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Effect of Ionic Strength on Oil Removal from Stainless Steel in the Presence of Ionic Surfactant

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Abstract: Contact angles of oil droplets on solid surfaces provide useful insight into surfactant cleaning behavior. Contact angles of hexadecane and MAR-TEMP® 355, an industrial quench oil, on stainless steel were measured for ionic surfactant solutions as a function of ionic strength. The ionic strength of sodium dodecyl sulfate (SDS) and cetyl trimethyl ammonium bromide (CTAB) solutions was modified by the addition of sodium chloride. Increases in the contact angle with additions of 1.0 mM and 2.5 mM NaCl were observed for the two oils in SDS and for hexadecane in CTAB. For the industrial quench oil, detachment occurred in CTAB concentrations above the critical micelle concentration; as a result, the equilibrium contact angle measurements were not measured. The critical concentration of CTAB decreased with increasing NaCl concentration. Oil-removal studies indicate that increasing ionic strength by as little as 2.5 mM can result in improved cleaning.

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A theoretical insight previously used to explain contact-angle behavior for a hexadecane-gold system is used to describe the results obtained with the current system.

Keywords: Surfactant, ionic strength, contact angle, aqueous cleaning

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INTRODUCTION

Many researchers have studied surfactant solutions over the years for a variety of reasons. During recent decades, surfactant use in detergency has been a focus of many research activities (1–5). A significant portion of this interest was brought about by environmental regulations on industrial cleaning and degreasing processes that eventually banned commonly used organic solvents. Aqueous surfactant solutions arose as a viable replacement for the banned organic solvents in many applications. Although tremendous progress has been made in substituting aqueous surfactant solutions for organic solvents in cleaning systems, a better understanding of the basic phenomena behind surfactant behavior remains to be needed; such an understanding will enable improvement of industrial aqueous-surfactant cleaning performance and so that they may be considered as viable choices for many applications where organic solvents are still in use. This switch from organic solvents to aqueous-surfactant solutions is often a more environmentally-acceptable choice.

Previous works sought to find a way to improve the cleaning ability of surfactant-based cleaning systems. Starkweather et al. (6–8) studied the effects of nonionic and anionic surfactant concentrations and pH on oil removal, MAR-TEMP® 355 (a commercial quench oil), from a stainless steel surface. Interfacial tensions of the aqueous/oil interface and the contact angle of the oil on the metal surface were used to measure the effects of varying the surfactant concentration and pH. Additionally they reported a directly proportional relationship between both surfactant concentration/pH and contact angle/oil removal. While holding surfactant concentration constant, an increase in the pH resulted in an increase in the contact angle and an increase in the subsequent removal of oil from the stainless steel surface. This trend also occurred when pH was held constant and the surfactant concentration was increased. An inversely proportional relationship was found between both surfactant concentration/pH and interfacial tension. An increase in either variable while holding the other constant resulted in a decrease in the oil/surfactant interfacial tension. The research of Starkweather and colleagues, which was in agreement with previous research by Carroll (2), suggests a strong relationship between the interfacial tension, the contact angle, and the oil removal from a surface.

Rowe et al. (9, 10) extended Starkweather's research incorporating both cationic and zwitterionic surfactants in the study of the effect of pH and investigated applied potential on MAR-TEMP® 355 removal from a stainless steel

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surface. These studies showed enhanced oil removal for aqueous anionic surfactant solutions at high pH. Increased oil removal for the aqueous cationic surfactant solutions were found at low pH. A range of -4 to $+4$ volts was used to study the effect of applied potential. Oil removal in the presence of a cationic surfactant was best at low negative applied potentials. The oil removal for the nonionic surfactant increased as the potential applied between the surface being cleaned and a reference electrode increased. The anionic surfactant removed oil best at both ends of the voltage range. As for the zwitterionic, Rowe et al. discovered that this particular surfactant exhibited the behavior of a cationic surfactant at the low range of applied potential, acted as a nonionic surfactant as the applied potential increased and behaved like an anionic surfactant at both ends of the applied potential range. The findings of Rowe el al. involving electrified interfaces were confirmed and extended by Morton et al. (11).

Morton et al. (12–14) also developed a model for the prediction of equilibrium oil droplet contact angles on solid surfaces immersed in aqueous surfactant solutions; this model has proven useful in predicting industrial cleaning equipment performance. This model applies classical thermodynamics, a relevant surfactant self-assembly modeling theory, and includes estimating of the impact of ionic strength and other systemic parameters on the prediction of the oil droplet contact angle. The model and related theory provide a foundation upon which to further understand and enhance industrial aqueous cleaning processes.

