



Distribution of Cross-linkers in Microgels obtained by Contrast variation in Small Angle Neutron Scattering

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Smart membranes have applications in wastewater treatment and separation[1]. These membranes show permeability depending on the external stimuli, such as thermoresponsive microgel-based membranes. Such membranes can be prepared by UV cross-linking, electron beam cross-linking, or chemical cross-linking. A free-standing membrane made of NIPAM microgels has been reported, which are cross-linked with a secondary UV-sensitive crosslinker HMABP. These free-standing thermoresponsive membranes are resistant above their VPTT(33°C) and show permeability of ions below it[2].

To understand the incorporation of HMABP in the microgel particles, we performed contrast variation Small Angle Neutron Scattering(SANS) experiments. We synthesized microgels with deuterated monomers (NIPAM/NIPMAM) and non-deuterated cross-linkers BIS and HMABP. We performed contrast-matching experiments to see how these cross-linkers are distributed within a microgel particle(Figure-1). With changing scattering length densities of the solvent, the particles show similar scattering behavior when the D-monomers are scattering, thus confirming that they form the microgel. Whereas when the deuterated monomers are matched, and the cross-linkers are highlighted (which is at 100%D₂O), there is a change in the scattering behavior which suggests an inhomogeneous distribution of the cross-linkers inside the microgels.

In the high-q region, both the NIPAM and NIPMAM-based microgels show similar scattering behavior at different solvent scattering length densities(except at 100%D₂O). But in the low-q region, the scattering behavior of NIPMAM-based microgels, the samples above 50%D₂O when the cross-linkers are highlighted, scatter very differently. This again suggests the inhomogeneous distribution of cross-linkers, which is different in NIPMAM- as opposed to NIPAM-based microgels.

It is hoped that these distributions of cross-linkers in microgels based on deuterated monomers can be used to describe the distribution of cross-linkers in non-deuterated microgels as these are comparable in size and also show similar thermoresponsive behavior with a VPTT between 33°C-44°C (depending on the monomer NIPAM/NIPMAM) and reach a fully collapsed state at 60°C. However, the scattering behavior is different when comparing the deuterated microgels with non-deuterated ones because, in a given solvent scattering length density(100% D₂O), the non-deuterated monomers are highlighted along with the cross-linkers while the deuterated ones are contrast matched with the solvent.

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