![ESRF logo]	**Experiment title:** Dendritic surfactants as new tools for palladium recovery and water-based nanocatalysis	**Experiment number**: MX1965
Beamlines: BM29	**Date of experiment**: from: 17/11/2017 to: 18/11/2017 29/11/2017 to 30/11/2017	
Shifts: 2/2	**Local contact(s)**: Petra Pernot	*Received at ESRF:*

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Proposal context: In this project, we aim at characterizing the physico-chemical and structural properties of dendritic surfactant self-assemblies for their capacities to extract palladium from electric and electronic equipment waste (WEEEs) with a direct use into catalysis of organic reaction. This project is part of a PhD thesis funded by labex CheMISyst that started in October 2016.

Report: The objective of the proposal submitted in 2017 was to study the physico-chemical properties of new dendronic surfactants in solution and understand the impact of their design (fluorinated or hydrogenated tail of variable length, size of the dendritic polar head and number of branches) on micelle form factor, structure factor, micellar behaviour in solution. Indeed micelle shape and interactions in solution (repulsive or attractive) may have an impact on the capacity of these surfactants to form stable micellar solutions for the recovery of palladium.

In the SAXS experiments performed in November 2017 on beamline BM29, we studied the behaviour (size, shape, aggregation number) of surfactants designed at University of Avignon, CBSA-IBMM[1]. Theses surfactants can be divided into six families depending on the nature of the hydrophobic tail (hydrogenated or fluorinated) and on the nature of the polar head (dendritic, simple w/wo spacer) **(scheme 1)** .
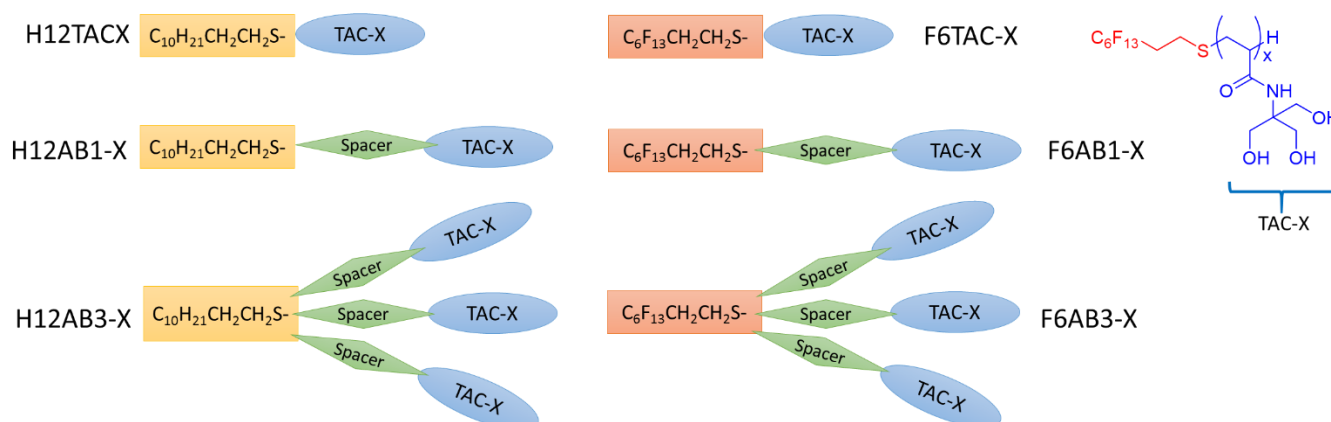

Scheme 1 : *Structure of hydrogenated and fluorinated surfactants developed by the CBSA team*

[1] K. Astafyeva, L. Somaglino, S. Desgranges, R. Salomir, A. Polidori, C. Contino-Pépin, N. Taulier, J. Mater. Chem. B, 3, 2892-2907 (2015)

Results: We first studied each surfactant in pure water in order to thoroughly characterize their physicochemical properties. SAXS experiments performed at different concentrations highlighted a concentration below which the micelles increase in size and above which self-assemblies are stable in shape (above 12,5 g/l for H12TAC9) but in weak repulsive interactions (**scheme 2.A**).

