removal

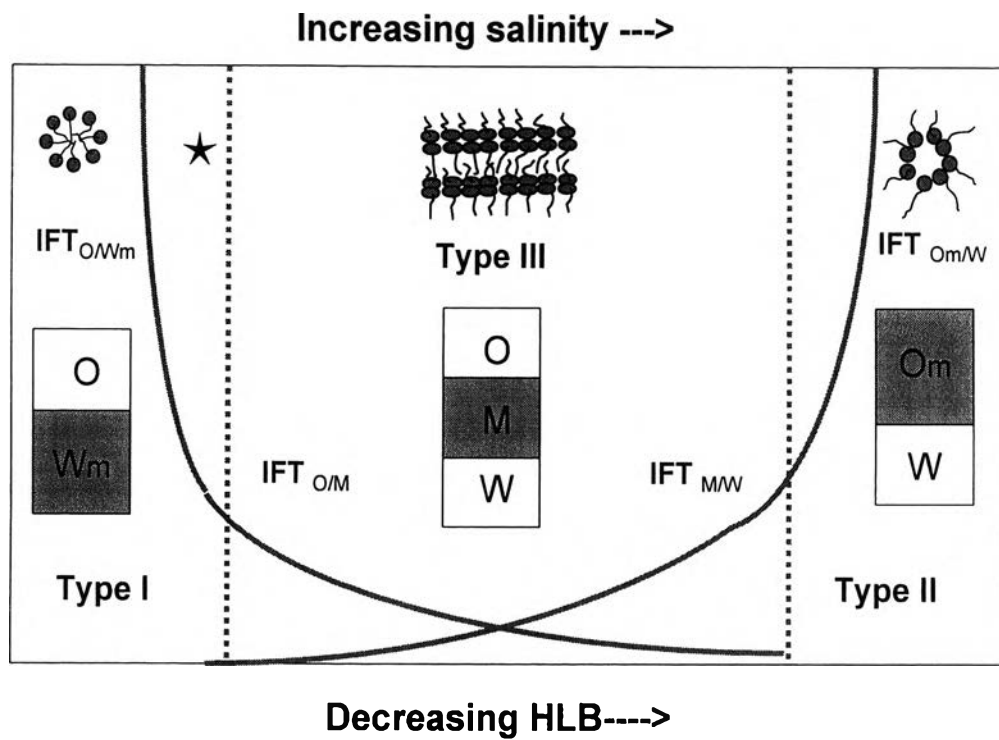


FIG. 1 Phase behavior showing IFT as a function of scanning variables, where O is oil; W is water; M is middle phase; Wm is O/W microemulsion; Om is W/O microemulsion; and \star is supersolubilization region.

