Experimental report

05/02/2017

Proposal: 9-11-1801 **Council:** 4/2016

Title: Rheo-SANS study of hydrogel mixtures during gelation under shear.

Research area: Materials

This proposal is a new proposal

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Samples: C30H28N2O5
C6H10O6
C19H22N2O5

Instrument	Requested days	Allocated days	From	To
D11	3	2	19/12/2016	21/12/2016

Abstract:

Small molecules known as low molecular weight gelators (LMWG) can self-assemble to form a network of fibres, which trap and hold a vast amount of water forming a gel. An increase in the acidity of the system can be used to trigger gel formation and during this process the way molecules are arranged in space changes as they assemble into fibres. We are particularly interested in gels formed from two types of LMWG. The behaviour of these soft solids when shear is applied is important both for real world applications and in the understanding of the structures formed. We will use small angle neutron scattering to investigate the sizes and shapes of structures formed within a solution of two LMWG while the gel is forming and while it is simultaneously sheared using a rheometer.

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Introduction Low molecular weight gelators (LMWG) self-assemble into hierarchical structures when gelled. Understanding these structures is complicated by the fact that gelation in water is a phase separation event, with insoluble fibrous structures being formed. Hence, the process of assembly is also extremely important. Critically, the properties of the gels are not only controlled by these primary structures, but by how they are arranged in space. We have synthesised an extensive library of peptide based LMWG, which form hydrogels using various different triggers, such as a pH change. We are specifically interested in understanding how the gels form, since our rheological data shows that this is a multi-step process. However, rheology alone does not explain what the structures are that are leading to the different stages in the evolution of the structure.

Experimental The LMWG were prepared as described elsewhere.[1] Solutions at 10 mg/mL were prepared in D_2O at high pD by the addition of one molar equivalent of NaOD (0.1 M), followed by stirring until the LMWG had dissolved.
SANS experiments were performed on the D11 diffractometer, a neutron wavelength of λ= 10 Å was employed at three different detector distances, D = 1.2, 8 and 40 m. This set-up corresponds to a Q range from 1.0 × 10-3 to 0.31 $Å^{-1}$. All spectra were normalised and corrected using the scattering of the empty cell. Scattering data were corrected for electronic noise and incoherent background subtraction and normalised by the intensity scattered for a 1 mm H_2O sample corrected by the intensity scattered from the empty quartz cell. The MCR501 rheometer was used, with the sample placed in the couette cell. To follow gelation, the trigger (GdL^2) was added, and then the solution immediately placed in the cell. A time sweep was then carried out at 0.1 rad/s and a strain of 1 s^{-1}. At the end of the experiment (about 8 hours), a strain or frequency sweep was carried out. For the viscosity data, a LMWG solution was added to the cell and a number of different shear rates were applied.

Results We were able to follow the gelation process of several hours and simultaneously collect rheological and SANS data. This should allow us to correlate the structure of the self-assembled aggregates and the overall rheology. Additionally, we were able to collect data on solutions of the LMWG at high pH and show that we were able to shear align the structures. This has provided key data, as it is possible to separate ourt the scattering from the network and that of the primary structures.

These data are currently being analysed and will be published in the future.

References

1. L. Chen, S. Revel, K. Morris, L.C. Serpell and D.J. Adams, *Langmuir*, **2010**, *26*, 13466-13471.
2. D.J. Adams, W.F. Frith, M. Kirkland, L. Mullen and P. Sanderson, *Soft Matter*, **2009**, *5*, 1856-1862.