Experimental report

29/09/2016

Proposal: **9-10-1462** **Council:** 4/2016

Title: Electric field induced adsorption of sodium dodecyl sulfate to a silica surface

Research area: Soft condensed matter

This proposal is a new proposal

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Samples: D2O and H2O
CH3(CH2)11SO4Na

Instrument	Requested days	Allocated days	From	To
D17	3	2	04/07/2016	06/07/2016

Abstract:

The proposed experiments will directly determine the influence of surface potential on the packing of surfactant molecules at a silica/solution interface. Competitive effects of hydrophobic interactions, solvation and molecular and ionic packing are all known to influence the structure and the amount of material in adsorbed layers. Neutron reflection measurements with an applied electric potential permit the determination of the role of potential and results will be compared with previous work where simulations have been made and studies where pH is used to change adsorption but also influences simultaneously the activity of molecules in solution.

Introduction

The aim of the experiment is to determine the influence of surface potential on the adsorption of surfactant molecules at silica/water interface. Due to the repulsive force between similar charges, the adsorption of anionic surfactants to surfaces with a negative potential, such as that of silica at pH 7, is known to be insignificant. In this experiment, we applied an external DC field to create a positive potential and study the forced adsorption to the surface.

Experiment and measurements

The sample holder for this experiment is a cell similar to our previous experiments [1] with modifications to apply the voltage. The solution is sandwiched between two silicon crystals; and silicon is sufficiently conducting to transfer charge through the circuit. Aluminium plates are in direct contact with the crystals and connected to the external circuit with voltage controlled by a source meter. The plates are separated from the frame of the cell by custom printed plastic sheets. Silicon crystals act as voltage dividers; therefore significantly higher voltage should be applied to the circuit in order to have sufficient potential across the dispersion [2]. More detailed estimates of the division of potential across the oxide and the bound layer will be made along the lines proposed recently by Hemmerle et al.

SDS in D_2O at 10 × the criticical micelle concentration (cmc) was prepared as the sample stock and diluted during the experiment using an HPLC pump. The surfaces were characterized in three contrasts of water and then 0.3 cmc SDS was injected into the cell (no effect of adsorption: reflectivity pattern similar to that of Si/D_2O). The measurements were then made in the following order:

1. V = + 250 mV was applied to one crystal while the other crystal was kept as ground; the structure were measured at 0.3, 0.6 and 1 cmc on both surfaces: no change was observed in the reflectivity pattern compare to the Si/D_2O interface.

2. At a fixed concentration of 1 cmc, the voltage was increased and the reflection was measured at +0.5 V, +1 V, +1.5 V and +3 V. Figure 1 shows these data. When 3 V was applied across the circuit, there was sufficient potential at the silica solution interface to change the adsorption. Figure 3 shows a model fit to the data that corresponds to the formation of a 20 Å layer of 20% SDS and 80% D_2O on Si/SiO_2. The adsorbed layer was found to remain after extensive rinsing with water and setting the voltage to zero.

3. Applying +3 V was seen to provide sufficient field to drive the molecules towards the interface. The effect of adding more SDS was tested by increasing the concentration up to 10 × cmc. Figure 2 shows data measured when applying +3 V to 1, 2, 5 and 10 × cmc SDS in D_2O. No significant changes were observed. This contrasts with a maximum adsorption found above the cmc [3] at an alumina/solution surface.

4. The voltage was increased to +5 V with a 10 cmc solution in the cell and significant changes were observed in the reflectivity pattern. A preliminary fit to the data (figure 3) shows the formation of a 30 Å thick layer that consists of approximately 50% SDS and 50% D_2O on silicon.

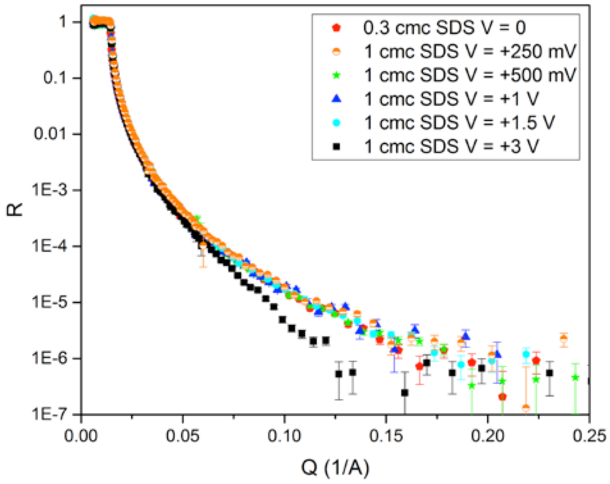

Figure 1. The effect of potential on adsorption of SDS at 1 cmc. Reflectivity data at V = +3 V (where changes are seen) is shown with a fit in Figure 3.

