Experimental report

01/09/2022

Proposal: **EASY-1060** **Council:** 4/2021

Title: Determination of thermoresponsive microgels and PEG-based nanoparticles form factor

Research area: Soft condensed matter

This proposal is a new proposal

Main proposer: **Yuri GERELLI**

Experimental team: Alessia PEPE
Erica DUGO

Local contacts: Lionel PORCAR

Samples: PNIPAM and PEG nanoparticles

Instrument	Requested days	Allocated days	From	To
D22	4	4	10/10/2021	11/10/2021

Abstract:

We propose to measure the changes in structure of thermoresponsive PNIPAM-based microgels above (40 °C) and below (25 °C) the well-known VPT. These data are necessary to clarify some of the structural features emerged from earlier SANS experiments performed in a low-resolution configuration at the ISIS neutron source. If successful, this experiment will allow the submission of a publication within the end of the year. During the same easy access experiment we plan to investigate the internal structure of PEG-Polyamine nanoparticles developed as drug delivery systems. We have performed SAXS, DLS and TEM measurements on these samples, and by SANS we plan to determine the relative arrangement on the two polymeric components. For this purpose, NPs with deuterated PEG will be prepared.

The need of an easy access is dictated by the limited number of samples to be measured.

Report for EASY-1060

During the experiment EASY-1060 we investigated the structure of thermoresponsive PNIPAM-based microgel measured above and below the well-known volume phase transition (VPT).

These experiments were performed to verify some unexpected structural features emerged from earlier SANS experiments performed in a low-resolution configuration on SANS2D at the ISIS neutron source. Samples were prepared using three different methods of inclusion of cross-linkers (all at once, drop-by-drop with two different rates) and tested before the experiment by dynamic light scattering.

The aim of the experiment was to verify if the preparation method could change the microgel morphology (especially the core-corona size ratio) while keeping constant the cross-linker density and polymer type.

SANS curves were collected on D22 at 313 K, 303 K and 293 K for protiated samples prepared in D_2O. The reduced datasets (by GRASP) were then analyzed using the analytical expression of the fuzzy sphere form factor summed with a Lorentzian for factor typically used to describe the mesh size in polymer networks. Typical fits are shown in Figure 1.

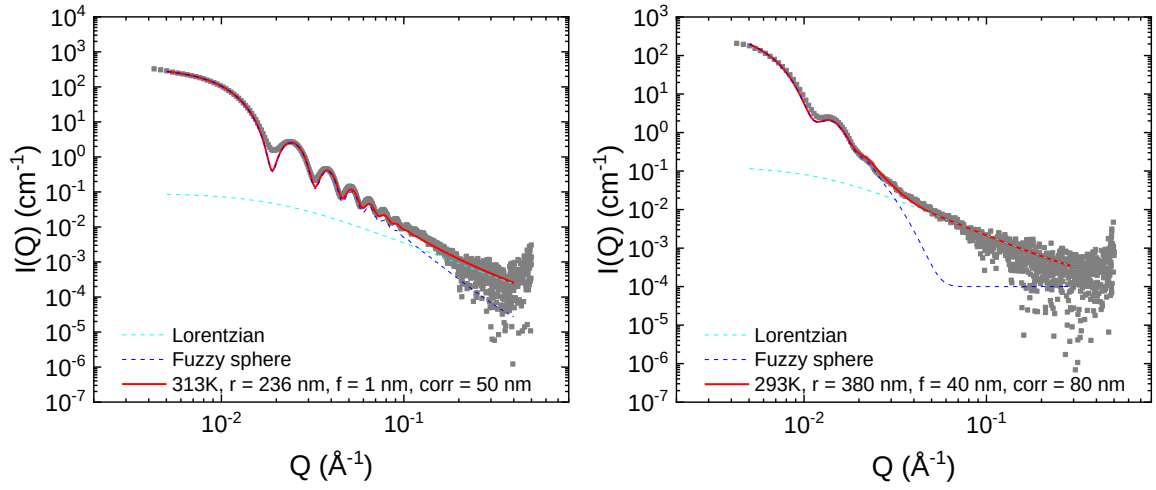

Figure 1. Form factor of a PNIPAM-based microgel measured at 313 K and 293 K. The total fit is shown in red while the fuzzy sphere and Lorentzian form factors are shown with blue and cyan dashed lines respectively.

The analysis contradicted the previous results obtained on SANS2D. The different preparation methods affect the overall size and the polydispersity of the microgel particles while the internal features remain almost constant in relative size.

Coarse-grained molecular dynamic simulations are being performed at the moment on the same systems and a manuscript should be submitted by the end of 2022.