Experimental report

13/07/2022

Proposal: 9-10-1701 **Council:** 10/2020

Title: SANS from foam in a (nano)ion-flotation extraction process: integration of a Foamscan apparatus on D33 spectrometer

Research area: Soft condensed matter

This proposal is a new proposal

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Samples: D2O
DCl
Sodium Dodecyl Sulfate
BRIJ-O10
Polyoxometalate SiW12040
sodium tungstate dihydrate

Instrument	Requested days	Allocated days	From	To
D33	2	2	23/06/2021	25/06/2021

Abstract:

Liquid foams structures are ubiquitous and at the basis of many applications for: environment (extraction via flotation, decontamination), detergency (often through foam stabilizers or anti-foaming agents), food, cosmetics, firefighting and crude oil recovering. An academic research is still required to understand the mechanical properties of the foams that strongly depend on the films structure at all the scales. However, the accurate structural characterization of the inter-bubble film with the distribution of the various chemical species within the film is often lacking during these investigations. These characterizations provide the information of the molecular and supramolecular interactions within the interfaces and between facing interfaces, required for understanding the viscoelastic properties of the films and for controlling the foam stability. The aim of this proposal is to use on selected exemples a modified Foamscan instrument that has to be implemented on D33 to record for the first time the simultaneous acquisition of mesoscopic and macroscopic foam information via (TOF-)SANS, imaging and conductivity techniques.

/Experimental report

01/02/22

Proposal: 9-10-1730 **Council:** 2021-10

Title: **Mechanisms of (de)stabilization ofan electrostatic foam**

Research area: Soft condensed matter

Main proposer: **Olivier DIAT**

Experimental team:

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Samples: BrijO10, D2O, SiW, SDS

Instrument	Requested days	Allocated days	From	To
D33	3	2	2021 June 23 at 9h to 25 at 9h00	

Abstract: When you try to produce a liquid foam, you may observe different behavior: a few shortlived bubbles, a fragile foam which rapidly disappears, or a more stable one lasting several hours. The fact that a foam forms at all is the result of several different mechanisms which tend either to produce and stabilize it or to destroy it.

In order to obtain a stable foam, it is evidently necessary to add to the liquid surfactants. During foaming, surfactants adsorb to the gas/liquid interfaces of each bubble. The electrostatic interaction, which is often significant because the interfaces are generally electrically charged, stabilizes the film by repulsive forces between its surfaces. These charged surfaces repel each other. However, for some foams, stability decreases by increasing the charge on the interface. The goal is therefore to study an electrostatic foam to understand the mechanisms of (de)stabilization of the foam.

Experimental Report

Two days is very short for this type of experiment that requires some time for setting, align and calibrate all the coupled techniques around foam studies.
We had still some computer crashes related to foam image capture (less than previous experiment in March but still that prevent us to complete all our objectives. The objectives were not those described in the proposal because this proposal was written before the results of the previous run in March and for which the data were not exploited. So this run was dedicated to re-performed what was carried out in March.
We decided also to change the protocol of foam generation because prior to SANS measurements, foams were studied using the FOAMSCAN apparatus. We so injected 40 mL of the foaming solution into the column in 10 mL-steps through the inlet at the bottom of the column, just above the frit. After each injection step, conductance was measured, resulting in a linear calibration curve (conductance versus liquid volume). Then, N2 was flowed at 50 mL/min through the porous frit. Once the column was filled by the foam after 4 min, the N2 flow was stopped leaving the foam under free drainage conditions during 75 min. The foam structure was captured using a camera focussing on the prism foreseen to this operation and SANS experiments were performed between two image captures, a transmission as well.
We developed i) a specific procedure to fit scattering data over a large intensity and q-vector range in order to extract quantitative information of specific surface areas, from Plateau border and thin inter-bubble as well, the liquid volume fraction through the micellar contribution and the film thickness, all these features as a function of time. We developed (part in collaboration with B. Dollet) also ii) a code for image analysis to extract information on bubbles sizes and distribution evolution, liquid volume fraction, Plateau border radius and deduced disjoining pressure.
Liquid volume fraction versus time was also extracted from Transmission data as well as from conductance measurement. All these data and their correlation in time and in space were discussed in a submitted article in Scientific Report in July.
Some of the figures of this article are given in the table below:

