ESRF	**Experiment title:** Fate of silver nanoparticles contained in wastewater and transfer in soils and plants	**Experiment number**: EV32
Beamline: ID21	**Date of experiment**: from: 26-6 2013 to: 1 7 2013	**Date of report**: Feb 1 2014
Shifts: 15	**Local contact(s)**:Hiram Castillo Michel	*Received at ESRF:*
Names and affiliations of applicants (* indicates experimentalists): Geraldine Sarret and Camille Larue, ISTerre Hiram Castillo Michel, ESRF		

Report:

Nanosilver is currently used in a large variety of consumer products. An important part of this contaminant ends up in waste water and is then retained in sewage sludge, which is applied on agricultural soils. This experiment aimed at studying the fate of Ag in a soil contaminated by real nanoAg-contaminated sewage sludge, their transfer in crop plants and the changes in speciation at the various steps of this pathway by µXRF and Ag L_{III}-edge µXANES.

The sludge is produced at EAWAG (Zurich) using a pilot wastewater treatment plant. The production of the sludge took a much longer time than anticipated, and the samples were not ready for the experiment. After discussion with the beamline scientist, we decided to use the beamtime for:

- Recording Ag and Fe reference spectra that will be useful for the project
- Performing complementary analyses on plants exposed to pristine nanoparticles and aged paints

Results obtained made it possible to finalize a publication a publication entitled "Fate of pristine TiO2 nanoparticles and aged paint-containing TiO2 nanoparticles in crops after foliar exposure" which is currently under review (Figure 1 and 2). Not only TiO_2-NPs pristine and from aged paints, but also TiO_2-MPs were internalized in lettuce leaves, and observed in all types of leaf tissues, both in extracellular and intracellular compartments. No change in speciation was observed, but an organic coating of TiO_2-NPs is likely. These results obtained on the foliar uptake of nano- and microparticles are important in the perspective of risk assessment of atmospheric contaminations. This experiment was also used to get complementary results on the root uptake of nanoparticles, and another article is currently in preparation.

We were able to perform the experiment on sludge, soil and plant samples later on using inhouse beamtime, in collaboration with Hiram Castillo Michel. Results showed that Ag was well dispersed in the sludge and soil (e.g., Fig. 1A). µXANES evidenced the prosence of Ag2S as major species, and an additional species was identified Fig. 1B). Analysis of isolated roots showed Ag in the root surface and inside roots tissues. µXANES analyses were also performed on roots. A new mode of acquisition of µXANES was tested, which uses the principle of full field mode. This mode storngyl increases the quality of µXANES spectra especially on very small spots.

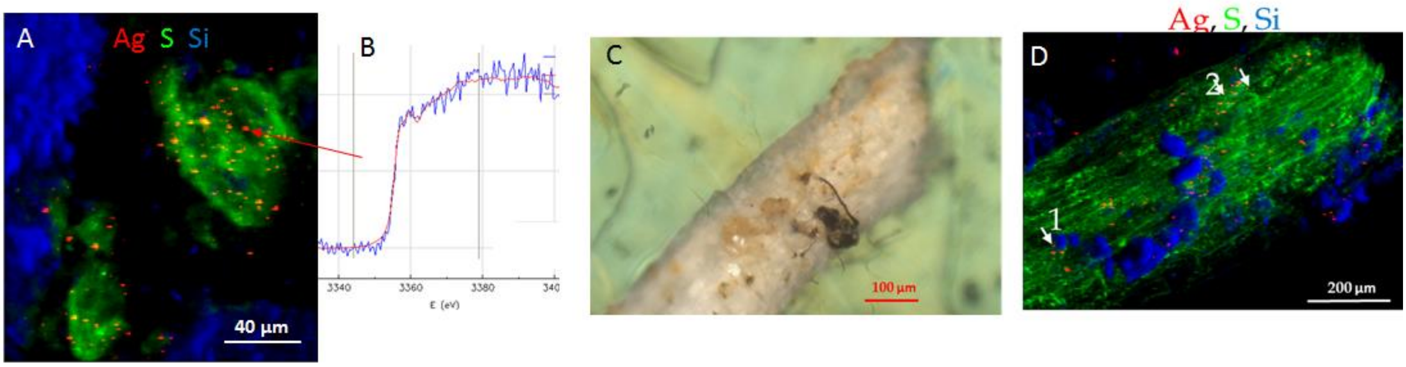

Figure 1A: µXRF map of a soil thin section. B: µXANES analysis of a selectid Ag-roch region. C: isolated root of rape grown in the soil. D: µXRF map of the root.

Publications

Larue C., Castillo-Michel H., Sobanska S., Trcera N., Sorieul S., Cécillon L., Ouerdane L., Legros S., Sarret G., 2014, Fate of pristine TiO2 nanoparticles and aged paint-containing TiO2 nanoparticles in lettuce crop after foliar exposure, J. Haz. Materials, 273, 17-26.

Larue C., Castillo Michel H., Sobanska S., Cécillon L., Bureau S., Barthès V., Ouerdane L., Carrière M., Sarret G., 2014, Foliar exposure of Lactuca sativa to silver nanoparticles: Evidence for internalization and changes in Ag speciation, *J. Haz. Materials*, 264, 98– 106.