Experimental report

23/04/2020

Proposal: **EASY-582** **Council:** 10/2019

Title: Flow-induced lamella-to-MLV transformation: flow-through vs. rheo-SAS

Research area: Soft condensed matter

This proposal is a new proposal

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Experimental team:

Local contacts: Sylvain PREVOST

Samples: 100 mM TDMAO, 200 mM hexanol

Instrument	Requested days	Allocated days	From	To
D11	12	12	21/01/2020	22/01/2020

Abstract:

Soft matter under flow is ubiquitous in both industry and biology, but not frequently investigated scientifically. In contrast to rheo-SAS with very well-defined shear and deformation, a flow-through setup is flexible and conceptually easy, but the mechanical stress on the sample is less controlled and homogeneous.

In order to complement previous measurements on the lamella to MLV-transition in the system TDMAO/cosurfactant/water done in our group (V4, HZB Berlin), we would like to measure two samples. We will then be able to systematically contrast our results from rheo-SAXS (ID02, ESRF) with the effects observed due to flow-induced shear, which we believe to promise insight not only on the system, but also further development in the analysis of flow-through data.

[1] Escalante, J. I., Gradzielski, M., Hoffmann, H., & Mortensen, K., Shear-induced transition of originally undisturbed lamellar phase to vesicle phase, Langmuir, 16(23), 8653–8663 (2000). http://dx.doi.org/10.1021/la000242c

1 Experiment details

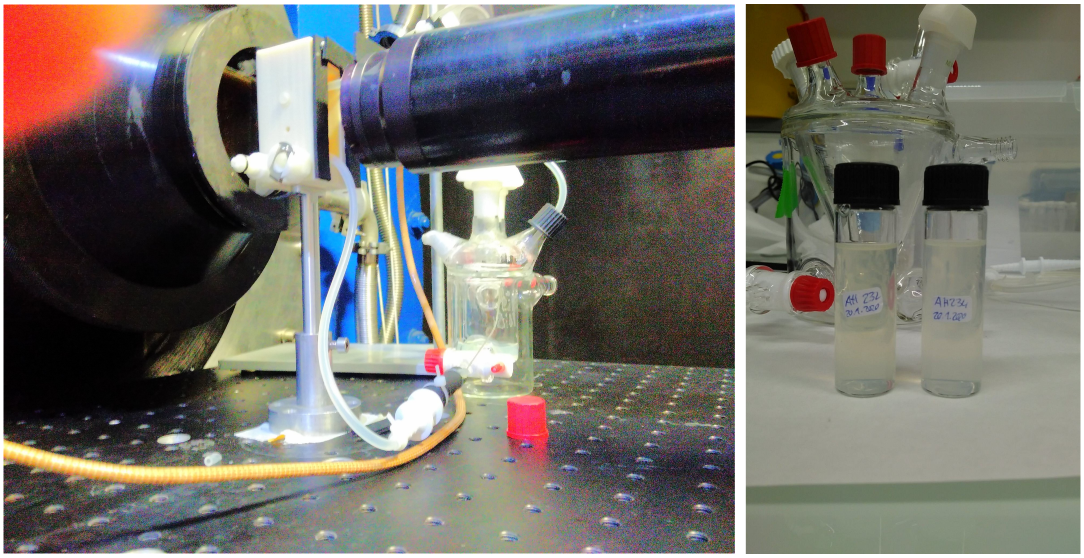

Figure 1: Left: Setup for the experiment for the sample with 220 mM 1-hexanol. Right: both samples (left: 200 mM, right: 220 mM 1-hexanol) in front of the conical reactor used for the left sample.

We observed the small-angle scattering of lamellar phases of TDMAO/1-hexanol/water (100 mM TDMAO, 200 and 220 mM hexanol respectively).

Sample to detector-distance was chosen to be 20.5 m, with a wavelength of 5 Å we obtained a q-range of $0.031\,\mathrm{nm}^{-1}$ to $0.399\,\mathrm{nm}^{-1}$.

