

## Experiment Report Form



<p><b>Experiment title:</b> Au supported nanoparticles for selective alcohols oxidation: an XAS/DRIFT/MS study of the synthesis and Au-PVA anchoring process</p>	<p><b>Experiment number:</b> CH4234</p>	
<p><b>Beamline:</b> ID24</p>	<p><b>Date of experiment:</b> from: 08 July 2015 at 08:00 to: 14 July 2015 at 08:00</p>	<p><b>Date of report:</b> 01/03/2015</p>
<p><b>Shifts:</b> 18</p>	<p><b>Local contact(s):</b> Giovanni Agostini</p>	

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**Report:**

Energy dispersive beamline ID24 was used for a first attempt to investigate the nucleation process of metal nanoparticles at ms time resolution. The experiment was performed with the stopped-flow/UV-Vis/EDE facility of ID24, which in turn was synchronised at the millisecond regime.

The experiment was performed on Au L3 edge (11919 eV), and Pd K edge (24350 eV) and a Stop Flow Cell available at ID24 was employed.

According to Scheme 1, a typical experiment involved the preparation of two water solutions (S1 and S2) to be mixed in the cell: the former containing the Pd precursor,  $\text{Na}_2\text{PdCl}_4 \cdot 2\text{H}_2\text{O}$ , or the Au precursor,  $\text{NaAuCl}_4 \cdot 2\text{H}_2\text{O}$ , and the latter with the polyvinyl alcohol (PVA) or polyvinylpyrrolidone (PVP) protecting agents. Then S1 and S2 were mixed and the metal/PVA ratio (wt/wt) has been varied in order to evaluate the effect of the protecting agent on the final metal size. The obtained solution S1+S2 (yellow color) will be mixed and after a

delay time mainly determined by the stop flow itself, a water solution containing  $\text{NaBH}_4$  as reducer (S3, metal/mixture= 1:100 mol/mol) was added and the measurement was carried out.

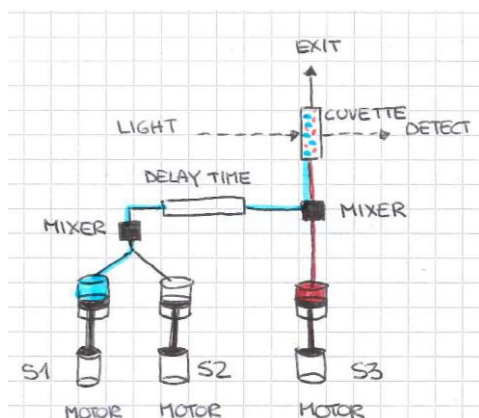


Figure 1. Schematic representation of the operation of the stop flow cell.

The experimental conditions, such as the concentration of the solutions, the S1/S2/S3 ratios, the acquisition time etc., were carefully optimised.

An example of typical spectra acquired in static conditions at the Pd K edge (24350 eV) is shown in Figure 2, section a, whereas a typical output of a kinetic measure is reported in section b of the same figure.

The same kind of acquired data obtained by monitoring the Au L3 (11919 eV) is shown in Figure 3.

