Hysteresis of conductivity in a micellar surfactant solution near the Krafft point

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Abstract: The specific conductivity of aqueous cetyltrimethylammonium bromide solutions has been investigated below and above the critical micelle concentration, in order to elucidate slow structural changes. Around the Krafft temperature (≈25 °C) the monomer solubility reaches the critical micelle concentration, and a significant increase in charge transport is recorded. When a temperature decreases, the micellar surfactant solution passes through the Krafft temperature, and a hysteresis phenomenon is observed with the appearance of crystals in a solution. We have scrutinized the conditions leading to this hysteresis and quantified some of the relevant parameters. We also outline a simple procedure that allows the "erasure" of such structural memory effects, which are potentially detrimental to the formation of adsorbed self-assembled monolayers from solution.

Keywords: surfactants; self-assembled monolayers; the Krafft temperature; conductivity; hysteresis.

INTRODUCTION

The amphiphilic structure of surfactant molecules can give rise to complex effects in solution. Even at small concentrations, surfactants can drastically change the properties of a solution.[1] Above a certain concentration, the critical micelle concentration (*cmc*), solvated monomers (surfactant molecules) self--aggregate into micelles and other more complex structures. This aggregation phenomenon has been studied by the various techniques, including surface tension and conductivity measurements.[2,3]

In this paper we focus on the cationic model surfactant, cetyltrimethylammonium bromide (CTAB), which readily adsorbs onto anionic substrates, such as silica,[4,5] oxide surfaces[6] or muscovite mica.[7,8] Previous work had suggested that the behaviour of CTAB is more complex than the behaviour of other comparable cationic surfactants.[7,9,10] The morphology and physical properties of the organ-

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ized CTAB films, for example muscovite mica, have been studied by different techniques, including the atomic force microscope,[7,8] X-ray photoelectron spectroscopy,[11] the surface forces apparatus[12,13] or electron microscopy techniques often applied in numerous field of science and technology.[14,15] Even at concentrations below the bulk *cmc*, monolayers or bilayers,[16] or small CTAB aggregates can form on the mica surface. Above the *cmc* CTAB tends to form cylindrical aggregates, or monolayers[4] and bilayers.[13] As a result of the numerous adsorption preparation and protocols described in the literature, variety of results have been obtained. Since the quality of adsorbed surfactant layers depends on the solution properties, there is a need to analyse properties of CTAB/water solution in more details. Additionally, to this observation, the variety of different self-assembled monolayers obtained for various designed experimental protocols described in our previous work,[17] confirmed a requirement to look closer into this system and to monitor the properties of the CTAB/water solution.

Low-concentration aqueous solutions of ionic surfactants essentially consist of solvated, dispersed monomers. The solubility, defined as the quantity of CTAB that can be dissolved before reaching saturation is negligible at temperatures below 20 °C.[18] Above the solubility limit the monomers coexist in equilibrium with a crystalline phase. One can observe a small but noticeable increase in solubility with increasing temperature in this regime. When the concentration of dissolved molecules reaches the critical micelle concentration (*cmc*), structures called micelles start to form at the so-called Krafft temperature. Consequently, this temperature is usually defined as the minimum temperature at which surfactants form micelles. Around the Krafft temperature, T_k, many physicochemical properties of the surfactant solution, such as the solution surface tension, or the conductivity reflect this transition.[19] The shape and stability of micelles present in the surfactant solution is determined by various factors, such as ionic and hydrophobic interactions as well as optimal head group area and tail volume.[20] Micelles can thus exhibit a different shape, such as spherical, cylindrical or lamellar. The existence of only spherical micelles in a CTAB solution at temperature 27 °C[21] has been observed. These spherical micelles exist over a concentration range (*cmc*–100*cmc*), and the significant changes in their shape have been detected at higher concentrations.[22,23] The number, *n*, of monomers in a micelle (the so-called aggregation number) is usually for spherical micelles between 50 and 100.[24] Based on the experimental results, it turned out that is possible to define one *cmc* for spherical micelles and another *cmc* for micelles of other shapes.[24,25] The dynamics of micelle formation and the subtle interactions between them have been studied in great detail.[26]

