Experimental report

08/01/2019

Proposal: **9-10-1558** **Council:** 4/2018

Title: Emulsification using Fire-fightingsurfactants

Research area: Chemistry

This proposal is a new proposal

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Experimental team: Christopher HILL

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Samples: Fluorocarbon Surfactants

Instrument	Requested days	Allocated days	From	To
D33	3	2	18/09/2018	20/09/2018
D22	3	0		
D11	3	0		

Abstract:

The aim is to understand how the addition of a common hydrogenated/deuterated oil affects the size of the self-assembled structure of three common industrial fluorotelomer surfactants used in fire-fighting foam formulations (Figure 1) using contrast variation SANS. These surfactants have already been studied individually and now will be studied in the presence of different concentrations of oil (cyclohexane), over both a range of surfactant concentrations which are commensurate with commercial fire-fighting foam formulations. Fire-fighting foams are generally employed on liquid fuel fires, therefore the degree of emulsification is of great interest to understand (as this can have adverse effects of foam stability (Figure 1)) and this experiments represents a model system close to practical application conditions in which we can study this effect. This is the fourth stage in a 3-year program, including SANS, so that F-carbon surfactants can be replaced by more environmentally-responsive low-F or hydrocarbon analogues. Chris Hill is a 3rd year PhD student fully funded by fire-fighting technology company Angus Fire (French subsidiary company, Eau et Feu).

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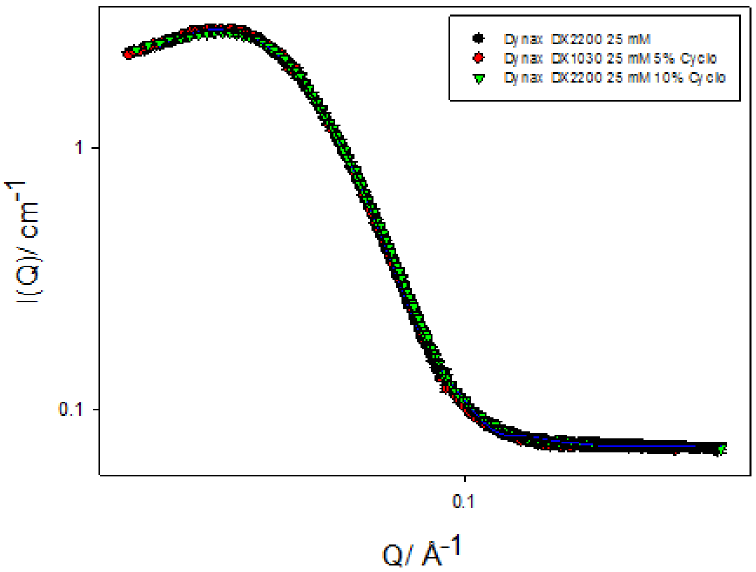
Instrument: D33
Dates of experiment: 18/09/2018 to 20/09/2018

Fire-fighting foam formulations are complex, multicomponent systems that contain mixtures of fluorocarbon (FC) and hydrocarbon (HC) surfactants. An example of a commonly used formulation is an Aqueous film-forming foams (AFFFs) which are employed on liquid fuel fires. Although the fires will be on hydrocarbon fuels, it is of interest to know how the FC surfactants interact with a fuel in terms of self-assembled structures both individually and as mixed FC/ HC systems in mimics of a real fire-fighting formulation.

The first part of the experiment was based on characterising the effect that adding a model oil (cyclohexane) had on the self-assembled structure of three commonly used fluorinated fire-fighting surfactants. The three FC surfactants (1 anionic, 1 non-ionic and 1 zwitterionic) were are mixed with cyclohexane (5 and 10 % v/v) at surfactant concentrations in D2O. Results for the non-ionic FC surfactant are shown below in Figure 1.

