

The effect of cosurfactants on sodium dodecyl sulfate micellar structures at a graphite surface

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Abstract

Sodium dodecyl sulfate (SDS) self assembles at hydrophobic graphite surfaces forming linear parallel hemicylinders with approximately 5 nm spacing between adjacent hemicylinders. In this paper, we report the effect of cosurfactants on the SDS surface micelle structures. It is known from the literature that cosurfactants influence the relaxation times of bulk phase micelles. Specifically, the relaxation times of SDS micelles increase dramatically when cosurfactants, such as non-ionic 1-dodecanol (C₁₂OH) or cationic dodecyl trimethyl ammonium bromide (C₁₂TAB) are added, resulting in the formation of stable micelles. Soft contact AFM imaging was used to determine if these increased relaxation times in bulk solution influence the formation of surface micelle structures at the graphite surface. Surface micelles formed by adsorption from SDS only solutions were significantly different from those surface micelles formed from mixed surfactant solutions. The addition of cosurfactants produced kinks and disrupted the otherwise highly ordered linear hemicylindrical SDS structures observed at graphite surfaces. In one case (SDS/C₁₂OH) swollen surface micelles with a spacing of more than 8 nm were observed. Finally, whereas surface micelles form instantaneously from SDS only solutions, the time taken for the equilibrium surface state to be established in the presence of the C₁₂OH cosurfactant was found to be more than 4 h.

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1. Introduction

The self-assembly of surfactants into spheres, cylinders and bilayers in solution is fairly well understood [1–3] and recent soft contact AFM imaging studies have helped visualize these surfactant aggregates at a variety of hydrophobic and hydrophilic surfaces [4,5]. The reported structures include spheres and cylinders on amorphous surfaces, such as silica [4] and highly aligned tubular, cylindrical and hemicylindrical, structures on crystalline substrates, such as mica and graphite [5,6], respectively. Especially, on the hydrophobic graphite surface the self-assembled surface micelle structures were found to be, highly ordered linear and parallel hemicylinders, for a variety of surfactants with differences in head group chemistry and molecular geometry [7–11]. The charac-

teristic dimensions of ~5 nm diameter have been explained by consideration of interfacial forces acting in the system [12].

The hemicylindrical structures observed at the graphite surface have been attributed to the epitaxial adsorption of the initial surfactant molecules in a tail to tail and head to head fashion determined by the crystallography of the graphite surface. In this context, it is important to understand how these surface-mediated micellar structures are influenced by the addition of a cosurfactant, which means competing forces must be considered in this otherwise homogeneous system. Wanless et al. [13] have studied the phase transition of the sodium dodecyl sulfate aggregates at a graphite surface, in the presence of 1-dodecanol. In that study, it was found that below the bulk critical micelle concentration (CMC), the shape of the surfactant structures is altered with the addition of the dodecanol but no changes were observed with the addition of dodecanol to SDS solutions above the bulk CMC. Below the

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CMC, the addition of dodecanol to SDS solutions resulted in two kinds of aggregate structures, swollen hemicylinders (periodicity of ~ 12 nm) and flat layers. Also, they observed some additional defects in the adsorbate structures, which could not be explained. Their study was limited to surfactant concentrations just around CMC. At very high surfactant concentrations, much above the CMC, some additional dynamic aspects of micelles that are described in the following section become very significant.

2. Relaxation times for micelles

A dynamic equilibrium exists between individual surfactant molecules in the bulk solution and the micelle entity. Additionally, the micelles themselves are constantly disintegrating and reforming [14,15]. The kinetics of micelle formation and breakdown has been studied using fast relaxation methods, such as temperature-jump, pressure-jump, ultrasonic absorption, shock-tube methods [16–18], etc. There are two relaxation processes involved in micellar solutions. The first is a fast relaxation time, τ_1 (generally on the order of microseconds), which is associated with the quick exchange of monomers between the micelles and the surrounding bulk solution. This process can be considered to be the collision between surfactant monomers and micelles. The second relaxation time, τ_2 (on the order of milliseconds), is related to the micelle formation and dissolution process. The long relaxation time constant τ_2 is hence an indication of the micelle stability in the solution, and hence controls the surfactant monomer flux available for the adsorption at interfaces.