The current paper investigates the effect of electrolyte addition and ties together the results found in the current research to the findings of Starkweather, Rowe, and Morton. A stainless steel surface similar to those used by Starkweather, Rowe, and Morton was chosen as the substrate. The contact angles of two different oils, hexadecane and MAR-TEMP® 355, on the stainless steel surface are measured for a cationic surfactant, cetyl trimethyl ammonium bromide (CTAB), and an anionic surfactant, sodium dodecyl sulfate (SDS). Oil removal experiments, like those performed by Rowe and Morton, again demonstrate the relationship between contact angle and cleaning. Simple conductivity tests were also performed to see if the critical micelle concentration (CMC) was being affected by the addition of electrolytes.

In a previous paper by Davis et al., the various types of wetting, adsorption, and cleaning methods for detergency were discussed in considerable detail (15). The current paper focuses more on the ability to use these experimentally determined contact angles to better understand the surface science phenomena. In a paper by Kwok and Neumann, an investigation into the ability to use contact angles to define surface phenomena was performed (16). It was proposed that the reluctance to study contact angles in past, present, and future research was and is due to three misconceptions.

The first misconception lies in the belief that contact angles are simple, easily measured and can be interpreted by anyone. However, contact angle data can be very problematic to both measure and use, therefore it is necessary when measuring and interpreting contact angle data to keep as many variables as possible constant. The second misconception is that contact angles show hysteresis, making equilibrium values unattainable and data useless to describe surface phenomenon. Kwok and Neumann show that a minimal amount of hysteresis caused by surface heterogeneity does not invalidate the fundamental assumptions upon which equilibrium contact angle data is based. The last misconception is that contact angle data provides information about intermolecular forces. Kwok and Neumann, as well as the research presented in this paper, caution using contact angle data to draw direct conclusions on surface tension and intermolecular forces (17).

A liquid droplet that exhibits both an advancing and a receding contact angle requires a more in-depth investigation than a symmetrical droplet with one unique contact angle. Many researchers have looked into the effect of surface roughness and heterogeneity on contact angle hysteresis. Research by Miwa et al. found that as surface roughness increased, higher contact angles were observed (18). Lam et al. found that contact angle hysteresis did occur to a degree on polished surfaces, however, advancing contact angles could be used to describe surface science while disregarding the receding contact angles (19). Katoh et al. (20) and Nakae et al. (21) found that contact angle hysteresis on rough or heterogeneous surfaces was linked to the wetting of the surface. Though this previous research illustrated the sensitivity of contact angle data due to hysteresis, investigations by Decker et al. concluded that contact angle hysteresis complicates the measurement and usefulness of the contact angle data (22).

As in the previous paper by Davis et al. (15), the contact angle of an oil droplet on a solid surface in an aqueous surfactant solution has been shown to mirror the behavior of surfactant adsorption isotherms proposed by Giles et al. (23, 24).

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EXPERIMENTAL

Materials

Two types of ionic surfactants were used in this study. The anionic surfactant sodium dodecyl sulfate (SDS) and cationic cetyl trimethyl ammonium bromide (CTAB) were chosen. The electrolyte sodium chloride (NaCl) was used to alter the ionic strength of the surfactant solutions. SDS and CTAB were purchased from Fisher Scientific of Fair Lawn, New Jersey. SDS was purchased at a purity of 98% and CTAB at 100%. The electrolyte, NaCl, was purchased from J.T. Baker Inc. of Phillipsburg, New Jersey at chemical grade. All water used to make the solutions was deionized. The materials shop at The University of Tennessee, Knoxville, provided the 304 stainless steel surface used in this study. A combination of hexane, acetone, and aerosol utility cleaner were used to clean the steel surface. All chemicals were purchased from Fisher Scientific. Hexane was purchased at HPLC grade and the acetone was reported by the manufacturer to have a purity of 99%. The aerosol utility cleaner was purchased from KELSAN, Inc., Knoxville, Tennessee. Two types of oil were used in this study. The organic oil hexadecane was purchased from Fisher at a purity of >98%. A blended petroleum distillate oil, MAR-TEMP® 355, was provided by Houghton International Incorporated. A VIS (Visible Range) cell, path length of 30 mm, was purchased from Spectrocell Inc. of Oreland, Pennsylvania. A Tantec CAM-PLUS contact angle meter, purchased from Tantec of Schaumburg, Illinois, was used to measure the contact angles. The instrument uses the Half-Angle™ Tangent line technique. The repeatability of the instrument is documented as $\pm 2^\circ$ and the accuracy as $\pm 1.5^\circ$. Basic ultrasonic cleaning experiments were performed using a Genesis™ Ultrasonic Generator system provided by Crest Ultrasonic of Trenton, New Jersey. Conductivity tests were performed using a Hewlett Packard E3632A DC Power Supply.

