ORIGINAL ARTICLE

Dissolution Study of Salt of Long Chain Fatty Acids (Soap Scum) in Surfactant Solutions. Part I: Equilibrium Dissolution

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Abstract Dissolution of calcium salt of a long chain fatty acid or soap scum is a major challenge for hard surface cleaners since soap scum forms when soap is exposed to hard water and has very low water solubility. In this paper, the aqueous equilibrium solubility of calcium octadecanoate (or calcium stearate) was measured as a function of pH as well as chelating agent (ethylenediaminetetraacetate disodium salt) and surfactant concentrations. Anionic, nonionic, and amphoteric surfactants were studied. The highest soap scum solubility was observed at high pH with an amphoteric surfactant. Under this condition, the chelant effectively binds calcium, and the stearate anion forms mixed micelles well with the amphoteric surfactant, which is in zwitterionic form at high pH.

Keywords Detergent formulation · Application of surfactants

Introduction

Soap scum is the calcium or magnesium salt of a long chain fatty acid. Many personal care products consist of soaps, which are salts of carboxylic acids or fatty acids. Soaps are unstable in hard water containing metal ions, especially calcium and magnesium, because soaps form insoluble precipitates with those metal ions. Insoluble soap scum

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D. R. Scheuing Clorox Technical Center, Pleasanton, CA, USA creates the well-known stain in the bathroom (e.g., ring around the bathtub). There have been extensive efforts to formulate bathroom cleaners and other hard surface cleaners to remove soap scum stains. Requirements for good formulation generally include effective, rapid removal of the scum with little or no mechanical force involved. One common approach in hard surface cleaners used for soap scum removal involves an aqueous solution of a chelant for calcium or magnesium complexation with a micelle-forming surfactant. Simultaneous chelation of calcium and mixed micellization of the fatty acid with the added water-soluble surfactant are hypothesized to be responsible for soap scum dissolution.

The relationship between the structure of different surfactants and performance in the equilibrium dissolution of soap scum in the solution containing chelating agent and the effect of solution pH are main focuses in this study. The solution pH can have an effect on both the effectiveness of the chelating reaction and the structure of the fatty acid due to protonation [1]. Generally, the solubility of soap decreases or the Krafft temperature increases as its alkyl chain length increases [2]. Mixture of soaps are more soluble than pure soaps [2].

Calcium di-stearate (Ca(C₁₈)₂), [CH₃(CH₂)₁₆CO₂]₂Ca or the calcium salt of octadecanoic acid was used as a model soap scum in this study. Actual soap scum can be a mixture of other metal salts (e.g., magnesium) or other fatty acids such as palmitic acid. Calcium stearate is normally considered "insoluble" in water, alcohol, and ether, but soluble in mineral oils and hot pyridine. The solubility of calcium stearate is only about 0.04 g/l of water at 15 °C [3]. Long chain fatty acid salts are weak ionizable surfactants [4] with the charge varying with pH. Calcium stearate has a pK_a of 4.5 [5]. So, at low pH (e.g., 2), the stearate anion protonates and forms nearly insoluble stearic acid.

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The solubility of stearic acid is only 0.0029 g/l of water at 20 °C [6]. At higher pH level, calcium cannot dissociate so the Ca(C₁₈)₂ remains mainly insoluble. At intermediate pH level, protonation is only partial so there is a mixture of stearate anion and stearic acid, both of which have low solubility. So, without some way to remove the calcium from the stearate or some aggregates like micelles to increase the solubility of the stearate anion or stearic acid in solution, Ca(C₁₈)₂ is nearly insoluble throughout a wide pH range.

The solubilities of either the nonionic stearic acid at low pH and the $Ca(C_{18})_2$ at high pH are so low that micelles do not form; i.e., as surfactant concentration increases, the solubility limit is reached before a CMC is reached. One strategy of increasing the solubility of $Ca(C_{18})_2$ at any pH is to add a "micelle promoting agent" [7], which forms micelles with which the protonated stearic acid or stearate anion can co-micellize. This is a common general strategy in increasing the solubility of surfactants. Since the tendency to co-micellize or form mixed micelles will depend on the charge of the added surfactant, we will study added anionic, nonionic, and amphoteric surfactants here.

