# Experimental report

13/07/2022

**Proposal:** 9-10-1675 **Council:** 4/2020

**Title:** Foam Flotation for ionic metal nano-clusters extraction and separation: Structure of the foam investigated using TOF-SANS

**Research area:** Soft condensed matter

**This proposal is a resubmission of 9-10-1643**


**Main proposer:** **Olivier DIAT**

**Experimental team:** Leonardo CHIAPPISI
Alban JONCHERE
Olivier DIAT
Pierre BAUDUIN
Coralie PASQUIER

**Local contacts:** Charles DEWHURST
Leonardo CHIAPPISI


**Samples:** D2O
DCl
BRIJ-O10
Sodium tungstate dihydrate¿
Sodium molybdate dihydrate

| Instrument | Requested days | Allocated days | From | To |
|---|---|---|---|---|
| D33 | 3 | 3 | 12/03/2021 | 15/03/2021 |


**Abstract:**

Ion flotation used in separation chemistry is a green alternative to classical solvent extraction process in order to concentrate within a foam, ionic species produced by acidic dissolution of metallic wastes or ores. We aim to study the structure of the foam, a fluid and multi-scaled 3D-structure as a function of its height in a dedicated flotation column and as a function of the the ion concentration in the bulk solution. This study will be carried out in stationary regime and under free drainage conditions. The results will be correlated with the ion extraction efficiencies to optimize the extraction process that will be exploited by an industrial company. Since foams are out-of-equilibrium and fragile systems, we propose to use the TOF mode, only available on D33, to cover a massive and suitable q range from 3.10-3 to 0.3 Å in one instrument setting and in operando.

# /Experimental report

01/02/22

**Proposal:** 9-10-1675 **Council:**

**Title:** Foam Flotation for ionic metal nano-clusters extraction and separation: Structure of the foam investigated using TOF-SANS

**Research area:** Soft condensed matter


**Main proposer:** Olivier DIAT

**Experimental team:** Max Hohenschutz
Pierre Bauduin
Philipp Schmid
Luc Girard
Alban Jonchère
Lamolinairie Julien

**Local contacts:** L. Chiappisi


**Samples:** BrijO10, D2O, SiW, SDS

| Instrument | Requested days | Allocated days | From | To |
|---|---|---|---|---|
| D33 | 3 | 3 | 2021 March 12 at 9h | to 15 at 9h00 |


**Abstract: Ion flotation used in separation chemistry is a green alternative to classical solvent extraction process in order to concentrate within a foam, ionic species produced by acidic dissolution of metallic wastes or ores. We aim to study the structure of the foam, a fluid and multi-scaled 3D-structure as a function of its height in a dedicated flotation column and as a function of the the ion concentration in the bulk solution. This study will be carried out in stationary regime and under free drainage conditions. The results will be correlated with the ion extraction efficiencies to optimize the extraction process that will be exploited by an industrial company. Since foams are out-of-equilibrium and fragile systems, we propose to use the TOF mode, only available on D33, to cover a massive and suitable q range from 3.10-3 to 0.3 Å in one instrument setting and in operando.**

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It was the first time that a specially designed column able to record simultaneously SANS data, the conductivity through foam structures and photo-image capture, all collection as a function of time, was installed on a SANS spectrometer (see photos in fig. 1 on D33). This combination of techniques are necessary to get structural correlation at different scales of the foam (from nm up to cm).
As usual, when an experiment is built for its first time, several issues appear, here some control-command computing related to NOMAD system that prevented us to perform a successful experiment (more than 10 server crashes per days, instability on the camera support). Nevertheless, we learnt a lot on carrying out these types of experiment.
This issue was a penalty because this type of experiment was designed to follow foam aging and thus once the system crashes at any time of the foam aging, the experiment has to be restarted from beginning with a phase of cleaning. So about ½ of the time was lost overall the experiments.