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METHODS AND PROCEDURES

Contact Angles

To ensure that the stainless steel disk was clean, it was first washed with a aerosol utility cleaner and then placed in a hexane bath and agitated for approximately 2 minutes. The disk was then rinsed with acetone and blown dry with pure air. Using a micropipette, the stainless steel disk was contaminated with a 1 μL droplet of hexadecane. The contaminated disk was allowed to sit for approximately 30 seconds before submerging in a solution to allow time for wetting of the surface.

The experimental set-up is the same that was used in the previous studies on a gold-coated surface (15). The aqueous bath was contained in the VIS cell. Approximately 20 mL of the desired 1 mM base SDS solution was added to the cell using a syringe. The contaminated disk was placed into the surfactant solution and plunged to the bottom of the VIS cell. The droplet remained undisturbed for 20 minutes after submersion in the aqueous solution to obtain the static contact angle.

After the static contact angle of the hexadecane droplet was determined, the stainless steel disk was removed from the SDS solution, rinsed off with water, washed with an aerosol utility cleaner, and placed in the hexane bath. The hexane bath was agitated to remove any remaining hexadecane. The stainless steel disk was then rinsed with acetone and blown dry with pure air. The contamination and contact angle procedure for hexadecane was then repeated for 1.0 mM SDS concentration. These procedures were repeated for SDS concentrations of 1.0 mM to 12.1 mM with the cleaning procedure described earlier between runs. After obtaining static contact angles for all salt-free SDS solutions, new solutions were prepared with the addition of 1.0 mM and 2.5 mM NaCl. The same contamination and contact angle procedures were performed for all solutions with the cleaning method performed between runs.

Once the static contact angle data for SDS solutions with and without NaCl were taken for hexadecane, the procedures were repeated for MAR-TEMP® 355. After all data for MAR-TEMP® 355 was taken, surfactant solutions of CTAB with and without 1.0 mM and 2.5 mM NaCl were prepared. The contamination and contact angle procedures were performed for all CTAB solutions for both hexadecane and MAR-TEMP® 355 with the cleaning method performed between runs. A variance in the procedure was used for MAR-TEMP® 355 droplets. These droplets had to remain undisturbed for 3 hours to ensure static contact angles.

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Ultrasonic Cleaning

Bench-scale ultrasonic cleaning tests were used to try to develop a relationship between contact angle behavior and oil removal from the surface. The procedure and instrument settings used by Rowe were repeated for the present study (10). A Genesis™ Ultrasonic Generator system provided by Crest Ultrasonic was used to mimic an industrial type of cleaning system. For these experiments both SDS and CTAB solutions were used with and without electrolytes. A stainless steel coupon similar to the stainless steel surface used for the contact angle experiments were used for the cleaning study. The stainless steel coupon was washed with a commercially available industrial cleaning agent and rinsed with deionized water. The coupons were then placed in an oven set at 120°C and allowed to dry for approximately 5 minutes. They were allowed to cool at room temperature, weighed, and the mass of each coupon recorded, **W**. The coupons were then submerged in MAR-TEMP® 355 for 5 minutes and the excess oil allowed to drain for 3 hours. The contaminated coupons were then reweighed and the mass recorded, **X**. Surfactant solutions of 3 mM, 6 mM, 8 mM, and 10 mM SDS solutions were prepared in a 2000 mL beaker and placed in the ultrasonic bath. The generator was engaged, set to 108 watts, and the bath allowed to degas for 5 minutes. A contaminated coupon was then placed in the surfactant solution for approximately 3 minutes. Visible excess water was removed from the coupon by gently blowing compressed air along the surface paying careful attention not to remove any oil. The coupon was then reweighed and the mass recorded, **Y**. The percent oil removed was calculated by to following equation:

$$\text{Percent Oil Removal} = \frac{(X - Y)}{(X - W)} \times 100 \tag{1}$$

Multiple coupons were run in each solution and the degree of cleaning measured. The SDS solutions were then remade containing 2.5 mM NaCl. The procedure was repeated and the percent oil removed calculated. The process was then repeated with 0.1 mM, 0.4 mM, 0.75 mM and 1 mM CTAB solutions with and without 2.5 mM NaCl.

