# Experimental report

01/12/2023

**Proposal:** **9-12-685** **Council:** 10/2022

**Title:** Solution structures of Oleo-Furan surfactant mixtures in the presenceof mono- and di-valent electrolytes

**Research area:** Soft condensed matter

**This proposal is a new proposal**

**Main proposer:** **Karen EDLER**

**Experimental team:** Karen EDLER
Laura DEEMING
Sylvain PREVOST

**Local contacts:** Olga MATSARSKAIA
Sylvain PREVOST

**Samples:** STFS/d-C12E6 in D2O with NaCl or CaCl2
STFS/C12E6 in D2O with NaCl or CaCl2
STFS/C12E6 in 75% D2O with NaCl or CaCl2

| Instrument | Requested days | Allocated days | From | To |
|---|---|---|---|---|
| D22 | 3 | 0 | | |
| D33 | 3 | 1 | 11/09/2023 | 12/09/2023 |

## Abstract:

In the detergent industry, anionic surfactants such as Sodium Dodecyl Sulfate (SDS) and Linear Alkylbenzene Sulfonate (LAS) dominate the market. Whilst LAS has been proven to be biodegradable, it is derived from petrochemicals using acid catalysts that cause low selectivity, corrosion of equipment and toxicity. This has led to a move towards investigation of biobased and renewable surfactants, such as the new class of anionic surfactants, oleo-furan sulfonates. However, the physico chemical properties of these surfactants have not yet been studied. This proposal is focused on characterizing a C14-tail oleo-furan sulfonate anionic surfactant that has been shown to have a high cleaning performance and high tolerance for hard water ions when alone in solution. Typical formulations however involve mixtures of surfactants; frequently anionics are mixed with non-ionics. In this experiment therefore we aim to investigate how the presence of mono- and di-valent electrolytes affects the morphology and composition of mixed micelles formed from this surfactant in dilute solutions with a model non-ionic surfactant.

**Experiment Report:**

Small angle neutron scattering data was collected to investigate the micellar composition of a three-component surfactant solution. The first two components were a standard anionic surfactant, Sodium Dodecyl Sulfate (SDS), and a standard non ionic surfactant, Hexaethylene glycol monododecyl ether ($C_{12}E_6$). The main component of interest was a linear anionic surfactant with a furan ring-based head group (Sodium-5-tetradecyl furan-2-sulfonate, STFS). Previous SANS studies have been undertaken at ISIS Neutron Source UK (RB 2220085) to characterise these compositions and mixtures in dilute solutions, exploring the role of how an aromatic furan ring structure in the head group may affect the packing and aggregation properties of a micellar solution. This experiment aimed to explore the effect of electrolyte addition of these systems, in particular the difference between mono- and di-valent salt addition on the bulk micellar properties.

Both the binary (STFS with SDS or $C_{12}E_6$) and ternary (STFS/SDS/$C_{12}E_6$) systems were run to understand whether one of the components would have a dominating effect on the micellar structure and composition when in the presence of electrolyte. Selective alkyl chain deuteration of SDS and $C_{12}E_6$ allowed for multiple contrasts of the same systems to be run, with STFS remaining as the h-surfactant. All experiments were run in 100% $D_2O$ so the contrasts arose only from the deuteration of the SDS and $C_{12}E_6$. The binary systems included three molar ratios (1:2, 1:1 and 2:1) and the ternary systems included seven molar ratios (1:1:1, 1:2:1, 1:1:2, 2:1:1, 1:2:2, 2:1:2 and 2:2:1). The samples were run in 1 mm Quartz Hellma cells on a standard SANS sample changer with a water bath used to regulate the temperature of the samples during the experiment. The samples were run on D33 with a rear detector distance of 13.3m and a front detector distance of 1.7m to give an overall q-range of 0.004-0.45Å$^{-1}$.