As described above, around T_k many physicochemical properties of the surfactant solution have changed and thus this temperature for the surfactant solution is very important.[27] It is interesting to note that the value of the Krafft tempe-

rature for CTAB/water solution is very different and is defined in the temperature range from 20[10] to 25 °C.[26] Therefore, one can conclude that the changes occurred in solution in a temperature range around T_k, rather than at a strictly defined one temperature value.

It is important to note that many CTAB monolayer preparation protocols apply the solution temperatures around 25 °C, without precise information on monitoring the temperature conditions during the experiment[28]. Using the solution concentration above or below the *cmc* and by the variety of defined preparation protocols, numerous different monolayer morphologies have been observed and described in the literature.[29] History and hysteresis effects of surfactant solutions have rarely been investigated.[30]

In order to follow the surfactant solution properties and to detect the structural changes in CTAB/water solutions, we have carried out the conductivity measurements. Our main intention was to investigate in detail the solution properties of CTAB due to better control of the adsorption process to form homogeneous self-assembled monolayers on muscovite mica. Following conductivity as a function of temperature, we were able to monitor the structural details in CTAB solutions and the behaviour of ionic species and their mobility in the surfactant solution. Therefore, it was possible to look closer into behaviour of this surfactant, especially around the Krafft temperature. Conductance has been measured for different concentrations of aqueous CTAB solutions, below and above the *cmc*.

MATERIALS AND METHODS

In the experiments we used cetyltrimethylammonium bromide CTAB (CTA$^+$Br$^-$), CH$_3$(CH$_2$)$_{15}$N$^+$(CH$_3$)$_3$Br$^-$, purchased from Fluka and ultra-pure water prepared using a Barnstead EASYpure™ batch-fed water purification system (resistivity 18.3 MΩ cm). To remove any contamination and to clean the glassware used in the experiments, we have applied piranha solution (preparation and using is described in the literature[17]). After piranha cleaning, all glassware was rinsed with purified water. For conductivity measurements, the CTAB solutions, above and below the *cmc* (8.9×10^{-4} M[31]), were prepared with ultrapure water.

Special attention was consecrated on temperature control in laboratory conditions, so that both the preparation of the solution and the conductivity measurements were carried out under defined conditions.

In order to ensure the same initial conditions in the experiments, the stock-solution was stored in a refrigerator at 5 °C usually 12 h before heating them up to the temperature used in the experiment. In the conductivity measurements, the temperature was changed (increased and decreased) ranging from 15 and 40 °C, and between 15 and 70 °C. Under certain experimental conditions, the precipitation of crystals in the solution is possible. To avoid this and to produce a uniform solution, we used a small magnetic stirring bar for the constant stirring during the conductivity measurements. The temperature was controlled using an external thermostat connected to a double-walled solution container. The measurements were organised in such a way that the temperature set-point was manually increased every 30 min for 2 °C. In the same time, the surfactant solution temperature was monitored by a thermometer (mercury

thermometer ±0.5 °C). The conductivity measurements was realised by a Portamess 913, instrument (Knick, Germany), with a measurement error <0.5 % of measured conductivity value. In the temperature range between 20 to 28 °C, the temperature was increased only in one-degree steps to detect the structural changes in the solution by more precise conductivity results. All measurements were repeated with the same solution, and with the new prepared solutions. The reproducibility was tested in both groups of measurements.

RESULTS

According to the experimental results in CTAB/mica adsorption process, we have noticed that influence of temperature can be one of the crucial.[17] Due to that, the behaviour of CTAB/water system as a function of temperature is very important. In order to detect the structural changes in CTAB solutions, we have realized conductivity measurements as a function of temperature. According to insights in our previous work,[17] the results presented here represent a comple- ment to them, giving additional and more precise information on the surfactant solution structure in the wide temperature range and under different experimental conditions.