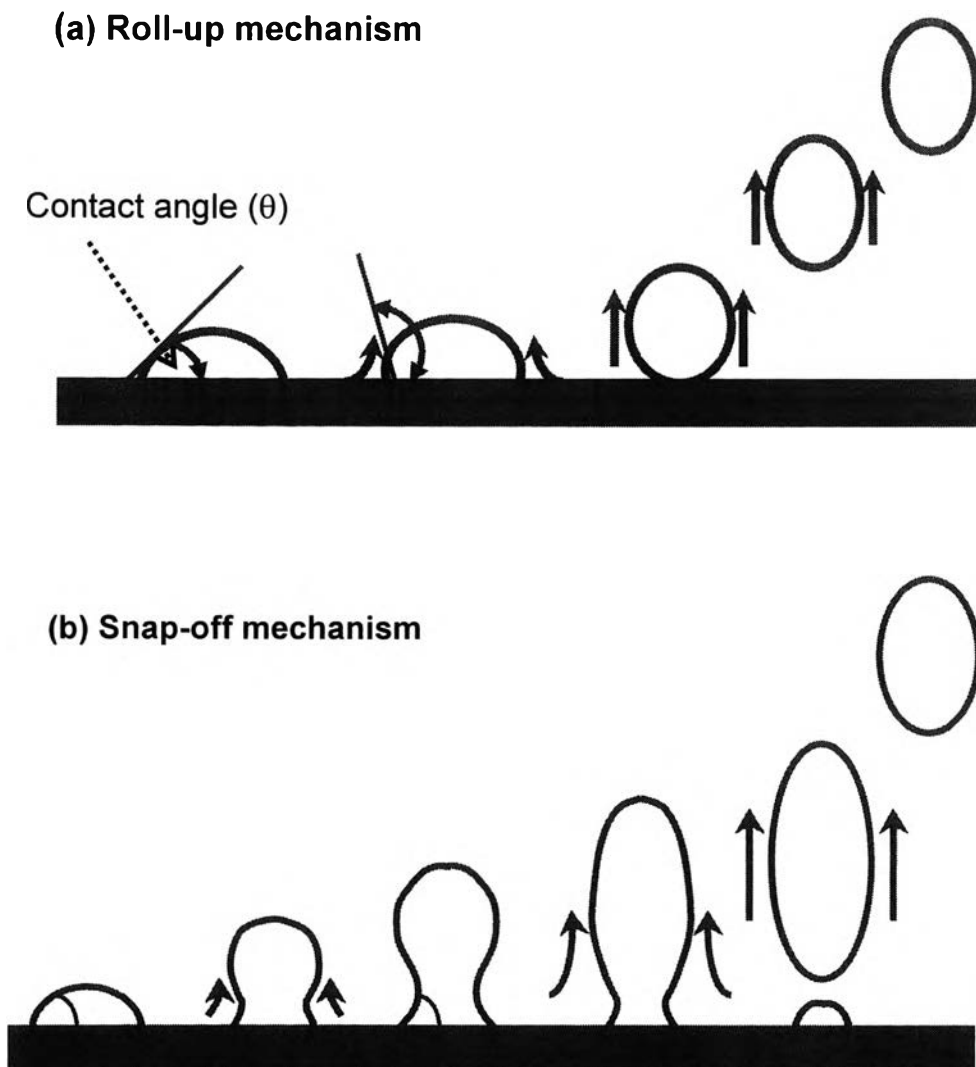


FIG. 2 Oily soil removal from substrate by (a) roll-up mechanism and (b) snap-off or emulsification-solubilization mechanism (14).

