Experimental report

04/10/2022

Proposal: 9-10-1697 **Council:** 10/2020

Title: Following surface freezing transition inside the foam

Research area: Soft condensed matter

This proposal is a new proposal

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Samples: surfactant (CTAB) + oil (tetradecane) + H2O+ D2O

Instrument	Requested days	Allocated days	From	To
D16	7	7	28/05/2021	31/05/2021
			28/06/2021	01/07/2021
			15/09/2021	17/09/2021

Abstract:

We want to measure a surfactant surface freezing transition at the air/water interface insitu in a foam to correlate the surface structure with foam stability. We will use a system composed of surfactant and oil. We will also follow the evolution of the thickness of the surface layer during the evolution of the foam structure. We need neutron scattering to obtain sufficient contrast of the interfaces by matching air and the solvent using a nul mixture of D2O:H2O (8:92).

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D16 – Viviana Cristiglio

Following surface freezing transition inside the foam

Anna Kharlamova, Chiara Guidolin, Stéphan Rouzière, Fabrice Cousin, Anniina Salonen

It is possible to stabilise foams with surfactant crystals. In particular we have previously shown that sodium dodecyl sulphate precipitated in the presence of a high concentration of potassium chloride can be an excellent foam stabiliser[1]. These foams are destabilised when heated to a temperature above the melting temperature of the crystals, which means that they are thermostimulable. Examples of crystal stabilised bubbles and the disappearance once heated are shown in Figure 1.

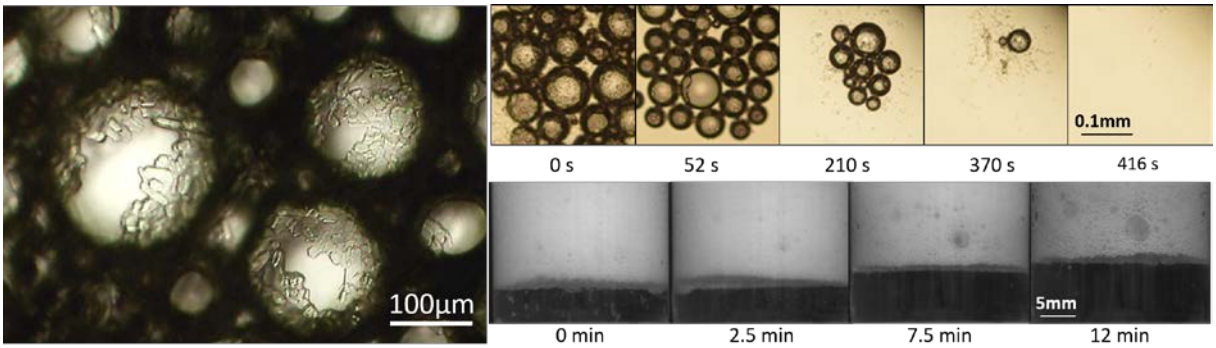

Figure 1 Surfactant crystals on bubble surfaces visualised under the microscope (left). Destabilisation of bubbles and foam thorugh heating (right).

During the experiments on D16 we explored the formation of the surfactant crystals on the bubble surfaces and their melting. This required experiments with four different q-ranges to span the range from the crystalline surfactant structure to the scattering by the bubble surfaces. The foams were generated just before filling the 1 or 2 mm glass capillaries, which were then placed in the temperature controlled sample holder. We used a fluorinated gas to slow down the evolution of the foams (this slows down strongly the increase of the bubble size through coarsening as the solubility of the gas is lower than that of air).

In Figure 2 the scattering from a foam made with deuterated SDS precipitated with KCl is shown. We can see the increase at low *q* characteristic of the scattering from the bubble surfaces. In addition we can see peaks from the crystalline structure of the precipitated SDS. The crystal structure of SDS has a lamellar spacing of 34 Å and there is no difference with deuterated and hydrogenated surfactants.

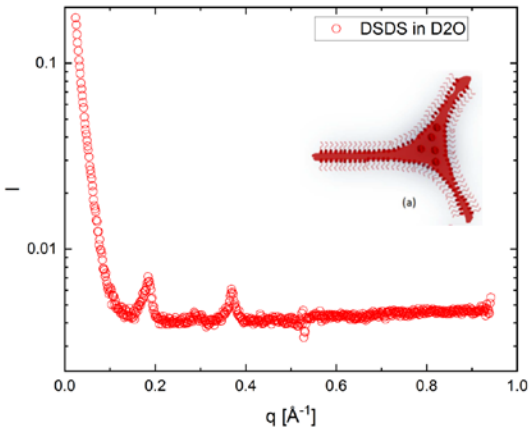

Figure 2 Scattered intensity as a function of q-vector for foam stabilised with DSDS precipitated in the presence of KCl.

We measured the crystals in the foams in a range of foams and varied the amount of salt used to precipitate the surfactant and the fraction of liquid inside the foams (very dry

foams to wetter foams). We also explored the evolution of the crystals inside the foams in time.

We also studied the foams in different contrast configurations. Using deuterated and hydrogenated surfactant and two different solvents (nul water, D2O) allowed us to highlight the adsorption of the crystals on the interfaces. Results for a single surfactant and salt concentration are shown in Figure 3.

