EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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:** Dynamic structure formation of particles and lipids at the oil/water interface	**Experiment number**: CH-6321
Beamline: ID10	**Date of experiment**: from: 2.3.22 to: 7.3.22	**Date of report**: 3.5.2022
Shifts: 15	**Local contact(s)**: Dr. Maciej Jankowski	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

During this beam time, we investigated the decane/water interface at ESRF beamline ID10 predominantly with grazing incidence small angle X-ray scattering (GISAXS), while cross-referencing particle immersion and packing with x-ray reflectometry (XRR) to ensure that the experiment is comparable to our previous results. A photon energy of 22 keV was used for best transmission to/from the oil/water interface and the double crystal diffractometer ensured alignment of the beam to the interface. The beam intensity was recorded with the Maxipix detector at 1010 mm distance to the sample. In addition to the regular setup, a manual slit was introduced directly in front of the sample cell for the GISAXS measurements to remove beam background that originated from the diffractometer.

In order to minimize scattering from the oil phase and from all other components of the sample cell, we designed a customized oil/water cell based on a design that we already tested at DESY in two previous beam times (Fig. 1). We evaluated monocrystalline CVD diamond (300 µm) and thin mica sheets (27 µm) as window material. Both materials were well suited, but the diamond windows gave an additional reflection that slightly superimposed the GISAXS pattern. Given the huge price difference between the two materials (about 2500 € per diamond window vs 2.50 € per mica window), we opted to continue to work with the mica windows. The cell geometry and windows were tailored to accommodate the beam footprint to minimize interaction of the beam with the cell walls. However, the rectangular shape of the new cell was a disadvantage compared to the spherical cell design that we used before because of the complex interfacial meniscus that was introduced by the rectangular walls. While we were able to record very good GISAXS patterns with the new cell, the complex meniscus complicated XRR measurements at very low q_z as shown below.

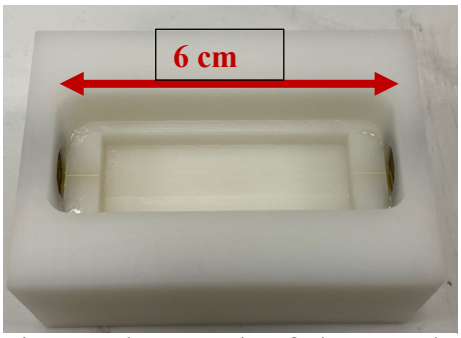

Fig. 1: Photograph of the sample cell. The water phase is filled up to the ledge to reduce meniscus formation. Note the beam damage in the middle of the ledges next to the windows.

After evaluation of the cell setup we were able to carry out most of our planned measurements as described in the original proposal. The experiments were designed around various aqueous nanoparticle dispersions and decane solutions containing lipids at different concentrations. Bare silica nanoparticles (TEOS) with diameters of 20 nm were used as well as the same particles with a coating of 3-aminopropyl triethoxy silane (APTES). Octadecyl amine (ODA) was dissolved in the decane phase with varying concentrations from 0.01 mM to 1 mM. The following measurements were carried out:

1. Initial alignment and adjustment of the cell and confirmation of the experimental setup quality by recording one complete XRR curve of the sample containing pure water and decane only. Evaluating and optimizing the sample environment for GISAXS measurements
2. One complete XRR curve with APTES particles and ODA at a fixed concentration, including radiation damage checks
2. static measurements: GISAXS reciprocal space maps for four ODA concentrations (fixed particle concentration) for both APTES and TEOS particles
3. time-resolved experiments: hourly XRR and GISAX measurements for four ODA concentrations (fixed particle concentration) for both APTES and TEOS particles

At least two samples of each type were prepared and studied to confirm the reproducibility of the results.

Fig. 2 shows selected results from GISAXS. On the GISAXS patterns, the emergence of an hexagonal packing of APTES particles at the oil/water interface could be observed with increasing concentrations of ODA. At the highest ODA concentration, the 2nd order peak becomes less sharp than the one at 0.1mM ODA, suggesting a worse in-plane order. This might be related to the slight hydrophobization of the particles that occurs at the highest surfactant concentration which we also observed at our previous measurements at DESY.

