



CHAPTER III

OIL REMOVAL FROM COTTON AND POLYESTER FABRICS USING MICROEMULSION SYSTEMS

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ABSTRACT Winsor Type I-III microemulsion phase behavior was studied for detergency application as a function of salinity. The microemulsion systems consisted of commercially available DOWFAX 8390, bis-ethylhexyl sulfosuccinate (Aerosol-OT), sorbitan monolaurate (Span 20), and propylene glycol. This research was conducted to examine the relationship between the phase behavior and the detergency of hexadecane and motor oil staining on cotton and polyester fabrics in the laundering process. The system exhibiting supersolubilization, as well as the middle-phase microemulsion systems, were studied for detergency. Spectrophotometric and reflectance measurements were used to quantify soil removal after washing. Maximum detergency was found to correspond to the optimal salinity for hexadecane but not for motor oil. From the detergency results, the middle-phase microemulsion system was more efficient than the supersolubilization system for hexadecane staining, whereas for motor oil staining, both systems did not differ significantly in terms of solubilization capacity and detergency performance. The detergency results revealed that soil removal of both hexadecane and motor oil from cotton fabric was better than for soil removal from polyester fabric

INTRODUCTION

Microemulsions are thermodynamically stable, homogeneous, mixtures of water and oil which are stabilized by a fairly large amount of surfactant. Microemulsion phases are defined to be in equilibrium with either excess oil or excess aqueous phase or both. Winsor classified such systems as Type I: oil-in-water systems with

excess oil; Type II: water-in-oil systems with excess water; Type III: “middle phase” systems with an excess of both water and oil and Type IV which is defined as a single-phase micellar solution (1,2). To promote phase transitions from Type I to Type II via Type III (and vice versa), altering the effective hydrophilic-lipophilic balance (HLB) of the surfactants is required as illustrated in Figure 1 (3). A Type I-III-II transformation can be achieved by varying one of the system variables (e.g., water/oil ratio, electrolyte, surfactants, cosolvent, organic additives, oil, temperature). For ionic surfactants, increasing electrolyte concentration reduces the electrical interactions of the ionic head groups causing the surfactant change from hydrophilic to lipophilic (4). The optimum interfacial tension (IFT) corresponds to equal volumes of water and oil solubilized in the microemulsion phase. At this salt concentration, the interfacial tension between oil and microemulsion equals that between water and microemulsion, and the corresponding salt concentration is referred to as the optimal salinity (5). With increase in temperature, the degree of hydration of a POE nonionic surfactant decreases and the surfactant becomes more lipophilic. The temperature at which the surfactant phase solubilizes equal volumes of water and nonpolar material is known as the phase-inversion temperature (PIT). In this research, the system salinity was adjusted while keeping other variables constant which is commonly known as a salinity scan (3).

The development of surfactant systems for oily soil removal has received much research attention in the past years. Formulating middle-phase microemulsion systems has been the popular approach to obtain superior detergent efficiency due to their notable advantages which are ultra-low oil-water interfacial tension, ultra-high solubilization capacity and spontaneous formation when the components are brought into contact (6).

It has been generally accepted that oily soils are removed mainly by the “roll-up” mechanism in which the contact angle that the liquid soil makes with the substrate is increased by adsorption of surfactant from the cleaning bath. The second mechanism is emulsification, in which partial drop detachment occurs when it is subjected to the hydraulic currents of the bath and the oily soil is suspended in the bath by solubilization mechanism. Nevertheless, the mechanisms of detergency are still controversial. The solubilization-emulsification mechanism was proposed to be pre-

dominant for oily soil removal from synthetic fabrics under low-temperature washing conditions for nonionic surfactant system (7). By using videomicroscopy technique, formation of intermediate phases (microemulsions or liquid crystals) was observed to be capable of solubilizing considerable oil (7,8).

Correlation of phase behavior and oily soil removal was studied by many researchers using nonionic and nonionic/anionic surfactant blends. For nonionic surfactants, *n*-dodecyl tetra- and pentaoxyethylene monoethers ($C_{12}E_4$ and $C_{12}E_5$) with *n*-hexadecane as the soil, the optimal detergency occurred when the temperature was near the PIT. It was also demonstrated that the soil components had a dramatic effect on the PIT. To attain high soil removal at the washing temperature, a minimum quantity of polar material in the soil is required (9). For enhanced oily soil removal, the optimum ratio of alcohol ethoxylates and alcohol ethoxysulfates surfactants was also determined. And the correlation between phase inversion temperature and optimum detergency was established (10). In addition, the removal of artificial sebum soil as a function of EO moles of the lauryl alcohol ethoxylates in combination with sodium salt of C_{12} linear alkyl benzene sulfonate (NaLAS) was investigated in the presence of soda ash and sodium tripolyphosphate (STPP). It was confirmed that incorporation of NaLAS requires compensation of the overall HLB by adding more hydrophobic (lower EO) alcohol ethoxylates (11). Adding electrolytes in the nonionic surfactant was also conducted to study the detergency at low temperature, it was found that PIT was depressed when the system contained NaCl, $CaCl_2$, Na_2SO_4 , sodium citrate and STPP (12).

