



<b>Experiment title:</b> EXAFS experiments for the structural analysis of Pumice-supported Pd-Rh and Pd-Ag bimetallic catalysts derived from Pd, Rh and Ag solvated atoms.	<b>Experiment number:</b> CH-392	
<b>Beamline:</b> BM08 GILDA	<b>Date of experiment:</b> from: September 4 <sup>th</sup> 1997 to: September 8 <sup>th</sup> 1997	<b>Date of report:</b> July 30 <sup>th</sup> 1998
<b>Shifts:</b> 13	<b>Local contact(s):</b> Francesco D'Acapito	<b>Received at ESRF:</b> <b>10 AOUT 1998</b>

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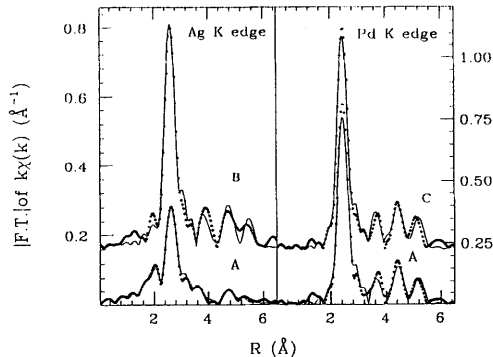
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**Report:**

The use of bimetallic Pd-Ag heterogeneous catalysts seems to be very promising in the reduction of nitrites and nitrates that pollute water to nitrogen; the addition of Ag avoids the further, undesired, reduction of nitrogen to ammonia. Silver-Palladium bimetallic catalysts, with different Ag:Pd ratios, were synthesised on pumice using metal ion reduction procedure. The reported XAFS experiments have been carried out to get more information about the structure of the investigated samples and, in particular, to check if the two metallic elements do mix or not to form an alloy. The XAFS measurements at the Ag (25.514 keV) and Pd (24.350 keV) K-edges were performed on the CRG italian beam line GILDA. The energy range was from 24.150 to 25.450 keV for Pd K-edge and from 25.460 to 27.000 keV for Ag K-edge. Beam energies were defined using a Si(3 1 1) double crystal monochromator that gives a resolution of about 1.5 eV at these energies. The dynamical sagittal focusing of GILDA [1] monochromator allowed to focus 4mrad of the horizontal beam divergence to achieve a small and intense ( $\sim 10^{11}$  ph/sec) spot at the sample.

This configuration is particularly suited for fluorescence XAFS measurement on diluted sample for which high photon fluxes are required. XAFS measurement were performed in transmission geometry, using two Ar filled ionisation chambers, on the three bulk references samples Ag-foil, Pd-foil and Ag<sub>2</sub>O-powder and in fluorescence geometry on the pumice supported catalysts. Fluorescence signals were detected using a 7-elements Ge multidetector. All XAFS spectra were recorded at 77°K to reduce the thermal disorder effect. From the XAFS analysis carried out using the GNXAS program package [2] and from auxiliary AWAXS experiments [3], no direct evidence of Ag-Pd alloying was found. The Pd-Pd interatomic distance in the bimetallic samples was in agreement with the reference. The analysis of the Ag K-edge pointed out the presence of two different Ag phases, oxide and metal. The Ag-O distance is longer than the Ag<sub>2</sub>O reference one and agrees with literature data relative to Ag implanted in silica [4]. The contraction of the Ag-Ag distance is likely due to the presence of small metal clusters. The figure draws some representative results of the GNXAS analysis



Dots: experimental FT  
 solid line: calculated FT  
 A: Ag 0.08%wt, Pd =.25%wt  
 B: Ag foil  
 C: Pd foil

## References :

- [1] S. Pascarelli, F. Boscherini, F. D'Acapito, J. Hrdy, C. Meneghini, S. Mobilio, *J. Synch. Rad.* **3** 147 (1996).
- [2] A. Filipponi, A. Di Cicco, CR. Natoli, *Phys. Rev. B* **52** 15122 (1995-I).
- [3] A. Martorana, A. Balerna, C. Meneghini, A. Longo, G. Pipitone, report of ESRF experiment n. CH-428.
- [4] D'Acapito, F., Gonnella, F., Cattaruzza, Pascarelli, S., E., Mazzoldi, P., Mobilio, S., *Nucl. Instr. and Meth. B* **120** 110 (1996).