18d Asphaltene-Stabilized Emulsion Characterization by Small Angle (Sans) and Ultra Small Angle Neutron Scattering (Usans)

Vincent J. Verruto and Peter K. Kilpatrick

Asphaltenes in model oil mixtures were prepared and mixed with water under high-shear conditions (15,000 RPM, 10 min) in a rotor-stator homogenizer. Small-angle (SANS) and ultra small-angle neutron scattering (USANS) were performed at the NCNR (NIST, Gaithersburg, MD) on the emulsified samples after a series of dilutions. The degree of deuteration of the continuous (oil) and dispersed (water) phases was engineered to be as close to contrast matching as possible so as to maximize the contrast of the thin nanometer-scaled asphaltenic film stabilizing the droplets. The scattering data were fit with a polydisperse core/shell form factor model. Two approaches regarding bulk asphaltene contributions to the scattering data were used before fitting with the core/shell model: (1) treating the bulk asphaltenes as scattering bodies (modeled with a polydisperse oblate cylinder form factor) and (2) assuming bulk asphaltenes only alter the bulk solvent scattering length density (SLD). Core/shell model fits of the SANS data yielded realistic parameters that describe the emulsions: $R_{\mathrm{core}} \sim 1.5\text{-}2\ \mu\mathrm{m}$, $\Delta \sim 30\text{-}60$ Å, and polydispersity ~ 0.3. From these fits we also ascertained the film composition (via scattering length density), and both asphaltene contribution approaches indicated significant solvent entrainment in the interfacial films (20-50% v/v) assuming equivalent bulk and entrained solvent compositions. Scattering intensity for samples in which the core and solvent SLDs were not closely matched was dominated by Porod scattering ofthe aqueous droplets (q^{-4} dependence) over the film scattering (q^{-2} dependence) in the SANS low-q regime. This, along with the inability to bridge the USANS and SANS data, highlights the inherent complexity in characterizing asphaltene-stabilized emulsions and stress the delicate nature of matching both mass density and scattering length density between the aqueous droplet and the organic solvent phases.