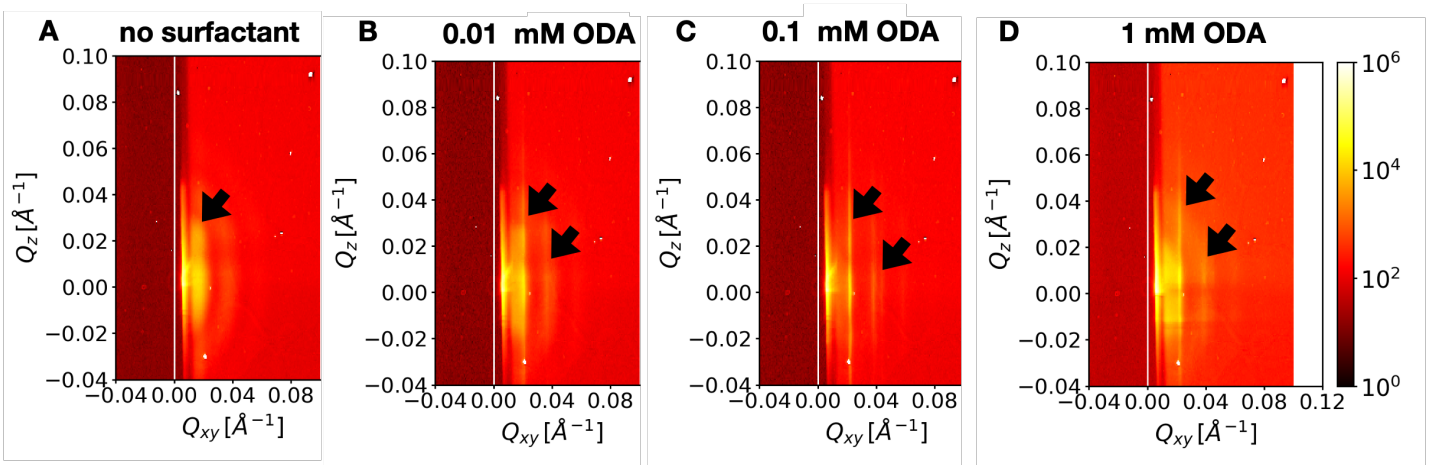

Fig. 2: GISAXS patterns from the oil/water interface: Effect of surfactant concentration on APTES SiO_2 nanoparticles distribution at the decane/water interface. (A) without surfactant, (B) with 0.01 mM, (C) 0.1 mM and (D) 1 mM of ODA.

The reflectivity of the same samples (Fig. 3) clearly shows increasing amounts of interfacially adsorbed nanoparticles with increasing surfactant concentrations, which corresponds to what we expect from this system. Note the irregularities at low Q_z which are particularly visible in Fig. 3C and which are most likely caused by the complex interfacial meniscus inside our cell and possibly also background radiation from the diffractometer, which make quantitative analysis of the XRR data difficult.

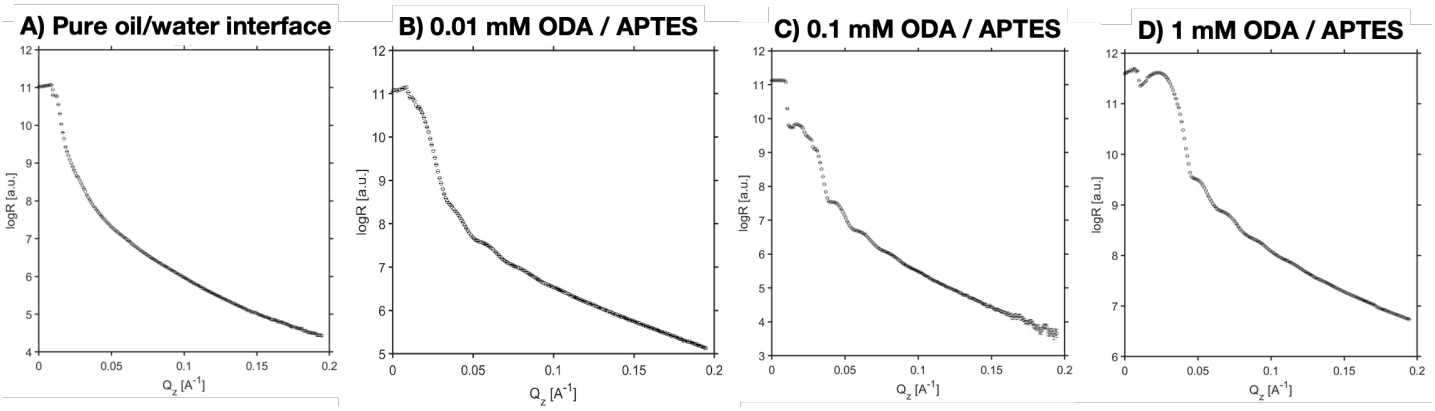

Fig. 3: XRR curves from (A) the pure oil/water interface, (B-D) varying ODA concentrations in decane on aqueous dispersions of APTES SiO_2 nanoparticles.