It turns out that each surfactant was used or will be used for an extraction of the palladium in order to provide enhenced-nanoreactors, which can be used for micellar catalysis. So we want to correlate the surfactant properties and their capacity for palladium recovery in order to understand how the surfactant interact with the metal during the proccess. Following that, we investigated the impact of palladium addition (1 equivalent) on the self-assembly in extraction conditions (nitric acid 2M) (**scheme 2.B**).
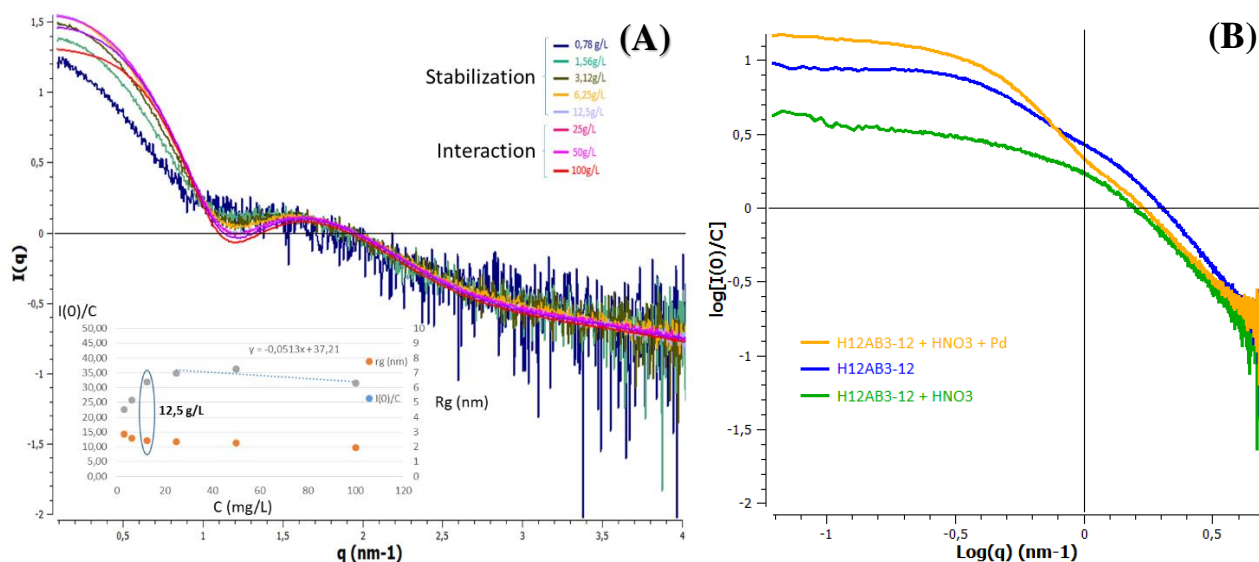
Scheme 2 : *(A) Effect of H12TAC9 concentration. (B) Extraction conditions of Pd using H12AB3-12 at 12,5g/L*

In order to explain our results, we simulated the electron density contrast of surfactant assemblies in different conditions (water, nitric acid and nitric acid with Pd) (**scheme 3**). Regarding the electron density profiles, we can claim that there is an interaction between our surfactant with both palladium and nitric acid.
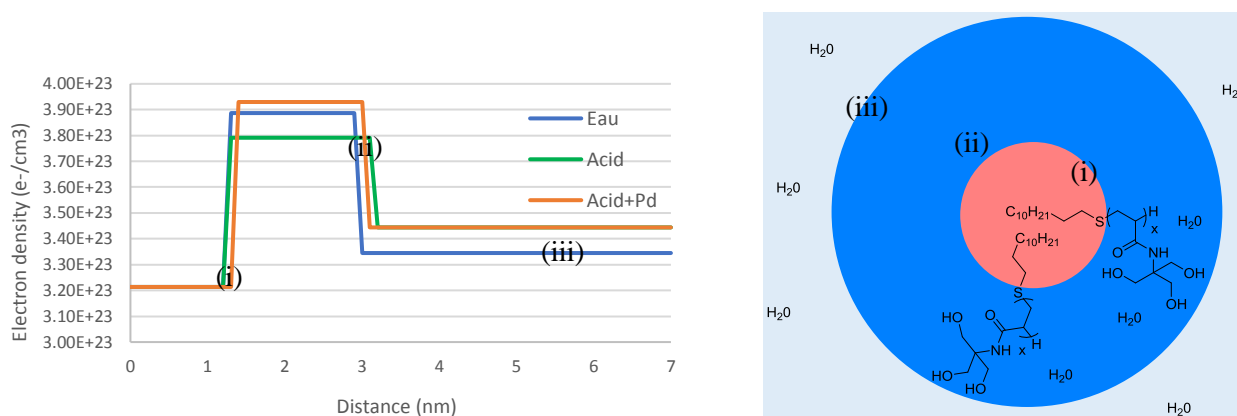
Scheme 3 : *Electron density contrast of H12TAC9. (i) Hydrophobic core. (ii) Hydrophilic shell. (iii) Solvent*

These first results were encouraging and were presented as a Poster during the UsersMeeting in February 2018. However, these results raise other questions : is there a maximum amount of Palladium in interactions with surfactants; can Pd interacts with surfactants without acid; is there an effect of Acid concentration on surfactant self-assembling; what are the effects of extraction conditions (organic solvent and nature of extractant) on surfactant assemblies?