Micellar relaxation kinetics shows dependence on temperature, pressure and concentration. Consider solutions of SDS, the CMC of which is 8 mM. The slow relaxation time, τ_2 , for SDS solutions increase from 0.01 s at 10 mM to a maximum value of 5 s at 200 mM [19]. It decreases again beyond that concentration. Experimental studies by Shah et al. have shown that this relaxation time has a big impact in many liquid/gas, liquid/liquid and solid/liquid interfacial phenomena involving surfactant adsorption [20–24]. At 200 mM SDS, minimum foamability, maximum single film stability, maximum single bubble volume and a minimum frequency of bubble generation were found. These phenomena were explained based upon the monomer flux to the newly created interfaces. If the micelles in solution are very stable, they cannot provide monomers fast enough to the interface, and thus the interfacial tension remains highest. Therefore, lower foamability, larger single bubble foam volumes and a minimum frequency of bubble generation were found.

Recently, the relaxation time studies were extended to mixed surfactant systems. For example, it has been shown that the stability of SDS micelles can be greatly enhanced by the addition of 1-dodecanol ($C_{12}OH$) [25]. The greater micellar stability is attributed to the strong ion/dipole interactions between the SDS and the dodecanol molecules. Even more significant was the effect of cationic alkylammonium

bromides on SDS micellar stability [26]. In this case, ion/ion interactions and charge neutralization between the surfactants causes the micelles to become even more stable.

The improved understanding regarding the dynamic aspects of mixed micelles and the pronounced effect of relaxation time on various interfacial properties prompted us to study how these relaxation times in the bulk solution would affect the surfactant aggregates at the surface. In this work, we investigated how the increase in relaxation times observed for micelles in bulk solution would affect the micellar structures at the graphite surface. Three different systems with significantly different relaxation times were studied, 100 mM sodium dodecyl sulfate (SDS) ($\tau_2 = 0.15$ s), 100 mM SDS + 5 mol% dodecanol ($C_{12}OH$) ($\tau_2 = 0.96$ s) and 100 mM SDS + 5 mol% dodecyl trimethylammonium bromide ($C_{12}TAB$) ($\tau_2 = 1.35$ s) were studied.

3. Materials and experimental methods

Sodium dodecyl sulfate (>99% purity), dodecyltrimethyl ammonium bromide (>99% purity) and 1-dodecanol (>98% purity) were supplied by Sigma–Aldrich Corp. (St. Louis, MO). The chemicals were used as supplied without further purification. The highly ordered pyrolytic graphite substrate (grade ZYH, Union Carbide) was cleaved with an adhesive tape before each experiment to remove the outermost layer and expose a fresh surface, free from contamination.

Three different solutions, 100 mM sodium dodecyl sulfate, 100 mM SDS + 5 mM dodecanol ($C_{12}OH$), 100 mM SDS + 5 mM dodecyl trimethylammonium bromide ($C_{12}TAB$) were considered. Deionized water (18 M Ω cm) was used in preparing the solutions. The molar ratio of the surfactant to cosurfactant being high (20/1), all the mixed surfactant solutions were transparent with no signs of precipitation.

Images at the graphite surface were captured using a Nanoscope IIIA atomic force microscope (Digital Instruments Inc., Santa Barbara, CA), using silicon ultralevers (Park Scientific, CA) with a nominal spring constant of 0.12 Nm $^{-1}$. All images presented are deflection images, captured at a scan rate of 12 Hz with very low integral and proportional gains.

Before taking the images, the graphite surface was allowed to equilibrate with the solutions of interest for at least 15 min to allow for the stabilization of the cantilever drift. Images were then captured with the force in the soft contact regime, details of which are explained elsewhere [5].

