Experimental report

24/10/2016

Proposal: **9-10-1468** **Council:** 4/2016

Title: Organogels with surfactants and halophenols

Research area: Chemistry

This proposal is a new proposal

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Samples: heptane - AOT - halophenols

Instrument	Requested days	Allocated days	From	To
D33	2	1	04/10/2016	05/10/2016
D11	2	0		

Abstract:

Contrast variation Small-angle neutron scattering (CV-SANS) will be used to probe structure of novel organogels made from surfactants and p-halophenols low molecular weight organogelators (LMOGs, Figure 2). Both the LMOG and the surfactant are commercially available. There is no literature on CV-SANS on these systems. Alongside this, the use of halophenols brings phase transition temperature of the gels to an appropriate temperature region (figure 3), and forms stronger gels than the hydrocarbon phenols previously documented 1-2, increasing their potential for use with CO_2-philic surfactants. These thermoresponsive structures will be explored around the ‘melting’ point, where a sharp decrease in viscosity is observed (Figure 3). Progress in this field has recently been reviewed by Peach et al.; this research has received external publicity through the UK government agency UK trade and Investment (UKTI)4 and is supported by an STFC funded studentship ST/L502613/1 - ‘Controlling fluid properties of dense CO_2’ and the G8 Research Councils Initiative on Multilateral Research Funding - G8-2012 - EP/K020676/1.

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***Instrument**: D33*
***Dates of experiment**: 04/10/2016 – 05/10/2016*

Contrast variation Small-angle neutron scattering (CV-SANS) has been used to probe structure of novel organogels made from surfactants using p-halophenols as low molecular weight organogelators (LMOGs, Figure 1). Both the LMOG and the surfactant are commercially available. There is no literature on CV-SANS on these systems. Alongside this, the use of halophenols brings phase transition temperature of the gels to an appropriate temperature region and forms stronger gels than the hydrocarbon phenols previously documented [1,2]. This increases their potential for use with CO_2-philic surfactants. These thermoresponsive structures have been explored around the 'melting' point, where a sharp decrease in viscosity is observed.

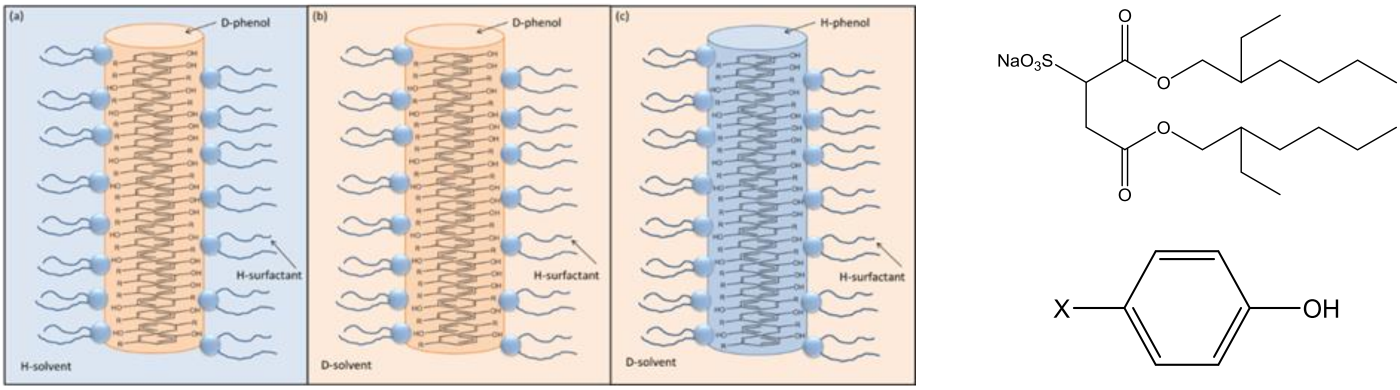

Figure 1 - Schematic of 'stacked' phenol structure with selective deuteration to give contrast between phenols, surfactant and solvent and structures of surfactant (Na(AOT)) and phenols (x=F, Cl, Br) used in this work.

