EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:
https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Deadlines for submission of Experimental Reports

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

Experiment Report supporting a new proposal ("relevant report")

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a *"preliminary report"),*
- even for experiments whose scientific area is different form the scientific area of the new proposal,
- carried out on CRG beamlines.

You must then register the report(s) as "relevant report(s)" in the new application form for beam time.

Deadlines for submitting a report supporting a new proposal

- 1st March Proposal Round - **5th March**
- 10th September Proposal Round - **13th September**

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report in English.
- include the experiment number to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** Surfactant mediated film formation of 2D materials at the air-water interface	**Experiment number**: SC 5354
Beamline: ID10	**Date of experiment**: from: 16.02.2023 to: 20.02.2023	**Date of report**: 24.03.2023
Shifts: 12	**Local contact(s)**: Oleg Konovalev	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Proposal Objective

In this proposal, we want to understand the precise role that the surfactant monolayers at the air-water interface play for the reaction dynamics of 2D polymers (2DPs) using surfactant monolayer-assisted interfacial synthesis (SMAIS). The final goal is to investigate the relation between different surfactant monolayers, the supramolecular pre-organization of monomers, and eventually the crystallinity of the resultant 2DPs. In this particular proposal, we will focus on the first two steps of the SMAIS process, the surfactant monolayer formation and the conformational orientation of the monomers at the surfactant layer. The formation of the surfactant is only the first step in the SMAIS process, but is crucial for the pre-organization of the monomer and eventually the polymerization of the 2DP.

Results

The first task before being able to analyze the acquired data was to convert it into 2D-maps in Q-space, which could be imported into our visualisation and analysis software WxDiff. With the help of Oleg Konovalov we were able to write a python code to convert the acquired data into binary files with quadratic pixel sizes, which allows the data analysis with WxDiff.

With the data conversion taken care of, we first focused on the monolayer assembly of the various tested surfactants. Within this report, we will turn our attention to the surfactants octadecylamine (ODA) and sodium dodecylbenzenesulfonate (SDBS) and mixtures of these two used in a small rectangular PTFE trough with water as subphase. The molecular structure of the two surfactants is shown in Figure 1a. The in-plane intensity profiles of the pure surfactants and various mixing ratios of the two are depicted in Figure 1b. There it can be seen that the position of the diffraction peaks is changing for different mixing ratios. For the pure ODA (Figure 1c left) we observe two diffraction peaks at 1.51 $Å^{-1}$ and 1.67 $Å^{-1}$. These peaks are very similar to other ionic surfactants like sodium oleyl sulfate and represent a 2D unit cell with a = 7.5 Å b = 5.0 Å and $\gamma = 90°$. The diffraction pattern of a mixture of ODA:SDBS with a ratio of 9:1 (Figure 1c middle) showed a significant change with peaks at 1.25 $Å^{-1}$, 2.16 $Å^{-1}$ and 2.50 $Å^{-1}$. These peaks can be indexed as the 01, 11 and 02 planes of a unit cell with a = 5.81 Å, b = 5.81 Å and $\gamma = 120°$. For the mixing ratios of 8:2 and 7:3 there is almost no change to the position of the diffraction peaks. The diffraction pattern then changes again significantly when using a ratio of 6:4 (Figure 1c right). We can now observe two peaks at 1.42 $Å^{-1}$ and 1.50 $Å^{-1}$. With only these two peaks visible

it is difficult to index them and assign a unit cell since there a multiple solutions that could result in this pattern. One possible solution with a = b that can explain the peaks is a = 4.93 Å, b = 4.93 Å and γ = 63.8°. For verification of this unit cell either additional measurements for example in a langmuir trough with defined surface pressure or theoretical calculations will be necessary. If we add even more SDBS to the ODA, e.g. 5:5 or 3:7, the diffraction signals completely disappear indicating the loss of structural order of the surfactant film. This seems to fit with the fact that the pure SDBS also does not show any diffraction peaks leading to the conclusion that the order in the layer is lost when the SDBS becomes dominant.