However, it was observed by some researchers that detergency was not enhanced at low interfacial tension conditions of the phase inversion region due to the formation and deposition of a middle oil/water/surfactant phase (13). Moreover, middle phase microemulsions may be accompanied by high viscosity (14) that could cause difficulty in soil removal. By considering Figure 1, the increased solubilization within the Type I near to Type III boundary region is referred as supersolubilization because swollen micelles at this point have a larger capacity for oil solubilization than normal micelles (3). Therefore, it is worthwhile to formulate an efficient micellar system, as close to the Type I-III boundary as possible (still inside the Type I area) to avoid the deposition of the middle phase.

According to the Chun-Huh relationship (15), solubilization parameter can be related to interfacial tension as shown in equation [1]:

$$S = \sqrt{\frac{C}{IFT}} \quad [1]$$

where, S = solubilization parameter; C = constant; IFT = interfacial tension.

From equation [1], minimum oil/water interfacial tension would be expected to correspond to maximum solubilization and hence optimum detergency might be expected at this point.

Alkyldiphenyl oxide disulfonates (ADPODS) has been produced under the trade name of DOWFAX surfactants. Due to their resistance to sorption and precipitation losses, DOWFAX components have been evaluated extensively for surfactant enhanced subsurface remediation (3, 16-20). However, their solubility enhancements are not as effective as others, various means to maximize contaminant solubilization were developed. Increasing alkyl chain length of DOWFAX components to adjust the micelle size produced slight increase in contaminant solubility. In combination with nonylphenyl ethoxylates, isobutanol, electrolyte, middle-phase microemulsions were produced, and the contaminant solubilities increased by one to two orders of magnitude over DOWFAX surfactants alone (19). Formulating alcohol-free ADPODS middle-phase microemulsion was achieved by using hydrophobic dioctyl sodium sulfosuccinate as a cosurfactant together with octanoic acid and L-tartaric acid as hydrotropes. The organic solubility was greatly enhanced when compared to micellar ADPODS surfactant systems (3).

Since particulate soils almost always occur with oily soils, the cleaning effect for both soils can be optimized by using mixture of anionic/nonionic surfactants in this research. In this study, formulating Type I to Type III microemulsion using Dowfax 8390 with bis-ethylhexyl sulfosuccinate (Aerosol-OT or AOT) and sorbitan monolaurate (Span 20) was studied for detergency purposes. It has been confirmed that surfactant structure is an important parameter to obtain a middle phase system therefore AOT was selected because it is one of the few surfactants that is able to form microemulsion (20) and Span 20 was incorporated in the formulation

in order to lower the HLB of the system. Propylene glycol as a hydrotrope was added to prevent phase separation and to minimize liquid crystal phase regions (22). Hexadecane was used as a model oily soil because it can represent the linear hydrocarbon part of the sebum (23) whereas motor oil represents a more hydrophobic, difficult-to-remove, soil. Practical detergency performance of the systems found to have high solubilization was evaluated and related to the interfacial tension and solubility parameter.

EXPERIMENTAL PROCEDURES

Materials. DOWFAX 8390 (36% active; mixture of phenyloxide mono-hexadecyl disulfonate and phenyloxide dialkyl disulfonate) and propylene glycol USP/EP were obtained from Dow Chemical Co. (Midland, MI). AOT (98% purity) was purchased from Fluka Chemie AG (Buchs, Switzerland). Span 20 was supplied by E.A.C Chemicals Ltd. (Bangkok, Thailand). Motor oil (Castrol GTX, SAE 10 W-30) produced by Castrol Heavy Duty Lubricants Inc. was also supplied by Dow Chemical Co. Hexadecane (99% purity) and Oil red O (Solvent Red 27, CI. no. 26125) were purchased from Aldrich Chemical Co. (Milwaukee, WI). Sodium chloride was purchased from Ajax chemicals (NSW, Australia). Butan-1-ol (99.4 % purity) and chloroform (99% purity) were obtained from Asia Pacific Specialty Chemical Ltd. (NSW, Australia) and BDH Laboratory Supplies (England) respectively. All chemicals were used as received without any further purification. For detergency test, cotton and polyester fabrics (100%) were purchased from normal commercial outlets.

Phase behavior studies. Microemulsion formation was carried out in a 20 mL test tube using DOWFAX 8390, AOT, Span 20 along with propylene glycol and NaCl. Equal volumes of aqueous solution and oil (5 mL each) were added in a test tube with Teflon screw caps. Each test tube was then shaken gently by hand for 3 min and equilibrated in a temperature-controlled circulating water bath at $30 \pm 0.1^\circ\text{C}$ until equilibrium was reached. Type I-III microemulsions and the volume of each phase were visually monitored by using a cathetometer, model TC-II, from Titan Tool Supply Inc. attaching to the digimatic height gauge, Model 192-631, obtained from Mitutoyo with 0.002 inch accuracy. The surfactant solubilization capacities were calculated in terms of solubilization parameter (5). The interfacial tension mea-

surements of the two and three phase systems were measured by using a Krüss spinning drop tensiometer (SITE 04, Krüss GmbH, Hamburg).