Conductivity Tests

As the surfactant solutions were prepared for the contact angle tests, portions of the solutions were placed in 4-oz. Clear Qorpak® bottles. The conductivity of water was first measured to create a baseline, and then the conductivity of the various surfactant solutions was measured. For SDS solutions, the conductivity meter was set at 10 volts. The conductivity meter was set at 30 volts for the CTAB solutions.

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RESULTS

Contact Angle

In Fig. 1, the relationship between SDS concentration and hexadecane contact angle is shown. As the surfactant concentration increases, the contact angle of the organic droplet increases. The contact angle data in this study appears to have only one unique plateau occurring near the CMC of SDS, which is approximately 8.2 mM (10). The contact angle data curve mimics the behavior of the expected surfactant adsorption isotherms. The data trend resembles a Giles S-2 type of surfactant adsorption isotherm, in which once adsorption of surfactant to the surface begins, additional adsorption of surfactant to the surface becomes easier. This agrees with the Giles interpretation of surfactant adsorption (23, 24). The effect of the addition of 1.0 mM NaCl and 2.5 mM NaCl are also shown in Fig. 1 over the range of SDS concentrations. It is observed from the graph that an addition of 2.5 mM NaCl, while holding the SDS concentration constant, results in an increase in the contact angle. However, the addition of 1.0 mM NaCl does not result in a statistically significant increase in the contact angle. Yet, in both cases, the addition of electrolyte does not seem to change the type of isotherm from that without electrolyte addition, S-2.

The effect of SDS concentration on MAR-TEMP® 355 contact angle droplets is shown in Fig. 2. As in the case of hexadecane, the contact angles of MAR-TEMP® 355 increase as the surfactant concentration increases. The data trend also resembles a S-2 type of isotherm, one plateau. The plateau occurs around 6 mM SDS. This is below the CMC of the surfactant SDS. The effect of the addition of 1.0 mM and 2.5 mM

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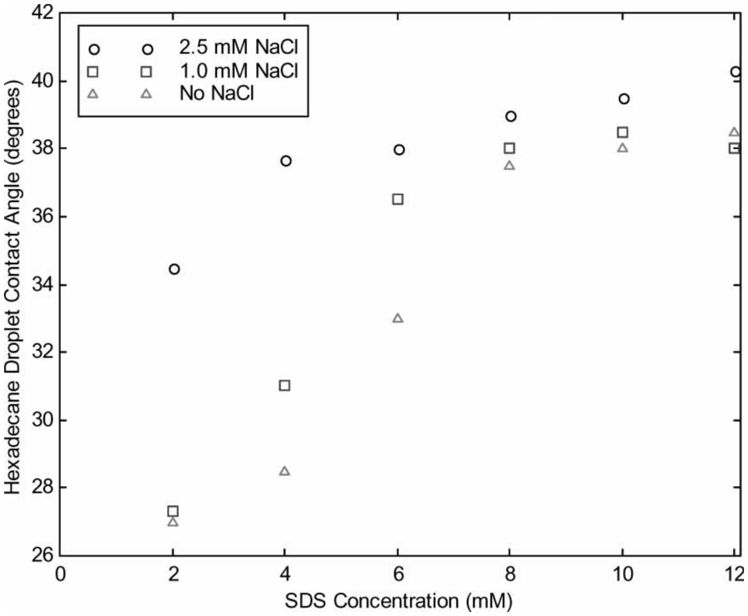

Figure 1. Hexadecane droplet contact angles on steel in SDS solutions.