4. Results

4.1. SDS only

The micellar structures observed at the graphite surface for adsorption of SDS from solutions with concentrations of

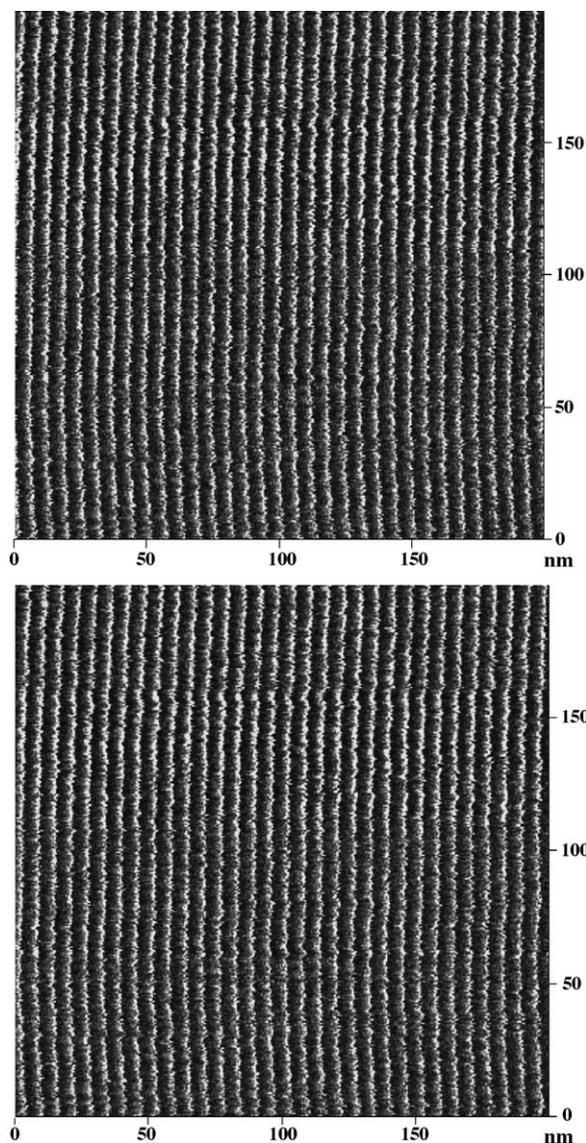


Fig. 1. AFM images of surfactant structures adsorbed at the graphite surface from sodium dodecyl sulfate (SDS) solutions. (A) From 4 mM SDS solution (below CMC) showing a spacing of 6.2 nm. (B) From 100 mM SDS solution showing a spacing of 5.3 nm.

4 and 100 mM are shown in Fig. 1. Periodic surfactant aggregate structures were observed at the graphite surface even at 4 mM SDS, well below the bulk CMC value of 8 mM. In contrast to significant changes in the slow relaxation times for bulk micelle formation, the surface micelles formed almost instantaneously both at high and low concentrations. The images, shown in Fig. 1, are similar to those previously reported in the literature [5,27] showing highly ordered linear and parallel hemicylindrical surface micelles. The shape of the surfactant aggregates did not show any change with an increase in concentration, and hence an increase in relaxation time. The only distinguishable change was the periodicity between the hemicylindrical structures. The periodicity between the hemicylindrical micellar aggregates observed for 4 mM SDS was determined as 6.2 nm, which is in good

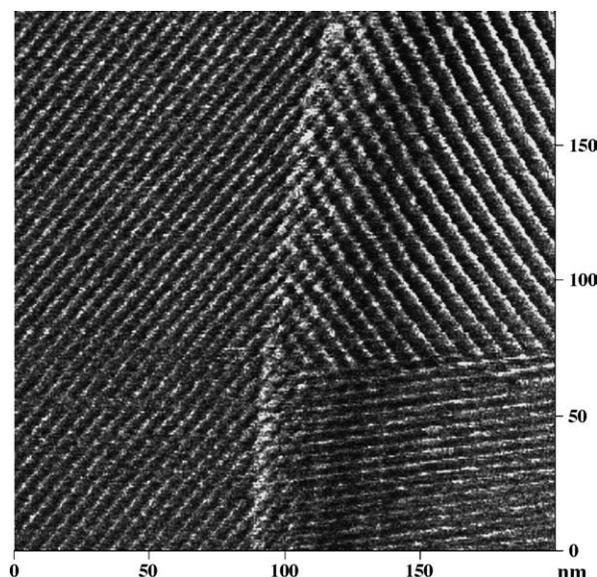


Fig. 2. The presence of steps at the graphite surface changes the alignment of the hemicylindrical surfactant aggregate structures. The above figure shows the existence of two such steps, where the orientation of surfactant aggregates is different on either sides of the steps.

agreement with the values reported in the literature [5,20]. The periodicity decreased to a value of 5.3 nm, for 100 mM SDS solution. This might be due to the fact that increasing the SDS concentration would lead to increased shielding of the head group charges meaning lesser electrostatic repulsions between the surfactant aggregates. Thus, the aggregates could be more closely packed.