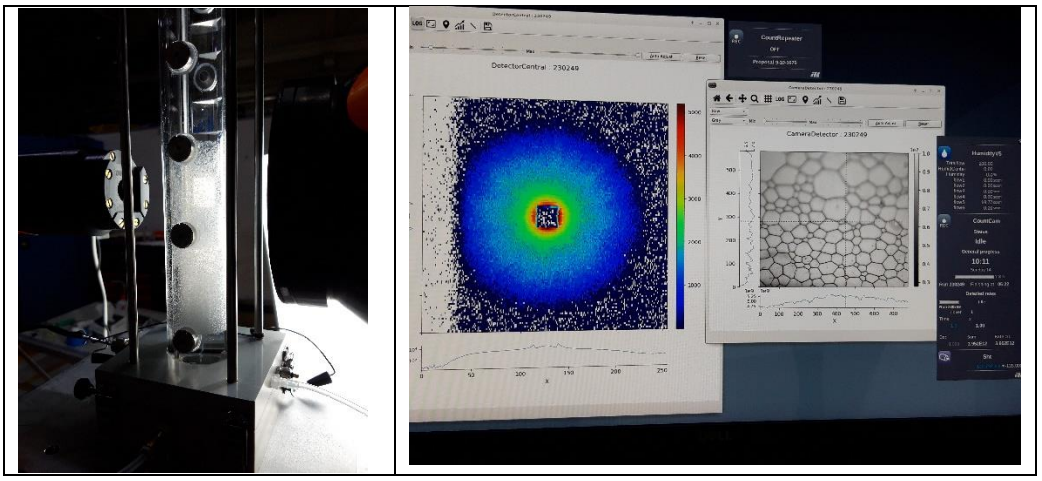

Fig. 1: Left: photograph of the "Teclis-type column" developed in quartz and settled on the sample table stages on D33. Right: visualisation on PC control screen of the SANS 2D data and image capture of a foam under aging.

The main part of the new device is a quartz column designed on the model of the FOAMSCAN from Teclis Scientific (France)and in which the foam will be formed. This cell is cylindrical (30 cm high with an internal diameter of 35 mmand a wall thickness of 2mm) and is always opened at the top. A flat face was machined in order to glue four prisms at different fixed heights allowing image capture of the bubbles in contact with the wall. Five pairs of electrodes were also integrated to measure the conductance through the foam at different heights. This cell is therefore compatible with the FOAMSCAN and can be used both in laboratory and on neutron diffractometer dedicated to small-angle scattering technique. The quartz cell is clipped onto a base via an O-ring to get a set "column and rigid support" tight but easy to dismount for efficient cleaning. The base is composed with a silica frit (pore size 10-16 µm, porosity P16 (ISO 4793)) and can be exchanged depending on the required bubbling size. The flux of injected gas is controlled by a digital Mass Flow Controller (MFC) from MKS INSTRUMENTS (Andover, Massachusetts, U.S.) with a flow range 0-100 mL/min for model GE50 series. When the foaming solution is injected into the bottom of the column, a slight suppression is maintained to avoid filling the pores of the frit with the solution (with a gas flow rate of 2 mL/min). The foams are then produced with a gas flow rate of 50 mL/min using nitrogen. On the diffractometer, the cell was fixed on a set of Y -Z stages in order to first center the cylinder in the neutron beam path and second to vary its Z-position so that the neutron beam passes through the cell at heights predefined by the position of the prisms and in order to be able to simultaneously capture photographs of the foam probed by neutrons.

We tested several foams composition such as:
Non-ionic Surfactant BRIJ-O10, 0.5 mM
Brij with SDS with and variable composition molar ratios 5/1 ; 2/1 ; 1/1 ; 1/2 (avec 20mM et sans sel)
Brij with HSiW nanoions: 5/1 (0.1mM de HSiW); ½ ; 1/6 ; 1/10 (5mM de HSiW) ; 1/20 ; 1/50
Non-ionic Surfactant C8G1 and C8G1 with HSiW

Even if the data are not really exploitable because, what was expected, was the data correlation between the different techniques and not individually, this first set of experiments were nevertheless pertinent to improve the settings of the cell and the neutron scattering acquisitions pour the next runs.
The previous published data on home-made quartz column were obtained using time-of-flight mode. It was decided with the local contact to come back to a monochromatic mode for a sake of simplicity in the scattering data management and normalization.