A chelating agent is sometimes added into cleaning products to prevent precipitation of active ingredients with metal ions naturally found in the hard water by forming a water soluble complex with the metal ions. In the system studied here, the chelating agent was added to complex with and promote the dissociation of calcium ion from the solid calcium stearate so the dissociated stearate anion can form micelles. There are several types of chelating agents or complexing agents such as phosphates and aminocarboxylates (e.g., EDTA) [8]. Ethylenediaminetetraacetate disodium salt, Na₂EDTA, was used here. It is a common complexing agent, which has four main active sites that can form a water-soluble complex with metal ions. One molecule of Na2EDTA can chelate one molecule of calcium ion stoichiometrically [9]. The effectiveness in metal complex formation depends on the equilibrium constants or



Fig. 1 Equilibrium solubility of Ca(C18)2 in SDS/Na2EDTA solutions

stability constants of the ligand with the metal. There are five possible forms of EDTA in the absence of calcium depending on pH, and there are two forms that can form complexes with calcium ions [9]. At high pH, the major species found is Y^{4-} , which is the most effective form of EDTA in chelating calcium. The molecule of EDTA in acidic solution is less effective than in a basic solution due to protonation of active sites on the EDTA.

At high pH, as calcium is removed from solid $Ca(C_{18})_2$, the stearate anion solubility increases to the point where all the solid dissolves, or it forms micelles, or forms precipitate with some other counterions (e.g., Na⁺) in solution in the absence of a co-surfactant acting as a micelle-promoting agent. The presence of a co-surfactant forming micelles further improves solubility synergistically with the chelant. At a low enough pH, the protonation reaction should cause complete dissolution of the solid calcium stearate to nearly insoluble protonated fatty acid, which can co-micellize (but doesn't have enough hydrophilicity in the nonionic head group to form micelles independently). So at low enough pH, the chelant should not have any effect on the equilibrium solubility of Ca(C₁₈)₂.

Experimental

Materials

Sodium dodecyl sulfate (SDS) (99+ % purity), obtained from Fisher Scientific (Fair Lawn, NJ), was further purified by recrystallization from water and then from methanol, followed by drying under a vacuum at 30 °C. Octyl polyglycoside (C₈APG) was obtained from Akzo Nobel and used as received. Disodium ethylenediaminetetraacetate (Na₂EDTA) (100% purity) was obtained from Fisher Scientific and used without further purification. Stearic acid (99% purity), obtained from Alfa Aesar (Heysham, Lancaster), was used without further purification. Calcium hydroxide (99.995% purity) was obtained from Sigma-Aldrich (St. Louis, MO). Absolute ethyl alcohol (100% purity) was obtained from AAPER (Shelbyville, KY). Acetone (A.C.S. grade) was obtained from Fisher Scientific. Dimethyldodecylamine oxide (DDAO) was obtained from Stepan and used without further purification. The NaOH and HCl were from Fisher Scientific (Fair Lawn, NJ) and were used as received to adjust the solution pH. Water was double deionized.

Methods

Calcium stearate, calcium salt of stearic acid, was prepared from stearic acid. Stearic acid was first dissolved in ethanol and then reacted with a clear solution of calcium



Fig. 2 Equilibrium solubility of $Ca(C_{18})_2$ in DDAO/Na₂EDTA solutions

hydroxide. The precipitate was filtered and rinsed with water, ethanol and acetone to remove excess calcium ion and unreacted stearic acid. Finally, the precipitate was dried in a vacuum oven at $30 \,^{\circ}$ C.

The equilibrium solubility of calcium stearate in anionic, nonionic, and amphoteric surfactants was measured at 25 °C. A series of surfactant solution was prepared at pH 4-11 using HCl and NaOH solutions. Sodium dodecyl sulfate (SDS) was used as an anionic surfactant. Octyl polyglycoside was used as a nonionic surfactant. Dimethyldodecylamine oxide (DDAO) was used as an amphoteric surfactant. An excess amount of $Ca(C_{18})_2$ was added into a mixed solution with other surfactants and Na2EDTA at the pH of interest. The $Ca(C_{18})_2$ was forced to dissolve by heating it up to above 70 °C or until most of $Ca(C_{18})_2$ was dissolved. Then it was equilibrated at the temperature of interest (25 °C) in a temperature-controlled water bath. The solution was allowed to equilibrate for at least 1 week with routine shaking to ensure that equilibrium was reached. The solution was filtered using a 0.22-micron Durapore hydrophilic membrane. Then the supernatant was analyzed for the concentration of $Ca(C_{18})_2$ by a gas chromatography (GC) (Varian 3300), using an SPB 20 column (Supelco) with a FID detector following derivatization.