Three different contrast conditions, each containing a fixed molar ratio of 1:1 Na(AOT) and p-phenol (0.10 M) were investigated. These samples all gave stable, self-supporting gels. D-phenol, H-surfactant and H-solvent were used to determine the location and distribution of p-phenol LMOG alone (Figure 1, a). A second contrast highlighted the surfactant only by using D-phenol and D-solvent with H-surfactant (Figure 1, b). Finally, the overall rod/fiber structure was determined with H-phenol, H-surfactant and D-solvent (Figure 1, c). The formation and destruction of the gel structure was observed as the temperature of the samples was methodically varied over the gel transition temperature. Scattering profiles where the halophenol species are varied can be seen in **Error! Reference source not found.**. The scattering profiles at obtained high T (above the gel transition temperature) have been fit to a spherical models using fitting program SASview after applying the Guinier approximation and determining R_g for the species. 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 ($scCO_2$), as these surfactants are analogous to many CO_2-philic surfactants[3,4]. Because of this, understanding gleaned from this experiment can directly influence the development of gels in $scCO_2$. Significant efforts have also gone into the design of CO_2-philic surfactants, and use of surfactants in supercritical CO_2 as viscosifiers. The development of viscosifiers and gelling agents in CO_2 is relevant in enhanced oil recovery (EOR) and carbon capture and storage (CCS)[5,6]**,** an area of research that has attracted significant research and media attention in the past. Furthermore, neither the full range of halogenated phenols, nor the use of contrast variation has previously been investigated; this set of experiments is unique in its use of extensive CV-SANS.

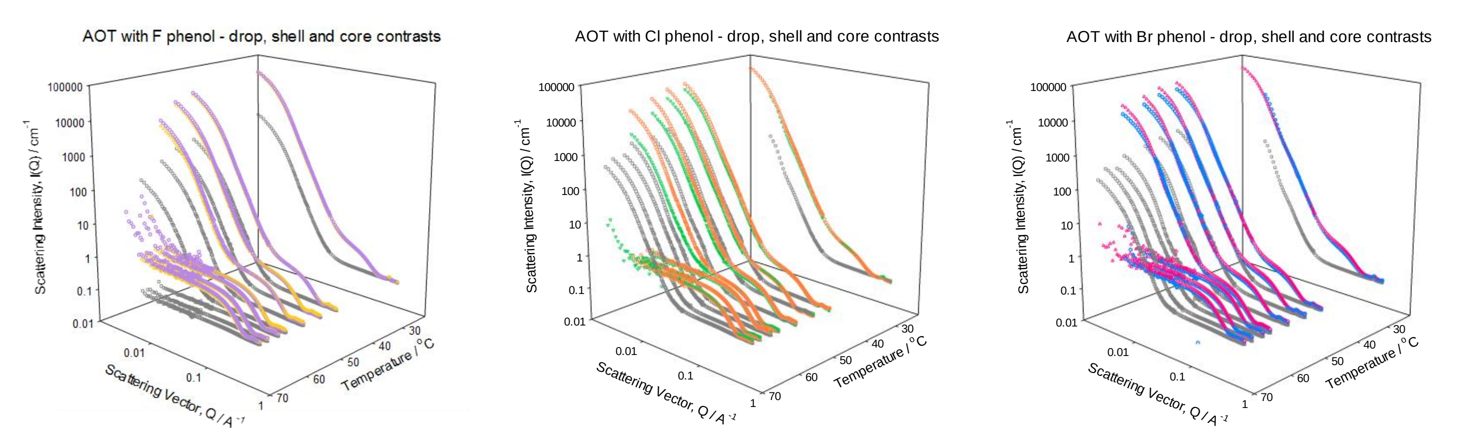

Figure 2: SANS profiles of Na(AOT) based organogels at high and low temperatures.

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