



	<b>Experiment title: SAXS probing of Janus Nanoparticles self-assembly</b>	<b>Experiment number:</b>
<b>Beamline:</b> ID2	<b>Date of experiment:</b> from: 29/01/2016 to: 01/02/2016	<b>Date of report:</b>
<b>Shifts:9</b>	<b>Local contact(s):</b> Dr Sylvain Prevost	<i>Received at ESRF:</i>
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## Report:

The initial proposal was about the self-assembly of gold/silica nanoparticles in solution. We were awarded some beamtime on another SAXS beamline (SWING at the SOLEIL synchrotron) and these experiments took place in summer 2015. Since we had performed most of our planned experiments during this beamtime, we decided not reproduce them and to focus on two other important topics in the framework of N. Castro's 3rd year PhD thesis:

- the formation mechanism of Janus CdSe/gold heterodimers,
- in situ probing of PbS nanocrystal nucleation and growth.

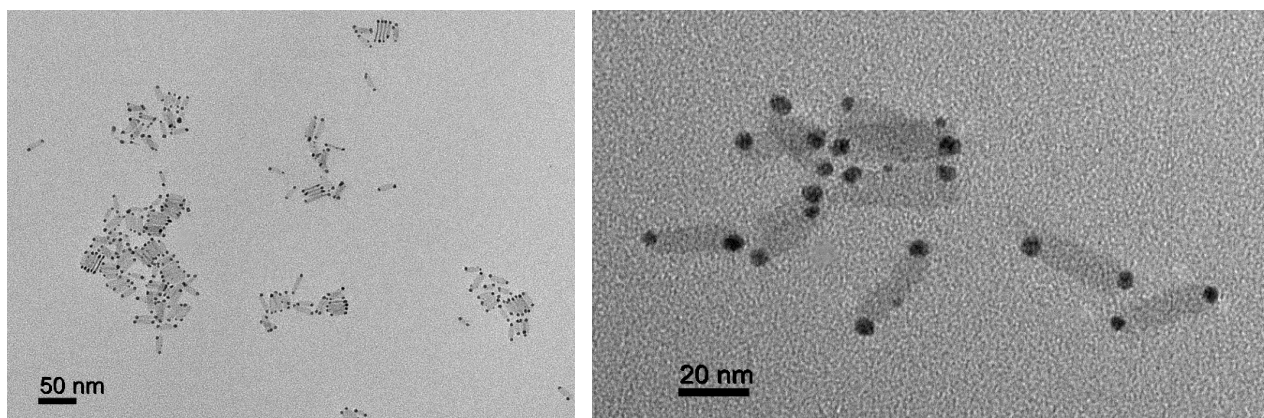
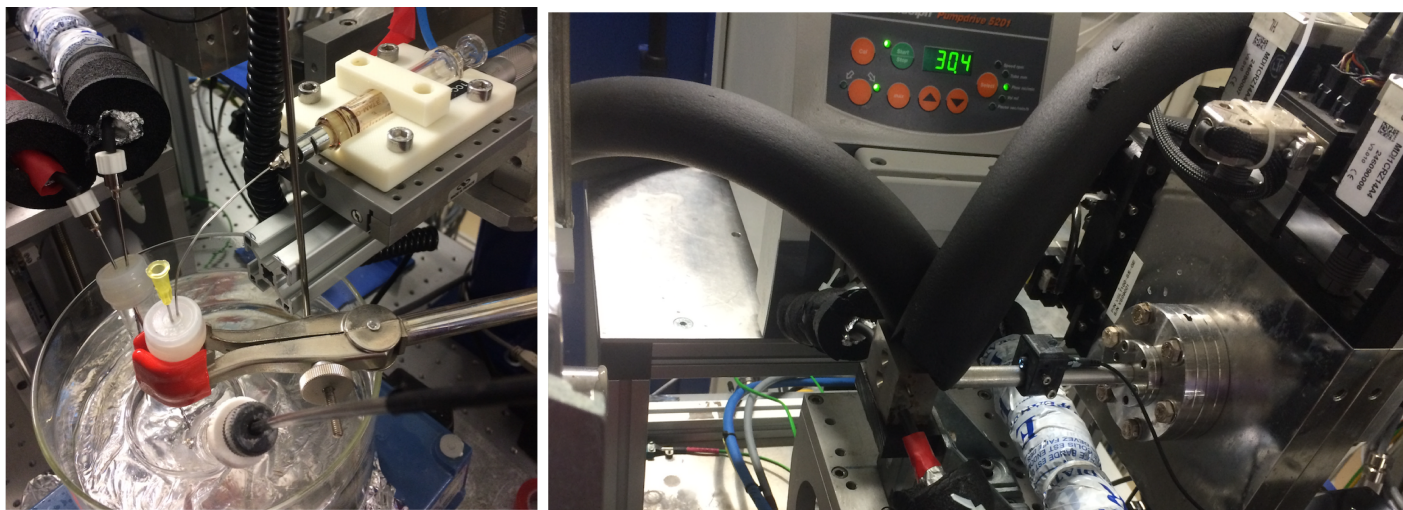


Figure 1: TEM images of CdSe-gold heterodimers

The first part of the experiment was dedicated to the Janus CdSe/gold (figure 1). These nanoparticles are obtained by adding gold salt and a reducing agent to a dispersion of CdSe nanoplatelets (NPLs). The kinetics is of the order of 10 minutes so it is impossible to look at such a fast kinetics on our lab instrument. The experimental set-up consisted of a round bottom flask which contained the NPLs dispersion in toluene. A peristaltic pump is flowing the reacting fluid through a capillary cell where SAXS/WAXS patterns were acquired during the reaction (figure 2). The reaction was triggered by the injection of the gold solution through a remote controlled home-built syringe injector. We explored different experimental configurations such as the quantity of added gold, the temperature, the dilution etc... Overall, we run 11 different experimental conditions with both high  $q$  (sample to detector distance of  $d=1$  m) and small  $q$  ( $d=5$  m). The experimental data are currently being treated by N. Castro and we expect to better understand the formation mechanism of this new type of particles soon.



*Figure 2: pictures taken the run showing the experimental set-up for in situ probing of janus nanoparticles and PbS nanocrystals. On the left: flask with injection syringe . The two black tubes on the left are for recirculating the fluid with the peristaltic pump. On the right: flow cell in which the reacting fluid is circulated. The peristaltic pump is visible in the back.*

The second part of the experiment was dedicated to formation dynamics of PbS nanocrystals. We use a new type of selenium precursors which enables the fine tuning of the precursors reactivity. These experiments are made in collaboration with Prof J. Owen's group at Columbia university. The goal here was to test the experimental set-up in order to ask for further beamtime. The experimental configuration was the same as described previously except that the temperature is higher in this case going from  $80^{\circ}\text{C}$  to  $130^{\circ}\text{C}$ . We tested 10 different experimental conditions and obtained overall good results. We noticed that strict air-free conditions have to be respected for the preparation of the lead oleate preparation since the solutions which have not been prepared in a glove box yielded aggregated samples. This will help us prepare the next run if time is granted for this project.

**Overall, our experiment went smoothly and the ID2 beam line worked perfectly.**