The proposed experiment had been to run all of these conditions at 10 mM surfactant concentration and 10 mM salt addition at 25°C, however after initial data showed no difference between mixtures run at both 25°C and 45°C for the ternary systems, and due to the limited beamtime awarded, all samples were run at 45°C for direct comparison with previously collected data. Previous results had shown STFS/SDS solutions precipitated at room temperature so using 45°C also ensured solubility. The table below lays out which sets were run during the experiment with a "set" including all molar ratios and all contrasts.

| | **Room Temperature (26 °C)** | **High Temperature (45 °C)** |
|---|---|---|
| **10 mM Binary STFS/$C_{12}E_6$ + 10 mM $CaCl_2$** | × | ✓ |
| **10 mM Binary STFS/$C_{12}E_6$ + 10 mM NaCl** | × | ✓ |
| **10 mM Binary STFS/SDS + 10 mM NaCl** | × | ✓ |
| **10 mM Ternary STFS/SDS/$C_{12}E_6$ + 10 mM NaCl** | ✓ | ✓ |
| **10 mM STFS + 10 mM $CaCl_2$ or NaCl** | × | ✓ |

The data will be fitted using the NIST SANS Analysis macros in Igor Pro, using the multiple contrasts obtained and several models will be trialled to describe micellar structures and interactions. Initial plots of the data (Figure 1a) show a trend of the monovalent salt (10 mM NaCl) screening the interactions between the micelles, flattening the structure factor peak and the divalent salt (10 mM $CaCl_2$) inducing larger elongated aggregates. The same trend occurs for the ternary system (Figure 1b) with NaCl appearing to screen the micellar interactions, as would be expected of a monovalent electrolyte presence. Parameters such as micelle size, composition and aggregation number of the micelles will be extracted for comparison to the same solutions but without the electrolyte presence.

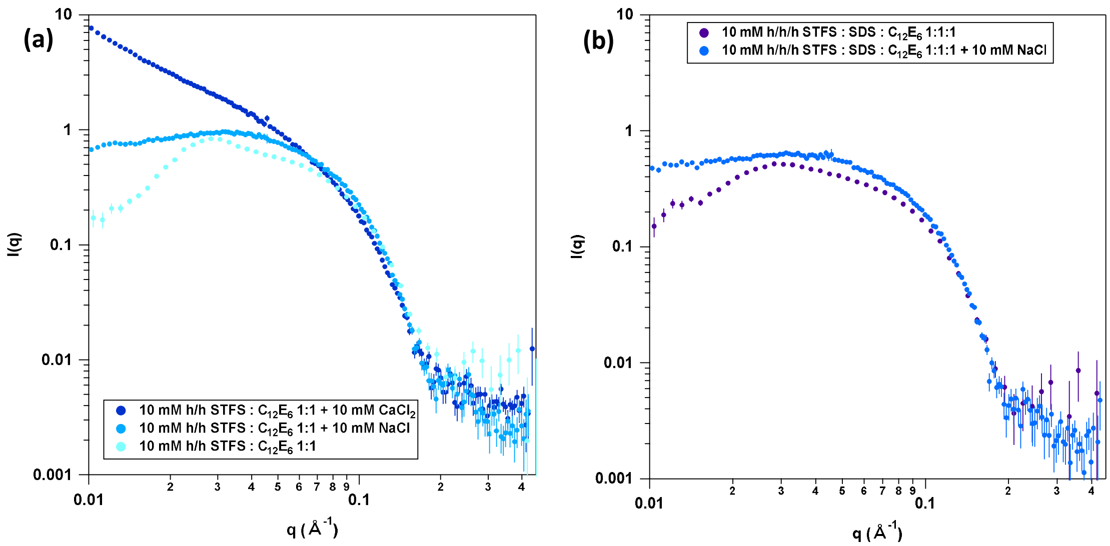


***Figure 1 – Initial graphs of SANS data for 10 mM Binary (a) and Ternary (b) mixtures in the absence and presence of added electrolyte***

This data will be incorporated into a paper on the mixtures of linear STFS with SDS and C12E6 which will become part of the PhD thesis for Laura Deeming.