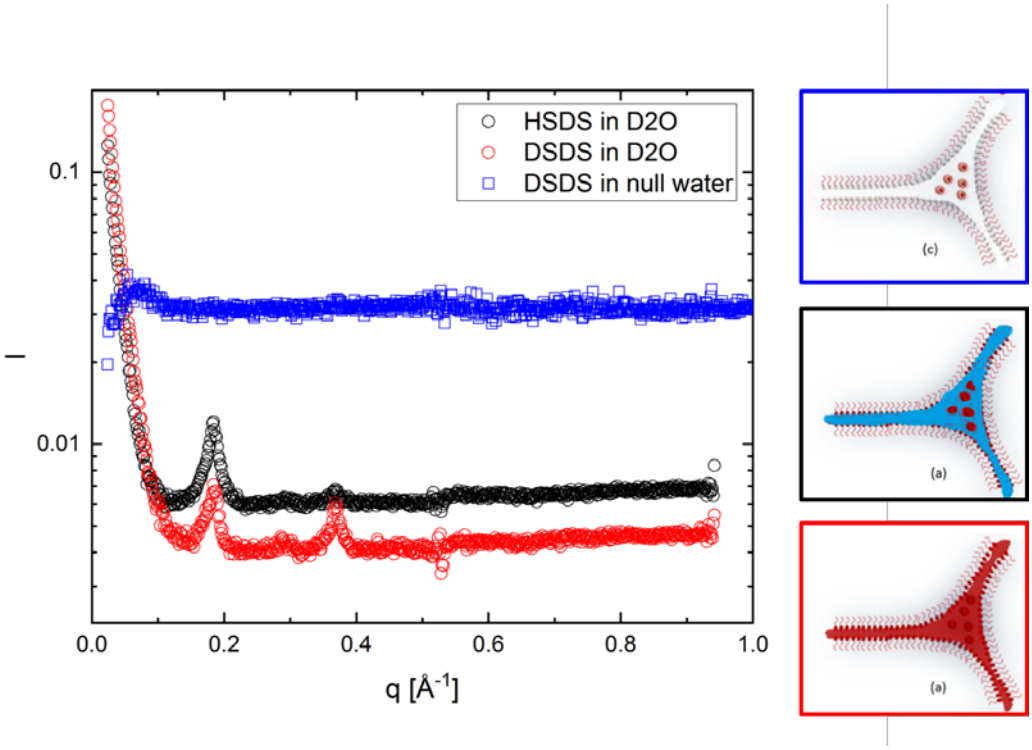

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Figure 3 Scattered intensity of foams stabilised with SDS precipitated with KCl in three different contrast conditions (details of the contrasts in the text).

We can notice the high incoherent scattering from nul water from the high fraction of H2O in the solvent. We notice that the scattering from the gas/liquid interfaces is effectively masked at the upturn at low *q* is no longer visible. No crystal structure peaks are visible, but they might can be hidden in the high incoherent background.

The scattering of HSDS in D2O is shown with the black points and of DSDS in D2O in red points. The intensity of the HSDS is higher due to the incoherent scattering of hydrogen in the SDS. The crystal lattice parameter remains unchaged between the HSDS and DSDS, however we notice a strong change in the intensity of the second peak. This is might be because the scattering cross-section of HSDS is close to that of air, and so the scattered intensity of the first crystal layer is much weaker than in the case of DSDS in D2O. However, this hypothesis requires further experimental verification.

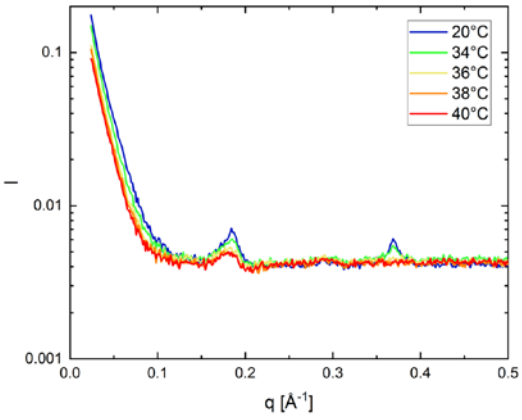

Figure 4 Scattered intensity as a function of q-vector for a foam stabilised with deuterated SDS precipitated in the presence of KCl at different temperatures shown in label.

We also explored the evolution of the foams when changing temperature. The crystals

melt if heated above the Krafft boundary. We heated the samples from 20°C to 40°C and measured the scattered intensity at the different temperatures. The scattered intensity at the different temperatures is shown in Figure 4 (starting in blue at 20°C and going towards red at 40°C). A gradual decrease of the surfactant precipitate peaks is observed as the crystals melt in the bulk. We notice that the first peak remains rather intense even at 40°C although the 2nd peak has completely disappeared, which might suggest that melting at the surfaces and in the bulk does not occur at the same time.

Foams with SDS and NaCl were also studied. The crystal structure is less visible in the example shown in Figure 5. The crystals are difficult to measure in the foams, although if bulk crystals are used the first peak becomes visible.

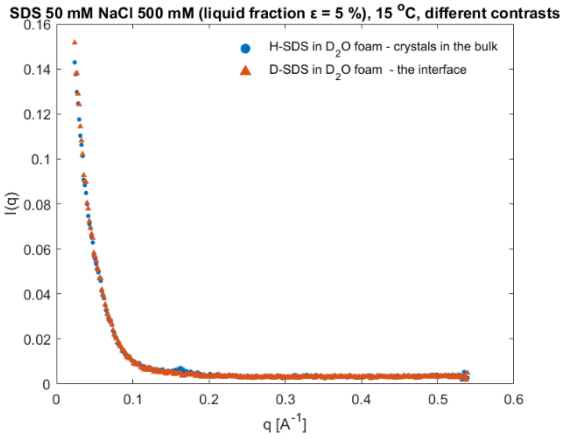

Figure 5 Scattered intensity as a function of q-vector for a foam stabilised with deuterated SDS precipitated in the presence of NaCl.

Conclusion

In conclusion we have studied surfactant precipitate inside foams, the results are interesting and we hope to write them up into a publication in the near future.