Samples were stored in flow-through chemical reactors for at least 5 hours before the experiment. From the sample container, the sample was pumped through silicone tubing of 2 mm ID, 4 mm OD to the flow-through cuvette used for observation by a Williamson model 150 peristaltic pump (6 rollers). The setup was chosen such that the peristaltic pump is situated after the cuvette in the direction of observation which is important for knowing the deformation. Peristaltic pumps perturb the well-defined flow rate as their working principle is technically a periodic squeezing of the tubing.

The length of the tubing from the chemical reactor to the cuvette was 20 cm, followed by 11 cm up to the pump and 27 cm through the pump and back to the reactor.

Flow rates were chosen according to a geometric series and their relation to the rpm of the pump calibrated using water and a Sensirion flow meter.

2 Preliminary results

We expected the sample with 220 mM to form vesicles more easily than the one with 200 mM hexanol.

We observed effects already at very low rpm, showing alignment of the lamellae with changing orientation (see fig. 2).

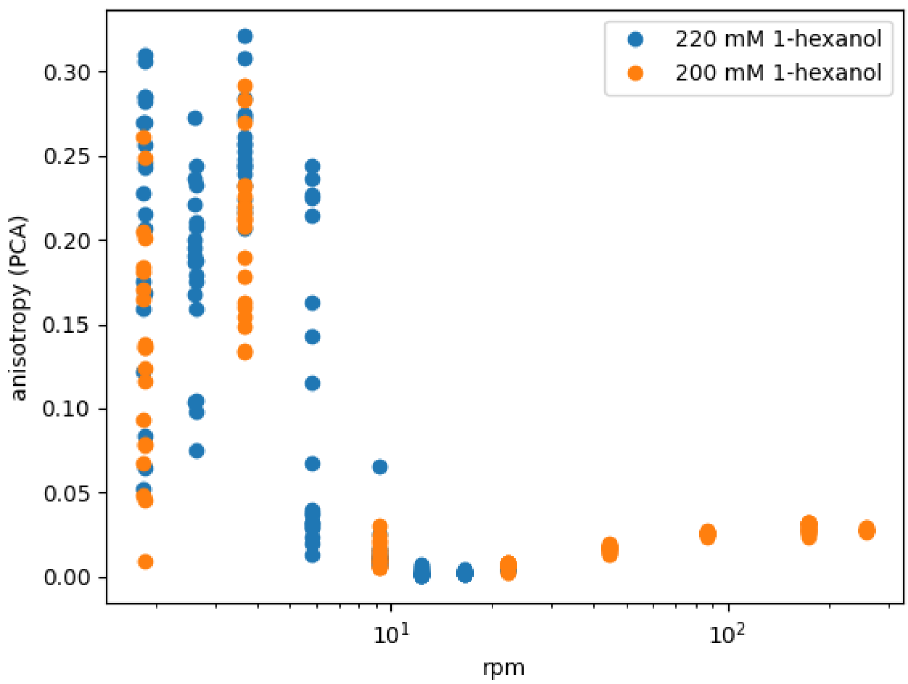

Figure 3: Anisotropy vs. rpm during the experiment. Especially at low rpm the signal changed over time, indicating that deformation, rather than shear, may be the relevant variable.

Despite the differences in experimental conditions, the anisotropy observed is qualitatively similar to results from rheo-SAXS.

Data analysis for this experiment is ongoing and requires taking into account the length of the tubing from the sample container (reactor) to the window of observation. This is because the deformation experienced by the sample observed in the cuvette is the result of its journey from the reactor to the cuvette.