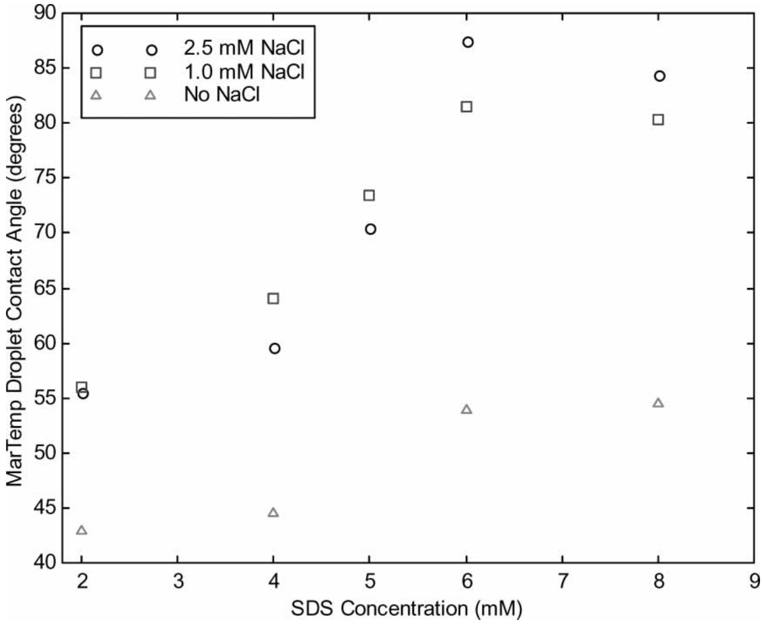

Figure 2. MAR-TEMP 355 droplet contact angles on steel in SDS solutions.

NaCl on the contact angles of MAR-TEMP® 355 oil droplets over the SDS concentration range is shown in Fig. 2. The addition of electrolyte to the surfactant system results in a substantial increase in the contact angle of the oil droplet, as surfactant concentration is held constant. As the concentration of the electrolyte increases, the contact angle of the MAR-TEMP® 355 oil droplet increases as well. The contact angle trends still resemble a S-2 type of isotherm.

The relationship between the CTAB concentration and the contact angle of hexadecane can be found in Fig. 3. As in the case of SDS, a shift in the contact angle is directly related to a change in the CTAB concentration. As the surfactant concentration increases, an increase in the contact angle is observed. Though the contact angle plateau is not as defined as in the case of SDS, it appears to be occurring around the CMC region of CTAB, reported to fall between 0.7 mM and 1.0 mM (10). As with all of the contact angle data preceding this one, the data trend resembles a S-2 type isotherm. The effect of the addition of 1.0 mM and 2.5 mM NaCl over the range of CTAB concentrations for hexadecane contact angles is also illustrated in Fig. 3. The same contact angle trend observed before the electrolyte addition is seen after the electrolyte addition. As the CTAB concentration is kept constant, an increase in electrolyte concentration results in an increase in the contact angle of the hexadecane droplet. The trend of the contact

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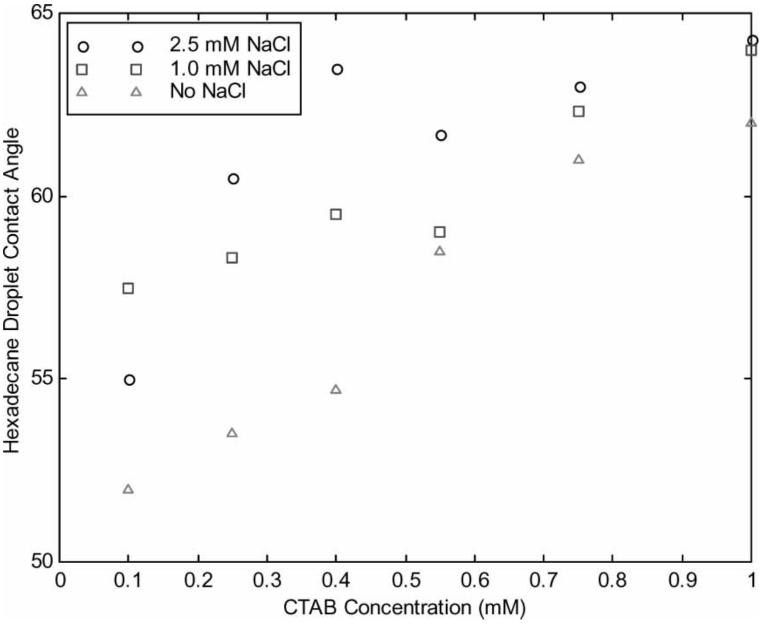

Figure 3. Hexadecane droplet contact angles on steel in CTAB solutions.

angle data is difficult to define due to the irregularity of the contact angle measurements. However, there is an overall increase in the contact angle data with electrolyte addition.

