Experimental report

11/03/2020

Proposal: 9-11-1925 **Council:** 4/2019

Title: Structure of cellulose acetate/polybutadiene mixed films at the air-water interface depending on composition

Research area: Soft condensed matter

This proposal is a continuation of 9-11-1868

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Local contacts: Philipp GUTFREUND

Samples: D2O
Polybutadiene
cellulose acetate
glutaraldehyde

Instrument	Requested days	Allocated days	From	To
FIGARO	4	4	07/10/2019	11/10/2019

Abstract:

The aim of the proposed experiment is to characterize the structure of mixed Langmuir monolayers formed at the air-water interface by two polymers, the cellulose acetate and the 1,2-polybutadiene, using neutron reflectometry. The structure of the mixed films as a function of the surface pressure and composition will be investigated in order to assess the mixing behavior. This study will provide a basis for the elaboration of 2D interpenetrating polymer networks, with a morphology controlled by the cross-linking reactions.

Standard Project

Experimental Report

Proposal title: Structure of cellulose acetate/polybutadiene mixed films at the air-water interface depending on composition		
Experiment number: 9-11-1925	**Date(s) of experiment:** from 7/10/2019 to: 11/10/2020	**Beamline:** FIGARO
Local contact(s): Philipp Gutfreund	**Date of report:** 5/03/2020	

Objective & expected results:

Structure of cellulose acetate/polybutadiene mixed films at the air-water interface depending on composition

The proposed experiment aims to study by neutron reflectometry the structure of cellulose acetate (CA) and deuterated 1,2-polybutadiene (PB) mixed monolayers spread at the air-water interface as Langmuir films, for three volume fractions of CA in the CA/PB mixed films (0.2, 0.5 and 0.7). The structure of mixed monolayers was characterized at three surface pressures under three different D_2O / H_2O mixed subphases, allowing alternately the extinction of CA, PB, and the mixed film if it is an ideal mixture. CA and PB pure monolayers have been studied in a previous experiment.

Results and conclusions of the study:

As example, Figure 1 presents the neutron reflectivity curves in $R(Q)Q^4$representation for a CA/PB monolayer at CA volume fraction 0.5 spread over the three studied subphases, as a function of the surface pressure.

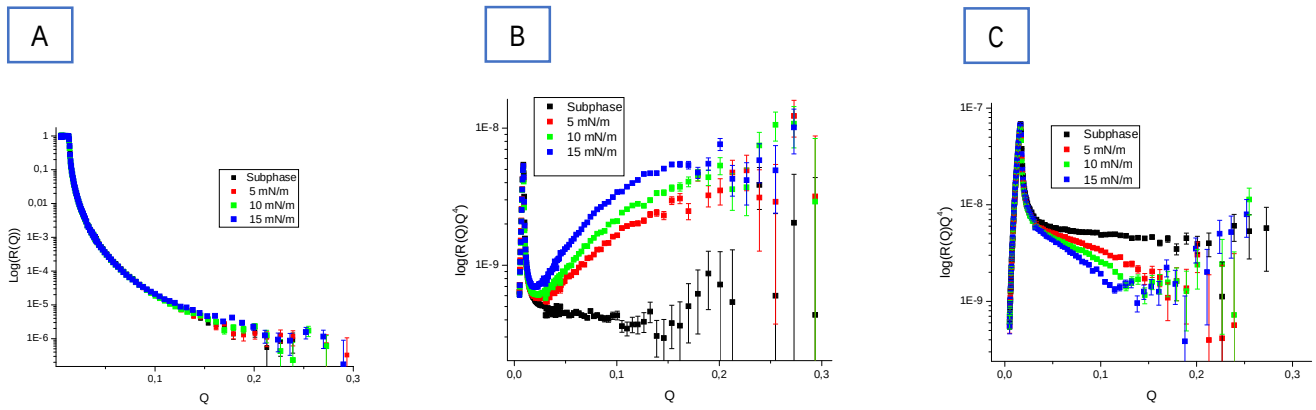

Figure 1: $R(Q)Q^4$ representation of the neutron reflectivity experimental data for CA/PB monolayer at CA volume fraction 0.5 at 3 surface pressures (5, 10 and 15 mN/m) spread over the 3 studied subphases. A. Homogeneous film-contrast matched. B. CA-contrast matched C. PB-contrast matched

Aurore software has been used to simulate the neutron reflectivity (NR) data. At 5 mN/m and CA volume fractions of 0.2 and 0.5, the mixed monolayer is vertically homogeneous and can be described by a single slightly hydrated thin layer (about 13Å-thick). In contrast, at the higher CA volume fraction (0.7), even at 5 mN/m, the NR curves cannot be fitted with a single layer. The used bilayer model indicates that the more hydrophilic CA polymer mainly occupies a thin layer at the surface of water while the more hydrophobic PB diffuse towards the air. Indeed, the SLD profile as a function of depth presented in Figure 2 evidences interdiffused layers with enrichment of PB at the interface with the air and of CA in contact with the water. By increasing the surface pressure to 10mN/m, only the lower CA volume fraction (0.2) leads to a single homogeneous layer. For the higher CA fractions, interdiffused layers with predominantly PB in the upper part of the layer and CA in the bottom part are observed. Finally at 15 mN/m, the same interdiffused structure is evidenced for the three studied volume fractions, with an increased enrichment of PB in the upper part of the layer with decreasing CA volume fraction.

These results are in good agreement with thermodynamical measurements, Brewster Angle Microscopy observations

and water advancing contact angle values obtained on monolayers transferred by the inverse Langmuir-Schafer method on Si/SiO2 substrates.

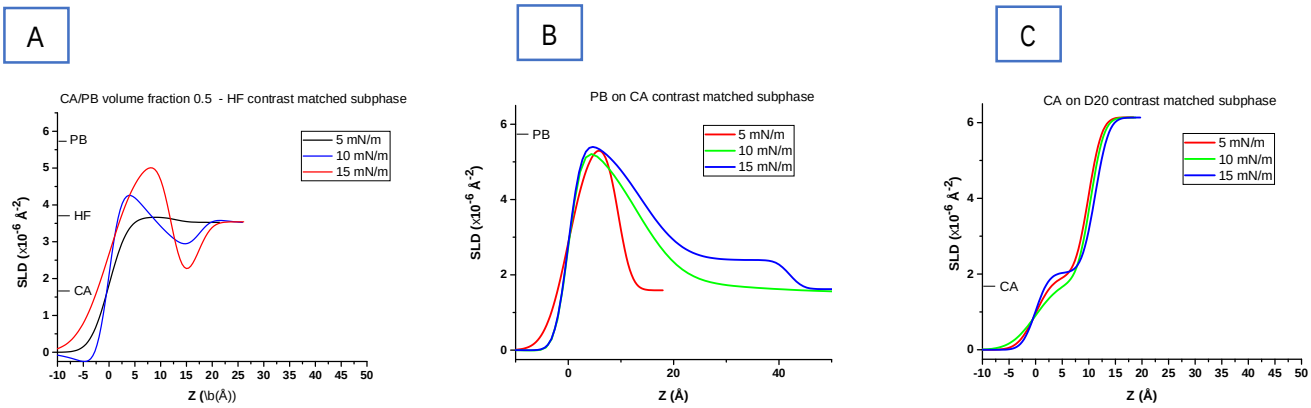

Figure 2: SLD profile as a function of depth for CA/PB monolayer at CA volume fraction 0.5 spread over a homogeneous film-contrast matched subphase (A) compared with the profiles obtained for pure monolayers : PB over CA-contrast matched subphase (B) ; CA over D2O (C)

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.

Publication(s):

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