Experimental report

19/08/2019

Proposal: 9-11-1890 **Council:** 4/2018

Title: Study of crystal-to-crystal transition using ultra-low crosslinked microgels and Rheo-SANS

Research area: Physics

This proposal is a new proposal

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Samples: pNIPAM suspension in D2O

Instrument	Requested days	Allocated days	From	To
D22	3	3	24/09/2018	27/09/2018

Abstract:

The interplay between softness, morphology and crystallization is one of the most challenging and fascinating problem that both theoreticians and experimentalists are dealing with in the last decades. Recently it has been shown that colloid suspensions, as well as microgel suspensions (soft and deformable object) present a two-step crystallization, mediated by a liquid phase. At the same time, it has been shown that star polymers, polymer chains tethered to a central microscopic core, are able to evolve from a crystalline lattice to another without the liquid phase mediated step. We propose to use ultra-low crosslinked (ULC) microgels, the softest microgels that can be obtained with precipitation polymerization, to probe if the presence of the liquid-mediated crystallization depends on the softness or on the morphology of the object. We choose to use ULC since they are the link between regular microgels and star polymer: they are spherical as regular microgel but present strong interpenetration at high concentrations. Rheo-SANS, under oscillatory conditions in the vicinity of the glass transition of the ULC-microgels will probe the crystal to crystal transition.

The aim of the experiment was to understand if the direct transition from a crystal lattice to another, without a liquid phase in between, is only related to the softness of the microgels or if the morphology of the object is playing a role in the nucleation process.

We have performed Rheo-SANS experiments under oscillatory conditions in the vicinity of the glass transition of the suspensions of ULC-microgels (3 different concentrations). The crossover frequency, ω_c, is the frequency above which the suspension behaves as a solid. The values of ω_c for the different concentrations have already been determined according to the measured viscoelastic spectra (Fig. 1C). For every concentration three strain sweeps experiments, using two different frequencies above ω_c, have been performed

From the analysis of the 2D images of the detector was not possible to observe Bragg peaks of the crystals even whether the in solutions crystals were clearly visible, Fig. 1. This can be due either to the fact that the first, and more pronounced peak, lies at q-values too low with respect to the one that D22 can probe. Another possibility was that the frequencies applied were too high to melt and reform the crystalline lattice. We are carefully considering how to address those problems and apply for a follow-up experiment. On one hand we are thinking to realize smaller ultra-low crosslinked microgels (radius ~100 nm, first Bragg peak ~ $6.3 \cdot 10^{-2}$ nm^{-1}); on the other we are thinking to let the samples rest more in the rheometer cell before starting with the oscillations.

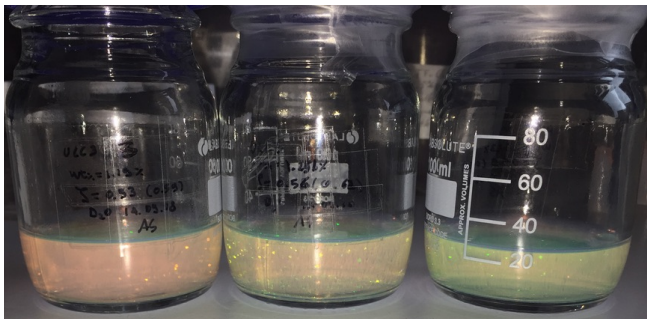

Figure 1 Image of the crystalline samples used during the experiments at three different concentrations.