# Experimental report

22/11/2018

**Proposal:** **9-11-1868**

**Council:** 4/2018

**Title:** Structure of polymers and 2D polymer networks monolayers at the air-water interface

**Research area:** Soft condensed matter

**This proposal is a new proposal**

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**Local contacts:** Philipp GUTFREUND

**Samples:** polybutadiene
deuterated polybutadiene
Deuterated Water
cellulose acetate
glutaraldehyde

| Instrument | Requested days | Allocated days | From | To |
|---|---|---|---|---|
| FIGARO Langmuir trough | 4 | 3 | 20/06/2018 | 23/06/2018 |

**Abstract:**

The aim of the proposed experiment is to characterize the structure of Langmuir monolayers formed at the air-water interface by two polymers, the cellulose acetate and the 1,2-polybutadiene, using neutron reflectometry. These monolayers will then be in-situ cross-linked by two different pathways and the resulting structural change will be investigated. This study will provide a basis for the elaboration of 2D interpenetrating polymer networks, with a morphology controlled by the cross-linking conditions.

# Standard Project

*Experimental Report*

**Proposal title:** Structure of polymers and 2D polymer networks monolayers at the air-water interface

| Experiment number: | Date(s) of experiment: | Beamline: |
|---|---|---|
| 9-11-1868 | from 19/06/2018 to: 23/06/2018 | FIGARO |

**Local contact(s):** Philipp Gutfreund

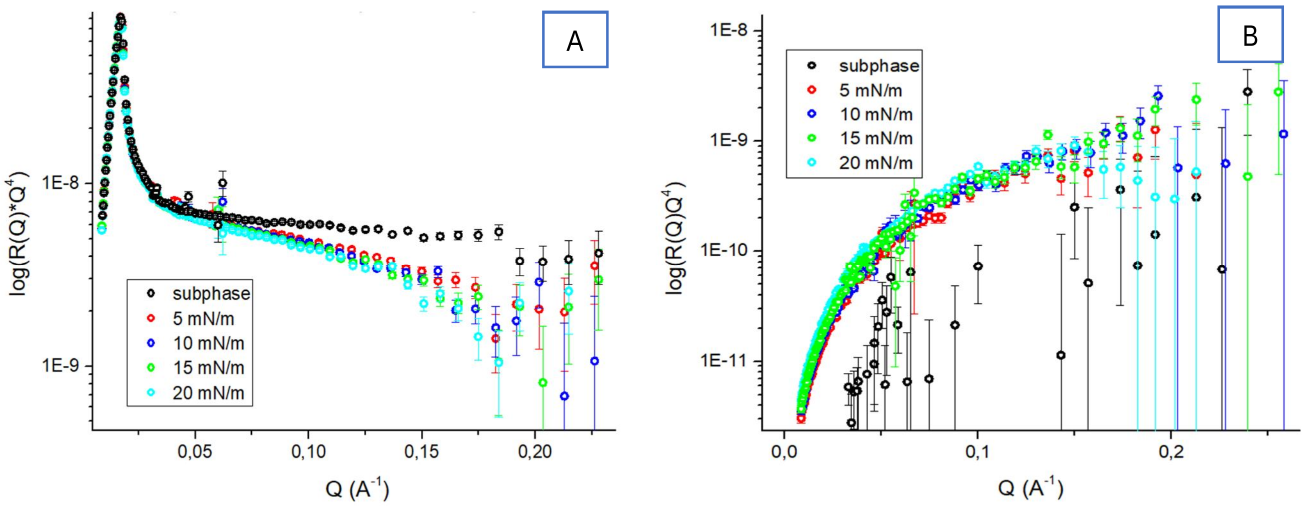
**Date of report:** 19/11/2018

**Objective & expected results**:

The proposed experiments aim to study by neutron reflectivity (NR) the structure of both an uncross-linked and cross-linked cellulose acetate (CA) monolayers at the air-water interface. The CA is cross-linked through an addition reaction in the presence of glutaraldehyde (GA) as cross-linker in the water subphase adjusted to pH 2. By using neutron reflectometry, the structure of monolayers was characterized at different surface pressures under two different $D_2O$ / $H_2O$ ratio to obtain two different contrasts.