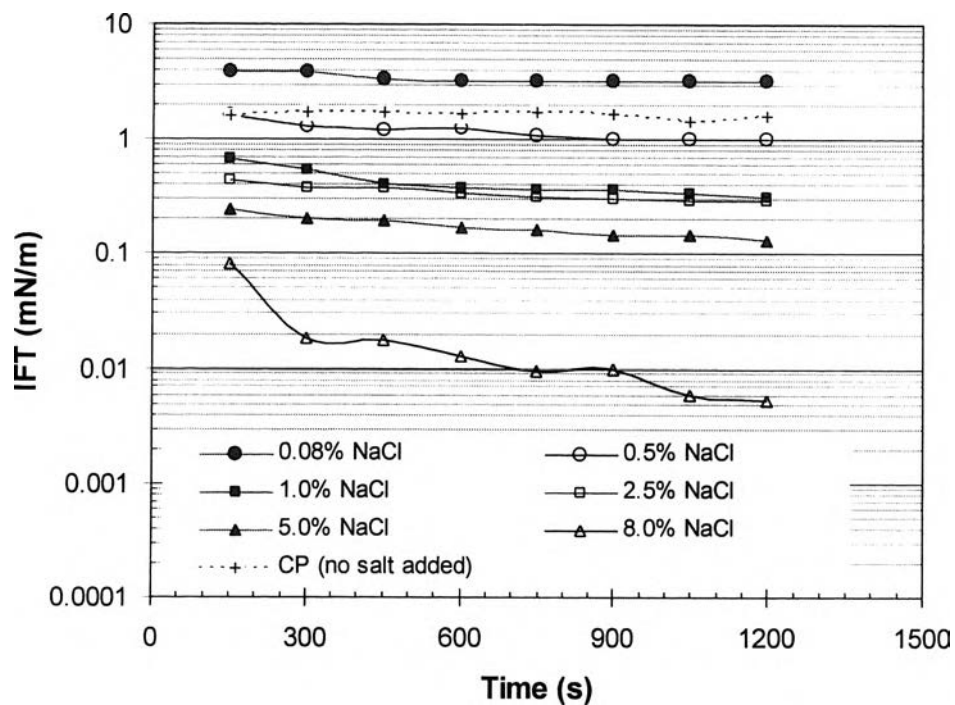


FIG. 3 Dynamic IFT of hexadecane and washing solution having 0.112 % active surfactant concentration at different salinities

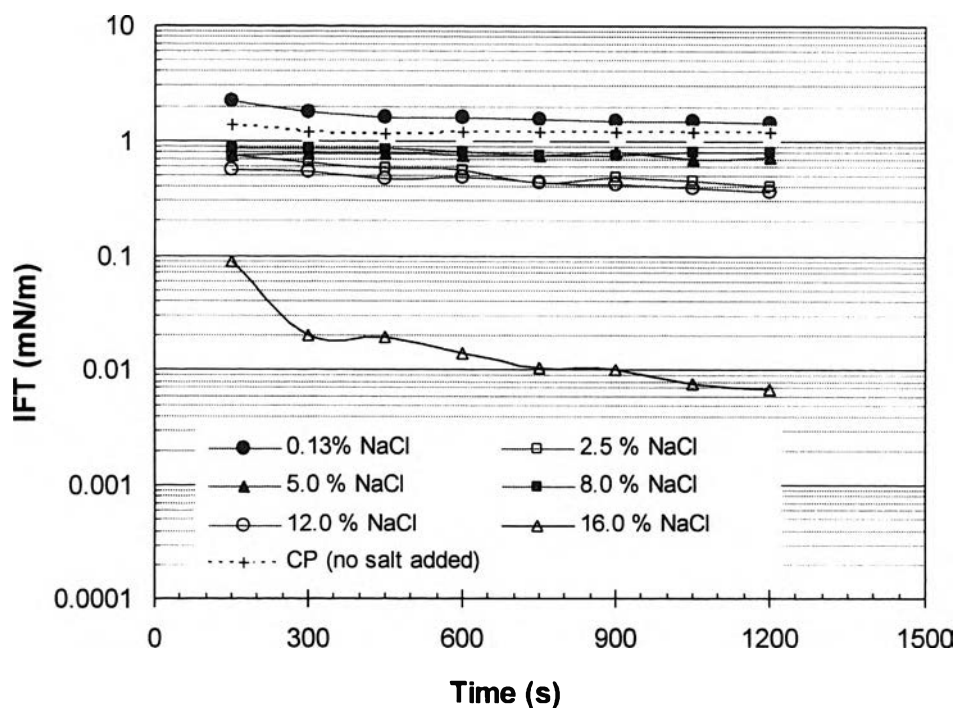


FIG. 4 Dynamic IFT of motor oil and washing solution having 0.112 % active surfactant concentration at different salinities

