Experimental Report template

Proposal title: In-situ measurement of the pressure develloped during ice templating using lipid bilayers as pressure probesglycolipids		**Proposal number:** SC-4479
Beamline: ID02	**Date(s) of experiment:** from: 14/06/2017 to: 16/06/2017	**Date of report:** 04/07/2017
Shifts: 6	**Local contact(s):** Thomas Zinn	*Date of submission: 04/07/2017*

Objective & expected results:

Freeze casting is a materials processing technique based on the segregation of solutes from ice during unidirectional freezing. Despite its simplicity, freeze casting has found extensive use in the development of a wide range of materials with precisely controlled macroporous morphologies. Some of the most recent uses of the technique have allowed to precisely shape materials as diverse as ceramic-based catalysts, biomimetic composite materials or encapsulation matrices capable of hosting living cells. However, important questions remain unanswered that may hold the key to a more widespread application of the technology. Among these, the pressure generated during the ice solidification on the segregated walls is critical to ascertain the technique's adaptability to the domain of biological encapsulation. To tackle this question we have used in-situ SAXS during the freeze-casting process of a swollen lamellar, as the evolution of the inter-lamellar distance can be used as an internal standard to obtain the pressure under which the lamellae undergo. In the meantime, we have used an in-situ pressure cell to apply a known pressure on the same swollen lamellar phase and correlate the interlamellar distance evolution with the data obtained by freeze casting. In the end, using the interlamellar distance as a standard, we should be able to precisely determine the pressure imposed by water during the freeze casting process. The expected results will be of major relevance to the scientific community working on the technique as they will provide an additional rational element to determine the applicability of the technique for pressure sensitive materials.

Results and conclusion

The experiment was divided into two separate parts. In the first one we have used the pressure cell available at the beamline. We have prepared a swollen lamellar lipid phase which was inserted in the capillary. A pressure between 0 bar and 1500 bar was applied to the system. The corresponding SAXS patterns were recorded for various pressure values. An evolution of the lamellar first order diffraction peak is observed with increasing pressure.

In the second part of the experiment, the pressure cell is replaced with a home-made freeze-casting device using a 2 mm flat (home-made) Kapton cell. Temperature is controlled between +20°C and -60°C using various freezing rates. The SAXS patterns are recorded every 5°C in the entire temperature range at different heights in the cell. Raw tretment of the data shows a marked effect of the temperature on the peak position both before and after the freezing point of water. Future work will try to correlate the evolution of the peak position in the pressure cell and in the freeze casting device.

Data treatment

The experiments have been done at 17.0 KeV and two distances were used: 2m and 8m. Calibration was fully done by the local contact to have normalized data in intensity and in q-values, given in nm^{-1}. Water was used normalize the data in intensity and data should be divided by the cell width (2 mm) to obtain I(q) in mm^{-1}. The data provided at the end of the experiment are not divided by the cell width. Masking was done to hide imperfections and beam stopper. All 2D

data were integrated at the beamline during the acquisition process and provided both in the *.edf and *.dat formats for further treatment. We did not observe beam damage on none of the samples. Typical acquisition times were in the order of 100 ms. 3 dark current spectra were acquired per sample. Generally, 5 spectra (in some cases 10) are recorded for each sample, exception made for the freeze casting dynamic experiments, for which only 1 spectrum-per-T was recorded.

Justification and comments about the use of beam time:

The use of the beamline was well-adapted to the case study. First of all, we needed the pressure cell developed at ID02 and the beamline scientists were highly cooperative to test it and set it up before our arrival. We started the measurements at about 4 pm and lasted the whole night.
For the freeze casting experiments, we needed fast acquisition times to follow the process and ID02 was well adapted for this task and allowed us to acquire about 100 spectra-per-minute on the fastest system. All spectra were of good quality.

Problems during beamtime:

We had no problem on the beamline and both the local contact and the entire staff was there to prepare the setups, make the beamline performing in the short time allowed for this experiment, and give us both technical and scientific advices to improve our data acquisition process and interpret them.
The only negative note concerned the 6 shifts that the committee allowed us. We asked for 9 shifts because we needed to change two setups and 6 shifts were very short in time. Despite the success of the experiment, we could not perform a wider exploration of the freeze casting conditions. The beamline staff agreed with us that 6 shifts were too short for this experiment and suggested us to propose a new experiment to acquire more data.