MX NON-BAG BioSAXS

Experimental Report template

Proposal title: pH-resolved in-situ study of biosurfactant-biopolymer colloidal solutions		**Proposal number:** MX 2311
Beamline: BM29 - BioSAXS	**Date(s) of experiment:** from: 27/01/2021 to: 29/01/2021	**Date of report:** 08/03/2021
Shifts: 6	**Local contact(s):** Petra Pernot	*Date of submission:* **09/03/2021**

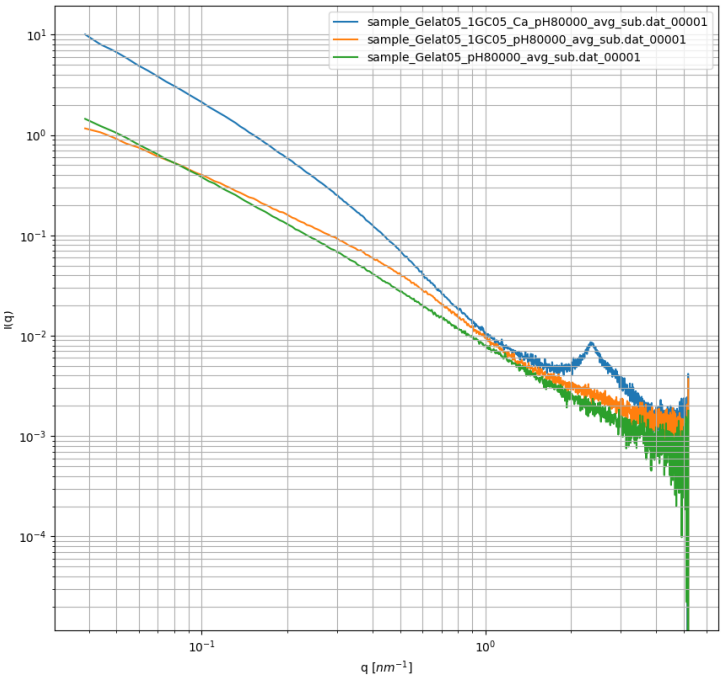
Objective & expected results:

Biosurfactants (BS) are compounds obtained from the fermentation of glucose and vegetable oil. These molecules, known since the 60's, are developed for their biodegradability and poor cytotoxicity, making them valuable alternatives to petrochemical surfactants. We have studied, in the past ten years, the solution self-assembly of BS in water. In more recent studies, we have started to investigate other properties, important for the development of soft materials, namely hydrogelation and complex coacervation. In the perspective of developing formulated consumer products using BS, we extend our approach to the study of interactions between BS and biopolymers, the latter being important macromolecules in the cosmetic and food industry. In the present run, we have combined both the study of new biosurfactants in solution and the interactions between selected BS and biopolymers, in order to establish the grounds for the future use of this class of low molecular weight compounds in biodegradable formulations.

Results and conclusion

The experiments have been done at 12.5 KeV, with a sample-to-detector distance of 2.83 m and AuBenh was used to calibrate the q-range. Water was used as solvent as well as calibrator of the intensity (0.016 cm^{-1}). We used the standard sample automatic environment available at BM29 and described in detail here:
http://www.esrf.eu/home/UsersAndScience/Experiments/MX/About_our_beamlines/bm29/beamline-setup/sample-changer.html
We mostly used 96 well-plates and 100 μL of each sample. For more viscous samples, we have used a direct injection using a 1 mL syringe and the Single Collect function of the software. The syringe is connected to a tube before the capillary.
We studied a large number of systems: 1) a set of 43 enginered sophorolipid and glucolipid derivatives, each at concentration from 1.25 mg/mL to 200 mg/mL, when possible. pH is not varied; 2) a set of samples containing glucolipid GC18:1-cis and various ions (Ca, Ag, Cu, Ba, Sr, Mg, Fe, Na). We varied concentration and pH (8 or 10); 3) a set of samples containing gelatine and fibers of GC18:1-cis.

Data acquisition occurred without problem, and we are at the moment analyzing the results. However, considering the large amount of data, we cannot actually forecast the ultimate outcome. We show below a typical set of SAXS profiles recorded on the glucolipid-gelatine mixture. The grren profile refers to gelatine alone, the organe profile refers to gelatine and glucolipid at pH 8, while the blue profile refers to gelatine and glucolipid in the presence of calcium, the latter providing a phase transition from micellar to fibrillar and enhancing the elastic properties of the solution. These results are under current study.



Data treatment

All samples have been treated at the beamline soon after acquisition. The CCD images have been integrated and correction for absolute calibration was done. Background (capillary + water) has been regularly measured and subtracted from the data. Ten measurements per sample have been recorded and averaged and background was collected generally every each three or five samples. Files were analyzed one by one to eliminate corrupted data (e.g., empty capillary) and then averaged. The acquisition time per sample was set to 1 s. Few cases of beam damage were observed and when that occurred, data were collected again manually at different parts of the injected solution. In summary, this beamline is perfect for the analysis of colloids in solution and revealed quite useful also for viscous solutions.

Justification and comments about the use of beam time:

The use of the beamline was necessary becasue of the high load of samples we had to analyze. Although the beamline is optimized for proteins, our solution were perfectrly adapted to the beamline characteristics, in particular its q-range. The automatic sample changer, the small volumes and software were crucial to record a large amount data in two days. The standard setup of the beamline make users save a lot of time which often derive from changing from one sample environment to another according to the previous user. The local stuff was also very helpful. The independent procedure to apply for beamtime is also very helpful.

Problems during beamtime:

We did not experience any trouble during the beamtime. Nevertheless, despite the big advantage of the automatic sample changer, one still needs to control all data simultaneously as various issues problems (e.g., viscous solutions, clogging, bubbles, poor self-cleaning procedure) can always occur.