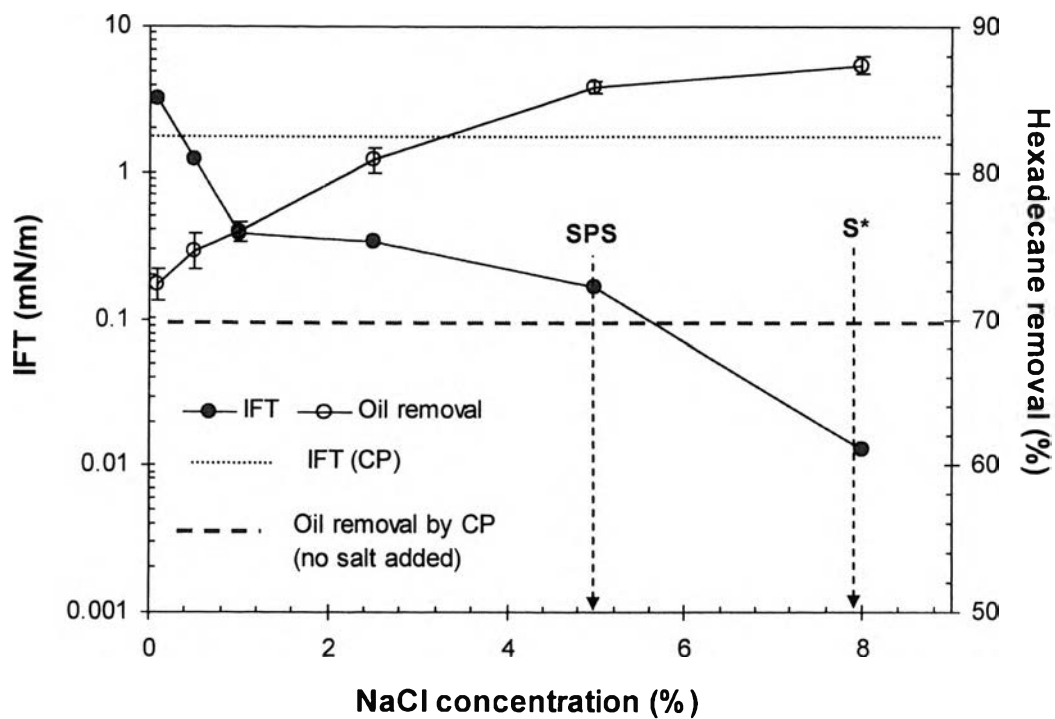


FIG. 5 Hexadecane removal and the interfacial tension between oil and washing solution at various salt concentrations using our formulation compared to commercial liquid detergent (CP); where SPS is the supersolubilization region and S^* is the optimum salinity at equilibrium from phase studies (total surfactant concentration = 0.112%)

