Experimental report

05/02/2018

Proposal: **INTER-350** **Council:** 4/2016

Title: Internal time on D11

Research area:

This proposal is a new proposal

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Samples: C6H11NO)n

Instrument	Requested days	Allocated days	From	To
D11	3	3	22/11/2016	25/11/2016

Abstract:

Internal pressure induced structure changes on a pDADMAC - p(NIPAM-co-MAA) system.

Introduction Microgels are an interesting topic for researchers, due to their different behavior compared to linear polymers. This applies especially to smart microgels, which react on external stimuli by changing their size. The change in their size is effected by the quality of the solvent, which changes the polymer chain conformation.
As the temperature induced shrinking is investigated in other works. This experiment concentrated on pressure dependent behavior of a p(NIPAM-co-MAA) microgel and how the pDADMAC concentration is effecting the phase transition and especially the changes in the internal structure of the system.

Results With the background of temperature dependent SANS measurements, the complexes with the initial charge ratio (icr) 0.5 and 2 were investigated with pressure at D11. Furthermore the microgel without PE was measured for comparison reasons. All samples were measured at 20 and 36 °C at different pressures up to 2500 bar in the 3 kbar cell.
The resulting curves of the microgel without pDADMAC can be seen in Figure 1. The curve without any pressure at 20 °C is taken from the temperature dependent measurements. Therefore it has a different background intensity. Both temperatures show that the pressure has an effect on the mircogel. The internal structure is changed at the lower temperature (20 °C) with higher pressure (2500 bar) and around the transition temperature (36 °C) with much less pressure (500 bar).

The equivalent measurements were performed for the microgel and PE complex with an icr of 0.5. The resulting curves are shown in Figure 2. At 20 °C this amount of pressure has no effect on the complex. In contrast to that at 36 °C only 100 bar change the curve completely and the complex precipitated during the measurement at 100 bar. When reducing the pressure to 0 bar the complex is resolved again.

The third sample, which was measured pressure dependent, is the complex with an icr of 2 (Figure 3). A clear pressure dependency can be seen for this sample. At 0 and

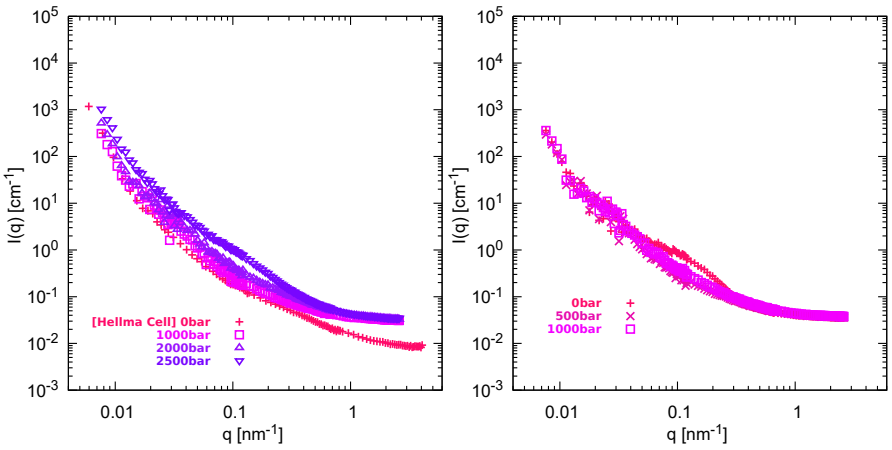

Figure 1: Pressure dependent SANS measurements of the microgel without PE at 20 (left) and 36 °C (right).

500 bar no minima can be observed. When the pressure is increased to 1500 bar a minima is forming, which is shifted to higher q values by increasing the pressure further.
At 36 °C the biggest change is found when adding only a small amount of pressure. The minima is not visible shifted during the whole measurement. Furthermore the shoulder is decreasing with pressure, which is indicated with the black arrow. To get this effect only a few 100 bars are needed. When the pressure is up to 500 bar the whole process is already through. The curve at even higher pressure (1500 bar) looks different, is justified with the fact that the complex precipitated. With this results it is assumed that the size of the complex at 36 °C is not effected strongly, hence the internal structure is changed when pressure is introduced. When the sample is under to much pressure it is not stable and precipitates.

Comparing the measurements at both temperatures, it is noticeable that at 20 °C the sample is stable even at much higher pressures and that the pressure effect is only visible at much higher pressures as compared to the sample at 36 °C. This is deformed with much lower pressure (100 bar).
All in all it can be said, that increasing the pressure decrease the solvent quality, which leads to a collapse of the samples.

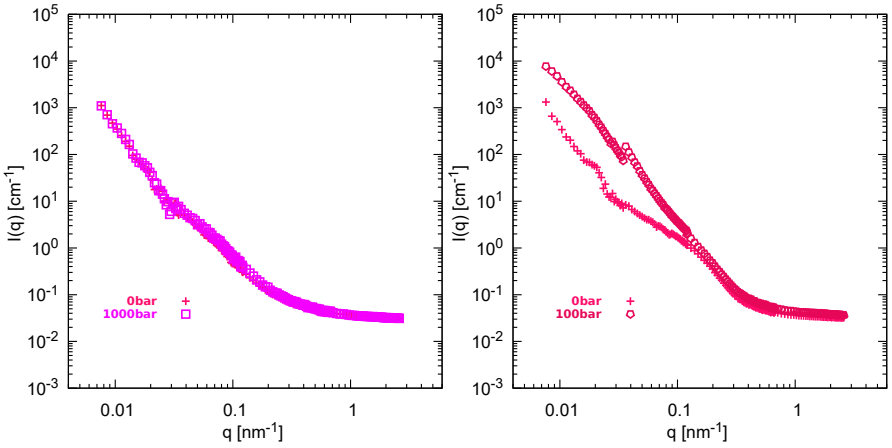

Figure 2: Pressure dependent SANS measurements of the complex with an icr of 0.5 at 20 (left) and 36 °C (right).

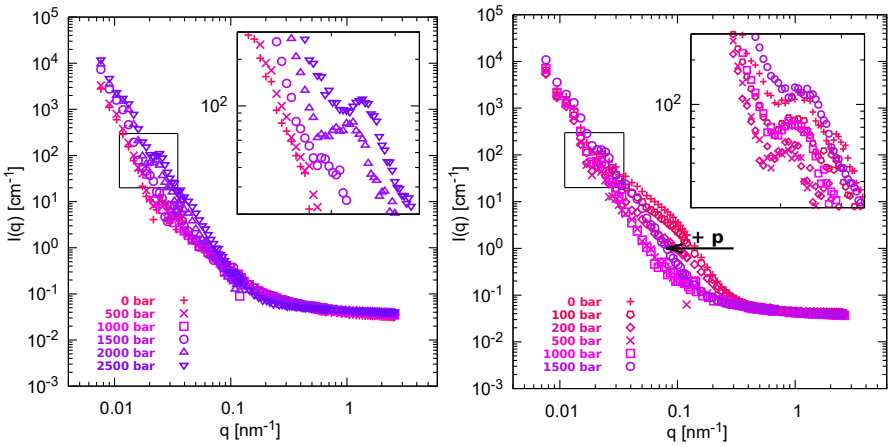

Figure 3: Pressure dependent SANS measurements of the complex with an icr of 2 at 20 (left) and 36 °C (right).