Experimental Report

11/09/2013

Proposal: **9-10-1328** **Council:** 10/2012

Title: Aggregation of surfactants in non-polar solvents

This proposal is a new proposal

Researh Area: Chemistry

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Samples: anionic surfactants - cyclohexane

Instrument	Req. Days	All. Days	From	To
D16	4	5	28/03/2013	02/04/2013

Abstract:

This aim of this project is to use D16 SANS to determine the structure of inverse micelles of counterion exchanged sodium dioctylsulfosuccinate surfactants (MAOT) near their critical micelle concentration (CMC). It is an unresolved question whether this property exists for nonpolar solvents, although different experiments have attempted to measure it. The understanding of the formation of inverse micelles is important for understanding charging in nonpolar solvents, as surfactant aggregates are thought to stabilize the ions formed. SANS measurements indicate that there may be a concentration below which NaAOT does not form inverse micelles in cyclohexane, and SAXS measurements will be performed with MAOT surfactants to detect low concentration aggregates with these surfactants. However, x-ray contrast only enables the counterion core structure to be observed. The proposed SANS experiments will enable the structure of the whole inverse micelle (H-surfactant in D-cyclohexane) and the hydrated core (D2O-hydrated H-surfactant in H-cyclohexane) to be determined, which will provide a comprehensive view of the inverse micelles and insight into the nature of the CMC in nonpolar solvents.

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Title :
Aggregation of surfactants in non-polar solvents
Instrument :
D16
Dates of experiment :
From : 28/03/2013 To : 02/04/2013

This experiment involved studying the structure of Ca^{2+}, Mn^{2+}, and Zn^{2+} analogues of sodium dioctylsulfosuccinate (Aerosol or AOT) in deuterated cyclohexane. These surfactants form inverse micelles in nonpolar solvents, with the polar ionic groups in the center and the branched alkyl tails extending into the solvent. The high-*Q* resolution of D16 meant that the core-shell region could be studied with high precision. These three metals were chosen because they are hydrated in salts and the rate of water exchange is high. By preparing the surfactants in D_2O rather than H_2O, it was possible to generate inverse micelles where the cores would be approximately contrast matched to the deuterated solvent. Several concentrations of surfactant were studied, as the concentration of surfactant approached the inverse micelle CMC of NaAOT. The SANS profiles for these three metals at the highest concentration (~10 mmol L^{-1}) are shown in Figure 1. The experimental curves have been fit to a monodisperse core-shell sphere model. The core radius is allowed to vary (expected for different metals), as are the core and shell SLD. The SLD of the alkyl shell would be expected to differ slightly from the expected value due to solvent penetration, and the SLD of the core would indicate how much the D_2O coordination modified the scattering curve. The fit SLD of the core is more similar to that expected for the metal ion alone rather than pure D_2O, indicating that the small amount of D_2O in the inverse micelle core is not sufficiently deuterated to match it to the solvent.

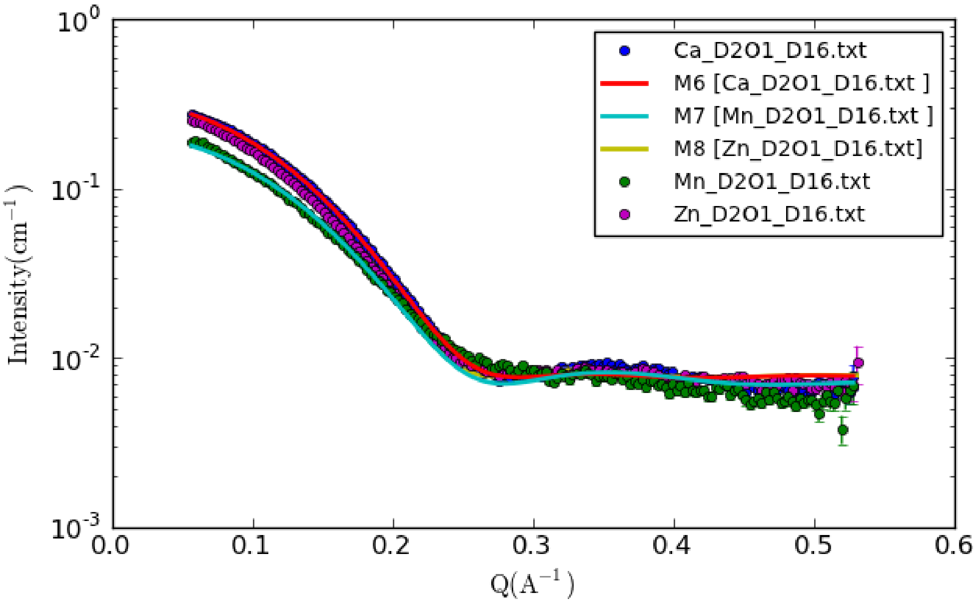

Figure 1. Scattering curves for metal exchanged AOT surfactant (with Ca, Mn, and Zn) in cyclohexane-D. The cores have been prepared with D_2O to highlight the shell scattering, but the amount of deuterium in the core is not sufficient to mask scattering from the particle core.