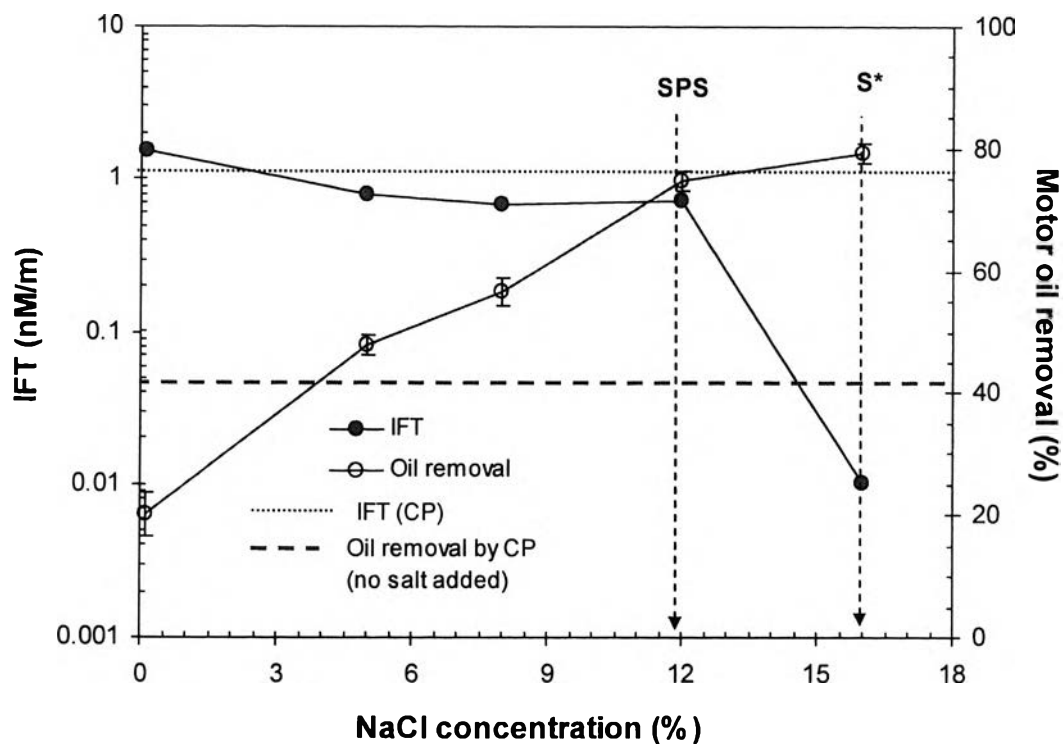


FIG. 6 Motor oil removal and the interfacial tension between the oil and washing solution at various salt concentrations using our formulation compared to commercial liquid detergent (CP); where SPS is the supersolubilization region and S* is the optimum salinity at equilibrium from phase studies (total surfactant concentration = 0.112%).

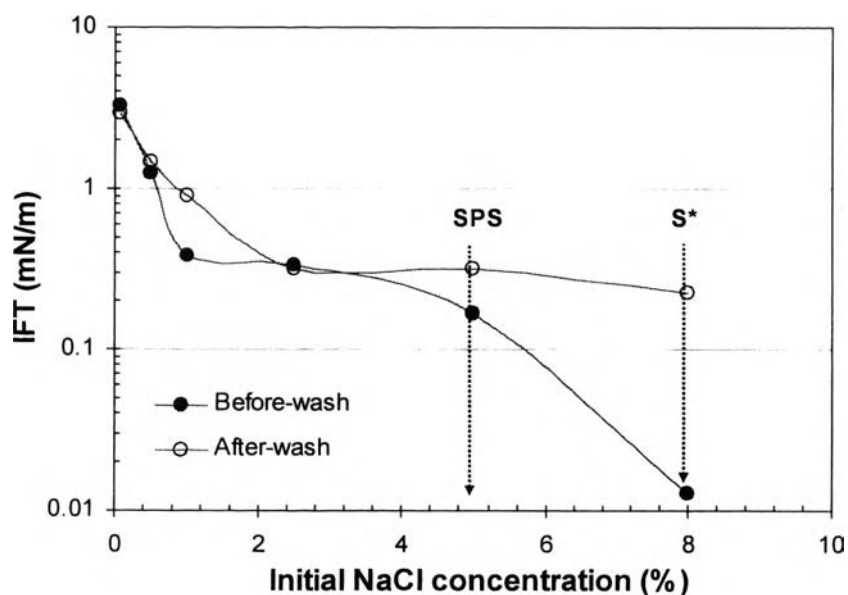


FIG. 7 IFT values of the washing solution and dyed oil before and after wash step for hexadecane at different salinities using our formulation; where SPS is the supersolubilization region and S* is the optimum salinity at equilibrium from phase studies (total surfactant concentration = 0.112% in the washing solution).