The periodic surfactant structures were found to extend unperturbed for lengths exceeding several microns at the graphite surface. Only the presence of a step or a grain boundary on the surface seemed to change the alignment of the surface aggregates, as shown in Fig. 2. The angle between the axes of the surfactant aggregates extending in different directions is approximately 60 or 120°, closely corresponding to the angle of crystallographic axes for the underlying graphite surface.

The SDS micellar structures at a graphite surface are described elsewhere in greater detail [27]. These results are revisited here to provide a reference point for the changes observed in the surfactant aggregate shapes for different relaxation times induced by the addition of cosurfactants.

4.2. SDS/C₁₂OH

No surface micelle structures were observed for the SDS/C₁₂OH system at shorter equilibration times. However, for longer equilibration times exceeding 4 h, very stable and highly distinguishable surfactant aggregate layers were formed on the surface, as shown in Fig. 3. A herring-bone kind of pattern is observed in this case compared to highly ordered linear structures observed for the SDS only system. Upon the addition of C₁₂OH to SDS, regular kinks are pro-

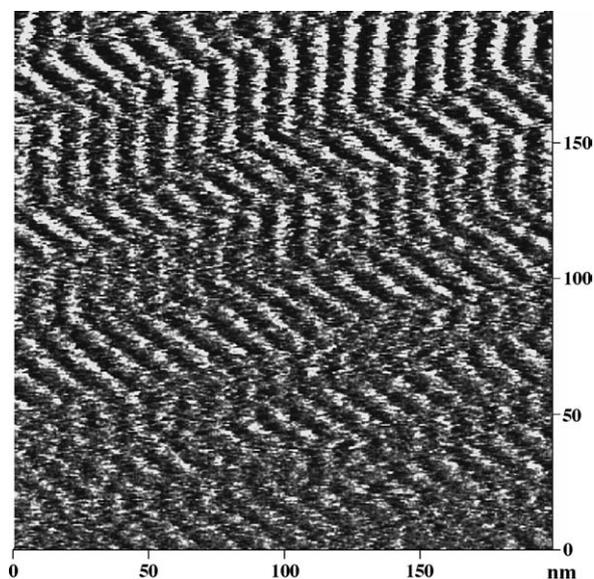


Fig. 3. The image shows the surfactant aggregate structures adsorbed at graphite surface from the 100 mM SDS + 5 mM $C_{12}OH$ mixed surfactant solutions. When compared to Fig. 1 (adsorption from SDS only solution), it can be seen that the surfactant aggregates are swollen. The spacing increased from 5.3 nm in Fig. 1 to 8.2 nm. Also, the highly ordered linear structures in Fig. 1 are replaced by a herring-bone pattern.

duced in the linear structures observed for SDS only system, turning the straight hemicylinders at the graphite surface into the herring-bone pattern. The long range ordering was no longer found but ordering was still persistent on a smaller scale. The herring-bone pattern seems to have periodic length of 30 nm. One other significant observation is that the spacing of the surfactant aggregates in this case (~ 8.5 nm) was significantly greater than that observed for the SDS only system (Figs. 1 and 3). In bulk solutions it has been observed that the ionic micelles swell slightly, increasing from ~ 4 to 5.2 nm, with the addition of long chain alcohols as the dodecanol molecules are solubilized by the micelles [28,29]. The increase in volume would result in an increase in the hydrophobic contribution to the packing parameter [1]. This increase in the packing parameter value suggests that micelles form aggregates of a lower curvature. A more significant effect is observed at the graphite surface, where the swollen hemicylinders have an even lower curvature as revealed by the increase in periodicity to 8.2 nm. There was another significant difference observed between the SDS only system and the SDS/ $C_{12}OH$ system. For the SDS only system, as mentioned previously, the surfactant aggregates were observed to be continuous over extended lengths and changed in direction only when encountered with a step or a grain boundary. But in the SDS/ $C_{12}OH$ system, the surfactant aggregates did not extend continuously but instead in some places featureless regions existed along side the herring-bone pattern. Once such example is shown in Fig. 4, where it can be seen that the herring-bone pattern of the surfactant aggregates is present in the middle, with flat regions existing on top and bottom of the image. The image was scanned from top to bottom and so