The relationship between MAR-TEMP® 355 contact angles in CTAB concentrations is illustrated in Fig. 4. The behavior of MAR-TEMP® 355 in CTAB surfactant solutions is different from the previous oil-surfactant systems. As seen in the figure, as the CTAB concentration increases, the contact angles increase but exhibit no plateau. The CTAB concentrations used in this study only cover a portion of the concentrations used in the previous studies due to the fact that above 0.55 mM CTAB, the oil droplets begin to detach from the steel surface. This detachment of the oil droplets made it impossible to measure a static contact angle. This behavior was not seen for MAR-TEMP® 355 in any of the SDS solutions. The effect of electrolyte addition to the MAR-TEMP® 355 contact angles in CTAB system is shown in Fig. 4. It is observed that as the concentration of electrolyte increases, the CTAB concentration at which the MAR-TEMP® 355 begins to detach is lowered. A color change of the oil is noticed with electrolyte addition. It was also observed that with the addition of electrolyte, the time required to begin the detachment was shortened. These contact angles shown in Fig. 4 represent the last CTAB concentration before the detachment occurred in the experiments.

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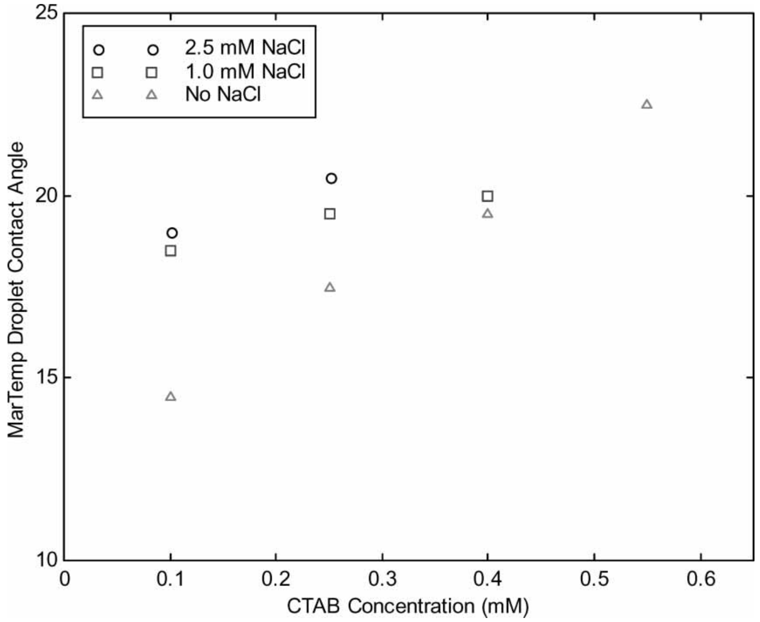

Figure 4. MAR-TEMP 355 droplet contact angles on steel in CTAB solutions.

Oil Removal

Figure 5 shows the results from the ultrasonic cleaning experiments performed to show a relationship between the effect of the addition of electrolytes on MAR-TEMP® 355 contact angles in surfactant solutions and the fractional amount of oil removed from the stainless steel surface. For SDS solutions, the results indicate that oil removal from a stainless steel coupon increased when 2.5 mM NaCl was added to all the original SDS concentrations. The data is not as smooth as the contact angle data previously measured and is most likely due to the fact that the method used to determine the percent oil removed from the surface is simple and imprecise. However, it is shown that the presence of electrolytes increased the oil removal.

In Fig. 6, the effects of 2.5 mM NaCl addition to the CTAB surfactant solutions are shown. As in the case of SDS, the addition of electrolyte to CTAB solutions increases the amount of MAR-TEMP® 355 oil removed from the stainless steel surface. The increase in oil removal coincides with the increase in the contact angles of the oil previously observed. The percent oil removal for MAR-TEMP® 355 in CTAB solutions is relatively the same as for the same oil in SDS solutions (Figs. 7 and 8).

DISCUSSION

A proposed mechanistic interpretation was presented in the previous paper by Davis et al. to describe surfactant adsorption on a gold surface (15). This