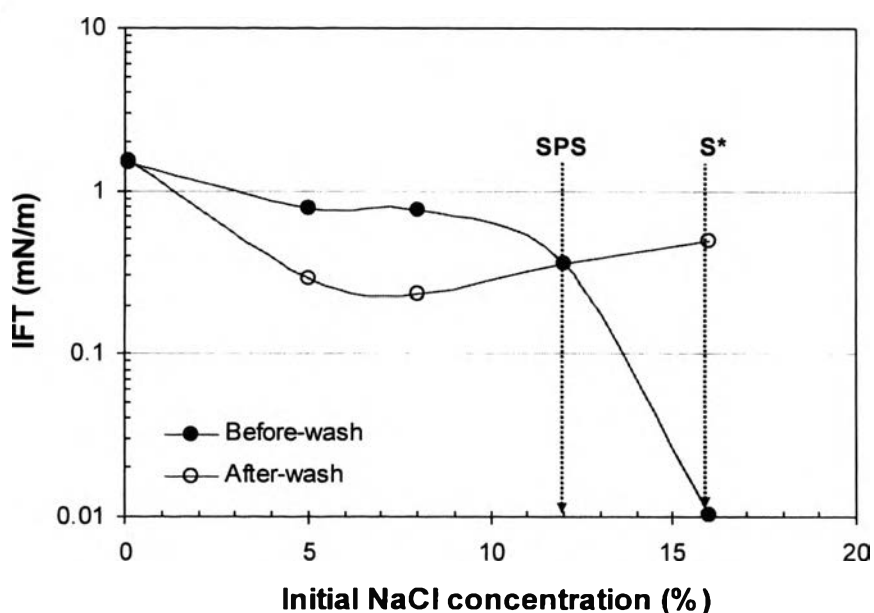


FIG. 8 IFT values of the washing solution and dyed oil before and after wash step for motor oil at different salinities using our formulation; where SPS is the supersolubilization region and S* is the optimum salinity at equilibrium from phase studies (total surfactant concentration = 0.112% in the washing solution).

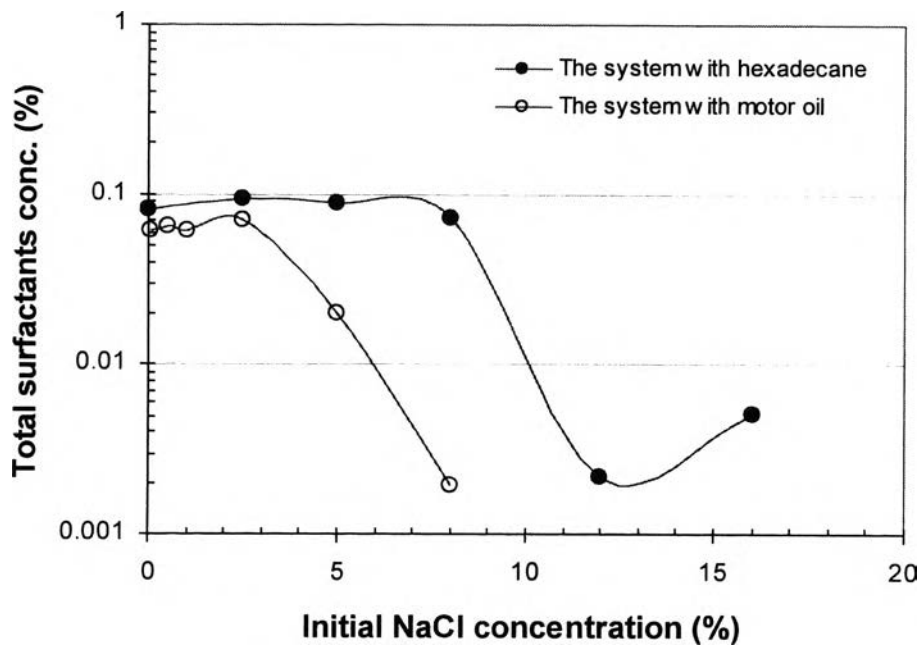


FIG. 9 Total concentration of AOT and ADPODS in the washing solution after wash step