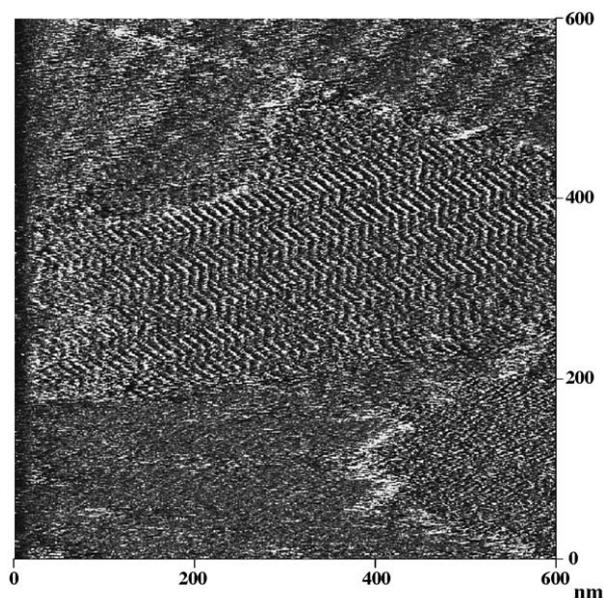


Fig. 4. A larger scale image obtained under the same conditions as Fig. 3 showing the existence of hemicylinders and flat layers on the graphite surface. The image was scanned from top to bottom, hence the presence of some distinguishable structures in the bottom right part of the image confirm that the flat layers are surfactant films and not the underlying graphite surface.

the presence of the patterns towards the bottom right of the image makes it very certain that the flat regions are indeed featureless surfactant films and not the underlying graphite surface. This is not entirely unexpected because even in bulk solutions, the addition of dodecanol to SDS solutions even in trace amounts induces some highly time-dependent phase transitions [30].

4.3. SDS/ $C_{12}TAB$

SDS/ $C_{12}TAB$ is the system with the greatest relaxation times but this was not reflected in the time taken for equilibrium micelle structures to form at the graphite surface. The surface micelles were observed almost as instantaneously (just allowing for system equilibration) as for the SDS only system. However, the shape of the surface micelles, shown in Fig. 5, is significantly different from the previous system, and the structures are more comparable to the surface aggregates observed for the SDS only system. The observed periodicity for this system was approximately 5.4 nm.

A large scale image observed for this system is shown in Fig. 6. Unlike the previous SDS/ $C_{12}OH$ system, the surfactant films seem to cover the entire surface. Even in bulk solutions the SDS/ $C_{12}TAB$ mixed surfactant solutions do not exhibit any kind of a phase separation with only thread like micelles existing in the solution [31,32]. Hence, the results at the surface seem to follow a similar pattern. Also in this case, the orientation of the surfactant aggregates inside a grain boundary was different from the surrounding terrace.

The various characteristics for all three systems are summarized in Table 1.

Table 1
The characteristics of surfactant systems used and surface aggregates observed in this study

Surfactant concentration	Relaxation time, τ_2 (s)	Time taken to form surface micellar structures	Periodicity of surface micelles (nm)	Electrical conductance (μ siemens)
100 mM SDS	0.15	Spontaneous	5.3	5838
100 mM SDS + 5 mM C ₁₂ OH	0.96	Around 4 h	8.2	5787.5
100 mM SDS + 5 mM C ₁₂ TAB	1.35	Spontaneous	5.2	6413.85