The properties of an aqueous ionic surfactants solution is determined by the mobility of dissolved ions within it and by the micellization process, what is usually affirmed by the conductivity experiments. Conductivity measurements are simple and accurate and thus widely used to determine the Krafft temperature of ionic surfactant solutions, such as CTAB.[32,33] In order to define the properties of CTAB/water solution and the structure of it, we have organized several groups of conductivity measurements. The experiments were begun with the conduct- ivity measurements carried out for different mole ratios of CTAB/water solut- ions, at nominal concentrations below and above the *cmc*, changing the tempe- rature in the range from 15 to 40 °C. A significant change was noticed in the conductivity results between 23.5 and 25.5 °C for all solution concentrations. It was also observed that solution behaves differently at the same temperature, depending on heating or cooling of the solution. This is confirmed by the adsorption result,[17] and by hysteresis in conductivity measurements detected in the complete thermal cycle for CTAB concentration above the *cmc*.[34]

To estimate the surfactant solution behaviour and stability of micelles at the temperature significantly above T_k, we increased the temperature of the solution up to 70 °C. This experiment was realised at a concentration of 30*cmc*, following the solution properties in the process of heating and cooling in the temperature range between 10 and 70 °C (Fig. 1). The results of conductivity measurement, presented in Fig. 1, suggest no significant structural changes in the solution in the temperature interval between 40 and 70 °C.

When we changed some of the experimental conditions, new additional obs- ervations have been made in the conductivity results. For example, if the same solution was successively heated at two different rates, we have detected that

these treatments have a big influence on the conductivity measurements. The results given in Fig. 2, point out the importance of the heating rate of the solution, usually neglected in the experiments.
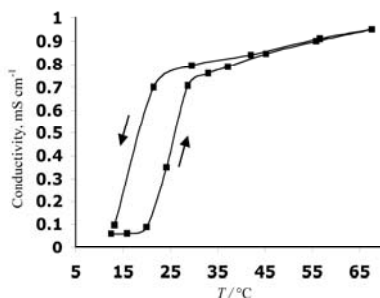

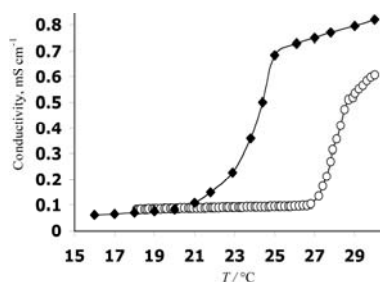Fig. 1. Hysteresis in conductivity of CTAB at concentration 30*cmc* in the temperature range 10–70°C.

Fig. 2. Conductivity *vs.* temperature curves of aqueous CTAB solution obtained at different heating rate. The onset of micelle formation appears at 27 °C for a higher heating rate (open circle), and at 21°C for a lower heating rate (solid rhombus); concentration of CTAB is 30*cmc*.

The structural transformation (*i.e.*, rise in conductivity) seems to occur at 27 °C for the higher rate (4 °C/h) and at 21 °C for the lower rate (2 °C/h). This difference clearly demonstrates the non-equilibrium character of most of these experiments and we would indeed expect to observe a hysteresis in this system upon cooling.

The fact that is important to emphasize is, that the measurements presented in Figs. 1 and 2, were realized by measuring the conductivity every 30 min, *i.e.*, without complete equilibration of the system. The equilibration was achieved by waiting at a constant temperature for certain time. In order to determine the equilibrium state of the micellar system, we stopped the temperature ramp at chosen temperatures between 15 and 35 °C. A representative example for the temporal evolution of the conductivity towards equilibrium is depicted in Fig. 3.

