



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://www.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 from 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.



	Experiment title: Crystallization of fat dispersions: characterization of crystal nanoplatelet thickness	Experiment number: A262943
Beamline: BM26	Date of experiment: from: 03/06/2022 to: 07/06/2022	Date of report: 05/09/2022
Shifts: 12	Local contact(s): Martin Rosenthal	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): Dewettinck, Koen - Ghent University, Food Structure & Function Research Group De Witte, Fien* - Ghent University, Food Structure & Function Research Group		

Report:

People taking place in the experiments

Lewille, Benny – Ghent University, Food Structure & Function Research Group

Penagos, Ivana – Ghent University, Food Structure & Function Research Group

De Witte, Fien – Ghent University, Food Structure & Function Research Group

Samples measured

The samples measured were pure fats (PO, AMF, HPKS) or suspensions consisting of these fats and various food related particles (concentrations varying between 10-60%). Additionally, sucrose esters were added as emulsifiers.

Research subject

Within the Food Structure & Function Research Group of Ghent University, the study of fats, and their polymorphic behaviour is a main research activity. Different X-ray scattering techniques can be used to elucidate the fat crystal network (see figure 1; left shows build-up of fat crystal network; right shows different X-ray scattering length scales and their use for elucidation of fat crystal network). Recently, a Xenocs Xeuss 3.0 was acquired via Hercules funding (Reference number AUGE/17/29). The main use of this equipment is to enable fast detection of fat polymorphs in food. One of the more advanced uses of SAXS is to obtain the crystal nanoplatelet (CNP) thickness from the peak shape. However, for lab scale equipment,

the peak shape is prone to peak broadening, which affects the results obtained for CNP thickness.

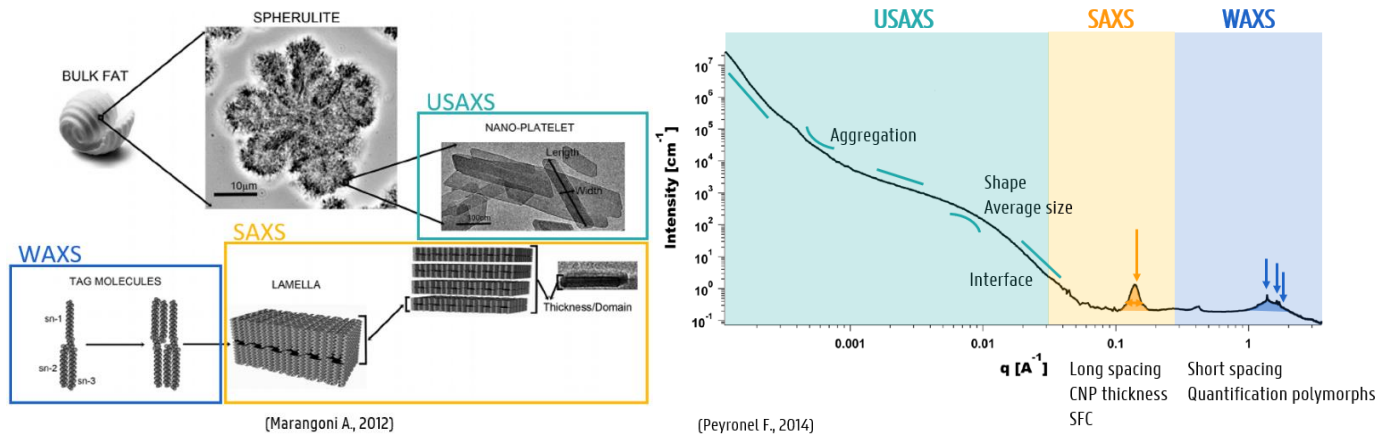


Figure 1: different X-ray scattering length scales and their use to elucidate the fat crystal network

Adaptations to the set-up described in the proposal

Samples were measured in two ways. Some adaptations to the original set-up were made, as the beamline does not have a lot of temperature controlled options.

A first set of samples was crystallized at a temperature of 0, 20 or 25°C. No thermostatic cabinets are available at BM26, therefore the PSCM lab was used. This is a serious drawback, as the temperature at the beamline is rather high and small samples do melt easily, which changes the polymorphic profile. The use of Kapton sheets to measure pre-crystallized samples allows to make efficient use precious beamtime.

The second set of samples was heated and cooled to 20°C in a capillary holder at the beamline. As such, crystallization during cooling (1 or 20°C/min) could be followed.

Experimental problems

We were able to execute most of the planned experiments, although some breakdowns in the beamline intensity were faced (including failure of the vacuum gauge that switched off the full synchrotron). The fact that data cannot be visualized as $I(q)$, and only as 2D image, makes it difficult to judge the quality of results on the spot.

Outcomes

Thanks to the experimental time at beamline BM26, peak broadening of the Xeuss equipment could clearly be investigated. Peak broadening was found to be twofold: on one hand, due to lab-scale equipment optics and on the other hand due to the composition/crystallite size of the materials used.

Other research findings are kept confidential not to compromise on any publications.

Publications

Submission of a publication from the data obtained during experimental session BM26 is expected, however, data must be put together with other experimental data from own laboratory (DSC, PLM, NMR, rheology...). Outcome expected end 2022.

Additional remarks

The research team would like to thank mr. Martin Rosenthal for the help provided during the research stay, especially since we were there 4 days of which 3 were weekend/holidays.