Calcium stearate was derivatized into methyl stearate by using a derivatizing agent, BF_3 -methanol (Alltech, Deerfield, IL). The derivatized solution was diluted by hexane before being analyzed by GC.

Results and Discussion

Chelant Only Systems

The equilibrium solubility of $Ca(C_{18})_2$ was measured in solutions of different types of surfactants with and without chelating agent (Na₂EDTA) at 25 °C and at varying pH. Figure 1 shows the equilibrium solubility of $Ca(C_{18})_2$ in 0.1 M SDS, 0.1 M Na₂EDTA, and a solution of 0.1 M SDS/0.1 M Na₂EDTA as a function of pH. Figure 2 shows the equilibrium solubility of $Ca(C_{18})_2$ in 0.1 M DDAO, 0.1 M Na₂EDTA, and a solution of 0.1 M DDAO/0.1 M Na₂EDTA as a function of pH. From both Figs. 1 and 2, the Na₂EDTA alone has no significant effect on $Ca(C_{18})_2$ solubility at any pH studied. Neither the protonated nonionic stearic acid (low pH) or the stearate anionic can attain a high enough solubility to form micelles without co-surfactant, even if free calcium is largely complexed. There is competition for calcium between the Na2EDTA and the precipitated $Ca(C_{18})_2$ and the extremely low K_{sp} of the latter maintains the solid $Ca(C_{18})_2$ even at very low free calcium concentrations in the presence of the chelant at high pH where the Na₂EDTA is most effective. At the lowest pH studied here (pH 4), the protonated form of the fatty acid and the solid $Ca(C_{18})_2$ will both be present. We did not attempt to ascertain the fraction of the solid present in either form since the focus of this study is the solubility of the stearate or stearic acid, neither of which was significant without co-surfactants. Table 1 summarizes the possible forms of $Ca(C_{18})_2$ that could be found at different pH levels. The stearic acid could form because of low pH by the protonation of stearate anion. The Na₂EDTA will also be protonated into less effective chelant forms at low pH. At intermediate pH, calcium is released by the

Table 1 Forms of soap scum, DDAO, ability of Na₂EDTA to complex calcium, and solubility of $Ca(C_{18})_2$ in surfactant/chelant system at different pH levels

рН	Dominant form of soap scum	Dominant charge on DDAO	Ability of Na ₂ EDTA to complex calcium	Solubility of $Ca(C_{18})_2$ in surfactant/chelant system		in em
_				SDS	DDAO	C ₈ APG
Low	Protonated stearic acid (nonionic)	Cationic	Low	Medium	Low	Low
Intermediate (6-9)	Protonated stearic acid (nonionic) and stearate (anionic)	Cationic and zwitterionic	Medium	Low	High	Medium
High	Stearate (anionic)	Zwitterionic	High	Low	Very high	High

protonation of $Ca(C_{18})_2$. The calcium is also complexed with chelant to some extent. The other molecules formed from $Ca(C_{18})_2$ are stearic acid and stearate anion. At higher pH, even though the calcium is complexed effectively, the dissolution of $Ca(C_{18})_2$ is still considered very low since the stearate anion has limited solubility, and at the temperature of study (25 °C), it cannot form micelles alone to increase its solubility.

Surfactant Only Systems

In the absence of additives, the CMC of SDS is 6.7×10^{-3} M [10]. Extrapolating from reported CMC values with added electrolyte [11, 12] to pure surfactant values, the DDAO at low pH where it is in cationic form has a CMC of 7.0×10^{-3} M, and the DDAO at high pH where it is in zwitterionic form has a CMC of 5.4×10^{-3} M. The dissolved calcium and stearate ions will reduce the CMC values below these pure surfactant values.