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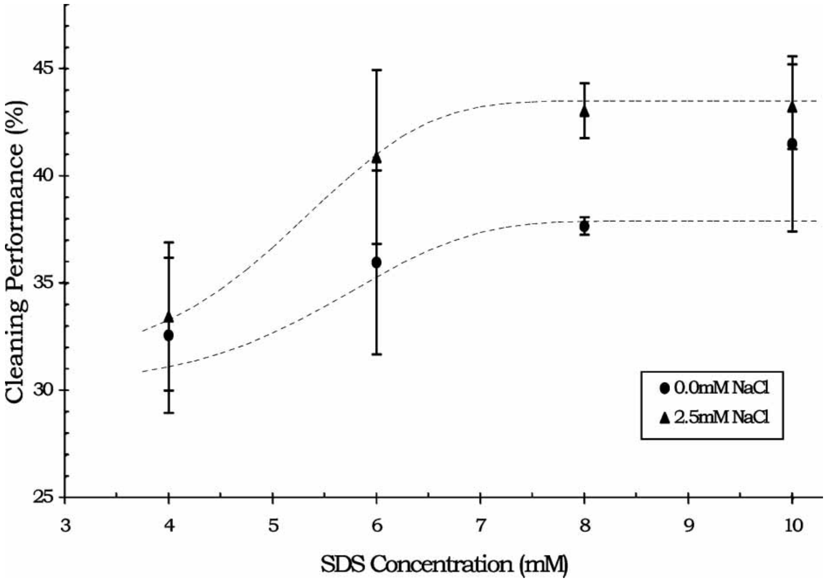

Figure 5. Effect of ionic strength on steel cleaning in SDS solutions.

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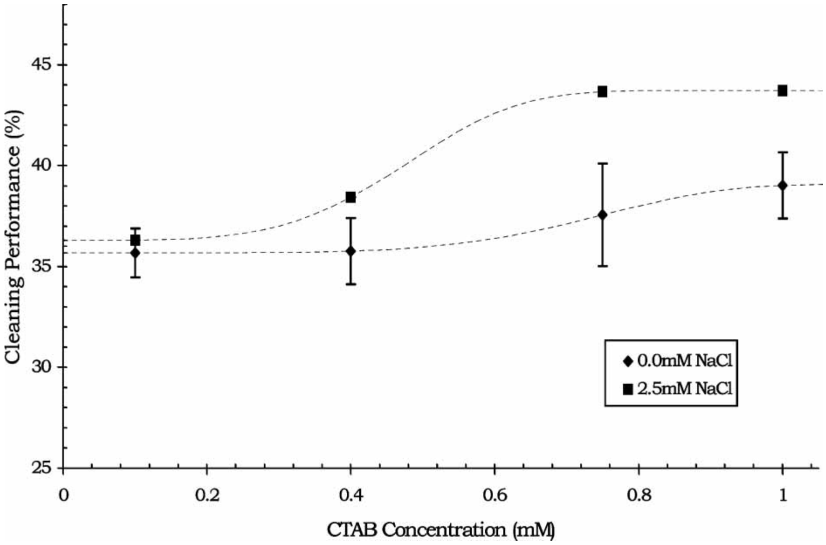

Figure 6. Effect of ionic strength on steel cleaning in CTAB solutions.

model is also used to describe the behavior seen in the present research using the same surfactants on a stainless steel surface. The initial increase in contact angle data is considered to be due to the adsorption of a surfactant monolayer on the solid surface, which competes with the oil phase for solid surface

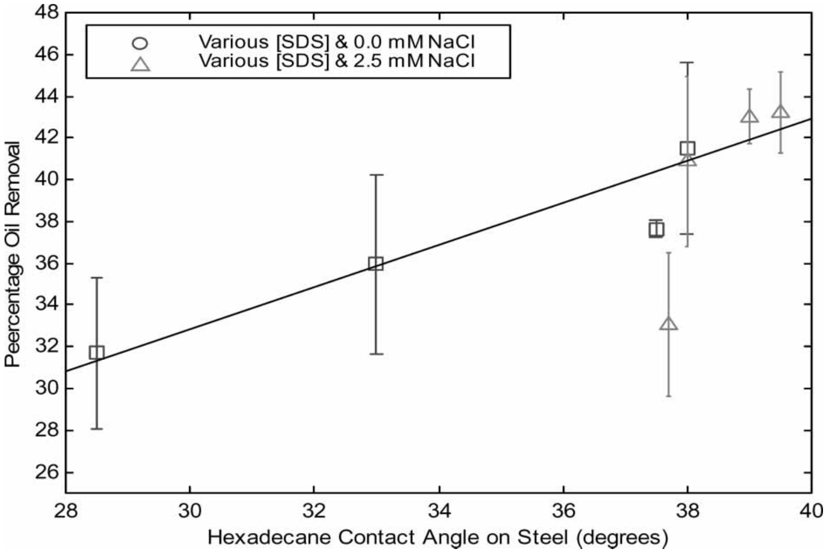

Figure 7. Correlation of SDS cleaning performance with hexadecane contact angle.