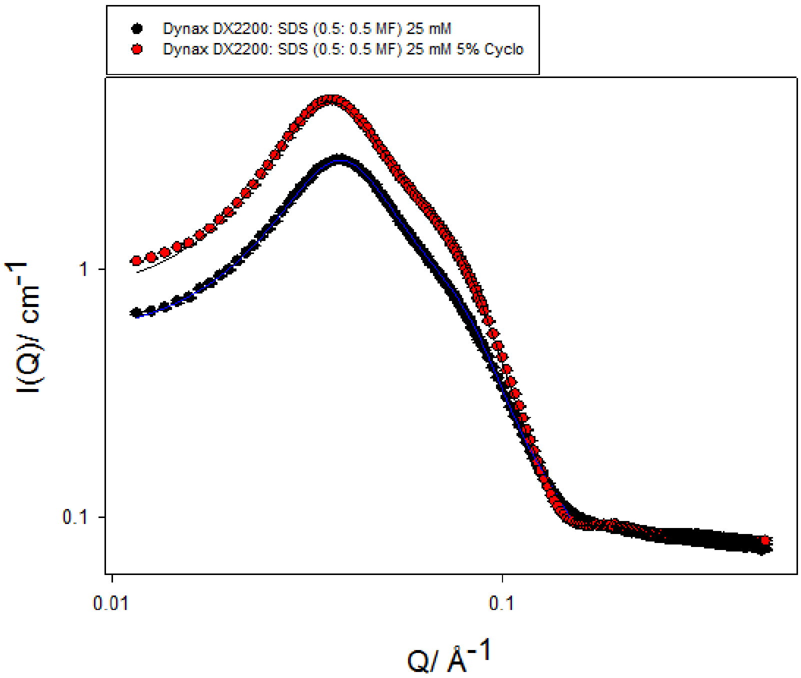


Sample	Fit	Background	Radius (Equatorial)/ (Å)	Radius (Polar)/ (Å)
DX2200 12.5 mM	Ellipsoid	0.061	66.5	30.1
DX2200 12.5 mM 5% Cyclohexane	Ellipsoid	0.061	66.9	29.2
DX2200 12.5 mM 10% Cyclohexane	Ellipsoid	0.062	66.4	29.5

Figure 1: Non-ionic Dynax DX2200 with and without D-Cyclohexane in a D2O medium. Surfactant concentration is 25 mM. Black lines are fitted models to the data. The table shows some of the parameters used to fit the data.

From the data presented in Figure 1, it is clear to see that there are no changes are observed in the self-assembled structure of the non-ionic FC surfactant on addition of the cyclohexane at both 5 and 10% v/v. This same behaviour was observed for all three of the studied FC surfactants. Although this was result is as expected, due to the lack of miscibility between FC and HC materials, it is important to have this information as a foundation for the second part of the experiment.

As previously mentioned, fire-fighting foam formulations are multicomponent systems that contain both FC and HC surfactants. The second part of the experiment was therefore concerned with characterising the effect that the addition of cyclohexane had on the self-assembled structures of FC/ HC surfactant mixtures. The HC surfactant used was SDS. The three FC were all mixed with h-SDS at three different mole fractions (0.9:0.1, 0.5:0.5, 0.1:0.9) in D2O. The surfactant/ cyclohexane concentrations were all kept the same as in the previous experiment for consistency. Initial results from the non-ionic FC/ HC mixed systems (0.5:0.5 mole fraction) with 5% Cyclohexane are shown below in Figure 2:



Sample	Fit	Background	Radius (Equatorial)/ (Å)	Radius (Polar)/ (Å)
0.5:0.5 MF No Cyclo	Ellipsoid	0.036	42.5	16.2
0.5:0.5 MF 5% Cyclo	Ellipsoid	0.053	41.1	21.2

Figure 2: Non-ionic Dynax DX2200/ h-SDS mixed system with and without D-Cyclohexane in a D2O medium. Surfactant concentration is 25 mM. Black lines are fitted models to the data. The table shows some of the parameters used to fit the data.

From initial observation of the above results, it is clear to see that there are some changes occurring on addition of the cyclohexane to the FC/ HC mixed system. The intensity of the scattering has increased and the profile has shifted to lower Q, suggesting swelling. This is what was determined from the fitted parameters as well. Analysis is still being carried out on the remainder of the data, which will make up a substantial chapter in my thesis and we hope to also write up this work for publication in 2019.