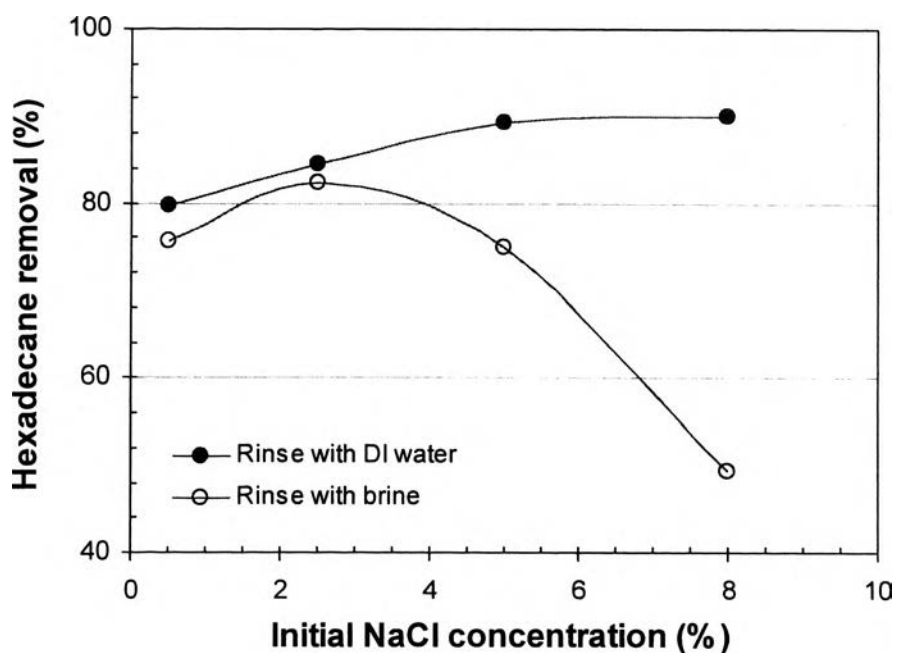


FIG. 10 Hexadecane removal in the washing process of with different salinities of rinse water compared to rinsing with D.I. water

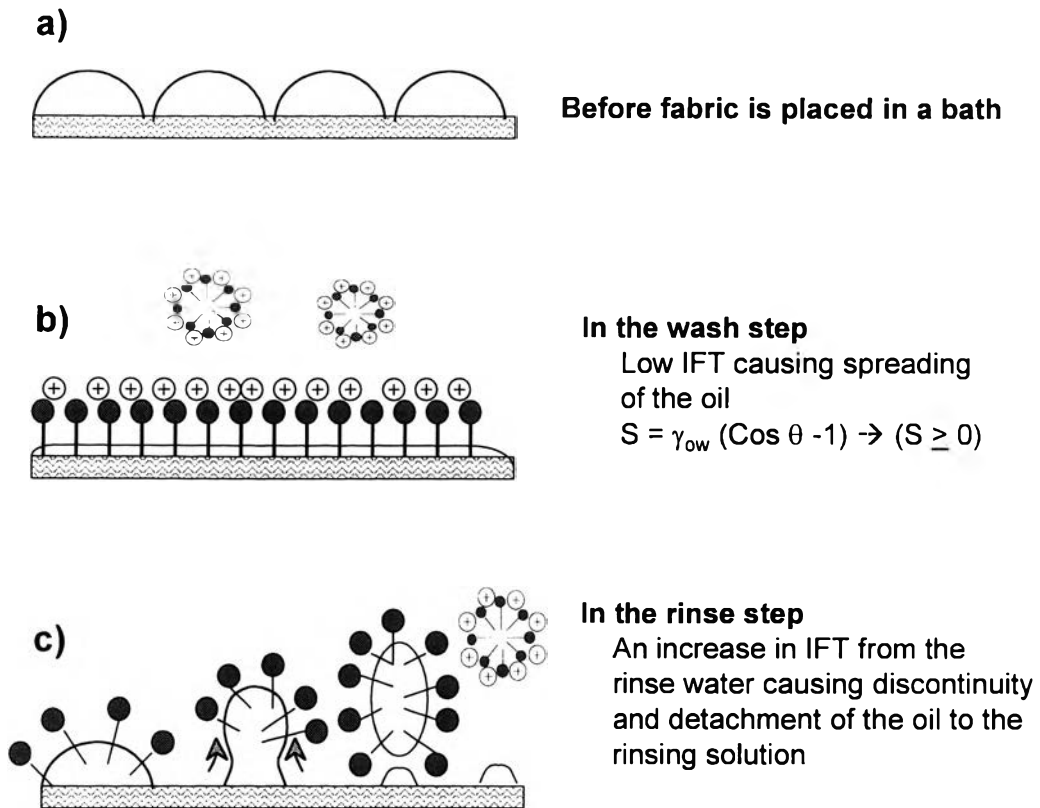


FIG. 11 Proposed mechanism of detergency at high salinities (the supersolubilization and the middle phase regions)