EUROPEAN SYNCHROTRON RADIATION FACILITY

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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

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

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.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal 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:** In situ investigation of structural changes in sheared metal-ligand bonded poly (ethylene oxide) melts	**Experiment number**: 26-02/811
Beamline: BM26B	**Date of experiment**: from: 19.02.2017 to: 23.02.2017	**Date of report**: 07.05.2018
Shifts: 9	**Local contact(s)**: Daniel Hermida Merino	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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The experiment 26-02/811 shared beamtime with experiments 26-02/815 and 26-02/808 with all three experiments utilizing a common setup comprised of a mobile stage and, a for the highly mobile setup modified rheometer. Rheometrical geometries made out of Kapton or aluminum with diamond windows were available and manufactured by FZ Jülich or TU Eindhoven, respectively. Installation of the setup and regular beam alignments reduced the beamtime available for measurements . Complications were encountered when optimizing the obtained scattering intensity and limited the number of measured samples. Having a more sensitive camera available would minimize those time losses.

The response of emulsion crystallized hydrogenated castor oil (HCO) suspensions after being subjected to shear was investigated by rheo-WAXS. The shear magnitude was stepwise increased with each shear rate being separated by cessation of flow. The scattering patterns after each shear rate are shown in Figure 1 and compared to the scattering pattern obtained of pure HCO.
The pure HCO as well as the HCO suspension show two clear peaks in the WAXS-domain at a spacing 3.91 and 4.43 Å (22.71° and 20° respectively) and one at 17.04 Å (5.18°) in the SAXS-domain. Comparing these results with the observations of Yang et al. [183], and other work on TAG's [139][86], these spacings correspond closely to the β (4.43 Å) and β' (3.91 and 17.04 Å) polymorphs. Looking at the acquired scattering patterns a transition from an homogeneous ring to an anisotropic scattering pattern becomes apparent, getting more pronounced with increasing shear rate. Moreover, for the two spacings in the WAXS-region, the anisotropy follows the flow direction, where in the SAXS-region the spacing becomes anisotropic orthogonal to the flow direction. These orthogonal anisotropic patterns confirm

that the observed shear thinning is due to alignment of the HCO crystals in the flow direction. The two anisotropic spacing patterns in the WAXS regime are related to the stacking of the HCO nanocrystals into rodlike crystal structures, as illustrated recently [139], together with the long length scale stacking of the orthorombic β' polymorph causing the 17.04 Å spacing line [156]. Apart from the particularity of the observed anisotropy due to alignment observed from intra-particle structural information, it indicates that the HCO crystals show the ability to align to the applied flow. In the HCO dispersions alignment within flocs thus also becomes highly probable. Due to the low concentrations, and the loss of beam intensity due the deflection in vertical direction, scattering patterns of 8 wt% dispersions themselves could not lead to a more robust proof of anisotropy.

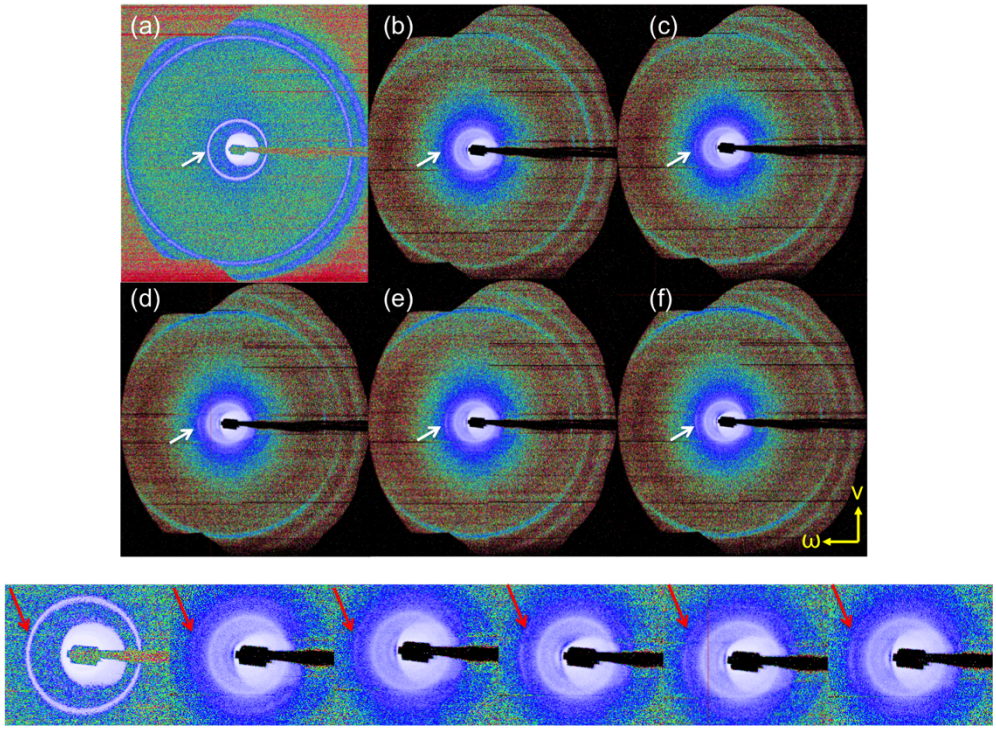

Figure 1: (top) Scattering patterns for emulsion crystallized HCO suspensions after different shear rates, with white arrow indicating the β'-spacing at small angles (a) reference case for HCO (b) at rest (c) after 1.04 1/s (d) after 26 1/s (e) after 52 1/s (f) after 104 1/s. (bottom) Zoom of scattering patterns to clarify (red arrows) the anisotropy at the β'-spacing at small angles.

The second system comprised mixtures of poly(ethylene oxide) melts mixed with transition metal salts. Due to low scattering intensities, the data was difficult to analyse and was lacking a certain degree of reproducibility.

[1] Yang, D., and Hrymak, A. N. Crystal morphology of hydrogenated castor oil in the crystallization of oil-in-water emulsions: Part I. Effect of temperature. Ind. Eng. Chem. Res. 50, 20 (2011), 11585–11593.

[2] Peyronel, F., Pink, D. A., and Marangoni, A. G. Triglyceride nanocrystal aggregation into polycrystalline colloidal networks: Ultrasmall angle X-ray scattering, models and computer simulation. Curr. Opin. Colloid Interface Sci. 19, 5 (2014), 459–470.

[3] Idziak, S. H. Powder X-ray Di raction of Triglycerides in the Study of Polymorphism. 2012.

[4] Sato, K., Goto, M., Yano, J., Honda, K., Kodali, D., and Small, D. Atomic resolution structure analysis of beta' polymorph crystal of a triacylglycerol: 1,2-dipalmitoyl-3-myristoyl-sn-glycerol. J. Lipid Res. 42, 3 (2001), 338–345.