Experimental report

14/08/2017

Proposal: 9-13-674 **Council:** 4/2016

Title: A SANS Study of the Shear-Induced Orientational Order of the Nematic Phase of Amyloid Fibrils

Research area: Soft condensed matter

This proposal is a new proposal

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Samples: RFRFRFRF

Instrument	Requested days	Allocated days	From	To
D22	0	2	07/09/2016	09/09/2016
D11	3	0		

Abstract:

SANS will be used to investigate the shear-induced alignment of the nematic phase of amyloid fibrils formed by designed octapeptides. The octapeptides will included sequenced and blocky peptides rich in arginine and phenylalanine such as RFRFRFRF and RRRRFFFF. In the former case, SAXS shows orientation under flow for a 1 wt% sample, however this was not controlled (it resulted from flow into a capillary delivery system at a synchrotron beamline) and there has been no prior systematic study of shear-induced orientation in such systems. SANS is well suited to this study because of the high contrast available using D2O as solvent as well as the elimination of beam damage (associated with longer X-ray experiments during kinetic shear-alignment measurements).

Report:

The bola-amphiphilic arginine-capped peptide RFL_4RF self-assembles into nanotubes in aqueous solution. The nanostructure and rheology were probed by *in situ* simultaneous rheology/small-angle scattering experiments including rheo-SANS.[1]

We used a combination of rheo-SANS (D22, ILL), rheo-GISANS (FIGARO, ILL) and rheo-SAXS (ID02, ESRF) techniques to examine orientation. Bulk alignment effects in a Couette (concentric cylinder) were examined by rheo-SAXS and rheo-SANS and structure-flow properties were investigated by simultaneous rheology/scattering measurements.

SANS data were obtained on beamline D22. Solutions of RFL_4FR were prepared using D_2O as a solvent in order to minimize incoherent background scattering from the buffer. Samples were loaded into a stress-controlled rheometer from Anton Paar (Paar Physica MCR 501) with a custom-made quartz Couette geometry (30 mm diameter, 0.5 mm gap) that allows for simultaneous measurement of the rheological behaviour and scattering patterns, termed rheo-SANS. Samples were subjected to shear at different shear rates in the range $\dot{\gamma}$ =0.1 s^{-1} to 1000 s^{-1}. The sample-detector distance was 17 m, 5.6 m or 2.5 m.

Selected SANS patterns are shown in Fig.1. Note that features from the form factor of nanotubes are visible, in particular the intensity maximum at q = 0.12 nm^{-1} which is associated with the nanotube form factor as discussed shortly. The overall degree of alignment observed in the radial SANS patterns is similar to that in the SAXS measurements at the same shear rate. The same loss of alignment upon cessation of shear was noted. The degree of orientation in the SANS patterns in the radial configuration was quantified by fitting azimuthal intensity profiles (in a band centered on q = 0.12 nm^{-1}) and extracting the orientational order parameter.

These results are included in our published paper on shear-alignment and rheo-SAXS/rheo-SANS on these peptide nanotubes.[1]

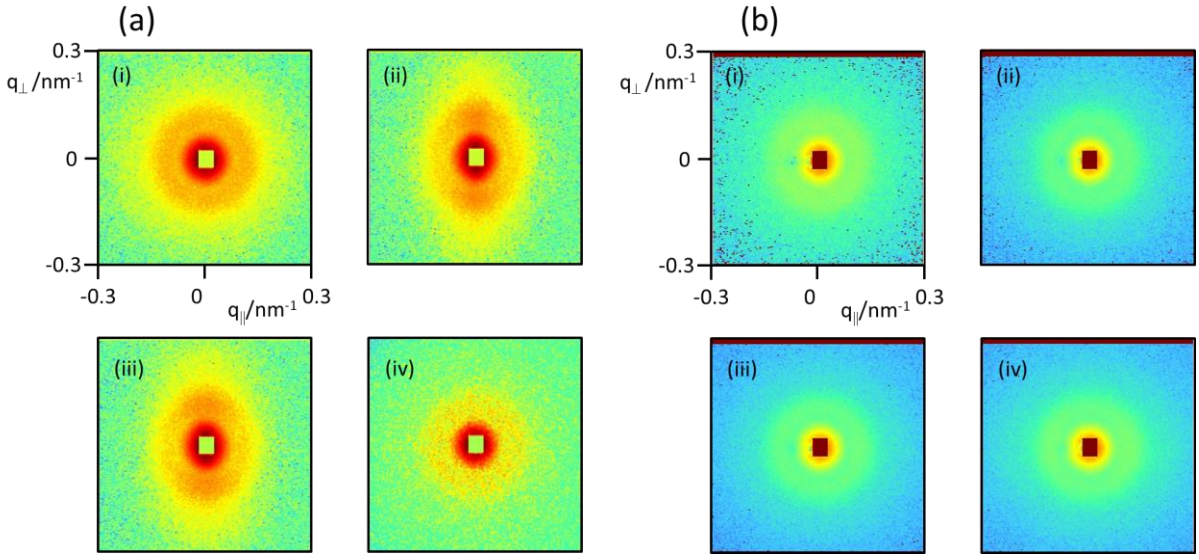

Fig.1. SANS patterns obtained from a 1 wt% solution of RFL_4FR in D_2O in (a) radial configuration. (i) Zero shear (ii) under shear at $\dot{\gamma}$ = 100 s^{-1}, (iii) under shear at = 1000 s^{-1},

(iv) Following shear at = 1000 s^{-1}. The shear direction is horizontal and the intensity scale is logarithmic. (b) tangential SANS patterns. (i) Zero shear, (ii) under shear at = 10 s^{-1}, (c) under shear at = 100 s^{-1}, (d) under shear at = 1000 s^{-1}.

References

1. Hamley, I. W.; Burholt, S.; Hutchinson, J.; Castelletto, V.; da Silva, E. R.; Alves, W. A.; Gutfreund, P.; Porcar, L.; Dattani, R.; Hermida-Merino, D.; Newby, G. E.; Reza, M.; Ruokolainen, J.; Stasiak, J., Shear Alignment of Bola-Amphiphilic Arginine-Coated Peptide Nanotubes. *Biomacromolecules* **2017,** *18*, 141-149.