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:** Fat crystallization in suspensions: the effect of particle hydrophobicity and dispersing agent	**Experiment number**: ME1606
Beamline: ID02	**Date of experiment**: from: 31/03/2022 to: 02/04/2022	**Date of report**: 05/09/2022
Shifts: 6	**Local contact(s)**: William Chevremont	*Received at ESRF:*

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
Hendrik, Nathaniel – Ghent University, Food Structure & Function Research Group
Rondou, Kato – 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 as reference) or suspensions consisting of these fats and various food related particles (concentrations varying between 10-60%). Additionally, sucrose esters were added as emulsifiers.

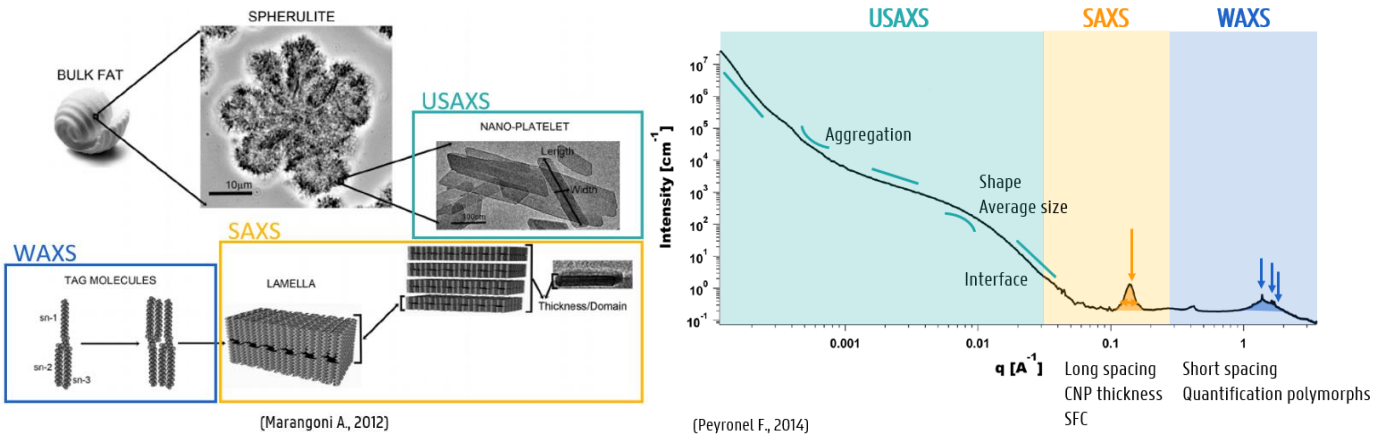

Figure 1: different X-ray scattering length scales and their use to elucidate the fat crystal network

Research subject

Within the Food Structure & Function Research Group, the study of fats, and their polymorphic behavior 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). Special interest is put into the investigation of the mesoscale structure, including nanoplatelet size and aggregation. Therefore, the use of USAXS is of great interest as information can be obtained after fitting of the results with Unified fit or Guinier-Porod model.

Set-up of the measurement

Samples were measured in two ways. A first set of samples was crystallized at a temperature of 0, 20 or 25°C. Samples were put between Kapton sheets to measure pre-crystallized samles to ensure efficient use of 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. The beamline is very well equipped with sample preparation material and can provide many different sample holders.

Experimental problems

We were able to execute most of the planned experiments, although some measuring issues were unforseen. As the samples are suspensions, containing particles with an average diameter around 30 µm, the very low USAXS region (StoD 31 m) got saturated and a new mask needed to be installed, cutting the lower edge of the USAXS results. This is a drawback in fitting the results as a lot of information is lost. Moreover, also in the higher q-region of USAXS information is lost due to the appearance of secondary scattering peaks. 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.

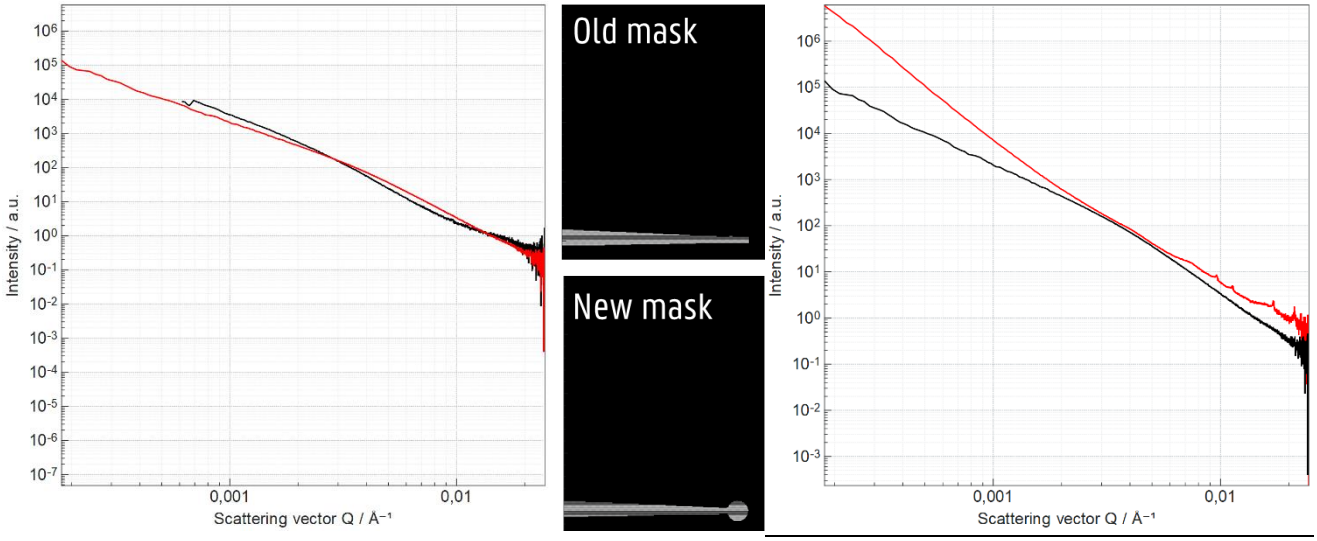

Figure 2: Left: red = result of USAXS with old mask applied, black = result of USAXS with new mask applied. Middle: shape of old and new mask applied. Right: red = sample with particles, suffering from secondary scattering, black = sample without particles, not suffering from secondary scattering.

Outcomes

Thanks to the experimental time at ID02, the possible use of USAXS for understanding crystallization of suspensions could be estimated. Nonetheless, the above described difficulties, the research has still proven to be successful. Further detailed research findings are kept confidential not to compromise on any publications.

Publications

Submission of a publication from the data obtained during experimental session ME1606 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. William Chevremeont for the continuous help provided during the research stay.