	Experiment title: <b>Post-monomolecular behaviour of pure and mixed Langmuir monolayers of semi-fluorinated alkane.</b>	<b>Experiment number:</b> SC2343
<b>Beamline:</b> ID10B	<b>Date of experiment:</b> from: 20-02-2008 to: 26-02-2008	<b>Date of report:</b> 03-09-2008  <i>Received at ESRF:</i>
<b>Shifts:</b> 18	<b>Local contact(s):</b> O. Konovalov	
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### Report :

Due to their unique properties (hydrophobic and lipophobic character, chemical and biological inertial) semi-fluorinated alkanes,  $C_nF_{2n+1}C_mH_{2m+1}$  (FnHm diblocks), appear as promising agent for medicinal use (i.e. ultrasonic imaging or synthetic lung surfactant<sup>[1]</sup>). It is then crucial to understand their self-assembling properties when they interact with water or phospholipid molecules. We previously demonstrated that such molecules display a vertical phase separation from other surfactant at the air/water interface<sup>[2]</sup>. Indeed, despite the lack of polar head group, semi-fluorinated alkanes are able to form stable Langmuir monolayers alone at the air / water interface<sup>[3]</sup>. However their structure was controversial. Using Grazing Incidence Small Angle X-ray Diffraction (GISAXD) on F8H16 monolayer at 3 mN/m surface pressure, we demonstrated the presence of a 30 nm diameter domains organized on an hexagonal 2D lattice<sup>[4]</sup>.

To understand the interaction at the origin of the auto organization of semi fluorinated alkane at the air/water interface, we performed Grazing Incidence x-ray scattering on Langmuir monolayers of F8Hm on the ID10B beamline. We used a monochromatized x-ray beam of energy 8keV (0.154nm). The beam was deflected downwards to impinge on the water surface with an incident angle of 1.98mrad. The beam sizes were fixed at 300μm horizontally and 100μm vertically. Scattered photons was detected by a vertical PSD (150mm height) located at 835mm from the center of the goniometer (COG). As collimator, we used two vertical slits of horizontal gap of 300μm and 500μm and located respectively at 260mm and 894mm from the COG. The sample was deposited in a home built gas tight Langmuir trough adapted to the goniometer and flushed by Helium gas.

The first part of the experiment was to explore the influence of the hydrogenated chain length on this structure<sup>[5]</sup> in order to understand interactions which lead to this huge difference on the lattice parameter. Figure 1 presents the GISAXS spectra of F8H14, F8H16, F8H18 and F8H20 deposited at the air/water interface. After indexation, the lattice parameter has been determined. It strongly changes with the length of the hydrogenated chain: a variation of 15% on the length molecule leads to a variation of 45% on the lattice parameter. This variation is still controversial and cannot be described only with Van der Waals interactions.

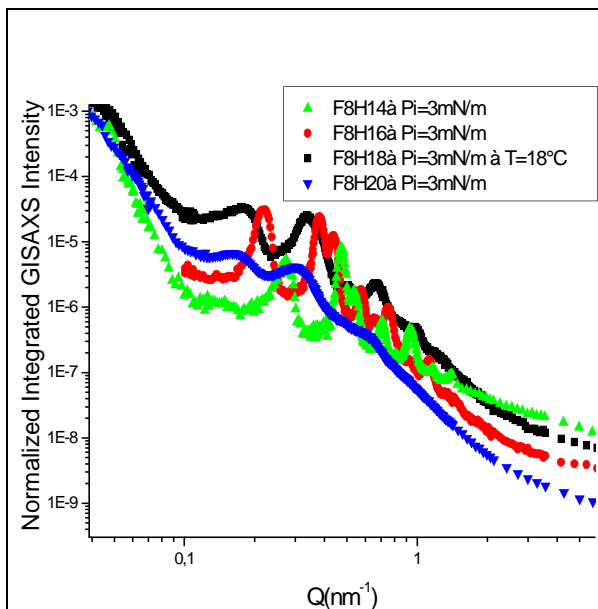


Figure 1 : GISAXS spectra measured on a monolayer of F8H14, F8H16, F8H18, F8H20 diblocks deposited at the air - water interface at surface pressure  $\pi = 3 \text{ mN.m}^{-1}$  and  $T = 18^\circ$ .

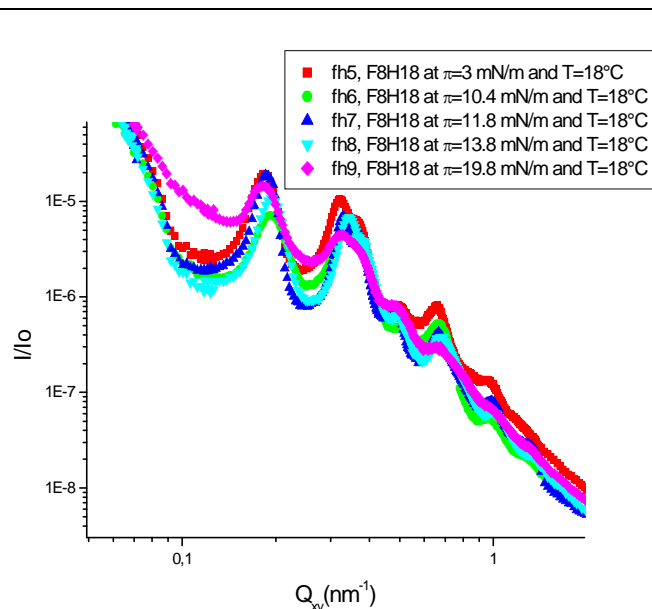


Figure 2: GISAXS spectra measured on a monolayer of F8H18, diblock at the air water interface at different surface pressures for  $T = 18^\circ\text{C}$ . (collapse appears at  $13 \text{ mN/m}$ )

The second part of this experiment was to study the transition to the third dimension, named the “collapse”, of pure FnHm molecules in Langmuir monolayers using GISAXS measurement directly on the water surface. Whereas a large self-assembled hexagonal structure of FnHm monolayer is observed at the air/water interface, it is not obvious that this organization is preserved when the film collapses in a multi-layers phase. Figure 2 presents GISAXS spectra of F8H18 diblock at the air / water interface for different pressures. One observes diffractions peaks on each GISAXS spectra indicating the presence of large domains of self-assembled FnHm molecules whatever the surface pressure. These first results indicate that these molecules are auto organized when the film is collapsing (i.e. multi layers phase). Up to our knowledge, this is the first time that organized micelles are evidenced during the “collapse”.

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[5] Experimental report SC 20 97