Fabric preparation. The pre-washed fabrics were cut into 10 x 10 cm swatches with the warp and weft direction carefully marked to assure that all specimens in the same test series were stained and graded with the same fabric orientation.

Soiling procedure. In this research, incorporation of an oil-soluble dye-tracer in the oily soil was adopted for determination of oily soil removal (24). Approximately 0.1 g. of oil-soluble dye (Solvent Red 27) having $\lambda_{\max} \sim 520$ nm in 100 mL of the oil (hexadecane or motor oil) was prepared for use as colored soil for the detergency experiments. The solution was filtered until clear. The colored soil was deposited onto the center of the fabric using an Eppendorf micropipette following the method as described in ASTM D 4265 (21). Each fabric swatch contains 23 mg of soil (1.35% of fabric weight). The soiled swatches were aged in a dark cabinet at room temperature for 1 day before the detergency test.

Laundering procedure. A Terg-O-Tometer (model: 7243ES, from Instrument Marketing Services, Inc., Fairfield, NJ) was used for washing. Three soiled swatches were washed in 1,000 mL of 0.16% w/w surfactant solution with a water hardness of 150 ppm $\text{Ca}^{2+}/\text{Mg}^{2+}$ (in a ratio of 2:1). This gave a surfactant to soil ratio of approximately 23.2:1, surfactant to cloth ratio of approximately 1:3.2, and cloth to wash-liquor ratio of approximately 1:196. The washing cycle lasted 10 min at 30°C, and two 3-min rinsing cycles followed at the same temperature. All samples were air-dried overnight before analyses.

Color measurements. Color changes of the fabrics upon soiling and washing were measured with a GretagMacbeth™Color-Eye®7000A spectrophotometer (Macbeth, A Div. of Kollmorgen Corp., New Windsor, NY). Prewash and postwash reflectance were recorded at 520 nm which is the characteristic wavelength for the dye. The difference in ΔR between the reflectance values of soiled and washed test fabrics was used to evaluate the detergency results (26,27). Readings were taken from the center front and back of each swatch along the warp direction and averaged for each treatment. Three layers of self-fabric were used below the test specimen when making color measurements.

Determination of residual oil. The quantity of residual soil was extracted from the fabric with butan-1-ol. UV/VIS spectrometer Lambda 20 (Perkin Elmer) was used to quantify the dye content in the solution. The residual concentration of the soil was calculated from the calibration curve of the control soil solution. To assure that the dye and the oily soil are removed by surfactant solutions in the same proportion in which they are loaded on the fabrics, the validation of this technique was done for both motor oil and hexadecane as described by Goel (22). The peak intensities of the recovered soil and the control soil solution were measured at 520 nm.

RESULTS AND DISCUSSION

Phase behavior studies. The surfactant concentration of Dowfax/AOT/Span 20 were 1.2:3.6:4.8, % wt by vol and 2:6:8, % wt by vol for hexadecane and motor oil, respectively. For these formulations, it is difficult to get homogeneous solution because of the inclusion of Span 20 which is insoluble in water. Therefore, replacing water by hydrotrope (propylene glycol) to prevent phase separation was required. It was found that at salinity values below 0.8 %, phase separation of surfactant solution occurred due to the low water solubility of AOT. Thus, salinity scans were started from 0.8 % wt/vol of NaCl and terminated when the middle-phase microemulsions were formed.

Scanning with NaCl produced the Type I-III phase transition (Figure 2) owing to the decrease in electrostatic charges which are screened by increasing counterion concentration. For hexadecane, the color of aqueous solution for Type I system changed from clear colorless to light blue translucent to hazy blue whereas for motor oil, the hazy light yellowish color was observed instead of light blue because the yellowish color of motor oil masked the blue color. The change in color indicated that surfactant micelle size increases due to higher oil incorporated into micelles (3), at this point it is referred to as supersolubilization. Supersolubilization systems were attained at 1.0 and 0.8 % w/v of NaCl for hexadecane and motor oil, respectively.

With an increase in propylene glycol concentration, lower oil solubilization was obtained for both hexadecane and motor oil systems as shown in Figure 3. On

the other hand, the optimal salinity was higher with increasing propylene glycol for motor oil but not significant different for hexadecane. It is noted that with different propylene glycol content, the salinity at which supersolubilization occurred was similar, only the optimal salinity was different. The decrease in oil solubilization is attributed to the incorporation of propylene glycol into the surfactant layer (22) and thus surface packing of the surfactant decreases. Because the solubilizing power of the surfactant depends on the number of the surfactant molecules per unit area of interface (2), therefore higher propylene glycol content leads to decrease in solubilization parameter for the oil. Another important observation was that macrophases occurred with motor oil which took longer time to equilibrate than the system with hexadecane which took only 1 day to reach the equilibrium. This may be because motor oil contains additives giving high viscosity-emulsion formation in detergent solutions (28). For motor oil, it took 1 month to equilibrate for the system with 15% propylene glycol and 1 ½ months for system with 10% propylene glycol.

