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:** Adhesion and interactions with hair biomolecules - a cosmetic perspective	**Experiment number:** SC-5276
Beamline: ID10	**Date of experiment:** from: 26 April 2022 to: 2 May 2022	**Date of report:**
Shifts: 18	**Local contact(s):** Oleg Konovalov	***Received at ESRF:***

Names and affiliations of applicants (* indicates experimentalists):

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Vorobiev Alexei, Uppsala University*

Luengo Gustavo, L'Oréal R&I

Report:

The planned experiments aimed at gaining more details about the in-plane structuring of different thiols self-assembled on gold-coated silicon blocks. They are used in the PhD project of Serena Cozzolino as mimetics of normal or damaged hair surface, to study adsorption of model surfactants and polymers (components of haircare products).

Two techniques were envisaged: X-ray Reflectometry (XRR) and Grazing-Incidence X-ray Diffraction (GIXD).

Most of the allocated time was needed to set up the appropriate configuration to perform experiments on the desired system. Gold, in fact, is not ideal for X-rays but it was needed to compare the results to those obtained by other techniques, and because it is the easiest way to have hydrocarbon chains *covalently* attached on the surface (necessary for experiments at the solid/liquid interface).

Measurements were done on test samples of bare or functionalized silicon, whose expected signal was known, to check the modified set-ups. Then they were performed on the actual samples, i.e., gold-coated silicon or glass chips functionalized with:

1. Octadecanethiol
2. 50:50 octadecanethiol:sodium 3-mercapto-1-propanesulfonate
3. Butanethiol
4. 50:50 butanethiol:sodium 3-mercapto-1-propanesulfonate
5. 2-methyl-1-butanethiol
6. 50:50 2-methyl-1-butanethiol:sodium 3-mercapto-1-propanesulfonate

Samples 1, 3 and 5 give hydrophobic surfaces with different chain packing, i.e., they mimic different types of "healthy hair" surfaces. Samples 2, 4 and 6, as hydrophilic, partly charged surfaces, are "damaged hair" models.

GIXD measurements could not give the expected results, as the ring due to polycrystalline gold covered other signals, as shown in Figure 1.

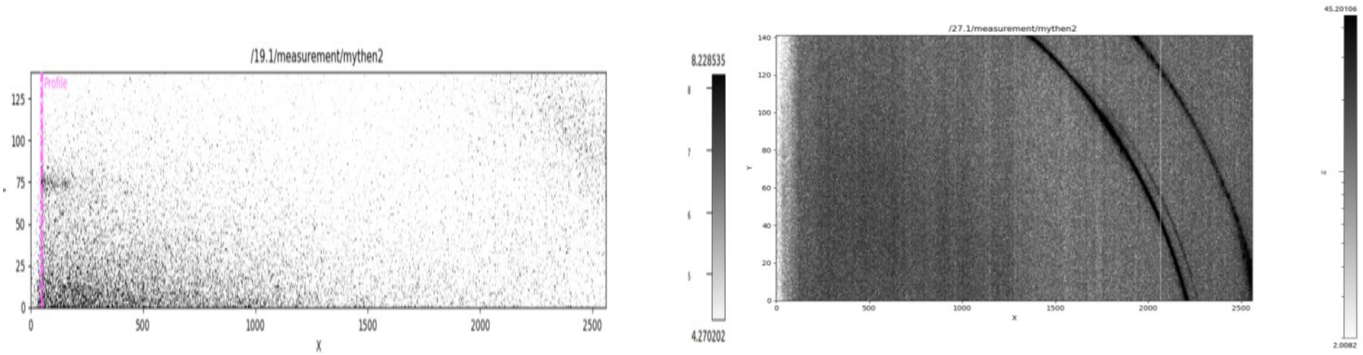

Figure 1. Left, the signal (at about half of the pink line) expected for a hydrocarbon chain on a silicon test sample. Right, the signal for Sample 1; the rings are present also on the bare gold surface, no signal due to the hydrocarbon chain is detectable

XRR measurements allowed the characterization of samples 2-6 in air (Figure 2), after optimization of the conditions to avoid beam damage. The first measurement with a standard set-up, in fact, immediately burnt the sample.

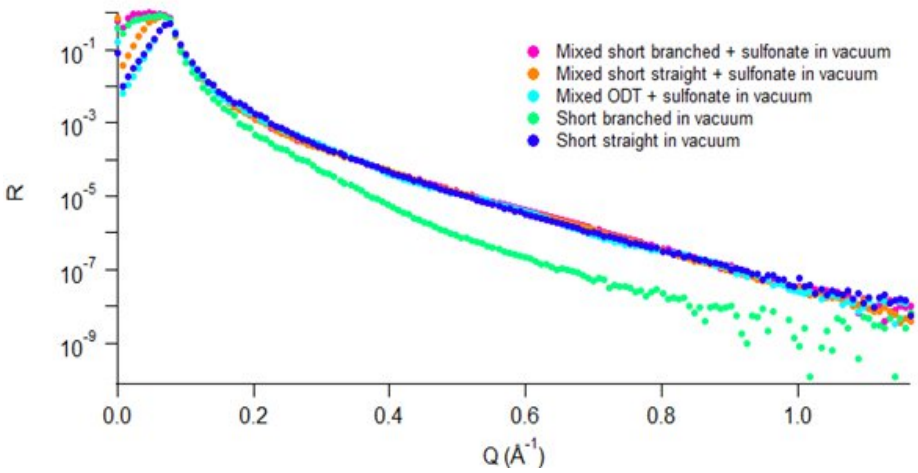

Figure 2. XRR curves on samples 2-6. The 3 chips produced for sample 1 were all damaged looking for the best settings for XRR measurements.

A different gold-coated substrate was in the meantime functionalized with octadecanethiol and measured by XRR. An experiment at the solid/liquid interface was also performed (Figure 3).

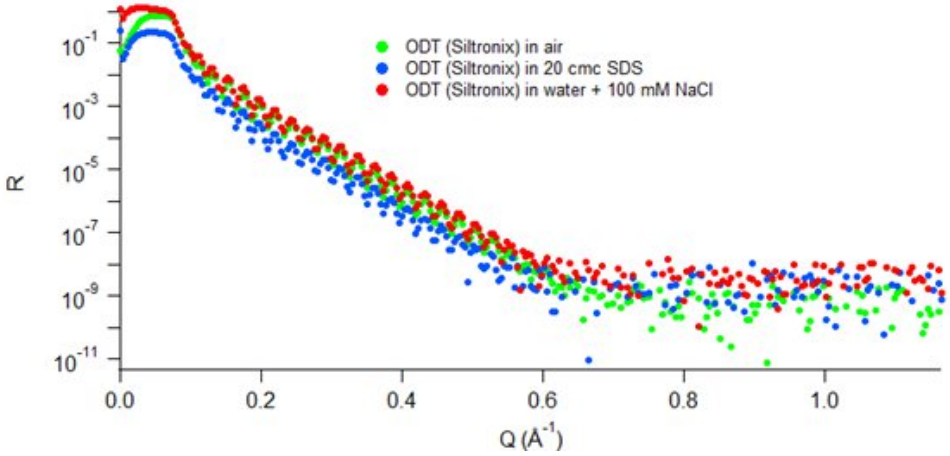

Figure 3. Sample 1 (on different substrate) in air and in water without and with a surfactant solution. The curves in water are overlapping as there is not enough contrast between the various species to distinguish between adsorbed surfactant layer and bulk aqueous solution.