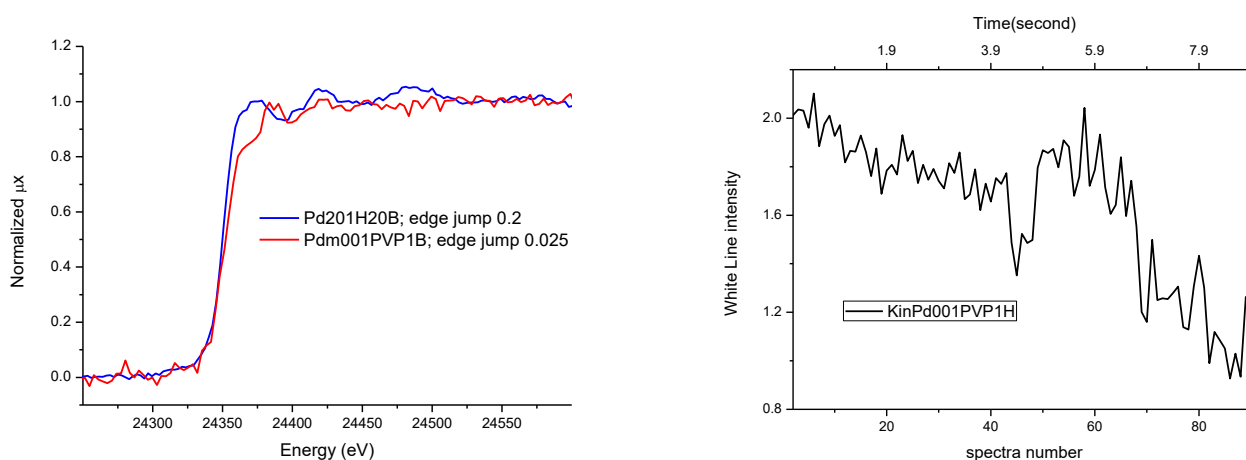


Figure 2. (Left) Spectra collected at the Pd K edge (24350 eV) of the S1 solution containing the Pd precursor (blue curve) and of the S1+S2 solution with PVP as protecting agent (red curve); (Right) Kinetic after the addition of S3 containing the reducer to S1+S2.

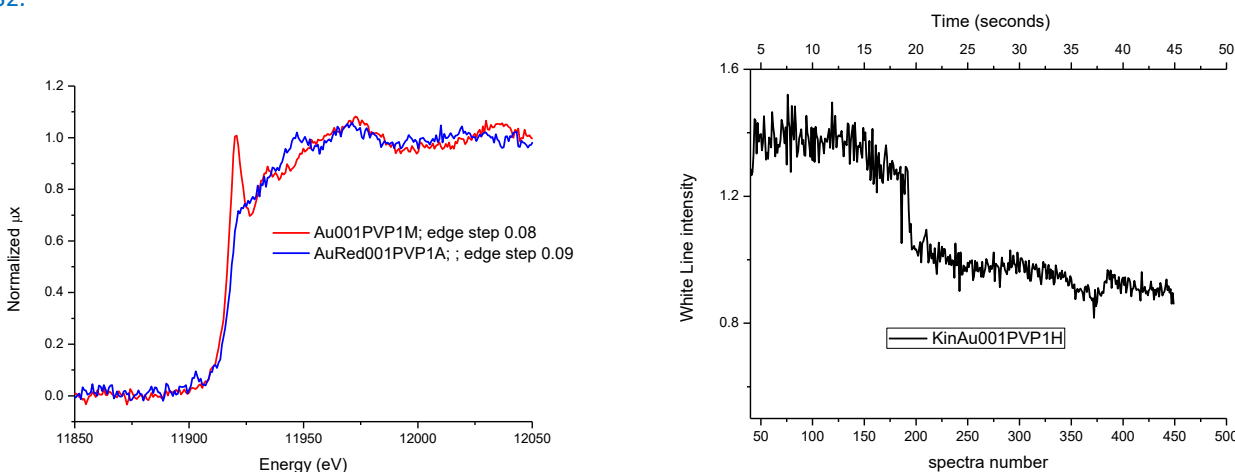


Figure 2. (Left) Spectra collected at the Au L3 edge (11919 eV) of the S1 solution containing the Au precursor (blue curve) and of the S1+S2 solution with PVP as protecting agent (red curve); (Right) Kinetic after the addition of S3 containing the reducer to S1+S2.

Despite the problems encountered due to the formation of bubbles after the addition of the S3 solution containing  $\text{NaBH}_4$ , these data demonstrate the technical feasibility of the experiment: the kinetics were determined for both Au and Pd nanoparticles in controlled experimental conditions.