In this study, solubilization parameter was used to determine the solubilization for oil and water, SP_o and SP_w have been defined for oil and water, as $SP_o = V_o/M_s$ and $SP_w = V_w/M_s$, where V_o , V_w , and M_s are the volumes of oil, water, and mass of surfactant, respectively. At optimum, where three conjugate phases coexist, the micellar phase contains equal volumes of oil and water. Therefore, $SP_o = SP_w = SP^*$, where SP^* is the solubilization parameter at optimum.

It was apparent that the oil type affected the oil solubilization capacity of the surfactants. Solubilization efficiency of the surfactants with hexadecane which is less hydrophobic than motor oil was almost twice the value for the system with motor oil as summarized in Table 1. We demonstrated that maximum oil solubilization was obtained with middle-phase microemulsion systems which is in good agreement with other studies (3,19,20). However, the solubilization for the oil (SP_o) did not differ greatly between supersolubilization and middle-phase microemulsion systems for motor oil. The systems exhibiting the highest solubilization parameters (systems a and c) were selected for interfacial tension measurement and detergency test.

Figure 4 illustrates the change in interfacial tension between $IFT_{m/o}$ and $IFT_{m/w}$, when the salinity was raised, $IFT_{m/o}$ was seen to decrease, while $IFT_{m/w}$

increased for both systems. The point where these two curves intersect inside the Type III region is defined as the optimum point and the value of IFT at the intersection where $IFT_{m/o} = IFT_{m/w}$ is denoted as IFT^* . For both motor oil and hexadecane, the optimal salinity obtained from the relationship between the solubilization parameter and salinity (see Table 1) was quite close to the one obtained from the plot between the interfacial tension and NaCl concentration as indicated in Table 2.

Correlation of phase behavior and detergency. In these initial studies, dosage of the surfactant solution was varied to evaluate the detergency. It was found that the cleaning performance was quite low and not different at each NaCl concentration. Therefore, the critical micelle concentration (CMC) was determined by measuring surface tension of the detergent solution at different concentrations. It was observed that the CMC was 0.16% surfactant for the system for motor oil and 0.10% for hexadecane. For comparison purpose, the same surfactant concentration was utilized in washing process which was 0.16%. Removal of hexadecane and motor oil from 100% cotton and 100% polyester fabrics were plotted versus %NaCl. As expected, for both fabrics soiled with hexadecane, highest performance in soil removal (Figure 5) was achieved at 1.6% NaCl which is the optimal salinity according to Figure 3. This finding indicates that micelles formation is required for enhancing soil removal. The largest difference in reflectance (ΔR) at 520 nm before and after washing was also observed at 1.6 % NaCl for the cotton fabric and 1.5% NaCl for polyester fabric. This finding infers that optimal salinity yielding the optimal interfacial tension corresponded closely to the optimal salinity yielding maximum detergency. Several authors related detergency maxima to phase inversion condition and some used composition. Results of our study are comparable to those of other researchers who found that the optimized system provides the highest hexadecane removal at optimum temperature or PIT in mixed ionic/nonionic surfactants system (10). Furthermore, the agreement between the optimum composition and optimum detergency was also observed (11-13).

Middle-phase microemulsion systems provided the highest detergency with 69.8% and 42.2% soil removal versus somewhat lower detergency of supersolubilization system with 62.7% and 33.8% soil removal from cotton and polyester fabrics, respectively. Ultralow interfacial tensions ($\sim 10^{-3}$ mN/m) at the optimal salinity pro-

bably plays an important role in the detergency enhancement by facilitating emulsification of oil and/or microemulsion phases into the surrounding water as reported by Miller and Azemar et al., (7,12).

Figure 6 shows the results of detergency with motor oil, the optimal detergency was found at 2.0 % salinity instead of 3.3% which is the optimal salinity. As observed in phase behavior studies that viscous middle-phase occurred, therefore this may make the soil removal more difficult than hexadecane. However, the difference in soil removal at 2.0% and 3.3% NaCl was only 0.5-0.7%, while the differences in soil removal between maximum detergency and supersolubilization systems were 3.7 and 4.7% for cotton and polyester fabrics, respectively. The reason for the small difference may be due to the insignificant difference in IFT throughout the salinity scan as shown in Figure 4(b). In addition, the IFT of this system was approximately 10^{-2} mN/m which may not be low enough to obtain high oil removal. Very low IFT might be required to achieve superior soil removal as suggested by Thompson (13). Moreover, Bourrel and Schechter (2) also state that the very low IFT is on the order of 10^{-3} mN/m.

For both oils, the dependence of fabric nature on detergency was observed. Hydrophobic oil tends to adhere strongly on the nonpolar substrate therefore it was more difficult to remove oily soil from polyester than from cotton which agrees with the results of Chi (28). In addition, the change in the color of the oil-soiled fabric as determined by ΔR correlated well with the actual amount of oil remaining on the fabric.

