Experimental report

01/02/2016

Proposal: 9-12-434 **Council:** 4/2015

Title: Organogels with CO2-philic surfactants

Research area: Chemistry

This proposal is a new proposal

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Samples: surfactants-hydrotropes-alkanes-water

Instrument	Requested days	Allocated days	From	To
D22	1	0		
D11	1	0		
D33	1	1	26/10/2015	27/10/2015

Abstract:

Contrast variation SANS will be used to probe structure in thermoresponsive organogels comprising of custom-synthesized surfactants (figure 5 a-c) and low molecular weight organogelators (LMOGs). These LMOGs (para-substituted phenols, figure 5 d) are a welcome change to the field due to their relatively low cost and commercial availability. They are markedly different from other LMOGs, which are both time consuming and laborious to synthesize. It is intended to test the proposed stacked phenol structure2-5 using contrast variation. Thermoresponsive structures will be explored around the pronounced ‘melting’ point, where a sharp decrease in viscosity is observed. Novel surfactants will be studied which have been specifically designed for low dielectric solvents such as supercritical CO2 (scCO2 figure 5 b-c). The structures of Co2+ and Ni2+ surfactant based gels will be investigated. This research is supported by the G8 Research Councils Initiative on Multilateral Research Funding - G8-2012 - EP/K020676/1 and an STFC funded studentship ‘Controlling fluid properties of dense CO2’. New results are included, in response to the suggestion on a previous submission 9-10-1428.

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***Instrument**: D33*
***Dates of experiment**: 23/10/2015 – 26/10/2015*

Recent work has shown that the addition of low molecular weight organogelators (LMOGs) in the form of p-substituted phenols (p-methyl phenol) can induce the formation of surfactant based thermo-responsive organogels using a partially fluorinated CO_2-philic sulfosuccinate surfactant, Na(CF2:AOT4), (**figure 1**). Contrast variation Small-angle neutron scattering (CV-SANS) has been employed to try and decipher the location of the phenol and surfactant in the gelled system (**figure 2**).

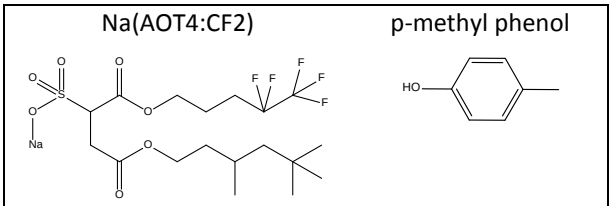

Figure 1: Structures of surfactants and phenols used.

When temperature is decreased, it is evident that there is a significant elongation of the micellar structure, indicative of the formation of the organogel. Elongation in these systems is key to the development of viscosifiers for supercritical CO_2 ($scCO_2$), due to the CO_2-philic nature of Na(AOT4:CF2), and has never been seen with LMOGs before. Studying and classifying surfactant assemblies in $scCO_2$ is incredibly experimentally challenging: samples need to be formed in-situ at high pressure (100-500bar) in a specially designed sample environment. We have devoted a significant amount of time to developing methodology and theory which allowed less beam-time intensive environments (surfactant/D_2O/oil) to act as proxies for water-in-carbon dioxide microemulsion systems. This promising area of research will be further explored through investigation of other partially fluorinated surfactants and substituted phenols (p-halophenols) that are known to give an organogel of increased strength. A manuscript for publication of this work is currently being prepared.

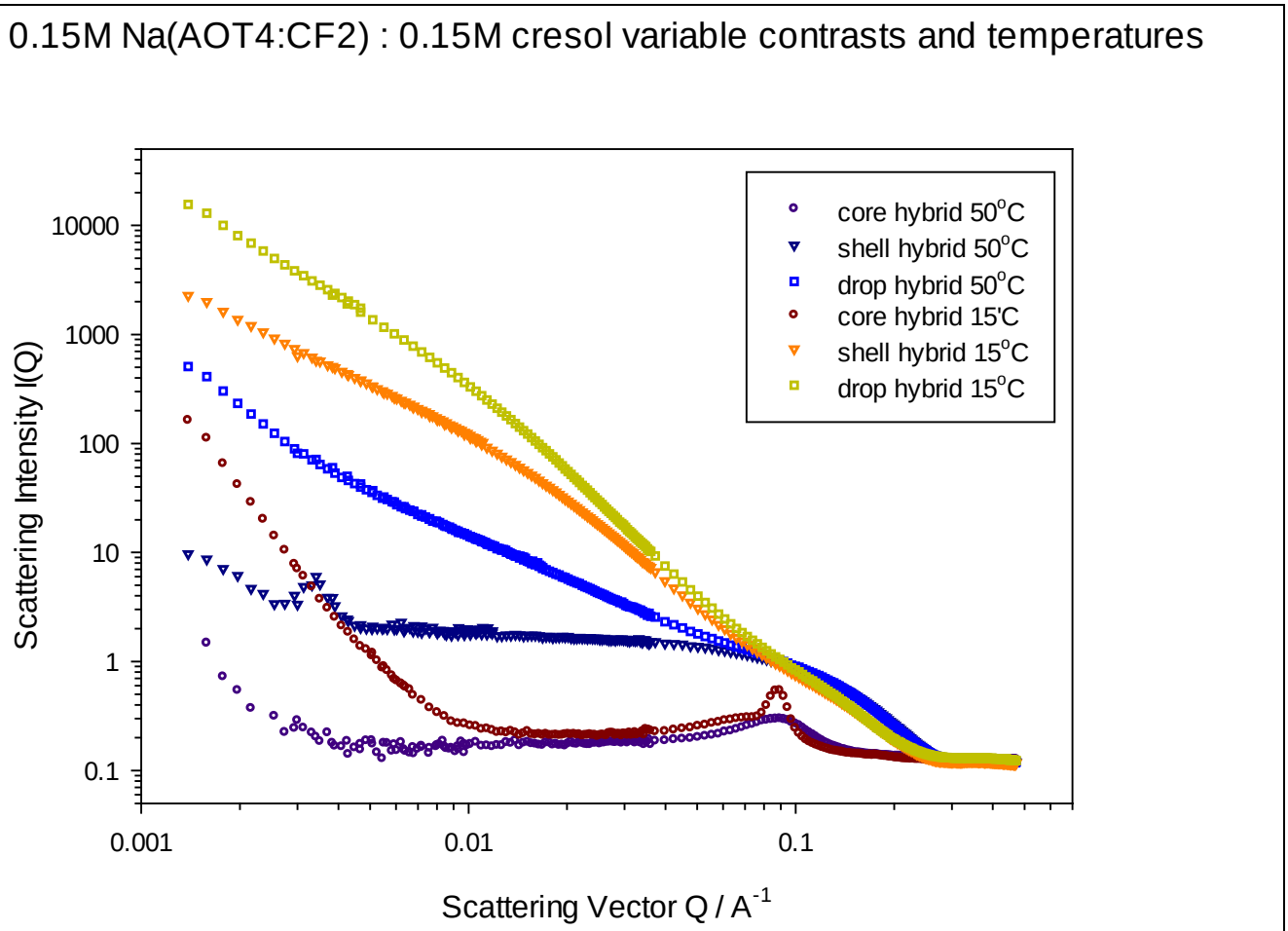

Figure 2: SANS profiles of partially fluorinated, CO_2-philic surfactant organogels at varied contrasts and temperatures.