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:** The effect of shear on crystallization in suspensions	**Experiment number**: ME1642
Beamline: ID02	**Date of experiment**: from: 21/06/2023 to: 26/06/2023	**Date of report**: 04/09/2023
Shifts: 9	**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

https://www.esrf.fr/UsersAndScience/UserGuide/ExperimentReports/Preparing_SubmittingYourReportFile

Report:

People taking place in the experiments

Fien De Witte, Ivana Penagos, Tom Rimaux and Griet Spaepen, all Ghent University, Food Structure & Function Research Group

Samples measured

The samples measured were pure fats (PO, AMF, HPKS as reference) and suspensions consisting of these fats and sucrose particles. Additionally, sucrose esters were added as dispersing agents.

Research subject

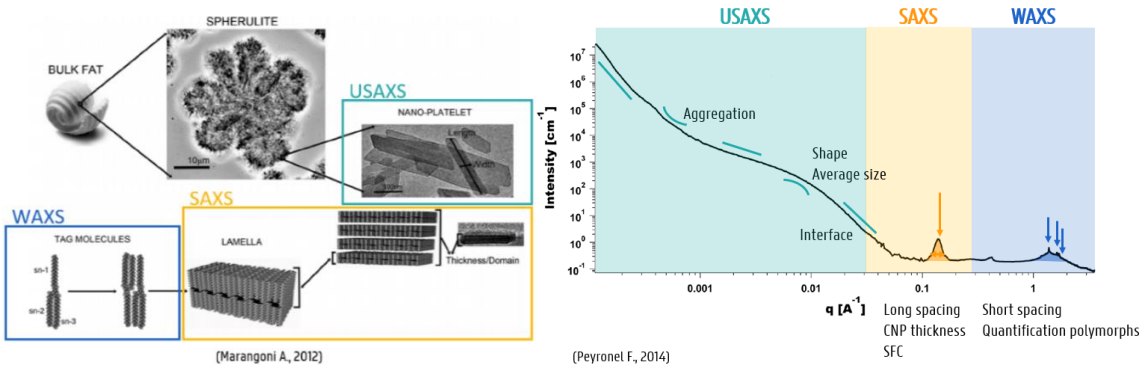

Figure 1: different X-ray scattering length scales and their use to elucidate the fat crystal network.

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 and related experimental problems

Samples were measured under shear, using a Linkam CSS450 equipped with nitrogen dewar for cooling (Figure 2 Left). Samples were loaded into the shear cell after which they were heated to 70°C. To make efficient use of time, the hutch was locked during this heating period of 10 min. Next, the samples were cooled at 20°C/min to 20 or 25°C and hold isothermally for 60 min. Shear was applied during the first 15 min of crystallization. The shear cell is designed with a 3-fold propeller (Figure 2 Right) that rotates when applying shear to the sample. When rotating, the three propeller ends block the beam for a short period of time. This is inherently related to the design of the shear cell. Measurements at the synchrotron facility are such short time frames that it was still possible to acquire data, which has proven unsuccessful on a lab-scale equipment. Both slow (1/s) and fast (50/s) shear rates were applied and both conditions have shown possible to obtain data. For statistics of the data, 10 short acquisitions were taken and then averaged. It will of course be necessary to check for every acquisition if it was blocked by the propellor, especially at the fast shear rate.

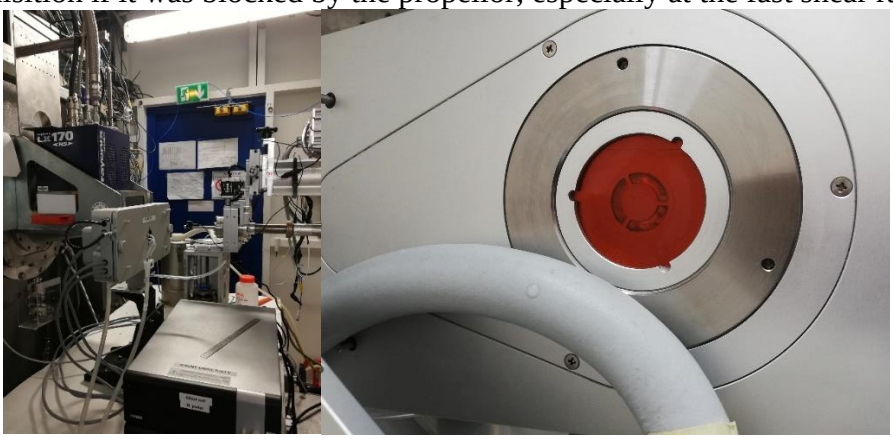

Figure 2: Left: experimental set-up of shear cell positioning in the beam. Right: design of shear cell propellor.

Outcomes & Publications

Publication for ME1642 will also be made, however, data must be put together with other experimental data from own laboratory (DSC, PLM, cryo-SEM…) which requires more time. Moreover, more effort will be put into the modelling part of the data. Detailed research findings are kept confidential for now not to compromise on any publications.

Submission of a publication from the data obtained during experimental session ME1606 is in progress, abstract can be found below.

Abstract: Despite some controversy, palm oil (PO) is still widely used for the production of all types of food products. Due to its triacylglycerol composition, PO is semisolid at ambient temperature, offering possibilities for many applications. In this study, PO was crystallized under three temperature protocols (fast (FC) and slow (SC) cooling to 20°C or fast cooling to 25°C) and followed for 1h isothermal time. A broad toolbox was used to fundamentally unravel the structural build-up of the fat crystal network at different length scales. Wide-angle and small-angle X-ray scattering (WAXS & SAXS) showed polymorphic transitions from α 2L to β’ 2L over time. Despite similar polymorphism and chain length structure, ultra-small-angle X-ray scattering (USAXS) showed clear differences on the mesoscale. Unified fit analysis showed radius of gyration (Rg) values of 728 Å, 1506 Å and 1689 Å for FC-20, SC-20 and FC-25, respectively. Guinier-Porod shape factor confirms the non-spherical structure of the platelets. The microstructure was visualized with polarized light microscopy (PLM) and cryo-scanning electron microscopy (cryo-SEM), showing clear differences in crystallite size, clustering and network morphology.

Additional remarks

The research team would like to thank mr. William Chevremeont and mr. Theyencheri Narayanan for the continuous help provided during the research stay.