ACKNOWLEDGEMENTS

The author (Porntip Pattayakorn) is deeply grateful to Unilever Thai Holdings, Ltd. for providing financial support for her study. Valuable advice on the work given by Prof. David A. Sabatini at the University of Oklahoma is gratefully acknowledged. Thanks are also due to Siam Singhapuntu and Nittaya Srisuwankul at Unilever Thai Holdings, Ltd. for help in detergency test.

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TABLE 1**Comparison of Different Microemulsion Systems at 30°C.**

System	Surfactants Dowfax/ AOT/ Span 20 (% w/v)	Propylene glycol (% w/v)	Salinity (% w/v)	Oil	Oil solubility (mL/g)	Description
a	1.2:3.6:4.8	15	1.00	hexadecane	2.38	Supersolubilization
a	1.2:3.6:4.8	15	1.59	hexadecane	3.17	Middle-phase
b	1.2:3.6:4.8	20	1.00	hexadecane	1.95	Supersolubilization
b	1.2:3.6:4.8	20	1.61	hexadecane	2.61	Middle-phase
c	2:6:8	10	0.80	motor oil	1.48	Supersolubilization
c	2:6:8	10	3.30	motor oil	1.63	Middle-phase
d	2:6:8	15	0.80	motor oil	1.36	Supersolubilization
d	2:6:8	15	4.50	motor oil	1.40	Middle-phase

TABLE 2

Comparison of interfacial tension and Salinity of Different Microemulsion Systems at 30°C.

System	Surfactants DOWFAX/ AOT/ Span 20 (% w/v)	Propylene glycol (% w/v)	Salinity (% w/v)	Oil	IFT _{mo} (mN/m)	Description
a	1.2:3.6:4.8	15	1.00	hexadecane	0.0137	Supersolubilization
a	1.2:3.6:4.8	15	1.52	hexadecane	0.0049	Middle-phase
c	2:6:8	10	0.80	motor oil	0.0158	Supersolubilization
c	2:6:8	10	3.10	motor oil	0.0126	Middle-phase

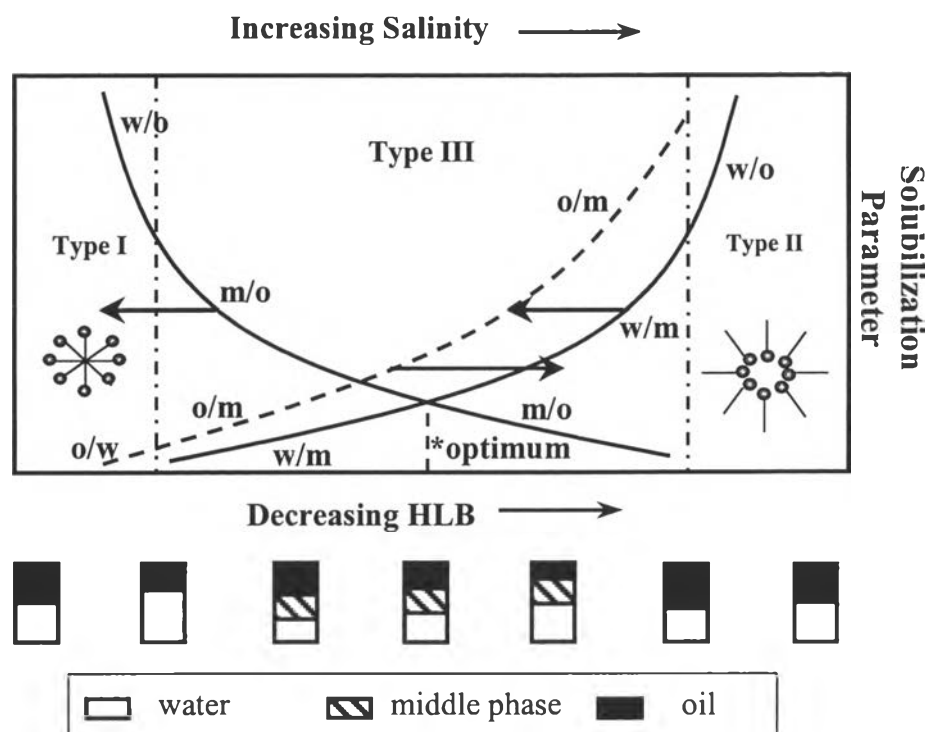


FIG. 1. Relationships of phase behavior, interfacial tension (IFT), and solubilization parameter. Abbreviation: HLB - hydrophilic-lipophilic balance; w/o - water/oil interface; m/o - microemulsion/oil interface; o/w - oil/water interface; w/m - water/microemulsion interface; o/m - oil/microemulsion interface.

