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:** Full-length characterization of triacylglycerols systems: effect of cooling and shearing rate	**Experiment number**: ME-1607
Beamline: ID02	**Date of experiment**: from: 02/04/2022 to: 04/04/2022	**Date of report**: 05/09/2022
Shifts: 8	**Local contact(s)**: William Chevremont	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Update 05/09/2022 – Preliminary Report
Certain findings have not been included due to confidentiality reasons.

During the visit to the ESRF in April 2022, two types of experiments were successfully performed.

Type A. Real-time crystallization

In this type of experiment, the triglyceride (TAG) dilution was added into a capillary and placed on the Linkam stage. The stage was set to 80°C for 15 minutes, and then the adequate crystallization profile was conducted (e.g., cool to X °C at Y°C/min). Time-resolved WAXS-SAXS and WAXS-USAXS were acquired during the phase transition. Table 1 presents the experiments that were successfully registered.

Table 1. List of crystallization experiments. FHRO: fully hydrogenized rapeseed oil, PS: Palm stearin, PPP: tripalmitin, SSS: tristearin

Experiment	Concentration (%w/w)	TAG	Rate (°C/min)	Final Temperature (°C)
A	10	FHRO	10	15
B	30	FHRO	10	10
C	30	FHRO	10	15
D	30	FHRO	1	15
E	30	FHRO	5	15
F	5	FHRO	10	15
G	10	PPP	10	15
H	30	PPP	10	15
I	10	PS	10	15
J	30	PS	10	10
K	30	PS	10	15
L	30	PS	1	15
M	30	PS	5	15
N	5	PS	15	15
O	10	SSS	10	15
P	30	SSS	10	15

With the results, the influence of the following parameters can be assessed:

- Type of TAG
- Concentration
- Degree of undercooling
- Cooling Rate

Additional techniques used and intended to be included in publications of this setup include polarized light microscopy and differential scanning calorimetry.

Due to the confidentiality of the results, no further declarations would be made regarding the findings. A part of the data of this experiment has already been discussed at international conferences (e.g., Edible soft matter, Wageningen, 2022). Publications in progress.

Type B. Static acquisition – pilot scale experiments

In this type of experiment, the samples were prepared in Belgium (Ghent University). Employing a pilot-scale scraped surface heat exchanger, samples were prepared with different shearing and cooling rates. These experiments are quite relevant from an application standpoint, as the setup mimics the industrial processing of shortenings. The samples were placed inside a metal ring with two Kapton tapes. For measurement, the metal ring containing the sample was placed on top of a Peltier cooled stage at the required measuring temperature (15°C). WAXS-SAXS and WAXS-USAXS were acquired.

Two different designs of experiments (DoE) were measured in line (Figure 1).

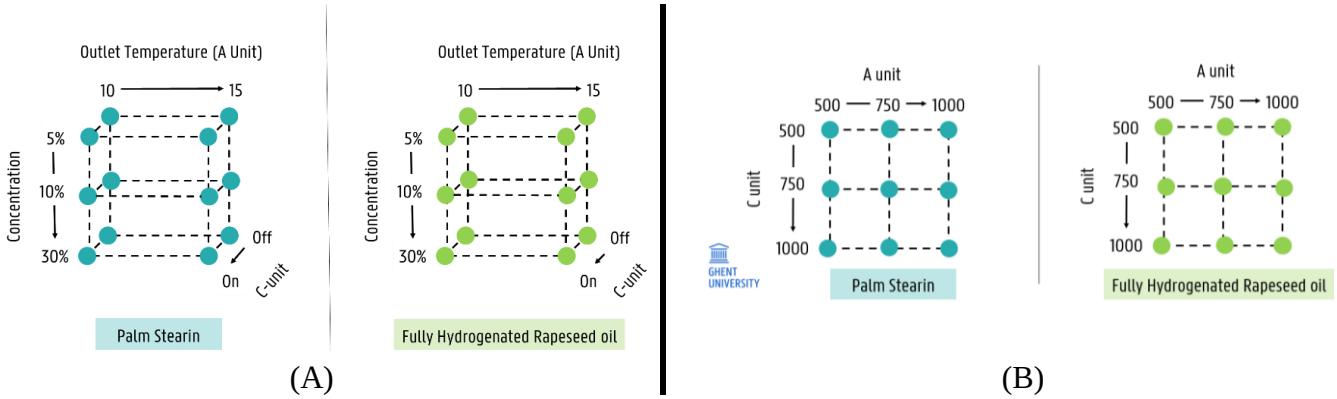

Figure 1. Designs of experiments used in Type B

With the results of the first DoE (Figure 1A), the influence of the following parameters can be assessed:

- Concentration
- Presence/absence of a pin worker (C unit)
- Outlet temperature – linked to the degree of undercooling.

With the results of the second DoE (Figure 1C), the influence of industrially relevant shearing protocols can be assessed:

- A unit (Scraped surface heat exchanger)
- C unit (Pin worker)

Both of which have a direct effect on cooling and shearing rate.

Additional techniques used in this setup include rheology, texture analysis, polarized light microscopy and Cryo-SEM. At the moment, advanced statistical methods are being used to address relationships between USAXS, SAXS and WAXS and Macroscopic product behavior.

Due to the confidentiality of the results, no further declarations would be made regarding the findings. Publications in progress.