# Experimental report

01/03/2021

**Proposal:** INTER-271 **Council:** 10/2016

**Title:** Internal time on FIGARO

**Research area:**

**This proposal is a new proposal**

**Main proposer:** Richard CAMPBELL

**Experimental team:** Angel PINEIRO

**Local contacts:** Erik WATKINS

**Samples:** Sodium Dodecyl Sulfate
Alpha-cyclodextrin

| Instrument | Requested days | Allocated days | From | To |
|---|---|---|---|---|
| FIGARO | 1 | 1 | 03/06/2013 | 04/06/2013 |

**Abstract:**

# EXPERIMENTAL REPORT FOR INTER-271

03/06/2013 to 04/06/2013

R. A. Campbell & Á. Piñeiro

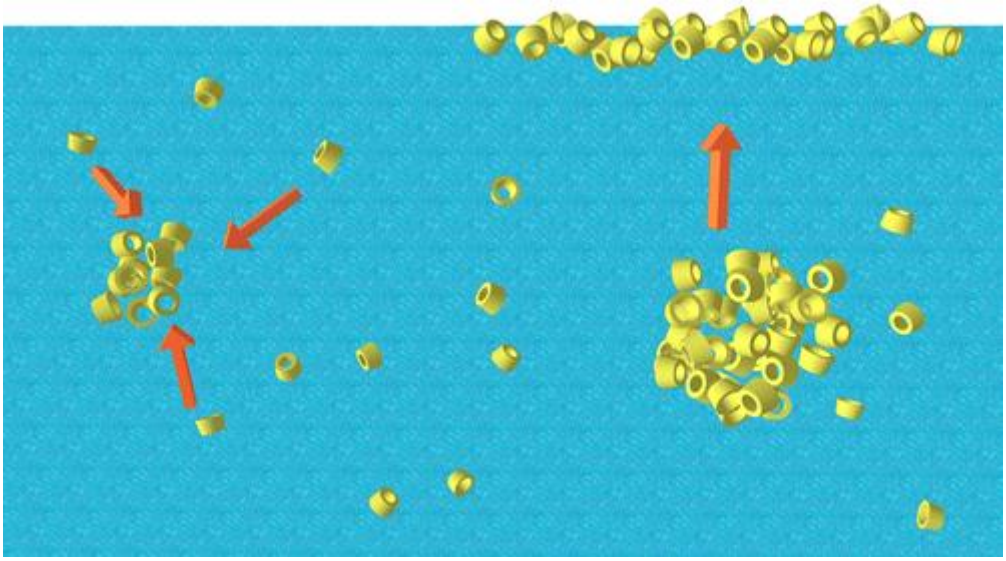
These data on the adsorption of cyclodextrin/surfactant mixtures, and pure cyclodextin, at the air/water interface contributed to two publications:

Hernandez-Pascacio, J.; Piñeiro, Á.; Ruso, J. M.; Hassan, N.; Campbell, R. A.; Campos-Terán, J.; Costas, M. "Complex Behavior of Aqueous alpha-Cyclodextrin Solutions. Interfacial Morphologies Resulting from Bulk Aggregation". *Langmuir* **2016**, *32*, 6682–6690.

http://dx.doi.org/10.1021/acs.langmuir.6b01646

*The spontaneous aggregation of alpha-cydodextrin (alpha-CD) molecules in the bulk aqueous solution and the interactions of the resulting aggregates at the liquid/air interface have been studied at 283 K using a battery of techniques: transmission electron microscopy, dynamic light scattering, dynamic surface tensiometry, Brewster angle microscopy, neutron reflectometry, and ellipsometry. We show that alpha-CD molecules spontaneously form aggregates in the bulk that grow in size with time. These aggregates adsorb to the liquid/air interface with their size in the bulk determining the adsorption rate. The material that reaches the interface coalesces laterally to form two-dimensional domains on the micrometer scale with a layer thickness on the nanometer scale. These processes are affected by the ages of both the bulk and the interface. The interfacial layer formed is not in fast dynamic equilibrium with the subphase as the resulting morphology is locked in a kinetically trapped state. These results reveal a surprising complexity of the parallel physical processes taking place in the bulk and at the interface of what might have seemed initially like a simple system.*

Luviano, A. S.; Hernandez-Pascacio, J.; Ondo, D.; Campbell, R. A.; Piñeiro, Á.; Campos-Terán, J.; Costas, M. “Highly Viscoelastic Films at the Water/Air Interface: Alpha-Cyclodextrin with Anionic Surfactants”. *J. Colloid Interface Sci.* **2020**, *565*, 601–613.



http://dx.doi.org/10.1016/j.jcis.2019.12.012

*This work showcases the remarkable viscoelasticity of films consisting of alpha-cyclodextrin (alpha-CD) and anionic surfactants (S) at the water/air interface, the magnitude of which has not been observed in similar systems. The anionic surfactants employed are sodium salts of a homologous series of n-alkylsulfates (n = 8-14) and of dodecylsulfonate. Our hypothesis was that the very high viscoelasticity can be system-atically related to the bulk and interfacial properties of the system. Through resolution of the bulk distribution of species using isothermal titration calorimetry, the high dilatational modulus is related to (alpha-CD)(2):S-1 inclusion complexes in the bulk with respect to both the bulk composition and temperature. Direct interfacial characterization of alpha-CD and sodium dodecylsulfate films at 283.15 K using ellipsometry and neutron reflectometry reveals that the most viscoelastic films consist of a highly ordered monolayer of 2:1 complexes with a minimum amount of any other component. The orientation of the complexes in the films and their driving force for adsorption are discussed in the context of results from molecular dynamics simulations. These findings open up clear potential for the design of new functional materials or molecular sensors based on films with specific mechanical, electrical, thermal, chemical, optical or even magnetic properties.*