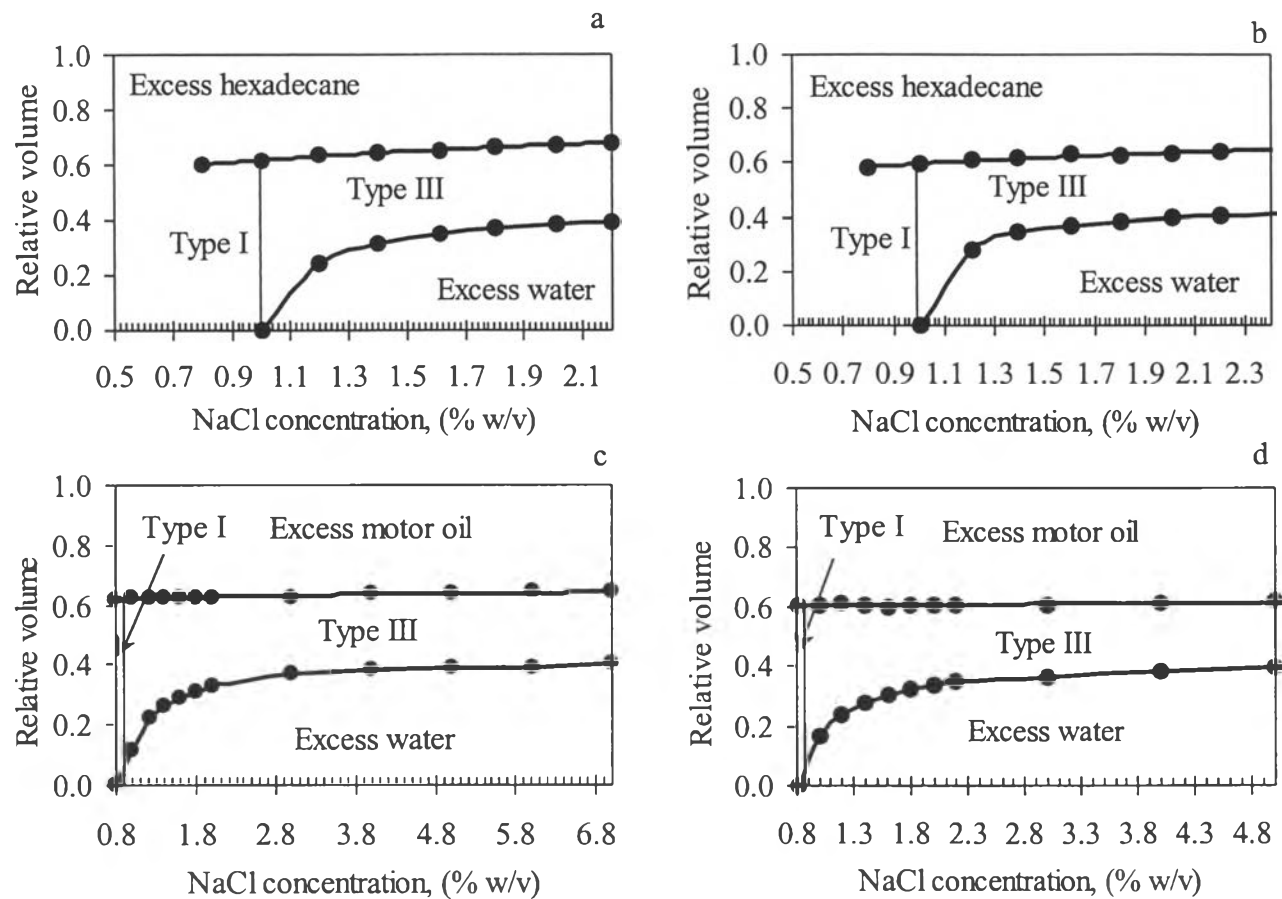


FIG. 2. Phase diagram of salinity scan on DOWFAX/AOT/Span 20/propylene glycol system at 30°C, for hexadecane: (a) 15 % w/v propylene glycol, (b) 20 % w/v propylene glycol, for motor oil: (c) 10 % w/v propylene glycol, (d) 15 % w/v propylene glycol.