The required equilibration time varied between minutes and weeks, depending on the equilibration temperature. The solution was cooled from initially 40 °C to the selected equilibration temperature, after which the relaxation of the system was followed by conductivity measurements. The temperature stability was better than ±0.05 °C. The set of final conductivities after equilibration is referred to as the experimental "equilibrium conductivity curve", shown in Fig. 4.
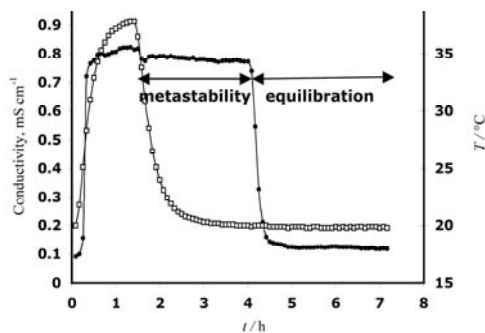
Fig. 3. Dependence of both, conductivity (solid circle) and temperature (open square) on the required equilibration time. The "equilibration curve" at 20 °C for aqueous CTAB solution, with nominal concentration of 30*cmc*.
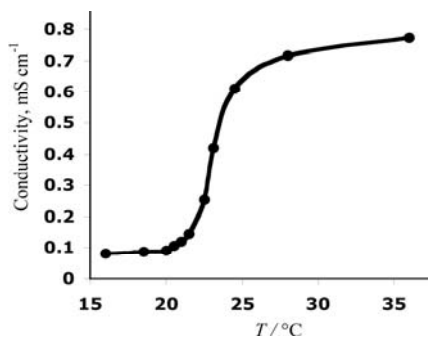
Fig. 4. An "equilibrium conductivity curve" is showing the conductivity as a function of temperature after thermodynamic equilibration; concentration of CTAB is 30*cmc*.

DISCUSSION

The observed conductivity measurements, presented here, indicate substantial conductivity increase of the surfactant solution around the Krafft temperature for concentrations above the *cmc*. The Krafft temperature is very important for the surfactant solution behaviour, due to big changes in the solution properties around it. For our experiments it is important fact that micelles are started to be formed at the Krafft temperature, when the solubility of the surfactant monomers become equal to the *cmc*. This connection between the dramatic change of surfactant solubility and the Krafft temperature have been studied very often and was described in the literature many years ago.[34] Above the Krafft temperature, the monomeric surfactant ions and counterions, as well as the significant number of micelles have the influence on solution conductivity. In these areas we can expect very small increase in conductivity due to monomers, and the big change in the conductivity is obviously consequence of the numerous micelles formed in the solution.[35]

In order to determine the behaviour of the solution at higher temperatures, at certain concentrations above the *cmc*, the temperature is increased further up to 70 °C, in some experiments At a concentration 30*cmc* we have observed monotonic increase in the conductivity with temperature increase above 40 °C (Fig. 1). These results indicate that there are no significant qualitative changes in the sol-

ution. The conductivity is affected by presence of monomers and the groups of monomers-micelles.

The measured conductivity depends on charge carriers, such as micelles and monomers. The significant influence on conductivity can also have the ratio between them, as well as changes of morphology of the micelles, their shape and mobility. Due to that, it is very interesting to understand the structure of the surf-actant solution changes as a function of concentration above the *cmc* and in the temperature range above the Krafft temperature. To answer this question we normalized the data by dividing the conductivity by the concentration, in units of *cmc* (Fig. 5).
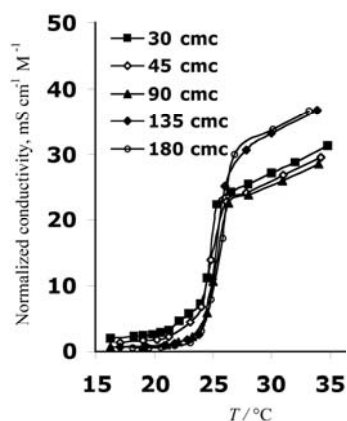

Fig. 5. Normalized conductivity *vs.* temperature curves for various different nominal concentrations of aqueous CTAB solution.

