DEUTERIUM NMR AND RHEOLOGY OF MICROGEL COLLOIDS AT AMBIENT AND HIGH PRESSURE

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Microgel colloids exhibit a polymer collapse transition resulting in a large reduction in colloid size at high temperatures or pressures. Our goal is to obtain a microscopic understanding of the internal structure and microscopic dynamics of microgels by examining the temperature and pressure dependence of the collapse transition. We have conducted a systematic study of how the nature of this collapse transition is affected by crosslink density (Cd). We used deuterium NMR (^{2}H-NMR) to probe the microscopic dynamics of cross-linked poly-N-isopropylacrylamide (p-nipam) chains, in microgel colloids, as a function of temperature and pressure. Four differently crosslinked microgels colloids were synthesized with deuteron labels on the nipam backbone (d3-nipam). Corresponding macroscopic properties of unlabeled colloids having the same crosslink densities were characterized by dynamic light scattering (DLS) and rheology. Rheological characterization as a function of temperature (*T*) and particle concentration (*c*), and for 4 crosslink densities, showed that the microgel viscosity decreases as temperature is increased, and that in the high *T*/low *c* regime, there is a collapse of the viscosity as a function of *T* and *c* when plotted against volume fraction: this yields a measure of the water content in the particles as function of *T.* ^{2}H-NMR spectra of the d3-nipam suspensions for all Cd indicated freely moving chains at low temperature and a nearly immobilized fraction above 35°C. This is consistent with DLS observations of a transition from swollen to collapsed colloids. ^{2}H-NMR spectra for the dry powder indicated totally immobilized segments in the particle. Nipam segments in the collapse phase of the d3-nipam suspension were more mobile than those in the dry powder. This suggests significant amounts of water in the collapsed phase, a finding consistent with the rheology observations. For the highest two values of Cd, microgel spectra showed the presence of an immobilized fraction of segments even in the swollen phase. Variable pressure NMR (up to 90 MPa) showed a slight increase in transition temperature with pressure for all Cd values studied.