In this study, the SDS and DDAO are present at least an order of magnitude above their CMC, so a high fraction (>93%) of these surfactants are present as micelles. As shown in Figs. 1 and 2, in the absence of chelating agent, both SDS and DDAO increased the solubility of $Ca(C_{18})_2$ slightly, with DDAO causing higher solubility than SDS at all pH levels. The SDS effect had little pH dependence except at pH below 5 where solubility of $Ca(C_{18})_2$ increased. The DDAO caused a monotonic increase in solubility in $Ca(C_{18})_2$ with decreasing pH, exhibiting an order of magnitude greater solubility at pH of 4 compared to SDS. At high pH (pH 7–11), the solubility of $Ca(C_{18})_2$ in DDAO is approximately five times higher than that in SDS.

The charge on the stearate/stearic acid molecule becomes less negative (high fraction of protonated or nonionic surfactant) as pH decreases as already discussed with a pK_a around 4.5. The effective pK_a can alter upon incorporation of the stearate into mixed micelles depending on the charge of the co-surfactant, increasing when co-micellizing with an anionic surfactant like SDS [13].

The DDAO is an amphoteric surfactant, which can exist in the form of a cationic or a zwitterionic surfactant depending on the solution pH as shown by the protonation reaction:

The pK_a of DDAO monomer is reported at about 5 [12], but the effective pK_a can be much higher in micelle form, even higher when co-micellized with an anionic surfactant [12]. Since 0.1 M DDAO is about two orders of magnitude above the CMC, the effective pK_a is that in micellar form. At the pH = pK_a , half of the surfactant is in cationic form and half in zwitterionic form. At pH below the pK_a , the cationic form dominates, and at pH above the pK_a , the zwitterionic form dominates. In addition to protonation of the stearate and the DDAO, the effectiveness of complexation of Na₂EDTA varies with pH as previously discussed, with higher pH being more effective.

In explaining the pH effects on $Ca(C_{18})_2$ dissolution, three effects dominate as pH decreases; the protonated nonionic stearic acid/anionic stearate anion ratio increases: the cationic DDAO/zwitterionic DDAO ratio increases; and complexation of calcium by Na2EDTA decreases. The effect of micellar composition on the effective pK_a of the stearate is probably a secondary effect in explaining trends. Charge repulsion of the head groups when anionic stearate is incorporated into an SDS micelle would inhibit comicellization of stearate with SDS, so the increase in solubility of $Ca(C_{18})_2$ at pH below 5 in a chelant-free SDS solution is due to a higher fraction of the fatty acid being in nonionic, protonated form. The highest synergism in mixed micellization is for a cationic/anionic surfactant mixture [14], explaining why the highest solubility in a chelant-free surfactant system is DDAO at low pH where it is below its pK_a and primarily in cationic form, co-micellizing with anionic stearate and (less synergistically) with protonated stearic acid. The synergism between a zwitterionic and an anionic surfactant is between that of an anionic/anionic and a cationic/anionic surfactant mixture [14], explaining why the solubility of $Ca(C_{18})_2$ at high pH (where the DDAO is primarily zwitterionic) is greater than that for SDS, but less than that of DDAO at low pH in the absence of chelant. Table 1 shows the ionic form of possible formed soap components and of micellar DDAO at low, intermediate, and high pH.

At low pH, the main soap scum component that forms mixed micelles with the added soluble surfactant is nonionic stearic acid. The formation of mixed micelles is expected to be most effective in either cationic (DDAO at low pH) or anionic surfactant micelles since cationic/nonionic or anionic/nonionic surfactant synergism is present.

At intermediate pH, stearic acid and stearate anion form mixed micelles with added surfactant. The DDAO is composed of both cationic and zwitterionic surfactants. The formation of mixed micelles when DDAO is used can be enhanced due to the mixed cationic (DDAO) and anionic (stearate) surfactants. However, the solubility of $Ca(C_{18})_2$ is lower than at low pH because stearate solubility is so low in equilibrium with $Ca(C_{18})_2$ when no calcium is complexed. Also, less protonated stearic acid is formed than at low pH.

At high pH, the soap scum mostly remains as an undissolved solid precipitate since the solubility of soap scum is very low. Even if there is any stearate anion, there is still an electrostatic repulsion between stearate and SDS in micelles so the solubility of $Ca(C_{18})_2$ is low. The DDAO is in completely zwitterionic form and would form micelles synergistically with stearate, but stearate solubility is so low in equilibrium with $Ca(C_{18})_2$ with no chelation.