We observed that the normalized conductivities essentially reduce to two different master curves, suggesting that there are two qualitatively different regimes of aggregation morphology in this CTAB solution. A possible explanation for the existence of a second master curve at higher concentrations (for concentrations 135*cmc* and 180*cmc*) is a change in the micellar shape. The change of micelles shapes at higher concentrations is already described in the literature and the explanation for this phenomenon is sometimes related to the existence of a secondary *cmc*.[31] At this concentration micelles change shape from spherical into more complex form, what for CTAB solutions was detected in the wide range[36]. From our experimental conductivity results such the noticeable changes were detected for concentrations above 90*cmc*, and are usually accompanied by significantly complex hysteresis.[17] A more precise explanations and description of obtained results from the point of the structural solution composition is difficult to bring, because only conductivity measurements are not enough.

There are different explanations in the literature as to how exactly the conductivity changes as a function of temperature, within the Krafft temperature phenomenon. One viewpoint proposes the existence of two characteristic points

on the conductivity curve, the Krafft point and the Krafft temperature.[37,38] The Krafft point is defined as the point where the concentration of the monomers becomes equal to the *cmc* and the micelles have started to form.

Instead of a distinction between a Krafft point and a Krafft temperature, some authors suggest the existence of a temperature range between them, the so-called "micelle temperature range",[39] claiming that this is a term that can more precisely explain the behaviour of surfactant solutions. But, from the literature overview of the Krafft temperature phenomenon, the difference between the Krafft point and the Krafft temperature is still a matter of debate.

The presence of hysteresis is observed in many self-assembling systems.[40] For such systems, there are not only important values of the parameters under which the phenomena occurring (such as temperature), but also the history of the system. These results indicate, for example, that the temperature of a stock-solution preparation may be very important for a following experiment.

Interesting observation in our conductivity measurements is that it is possible to realize the reproducibility in the conductivity results. A necessary condition that provides to "erase" memory effects in the solution is the process of cooling of the solution, what we have realized by storing the solution in a refrigerator (usually more than 10 h at 5 °C). Following this procedure, exactly the same conductivity results have been observed with the same solution after storing the solution in a refrigerator.[34]

At this point we would like to draw the reader's attention to the fact that the system exhibited a significant increase in conductivity fluctuations at a well-defined temperature. The evolution of the conductivity towards equilibrium at this particular temperature of 23 °C for aqueous CTAB solution with nominal concentrations of 30*cmc* is shown in Fig. 6. Here, the conductivity revealed a more complex, irregular behaviour, with longer equilibration and relaxation times than at the other temperatures, presumably due to the very significant slope of the conductivity-temperature curve at this temperature (*c.f.*, Fig. 5).
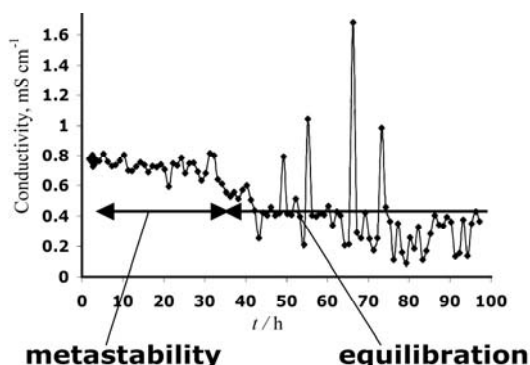

Fig. 6. The conductivity "equilibration curve" at 23 °C for aqueous CTAB solution with nominal concentrations of 30*cmc*.

As mentioned above, in many experiments in the conductivity measurements the temperature was changed after 30 min, and the conductivity was recorded without temperature equilibration. A very important question is how long one has to maintain the solution at a certain temperature to reach the equilibrium?

For the sake of simplicity, we have reduced this discussion to the cooling part of the problem, as shown for the typical down-equilibration in Fig. 3. In some cases, equilibration required waiting times of several days and at temperatures close to the Krafft temperature, the equilibration times became indeed very long (Fig. 6).