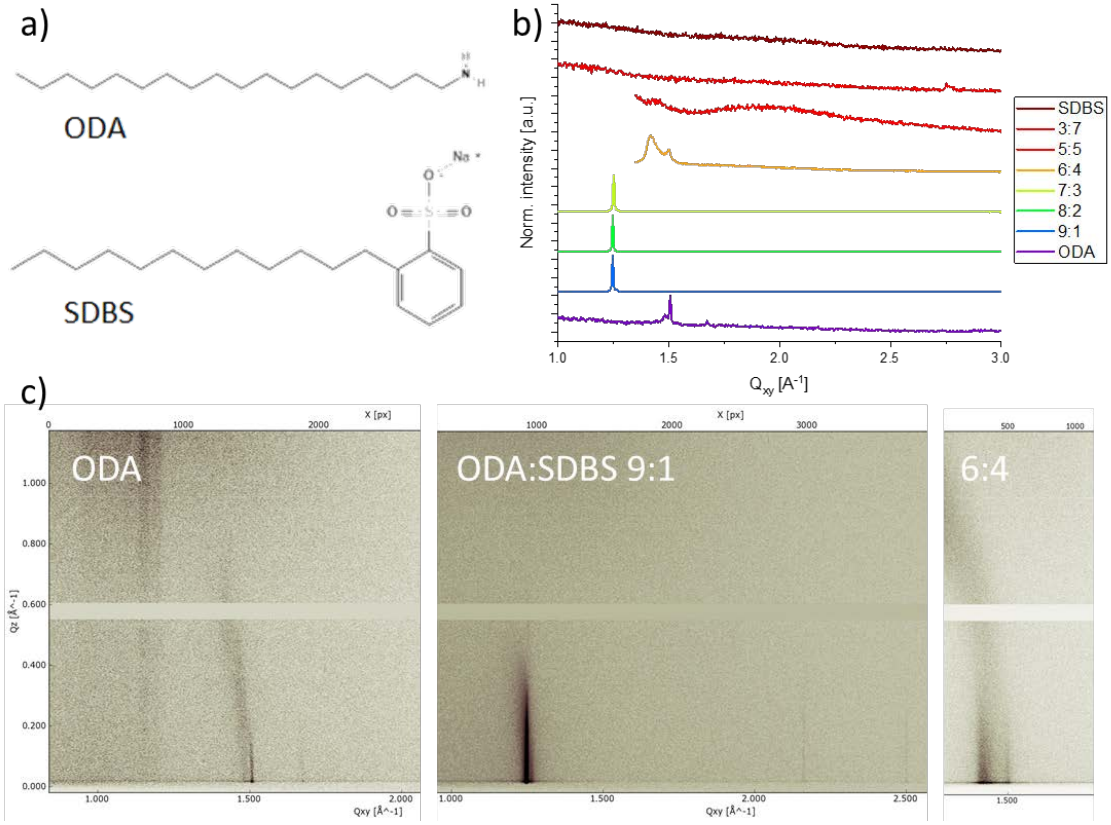

Figure 1: a) Chemical structure of the two surfactants octadecylamine (ODA) and sodium dodecylbenzenesulfonate (SDBS), b) in-plane intensity profiles of the pure surfactants and various mixing ratios of the two and c) 2D GIWAXS images of pure ODA and two different mixing ratios to show the changes in the diffraction patterns.

Outlook

Further analysis of the collected data is currently ongoing with a focus on the monomer assembly. Now that we have seen that we are able to relaibly collect data for various monolayers of surfactants and the assembly of some of our monomers we are planning to submit a proposal that builds on the results of this beamtime and focuses on the polymerization after the surfactant mediated assembly of the monomers. We expect to be able to publish the combined results in a high-impact journal due to the novelty of studying the polymerization process of 2D polymers by in-situ GIWAXS experiments.