Surfactant-Chelant Systems

In the presence of Na₂EDTA, the highest solubility is with DDAO at high pH (Figs. 2, 3, 4), where it is several orders of magnitude greater than in the chelant-free system (Fig. 2). The solubility increases monotonically with pH, the opposite trend than for the chelant-free system. So as long as there are zwitterionic or cationic micelles with which the stearate can co-micellize, the complexation of calcium by a chelant is crucial to improving solubility of the Ca(C₁₈)₂, and the increasing effectiveness of complexation of calcium by Na₂EDTA at high pH dominates pH effects, leading to maximum solubility at high pH. The



Fig. 3 Equilibrium solubility of $Ca(C_{18})_2$ in solutions of 0.1 M of SDS/0.1 M Na₂EDTA and 0.1 M C₈APG/0.1 M Na₂EDTA



Fig. 4 Equilibrium solubility of $Ca(C_{18})_2$ in solutions of 0.1 M SDS/ 0.1 M Na₂EDTA, 0.1 M C₈APG/0.1 M Na₂EDTA, and 0.1 M DDAO/0.1 M Na₂EDTA

lower solubility for the DDAO/Na₂EDTA system compared to DDAO only at pH 4 (Fig. 2) defies apparent explanation since the chelant might be expected to have no effect on solubility at low pH, but not to cause it to decrease.

For the SDS system, the synergism for the chelant/surfactant mixture increases as pH decreases (Figs. 1, 3) in contrast to DDAO. Here, the more efficient co-micellization of the nonionic, protonated stearic acid into the anionic SDS micelles relative to co-micellization of anionic stearate is more important than the decrease in complexation effectiveness of the chelant as pH decreases. However, it is important to note that the maximum solubility of $Ca(C_{18})_2$ in the SDS/chelant system is over an order of magnitude less than for the DDAO/chelant system, so lack of effectiveness of chelation at low pH greatly inhibits freeing of the stearic acid/stearate molecule from the solid $Ca(C_{18})_2$ to permit them to co-micellize.

More limited studies were carried out using the nonionic surfactant C₈APG compared to SDS and DDAO as shown in Figs. 3 and 4 for the C₈APG/chelant system (chelantfree C₈APG was not studied). The solubility of Ca(C₁₈)₂ in the C₈APG system shows increasing solubility of $Ca(C_{18})_2$ with increasing pH as with the DDAO system, although at high pH where maximum solubility is observed, total solubility is about a factor of five less for C₈APG than DDAO. Since anionic/nonionic mixed micelles are less synergistic than anionic/zwitterionic mixed micelles, the stearate/ C_8APG synergism is less than the stearate/DDAO at these high pH conditions, explaining this large amphoteric surfactant effectiveness. Aiding this performance difference is that the DDAO has a much larger hydrophobe than the C₈APG, forming micelles that co-micellize more effectively. Similarly, at low pH, the SDS system shows higher $Ca(C_{18})_2$ solubility than the C₈APG system, explainable by the greater synergism of the nonionic/anionic mixed micelles composed of protonated nonionic stearic acid/ SDS compared to the nonionic/nonionic protonated stearic acid/C₈APG mixed micelles. Table 1 summarizes the effects of pH on the chelation, surfactant structure and equilibrium solubility of the soap scum in surfactant/chelant systems.

Hydrolysis of SDS

Since equilibration experiments took 1 week at a pH as low as 4, the possible effect of the well-known hydrolysis reaction of SDS (to dodecanol) [15–17] on our results needs to be addressed. At 25 °C and pH 4 above the CMC, using hydrolysis rate constants [15], the reaction conversion is approximately 99% of that at equilibrium. However, at equilibrium, the reaction proceeds to such a small level as to be undetectable [15]. So the reaction is controlled by equilibrium, not kinetic considerations. At pH levels below about 3.5, hydrolysis can be significant.

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John F. Scamehorn has a BS and MS from the University of Nebraska and a PhD from the University of Texas. He has worked for Shell, Conoco, and DuPont, and is currently a Professor emeritus at University of Okklahoma, where he is director emeritus of the Institute of Surfactant Research. He has been working on surfactant systems and applications to consumer product formulation and separation processes.

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David R. Scheuing received his BS from the University of Illinois at Chicago and is currently Research Director in the Advanced Technology group at the Clorox Company. His research interests include the application of vibrational spectroscopy to the characterization of self-assembled colloidal systems and their interactions with solid surfaces.