Upon cooling, we observed a delay time or "memory effect" before changes became apparent in the conductivity. This effect strongly depended on temperature. It is interesting to note that the system resides in such a metastable state during a well-defined time prior to the relaxation. The linear dependence of the time needed to reach an equilibration *vs.* equilibration temperature is depicted in Fig. 7. The extrapolation of obtained dependence to low temperatures can be used to predict how rapidly one can "erase" the structural memory.
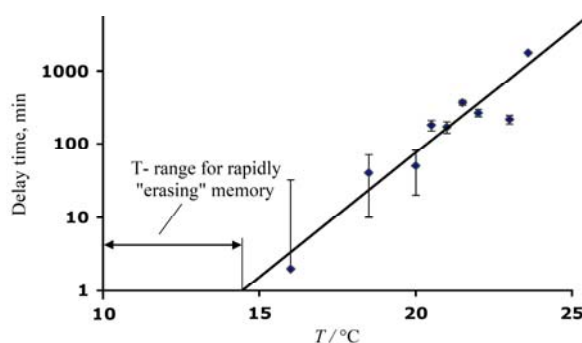

Fig. 7. The dependence of the time to equilibration on the equilibration temperature.

This behaviour is reminiscent of a nucleation process or an activation barrier for the disintegration of the micelles. This observation has practical consequences for the use of CTAB solutions. It shows that equilibration times of several hours or days are required when working around ambient. It also shows the existence of a structural memory effect, which can be "erased" within reasonably short times at temperatures below 15 °C.

CONCLUSIONS

Conductivity measurements of CTAB solutions at various temperatures and concentrations were carried out, which allowed to draw conclusions about their structural properties. The solubility of CTAB is a lot different in two areas, below and above the Krafft temperature. Due to low solubility of CTAB below

the Krafft temperature, we can consider that there is equilibrium between surf-actant monomers and the solid phase.

During the heating at temperatures in vicinity of the Krafft temperature, conductivity of CTAB solution increases sharply with temperature increase due to enhanced solubility of CTAB. Above the Krafft temperature, the slope of con-ductivity *vs*. temperature curve decreases indicating monomers aggregation and micelles formation. When the micellar solution is cooled below the Krafft tempe-rature, crystals will precipitate and the solution becomes visibly turbid. A repro-ducible conductivity hysteresis is detected in heating–cooling cycles. From the conductivity results we can signify that two additional observations, such as time-effects and the thermal history of CTAB solution, are very important for the appropriate preparation and utilization of these surfactant solutions. From a sys-tematic study of thermal equilibration, the characteristic time intervals have been determined at different equilibration temperatures. It is clear that history and memory effects are important when working with these solutions and the time required to "erase" such memory effects and obtain a reproducible solution struc-ture is highly temperature dependent.

И З В О Д
ХИСТЕРЕЗИС ПРОВОДЉИВОСТИ У МИЦЕЛАРНОМ РАСТВОРУ СУРФАКТАНТА У ОКОЛИНИ КРАФТОВЕ ТЕМПЕРАТУРЕ

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Структурне промене воденог раствора цетилтриметиламонијум-бромида (CTAB) изучаване су мерењем специфичне проводљивости раствора сурфактанта у функцији температуре, и то испод и изнад критичне мицеларне концентрације. Повећавањем температуре раствора сурфактанта уочено је да око тзв. Крафтове температуре ($\approx$ 25 °C) растворљивост мономера достиже критичну мицеларну концентрацију, када је и детектован значајан пораст броја наелектрисаних честица у раствору. При смањењу температуре, раствор сурфактанта пролази опет кроз Крафтову температуру и уочена је појава хистерезиса при праћењу проводљивости, уз присуство кристала у раствору. Преиспитани су услови који доводе до појаве хистерезиса и идентификовани неки од релевантних параметара. Приказана је такође и процедура која омогућава "брисање" таквих структурних меморијских ефеката, који су потенцијално нежељени у поступку адсорпције танких филмова из оваквих раствора.

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