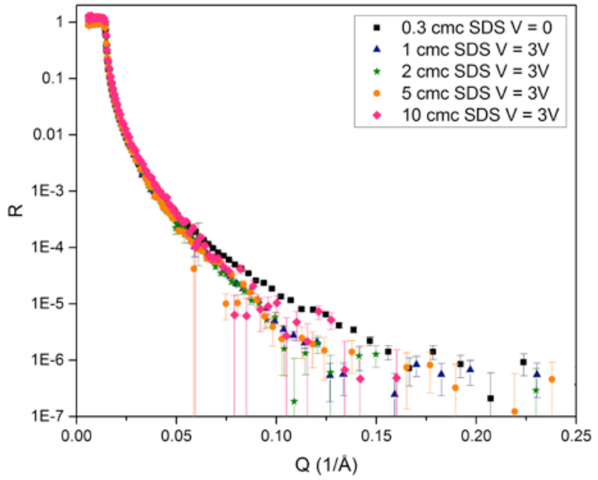

Figure 2. The effect of concentration on the adsorption of SDS at an applied potential,V = +5 V.

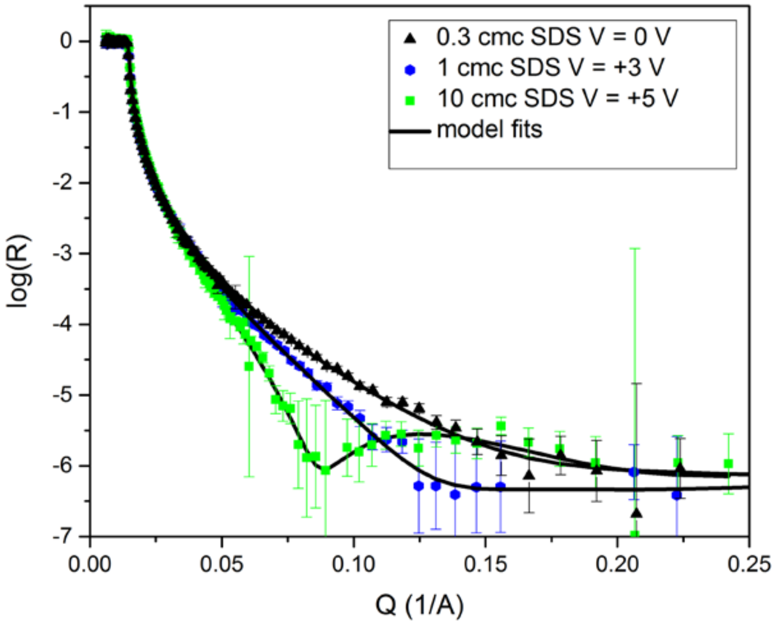

Figure 3. Data points and model fits for Si/SiO_2/water-SDS solution when:

No field is applied to the SDS solution (same as silicon/D_2O scan): red triangles.

+3 V is applied to 1 cmc (similar to that of 2,5 and 10 cmc at V = +3 V: dark blue circles.

V = +5 V is applied to 10 cmc SDS: light blue squares.

Conclusions:

1. SDS surfactant, which on its own does not adsorb to negatively charge surfaces, was strongly absorbed to a silica layer on a silicon crystal under the influence of applied external electric field.

2. The adsorption above the cmc did not increase with concentration. This contrasts with the behaviour seen at alumina surfaces [3]. Further studies are required in order to investigate the variation of adsorption below the cmc at sufficiently high voltage (~ 3V).

3. On increasing the voltage to +5 V, the surface coverage increased more than three times.

4. Switching off the voltage, and rinsing with water and ethanol did not remove the layer. Further studies are required in order to investigate whether the adsorption is irreversible under reversed potential.

References

1. A. R. Rennie et al. Rev. Sci. Instrum. 2015, 86, 016115.
2. A. Hemmerle, et. al. Phys. Rev. Lett. , 2016, 116, 228101.
3. N. Li, et al. J Colloid Interface Sci. 2016, 471, 81-88.