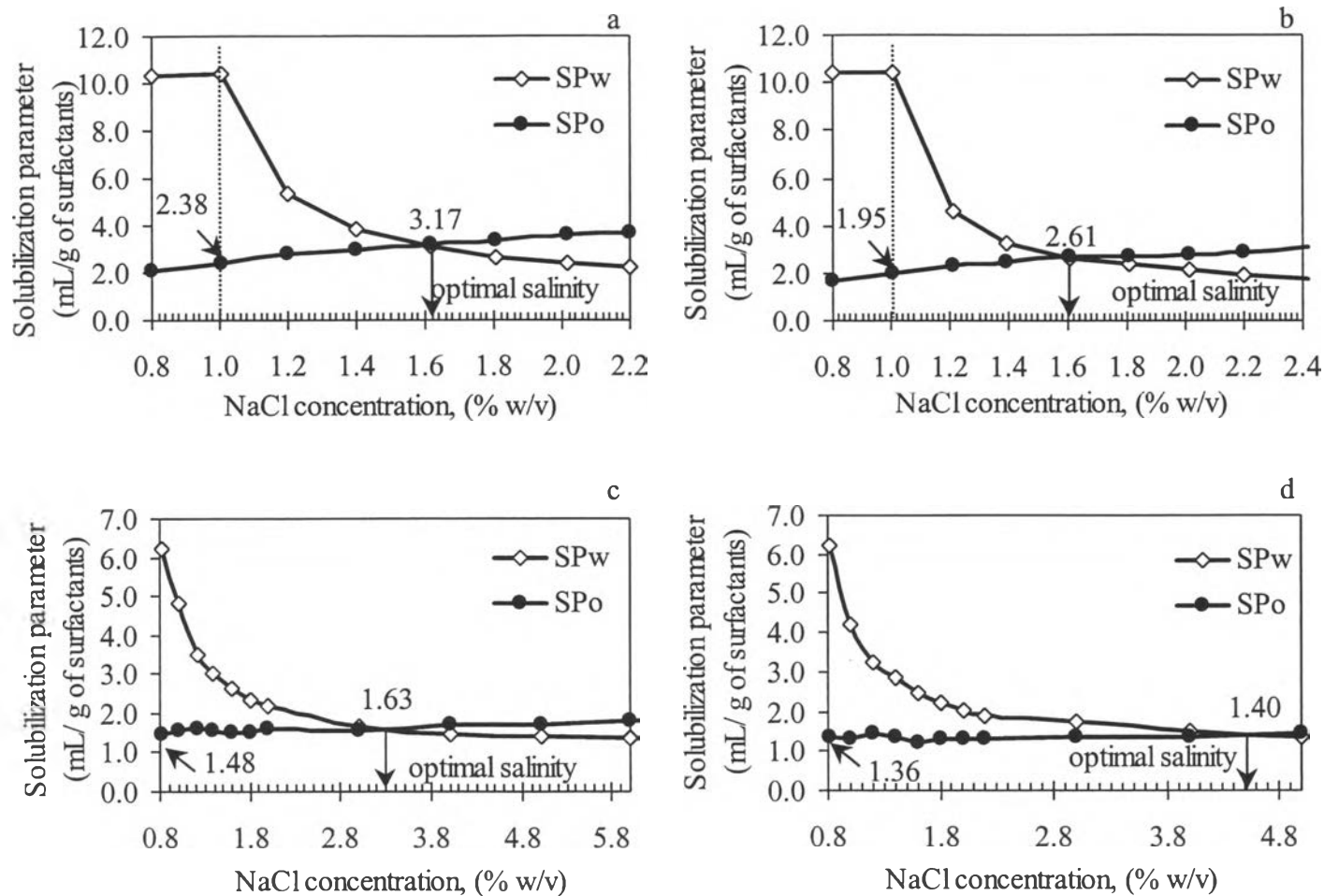


FIG. 3. Solubilization parameter as a function of NaCl concentration for hexadecane: (a) 15 % w/v propylene glycol, (b) 20 % w/v propylene glycol, for motor oil: (c) 10 % w/v propylene glycol, (d) 15 % w/v propylene glycol.

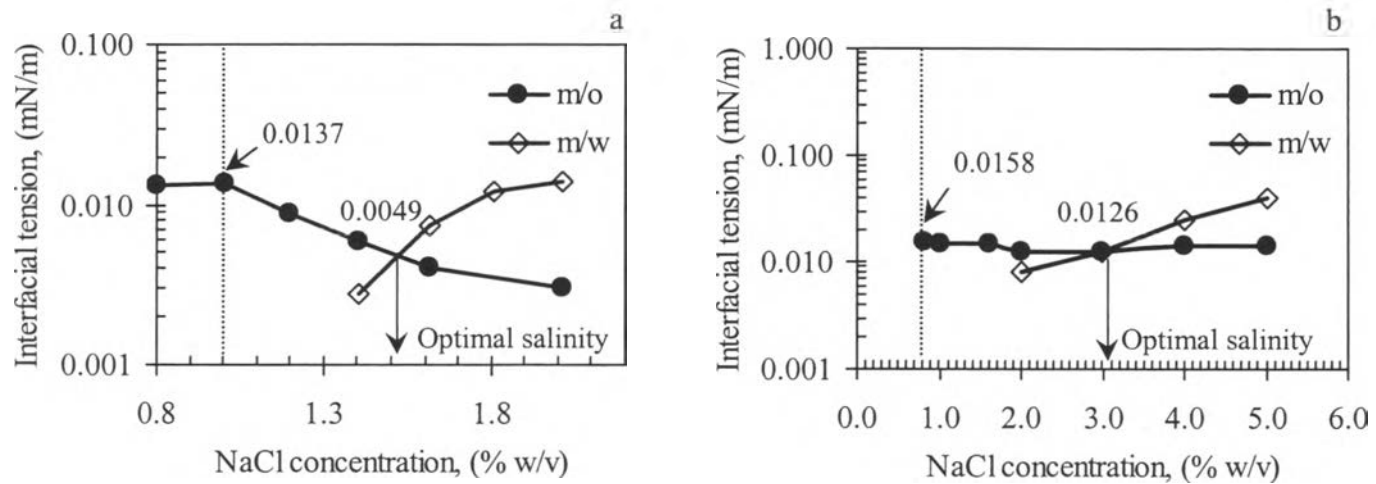


FIG. 4. Interfacial tension as a function of NaCl concentration for: (a) hexadecane, 15 % w/v propylene glycol, (b) motor oil, 10 % w/v propylene glycol.

