ESRF	**Experiment title:** Glass and jamming transitions in soft particles made of PNIPAM and Poly(acrylic acid)	**Experiment number**: SC-5349
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Report:

Small Angle X-ray Scattering (SAXS) measurements have been performed on microgels composed of Interpenetrating Polymer Networks (IPN) of poly(N-isopropylacrylamide) (PNIPAM) and poly(acrylic) acid (PAAc), thermo- and pH-responsive polymers respectively, in aqueous suspension. Measurements were carried out at different concentrations and temperatures across the typical volume phase transition (VPT) of particles from a swollen to a shrunken state that happens around 305K [1]. A recent rheological study performed by our group [2], in fact, has revealed the existence of a liquid-glass and a glass-jammed transition, depending on concentration, for an IPN microgel with high PAAc content. Hence, the aim of the experiment was to probe the structure as a function of concentration and temperature across the VPT to characterize the aforementioned transitions from a liquid to an arrested state. Measurements were performed also with Au nanoparticles (Au-NPs) to increase the scattering contrast. During the experiment SC-5349 we investigated an IPN with 32% of PAAc with a hydrodynamic particle diameter D = (1100 ± 100) nm and a PNIPAM microgel at different concentrations in the range C_w = (0.2-10) % and temperatures in the range (20-48)^{0}C. Particular attention has been devoted to forms factors measurements for all the investigated samples and temperatures to obtain optimal results. We used

2mm capillaries for samples with low viscosity and a cell with mylar windows for extremely viscus samples. We used a beam with 12.2 keV energy and an exposure time of 0.1s. In Fig.1 a) an example of static structure factor at T=293k and at different concentrations is shown, the first visible peak is at around Q=0.0055 nm^{-1} corresponding to a distance $d=2\pi/Q \sim 1140$ nm comparable with the particle diameter D = (1100 ± 100) nm, an evolution of the second peak with concentration is also observed signature of a changing microscopic structure in the sample. These preliminary results that highlight the existence of different structures, seem to support recent rheological measurements that pointed out different rheological regimes, characteristic of different states. Fig.1 b) reports the temperature evolution of the S(Q) across the VPT for a high concentration sample at C_w = 10 % that, by eye, appears in a "jelly state" at room temperature. In this case a transition between two different arrested state is found around T~28^{0}C.

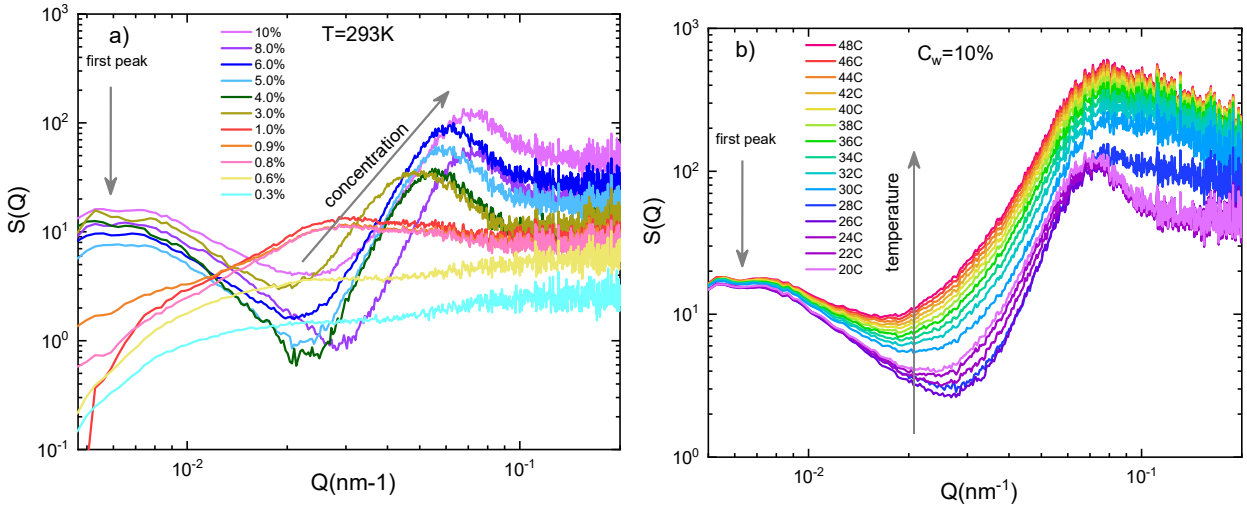

Fig.1 Static structure factor for an IPN microgel with 32% PAAc **a)** at T=293k and at different concentrations. **b)** at C_w = 10 % and at different temperatures across the VPT.

[1] X. Xia & Z. Hu, "Synthesis and Light Scattering Study of Microgels with Interpenetrating Polymer Networks", *Langmuir* **20**, 2094 (2004).

[2] S Franco, E Buratti, V Nigro, E Zaccarelli, B Ruzicka, R Angelini. Glass and Jamming Rheology in Soft Particles made of PNIPAM and polyacrylic acid. *Int. J. Mol. Sci.* 22 (8), 4032 (2021).