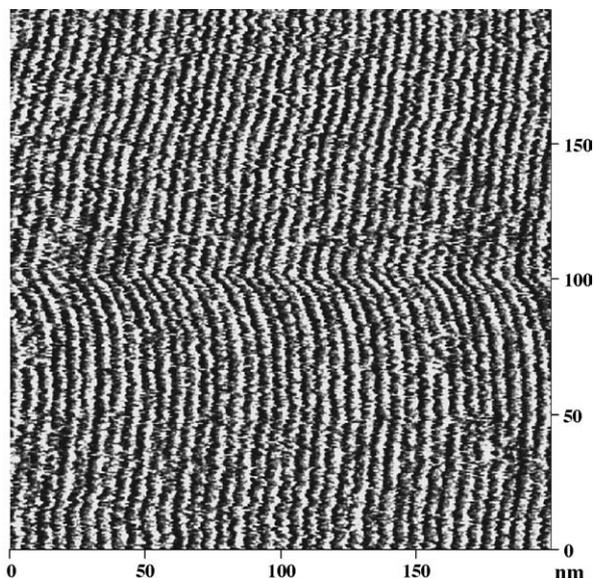


Fig. 5. The image shows the surfactant aggregate structures adsorbed at graphite surface from the 100 mM SDS + 5 mM C₁₂TAB mixed surfactant solution. There are more kinks introduced into the linear structures observed in Fig. 1. However, the structures do not deviate as significantly as in the case of the SDS/C₁₂OH system (Fig. 3). Also, the spacing decreased to 5.2 nm when compared to 5.3 nm for SDS only and 8.2 nm for SDS/C₁₂OH systems.

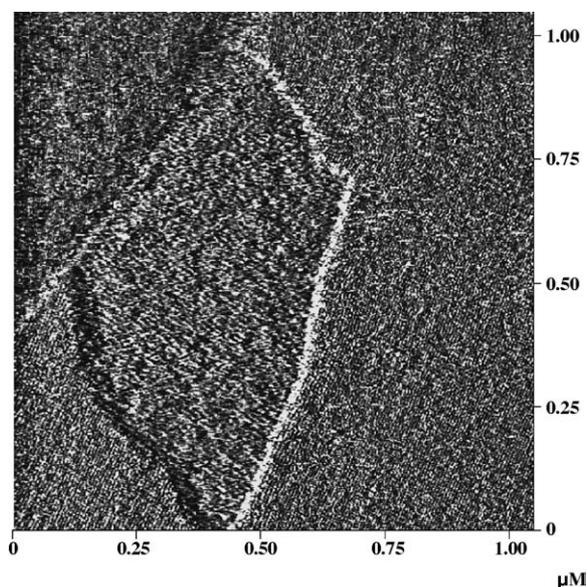


Fig. 6. A large scale image obtained under the same conditions as Fig. 5. The surfactant films are continuous showing no signs of any two-phase regime as in the case of the SDS/C₁₂OH system (Fig. 4). It can also be seen that the alignment of the aggregates inside the grain in the middle of the figure is different from surrounding terrace.

5. Discussion

Before discussing the surface micelle structures observed for the mixed surfactant systems it is important to examine the surface micelles observed for a single surfactant system. Several investigations, using soft contact AFM have revealed that the graphite crystal lattice serves as a template for the organization of surfactant chains and is the defining feature of the surfactant micelle morphology. This central role of the lattice has since been confirmed for a variety of surfactants with wide differences in head group chemistry and molecular geometry [7–11]. Images for the SDS system (Figs. 1 and 2) show linear, parallel aggregates oriented perpendicular to an underlying symmetry axis and spaced apart by a little over twice the surfactant molecular length. These findings are consistent with hemicylindrical structures, wherein molecules adjacent to the surface are arranged tail to tail along parallel strips, with tail groups oriented parallel to a graphite symmetry axis. These initially adsorbed molecules then act as nucleating sites for the growth of the hemicylindrical structures, with further adsorption of surfactant molecules. A schematic for this structure is shown in Fig. 7. It is expected that the spacing between the hemicylinders is determined by the substrate and interfacial forces acting on the system. Only the presence of a step or a grain boundary on the surface could alter the orientation of the hemicylinders (Fig. 3). In other words, when presented with a physical constraint on the surface, the hemicylinders are terminated in one direction and then continue in another direction depending on the orientation of the crystallographic axis of the new grain.