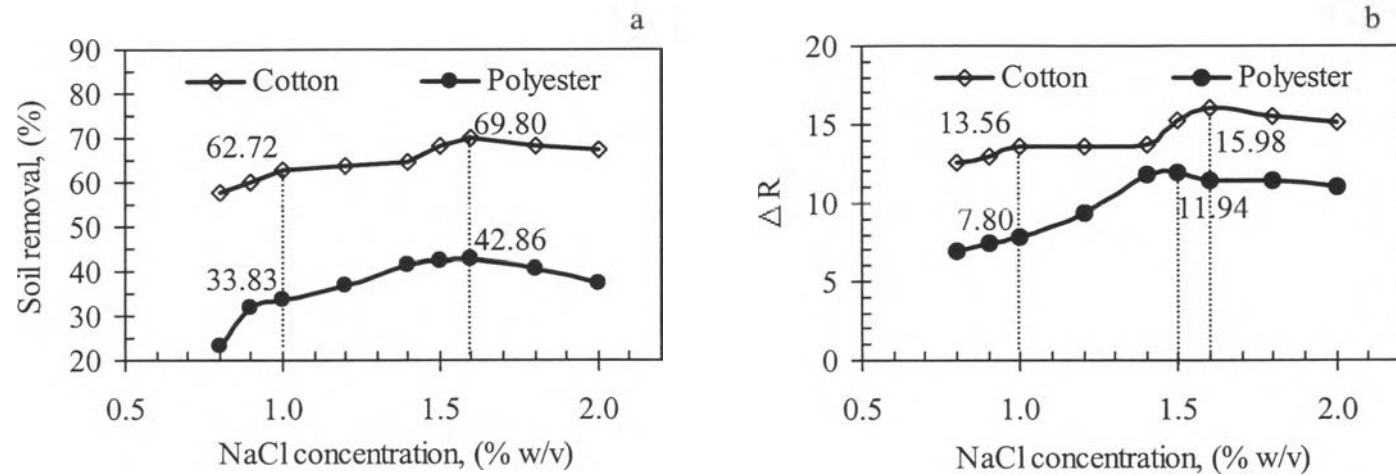


FIG. 5. Removal of hexadecane from cotton and polyester fabric based on: (a) spectrophotometric measurement plotted versus NaCl concentration. (b) the difference in reflectance values at 520 nm before and after washing (ΔR) plotted versus NaCl concentration.

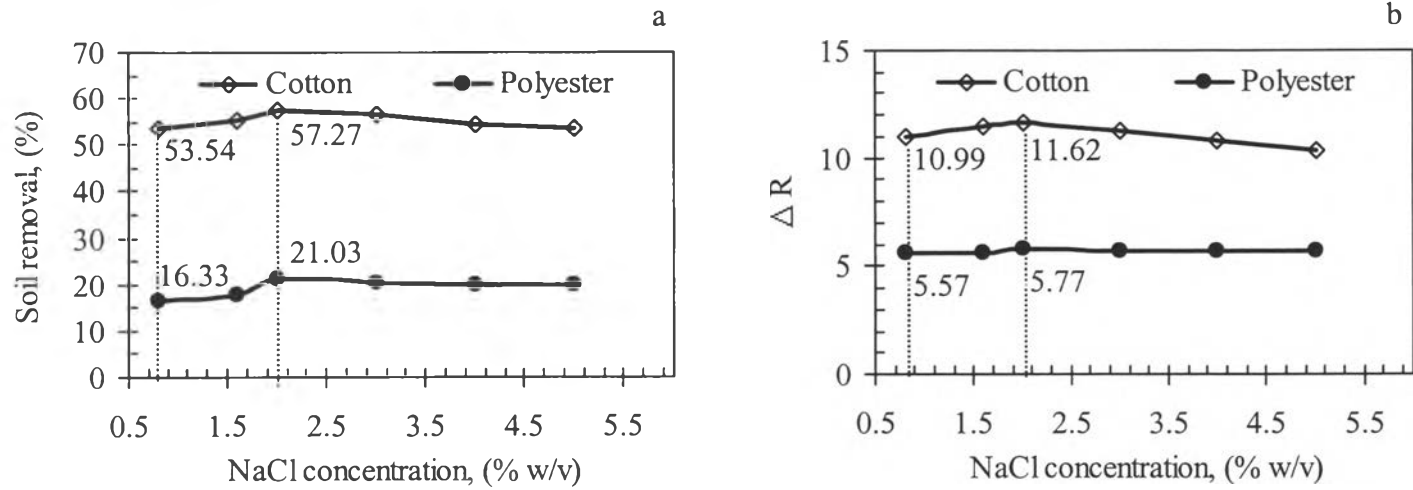


FIG. 6. Removal of motor oil from cotton and polyester fabric based on: (a) spectrophotometric measurement plotted versus NaCl concentration. (b) the difference in reflectance values at 520 nm before and after washing (ΔR) plotted versus NaCl concentration.