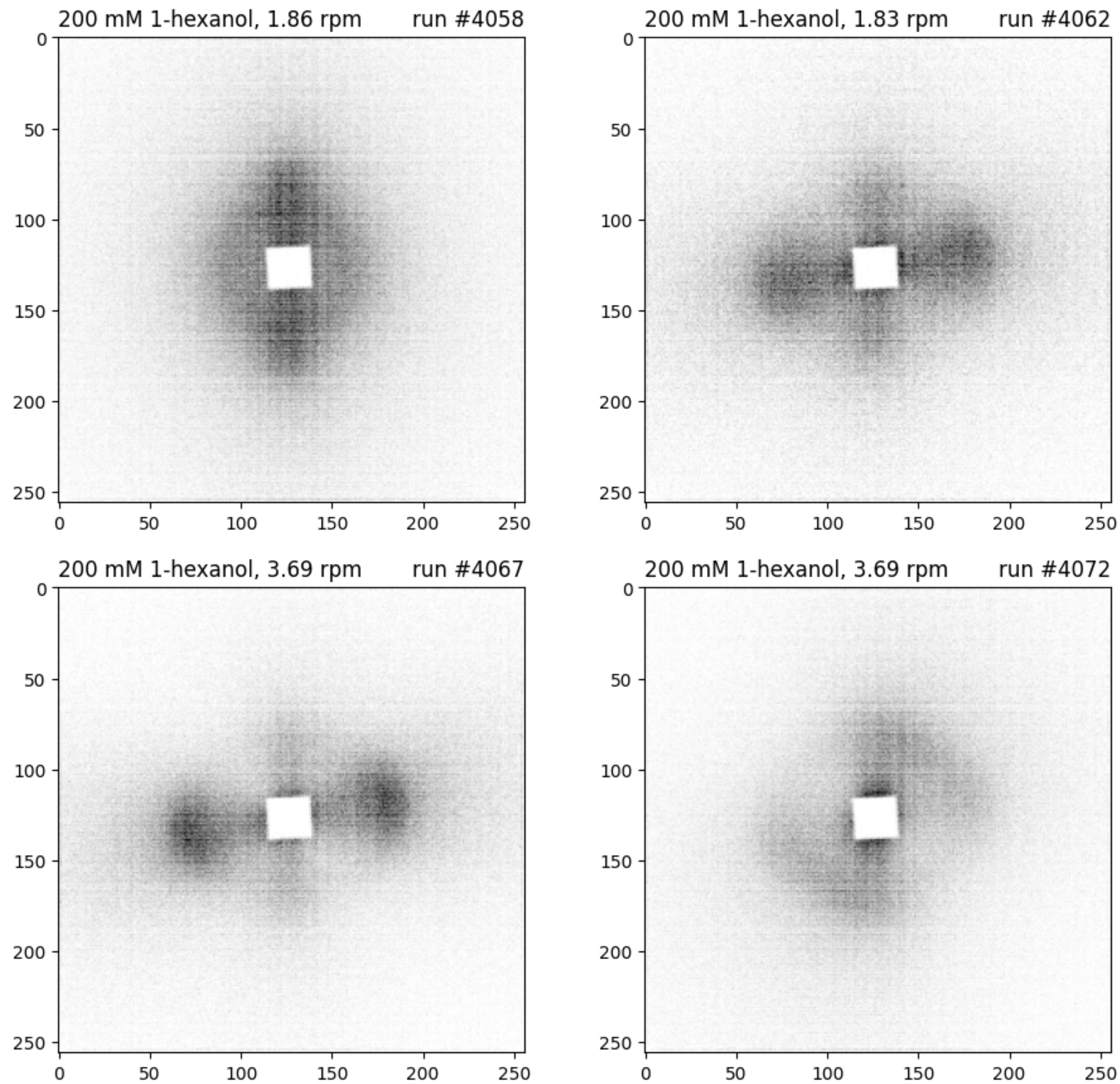

Figure 2: Examples of raw data showing anisotropic scattering at low rpm, averaged over 1 min each. We observe the transition from 1d-alignment to a clover-like pattern, which smears out with further deformation.