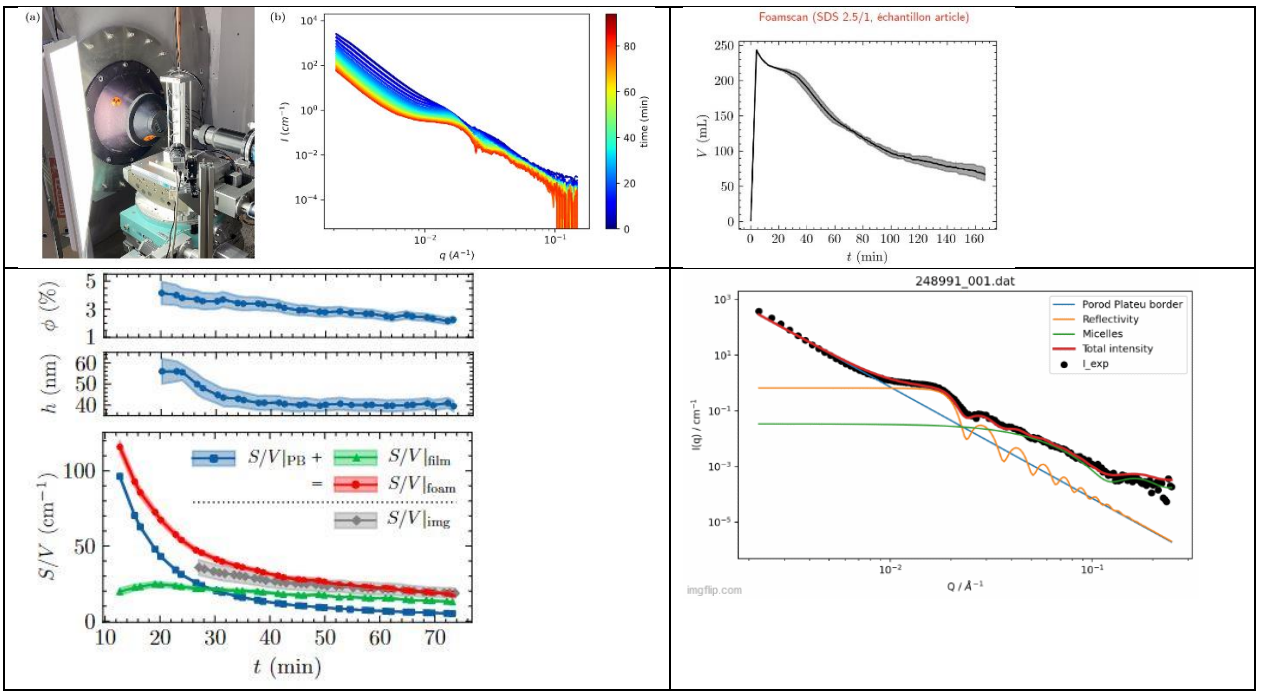

Fig. 1: Left top: photograph of the "Teclis-type column" and azimuthal average data versus time. Right top: foam height evolution under free drainage. Left bottom: evolution of the liquid volume fraction, inter-bubble film thickness et specific surfaces areas from Plateau borders, films and their sum as a function of time all these parameters being extracted from SANS curve shown in bottom right adjusted with a quantitative (in cm-1) model (A new one).

Two important points: 1) for the first time we can distinguish between specific surface areas (SSA) from Plateau borders and from films independently and compare their mutual contribution to the total SSA that can also be obtained via image analysis.

2) We can determine for the first time from operando image analysis the disjoining pressure from Plateau border curvatures and then we succeed to correlate its time evolution as a function of film thickness variation and determine two modes of relaxation in foam and free drainage.
All these analyses were carried out on our reference system: Brij with SDS with molar ratios of 5/1.
We had time to reproduce this experiment to be able to publish this quantitative analysis.
The experiments carried out with nano-ions (the objectives of the previous proposal 9-10-1675) were not finalized.
We kept 6 hours for collaborators because the objectives of this thesis program was also to deliver a foam cell that can be used by other interested team.
A group from Darmstadt university (group of R. von Klitzing) came with foam systems stabilized with protein. We realized that the cleaning procedure developed for our surfactant systems has to be changed and optimized because protein are more difficult to remove from quartz surface to be sure that a FOAM experiment is not influenced by the remaining chemical compounds from the previous one. The best will be to have another exchangeable cell to not loose time during the cleaning procedure (every hours or 1hours and half) – 20mn of cleaning!