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:** Dynamics of Oil/Water Interface Caused by Two-Dimensional Gelation: A New Challenge with GI-XPCS	**Experiment number**: SC 4654
Beamline: ID 10	**Date of experiment**: from: 09.02.2018 to: 13.02.2018	**Date of report**: 22.02.2018 *Received at ESRF:*
Shifts: 12	**Local contact(s)**: Yuriy Chushkin	

Names and affiliations of applicants (* indicates experimentalists):

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Report:

GI-XPCS performed at the oil/water interface still remains an open challenge since this technique application is not available anywhere else but it is rather an expertise of the beam line ID 10.

The primary goal of the proposed experiment is to quantitatively investigate the sol-gel transition kinetics of sodium alginate caused by the diffusion to the interface of calcium chloride nanoparticles suspended in oil and surfactant by means of GI-XPCS.

The change in the interface dynamics was followed by monitoring the time evolution of the autocorrelation function: $g^2(q,t) \sim exp[-2(t/\tau_q)^\gamma]$ of silanized SiO_2 nanoparticles (Ø ~ 100 nm) confined at the interface. The experiments were carried out in a sample chamber designed by O. Konovalov (ID 10) specially designed for experiments at the oil/water interface and setting the beam energy to 22 keV in order to overcome the strong radiation adsorption due to the presence of the oil.

Systematically, we studied the influence of the polymer concentration on the reaction kinetics by testing sodium alginate 1 and 1.5 wt%. Figure 1a shows a typical q-space map of SiO_2 nanoparticles confined at the oil/water interface recorded for 1 wt% sodium alginate using a 2D detector (Maxipix). The intensity profile along q_y is integrated between the two dotted black lines on the q-space map.

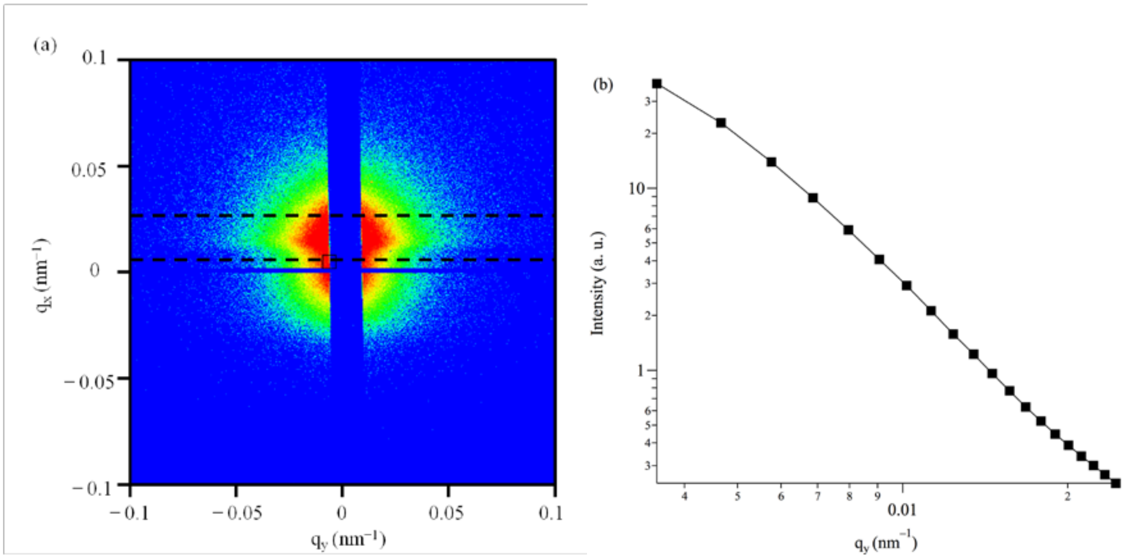

Figure 1. (a) *Reciprocal intensity map of 1 wt% polyalginate at the oil/water interface. (b) Intensity (static) 1D profile after integration along* q_y .

To extract detailed dynamics information, the autocorrelation function of the intensity fluctuations in the scattered signal was applied to quantify the temporal evolution of the microscopic reorganization at nanometric length scale. The dynamics can be related to the intermediate scattering function through the Siegert relation: $g^2(\mathbf{q},t) = 1 + A(\mathbf{q})|f(\mathbf{q},t)|^2$ which requires the system to be ergodic. This requirement can be extended to out-of-equilibrium systems such as gels, as long as area detectors are used. The obtained results for alginate 1 wt% are presented in Figure 2.

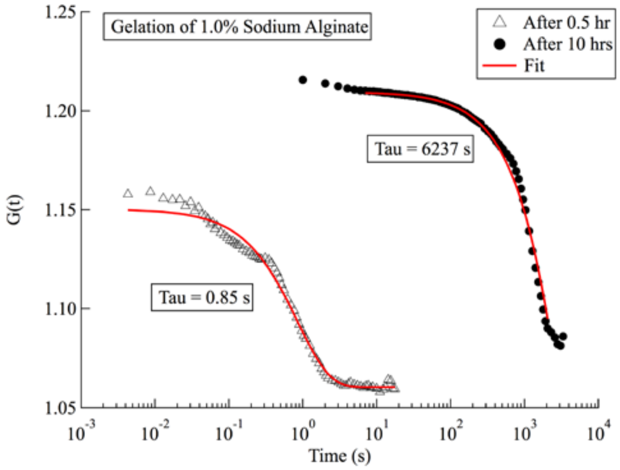

Figure 2. *Autocorrelation function of sodium alginate 1 wt% and* $CaCl_2$ *nanoparticles in sunflower oil after 30 minutes (open triangles) and 10 hrs (solid circles) of reaction for* $q = 0.0047\ Å^{-1}$ *. The solid red lines represent the results of the exponential fit.*

The results of the exponential fits of the autocorrelation function show that the characteristic decay time “tau” increased of a factor $7 \cdot 10^3$, when an early and late reaction stages are compared. These findings suggest that the dynamics of nanometric domains at the oil/water interface slows down as the sol-gel transition proceeds.

Further experiment and data analysis are needed to shed light on the impact of polymer and cross-linker concentration on the reaction dynamics at the oil/water interface.