Changes in direction of the hemicylindrical surface micelle structures observed for the mixed surfactant systems are not so easy to explain. It may be that after some accommodation of C₁₂OH in the SDS surface micelle and a swelling to 8.5 nm that a surface micelle saturation point is

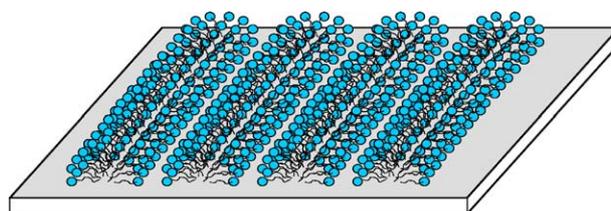


Fig. 7. Schematic of the hemicylindrical micelle structures at a graphite surface. The surfactant molecules initially adsorb in a tail to tail and head to head fashion along the crystallographic planes of the graphite surface. These molecules then act as the nucleation sites for the further adsorption and hence the growth of the surfactant hemicylinders.

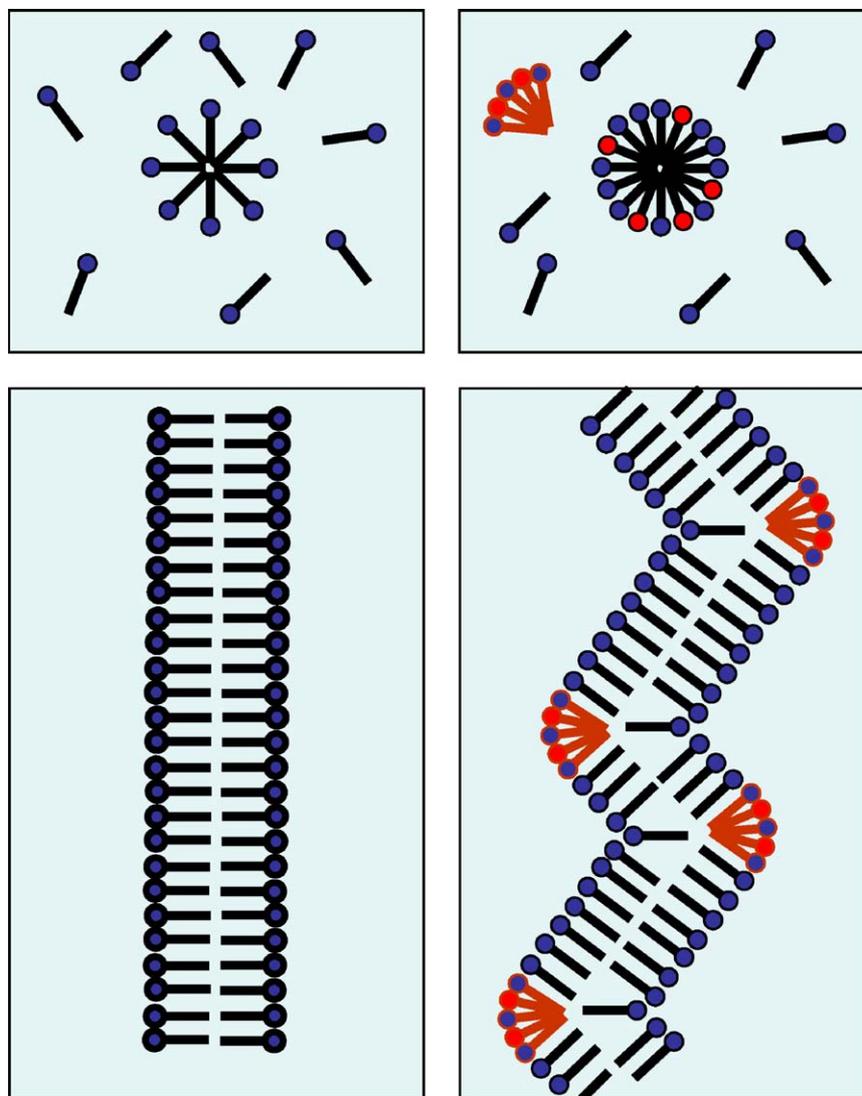


Fig. 8. Schematic of the molecular arrangements of surfactant molecules in the bulk and at the surface in the absence and presence of cosurfactant molecules.