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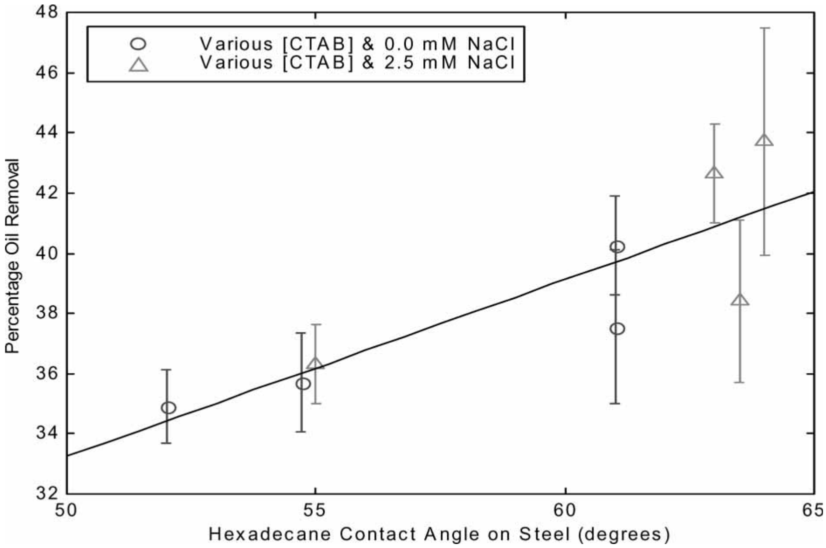

Figure 8. Correlation of CTAB cleaning performance with hexadecane contact angle.

adsorption sites. As the surfactant concentration increases, surface adsorption increases up to the point where the repulsive forces between the absorbed surfactant head groups prevents any additional adsorption to the solid surface. Further, increasing the surfactant concentration results in random surfactant adsorption with interaction between the tail groups. Eventually, the adsorption becomes less random and more ordered, with the tail groups of surfactant molecules in solution oriented parallel to the tail groups of surfactant molecules absorbed to the surface.

The adsorption of surfactant molecules onto the surfactant molecules already adsorbed to the stainless steel surface creates a bi-layer. The bi-layer adsorption continues until the CMC of the surfactant is reached. Above the CMC of the surfactant, the addition of the surfactant only aids in the formation of micelles resulting in a constant competition between the adsorbed surfactant and the oil phase resulting in a constant contact angle (15). The belief that a condensed surfactant bi-layer exists is supported by literature. Burgess et al. found evidence of a condensed bi-layer of SDS on a gold surface at high charge densities (25). It is believed that the increase in the contact angles observed with the addition of electrolytes to the surfactant solutions is due to a reduction in the repulsive forces between the adsorbed surfactant head groups. This allows additional surfactant adsorption in both the monolayer and bi-layer with increased competition between the adsorbed surfactant and oil phase.

CONCLUSIONS

In this paper, further investigations were performed to study the effects of electrolyte addition on surfactant cleaning behavior on a stainless steel surface. The same cleaning system used in the gold-coated glass study, Davis *et al.* (15), was utilized for a stainless steel surface and hexadecane, a pure oil, and MAR-TEMP® 355, a blended oil. The contact angles of both oils on the stainless steel surface were taken for the anionic surfactant sodium dodecyl sulfate (SDS) and the cationic surfactant cetyl trimethyl ammonium bromide (CTAB). The electrolyte sodium chloride (NaCl) was used at concentrations of 1.0 mM and 2.5 mM to alter the electrolyte concentration. Concentrations above and below the CMC of each ionic surfactant were used. The addition of non-surfactant electrolyte to the system does not change the shape of the contact angle response. The addition of electrolytes to the system is believed to reduce the repulsive forces between the adsorbed surfactant allowing additional adsorption potentially resulting in compressed monolayers and/or bi-layers. The increase in the adsorption of surfactant head groups to the solid surface reduces the available surface area for the oil to adsorb, so an increase in the contact angle should correlate to an increase in oil removal. This assumption was confirmed with basic cleaning tests showing that the aqueous surfactant cleaning solutions with electrolyte removed more oil from a stainless steel surface that those without. Introducing non-surfactant electrolytes at low concentrations to aqueous surfactant cleaning systems to create a more environmentally friendly industrial cleaning process has been shown to be a possibility for certain applications.

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