Standard Project

Experimental Report template

Proposal title: Structure-Property Study in Biosurfactants Hydrogels using Rheo-SAXS		**Proposal number: SC-4976**
Beamline: ID02	**Date(s) of experiment:** from: 05/03/2021 to: 07/03/2021	**Date of report:** 15/03/2021
Shifts: 6	**Local contact(s):** Thomas Zinn	*Date of submission:* **15/03/2021**

Objective & expected results:
The goal of this proposal was to study the structural and elastic properties of a biosurfactant-based hydrogels according to physico-chemical conditions. From prior rheology experiments, we can show that the elastic properties of the hydrogel have a temperature dependence drastically different according to the chemical condition of the bulk (1 or 2). This run was conceived in order to prove that the similar structural behaviour of the biosurfactant at room temperature could reach different structure at high temperature according initial chemical environment.

Results and the conclusions of the study (main part):

The Thermo Haake rheometer provided at the beamline is coupled to the SAXS beamline. The sample is contained in a polycarbonate Couette cell. The signal of the cell containing mQ-grade water are recorded for background subtraction and the sample thickness traversed by the beam is used to the calculation of the absolute intensity. The beam center is measured X= 917.413; Y= 995.773. The temperature is set at 25°C, unless otherwise stated. The energy of the beam is set at 12.28 KeV and a sample-to-detector distance of 1.500 m. The signal of the Eiger 4M (Dectris) detector, used to record the data, is integrated azimuthally at the beamline using the software Foxtrot and in order to obtain the I(q) vs. q spectrum after masking systematically wrong pixels and the beam stop shadow. The rheology and SAXS experiments are launched at the same time, with a possible ± 5 s difference due to the manual nature of the operation and the launching of rheologic experiment.

We analyzed a series of samples containing different concentration of a microbial biosurfactant in different aqueous environment at various pH values. Typical rheology experiments involving G' and G'' evolution with frequency, time and temperature are performed for selected samples.
A typical result is given in Figure 1. This system concerns the temperature-resolved *in situ* evolution of G' and G'' for different chemical environment/biosurfactant system. The G' and G'' are found at an initial value. Upon change of temperature, the corresponding G' and G'' are differently altered. The corresponding SAXS profiles show both the structural behaviour as a function of temperature and the reversibility of the structuration. If these data will be analyzed further, we could show that the structural evolution show very differents aspects according the initial chemical environment.

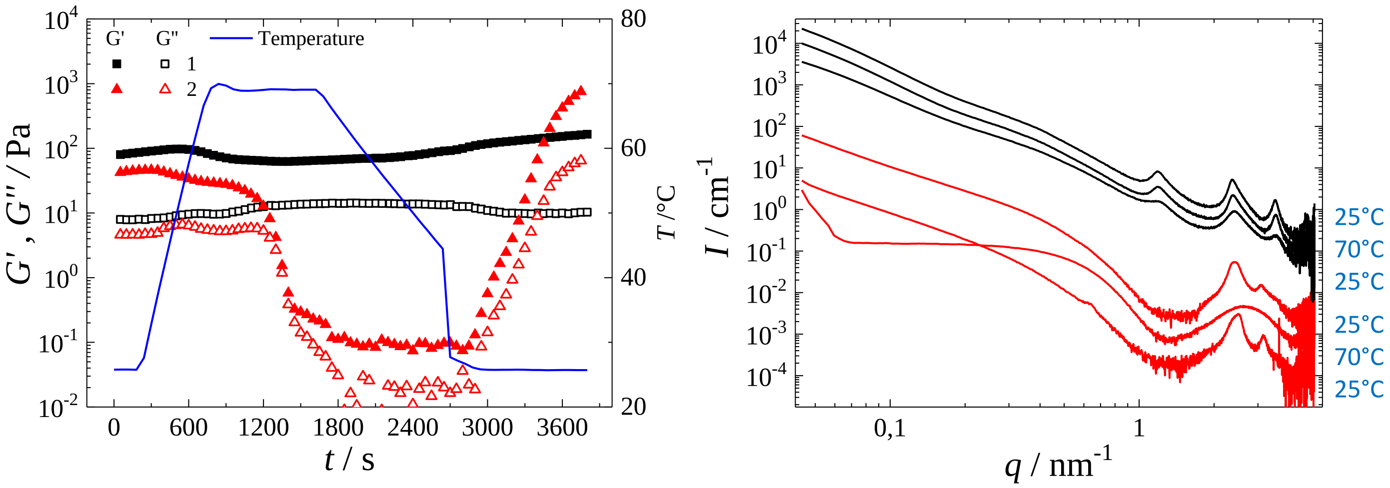

Figure 1

Data treatment

The SAXS signal is treated at the beamline soon after acquisition using the SAXSUtilities software. The rheology signal is acquired and monitored with the Thermo Scientific HAAKE RheoStress software. The 2D SAXS images are integrated, corrected for absolute intensity and background subtracted. Background (Couette cell + water) has been measured at different temperatures.

Justification and comments about the use of beam time:

The use of the beamline was justified by the appropriate q-range, which matched our needs, and by the precence of the in-house rheometer. The amount of beamtime, 6 shifts was totally adapted, as we could analyze all our samples.

Problems during beamtime:

We did not experience major problems during the beamtime. The beamline was well prepared prior to our arrival and assistance was guaranteed by the local contact all along. The only problem we did experience was related to the temperature control of the rheometer, as it was not possible to impose a rate of the temperature ramp and the temperature measured was with an uncertainty of +- 2°C due to long equilibration between the controlled bath temperature and cell. A Pelletier device could reduce such disadvantages. Two different external baths were then used, one for heating, the other one for cooling, but this method unfortunately produced a break in data acquisition and made the rheo-saxs-temperature correspondence imprecise.
Additionally, the presence of 1 single user per experiment due to Covid restrictions added some difficulty to perform the experiment under the best conditions possible, although the personnel of the beamline team was very helpful.