reached and the $C_{12}OH$ is excluded from the surface micelle causing termination of the linear structure. Another possible explanation can be provided considering the dynamics of surfactant solutions. Surfactant solutions above CMC are a dynamic equilibrium between monomers, submicellar aggregates and micelles. The cosurfactant molecules, being smaller in number, adsorb randomly as individual monomers or as submicellar aggregates on the surface. These molecules then act as wedges disrupting the ordering of the SDS hemicylindrical micelles in the same way as a grain boundary or physical imperfection. This hypothetical mechanism is shown schematically on Fig. 8.

It can also be speculated that relaxation times of micelles are related to the shape of the surfactant aggregate structure. In a previous work [13], Wanless et al. have reported swollen hemicylinders and featureless layers when dodecanol was added to SDS solutions below CMC. However, the addition of dodecanol did not have any effect on the surface micelle structures of SDS above the bulk CMC concentra-

tions of SDS and it was suggested that this might be due to the solubilization of the dodecanol inside the bulk phase micelles. The concentration of SDS (100 mM) used in our study is much higher than the bulk CMC value (8 mM) but still swollen hemicylinders could be observed. This is related to the relaxation times of the micelles. In systems with short relaxation times the molecules may not have time to reorganize themselves, and hence the dodecanol can be expected to be solubilized entirely in the bulk phase micelles. But as the relaxation times are increased the dodecanol molecules have enough time to be solubilized into the surface micelles. The swollen hemicylinders in Fig. 3 indeed represent this situation. If the periodicity of the hemicylinders can be taken as the size of one surface micelle, then in the case of the SDS only system, where the spacing is 5.3 nm, the calculations based on the hydrocarbon chain width (0.6 nm) and a packing area of $0.42 \text{ nm}^2/\text{molecule}$, there are approximately seven SDS molecules in one section of the hemicylinder. But in the case of the SDS/ $C_{12}OH$ system, the periodicity of the surface

micelles increased to 8.2 nm. Assuming that everything else remains the same, this increase in size can be attributed to the solubilization of the smaller dodecanol molecules having a packing area of 0.21 nm²/molecule. The composition of the mixed surfactant micelles remains unknown.

For the adsorption of SDS at the graphite surface, the surface micelles are formed instantaneously. So, now if the relaxation times of micelles in the bulk change, the monomer fluxes available for adsorption are less, and hence the adsorption kinetics should also be different. This indeed is the case for the SDS/C₁₂OH system, where it took approximately 4 h to obtain stable surface micelles. It was not the same for the SDS/C₁₂TAB system, where longer times would be expected, because of a greater relaxation time in the bulk. However, the SDS/C₁₂TAB surface structures are formed as rapidly as those formed for the SDS only system, i.e. instantaneously. Although, the relaxation times are greater for the SDS/C₁₂TAB mixture in solution, when provided with an interface, the stronger electrostatic attraction forces between the oppositely charged surfactants give way to the faster accommodation of the molecules at the surface and hence to faster kinetics. To check this postulate the electrical conductance of the solutions was measured. The results, presented in Table 1, show that the electrical conductance is the lowest for the SDS/C₁₂OH system, meaning that least number of monomers is present in that system.

6. Conclusions

The surface micelle structures for mixed surfactant solutions of high relaxation times have been studied. The addition of cosurfactants induced some significant changes in the continuous parallel hemicylindrical surface micelle structures observed for SDS self-assembly at graphite surfaces. The addition of a cosurfactant had the same effect on the surface micelle as structures as graphite surface defects, which perturb the alignment of the surface micelle structures. The orderliness of the hemicylinders observed for the SDS only system was replaced by a herring-bone pattern for the SDS/C₁₂OH system. However, in the case of the SDS/C₁₂TAB system, the structures were more similar to the SDS only system. Also, the time taken for the equilibrium surface state to be established was found to be significantly influenced in the case of the C₁₂OH cosurfac-

tant, when compared to either SDS only or SDS/C₁₂TAB systems.

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