**Results and the conclusions of the study**:

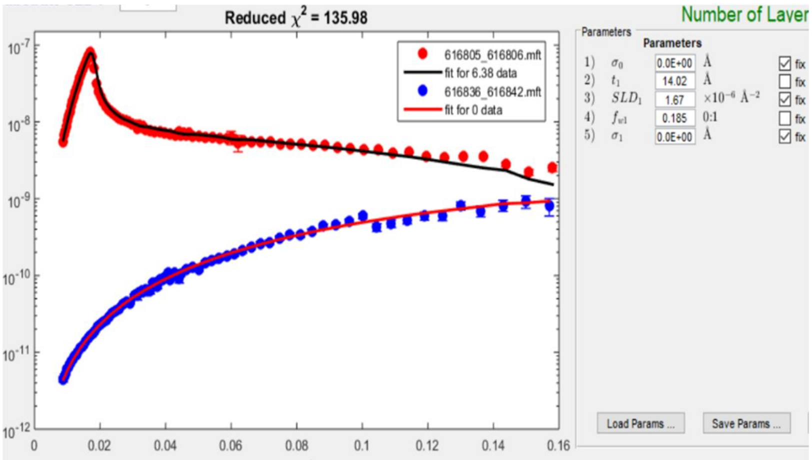
The CA monolayer which SLD is $1.67.10^{-6}$ Å$^{-2}$ has been spread on $D_2O$ subphase and on the Air Contrast Match subphase (8,1%D20 / 91,9%H20) (SLD 0 Å$^{-2}$) to assess precisely the monolayer structure. Those 2 subphases adjusted to pH2 have been tested with and without $10^{-2}$ M of glutaraldehyde (GA). CA monolayers on the acidic subphases have been studied at four surface pressures, 5, 10, 15 and 20 mN/m, corresponding to different monolayer densities, which leads to slight changes of the reflectivity profiles. However, with the equivalent subphase containing the crosslinking agent, only three surface pressures were probed 5, 10, and 20 mN/m.




*$R(Q)Q^4$ representation of the experimental data of neutron reflectivity profiles for CA monolayers spread over the 2 studied subphases. A. D20 subphase pH2. B. Air Contrast Match subphase pH2*

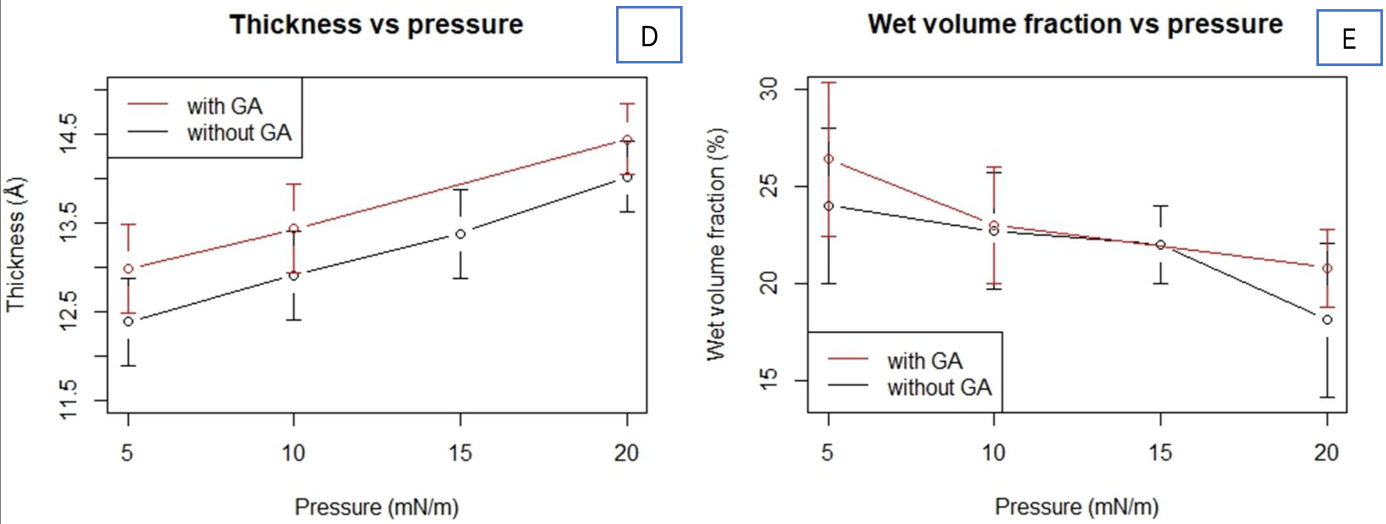
Aurore software has been used to simulate the neutron reflectivity data. The above NR curves (Figures A and B) recorded at different surface pressures for two different subphases were simultaneously and successfully fitted using a single thin layer without any roughness at both interfaces (see example in Figure C). Both thickness and water fraction in the monolayer have thus been determined thanks to the minimization process applied on both subphases at the same time. Both parameters have been estimated with a reliable uncertainty[1] (NR curves have been cropped to discard statistically irrelevant data points).

C




*Experimental (symbols) and simulated (lines) reflectivity profiles for CA monolayer compressed at 20 mN/m on D20 and Air Contrast Match subphase at pH2. Fits were obtained according to the parameters listed on the right side*

Figures D and E show the CA monolayer thickness and the water fraction evolution as function of the surface pressure respectively, with or without GA in the water subphase. The values are reported with the confidence intervals for each surface pressure. As the CA monolayer density increases, the thickness increases and the water fraction in the monolayer decreases. The GA crosslinked monolayer shows the same behaviour except a slightly higher thickness than that of the CA monolayer over pure water.




*Evolution of the thickness (D.) and the wet volume fraction (E.) as function of the surface pressure for a CA monolayer spread over an aqueous subphase with and without GA*

**Justification and comments about the use of beam time**:

The FIGARO beamline is perfectly suitable to determine the vertical structure of very thin polymer Langmuir monolayers and detect the changes due to polymer cross-linking .

**Publication(s)**:


Y. Gerelli**,** Aurore: new software for neutron reflectivity data analysis », *J. Appl. Cryst.* (2016). **49**, 330-339