Standard Project

Experimental Report template

Proposal title: fate of silver contained in sewage sludge after application on a cultivated soil		**Proposal number:** 20130945
Beamline: BM30B	**Date(s) of experiment:** from: 25 June 2014 to: 30 June 2014	**Date of report:** Sept 2014
Shifts: 15	**Local contact(s): Isabelle Kieffer**	*Date of submission:* *Sept 2015*

Objective & expected results (less than 10 lines):

Silver NPs (AgNPs) are one of the most prevalent metallic nanoparticles in consumer products due to their antimicrobial activity. However they are easily leached from them, for example from clothes during laundry washing, then ending in the wastewater treatment plants (WWTP). More than 90% of wastewater Ag-NPs are retained in sewage sludge. In France, 40% of sewage sludge is applied on agricultural soils as a fertilizer. Recent studies have identified silver sulfide (Ag_2S) NPs as major Ag species in sludges. This compound has a low solubility in its macrocrystalline form but the solubility of the nano form is not known and no information is available about its fate in amended soils where soil components and plant root exudates could modify their chemistry. The main objective of this work was to study Ag speciation in a sludge after introduction of Ag in the treated effluent water, and in a sludge-amended soil after culture with rape (monocot) or wheat (dicot), and without plant.

Results and the conclusions of the study (main part):

Sludge contaminated with 400mg AgNPs·kg^{-1} was produced by spiking PVPAgNPs into a pilot WWTP and then mixed with an agricultural soil in a 1/10 ratio. The speciation of Ag was studied in the sludge and in both the total and fine fraction (<2 µm) of the amended soils before and after 4 weeks of plant culture (and without plant) by Ag K-edge bulk EXAFS spectroscopy. The sludge and soil samples were lyophilized, ground and pressed to prepare 5 mm diameter pellets. The spectra of the following Ag standards were also recorded: Ag-GSH, $C_5H_{10}AgNS_2$, Ag_3PO_4, Ag-malate, AgO, Ag_3PO4, PVPAgNPs and different types of Ag_2S.

Results showed that Ag was mainly present in the sludge as Ag_2S of low crystallinity (Figure 1).

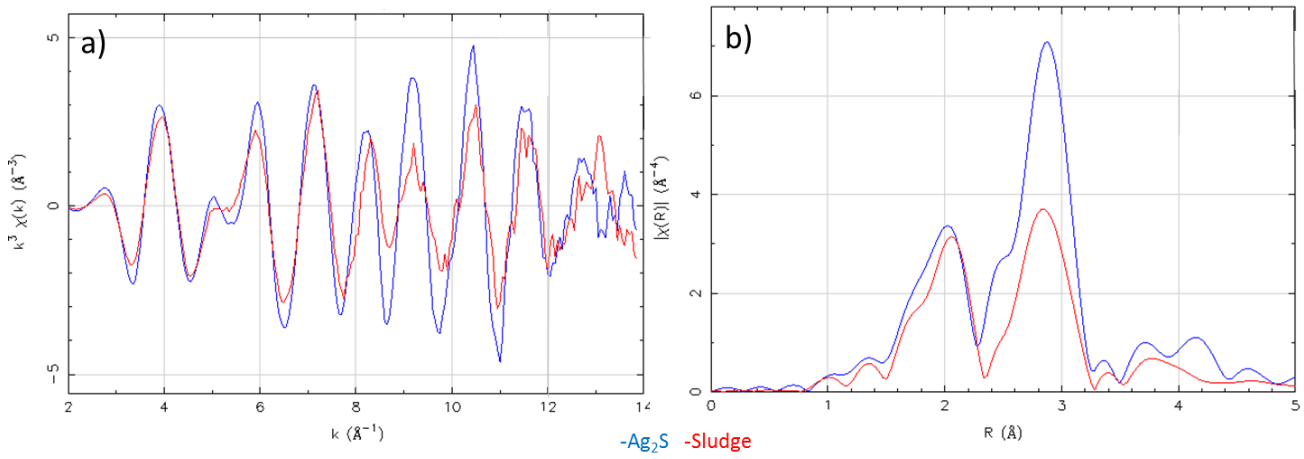

Fig.1. Ag K- edge EXAFS spectra a) and radial structure functions b) of Ag_2S standard and the polluted sludge.

Figure 2.a) shows the Ag K-edge spectra of the sludge and the soils fine fraction at the end of the experiment and the Linear combination fitting (LCF) for these samples. LCF for the sludge and soils showed that besides Ag_2S, secondary forms were present.This result suggest that Ag_2S present in the sludge is not as insoluble as generally considered based on the solubility constant of Ag_2S in its macrocrystallline form. Differences were also observed between unplanted and planted soil, suggesting a role of the rhizospheric activity.

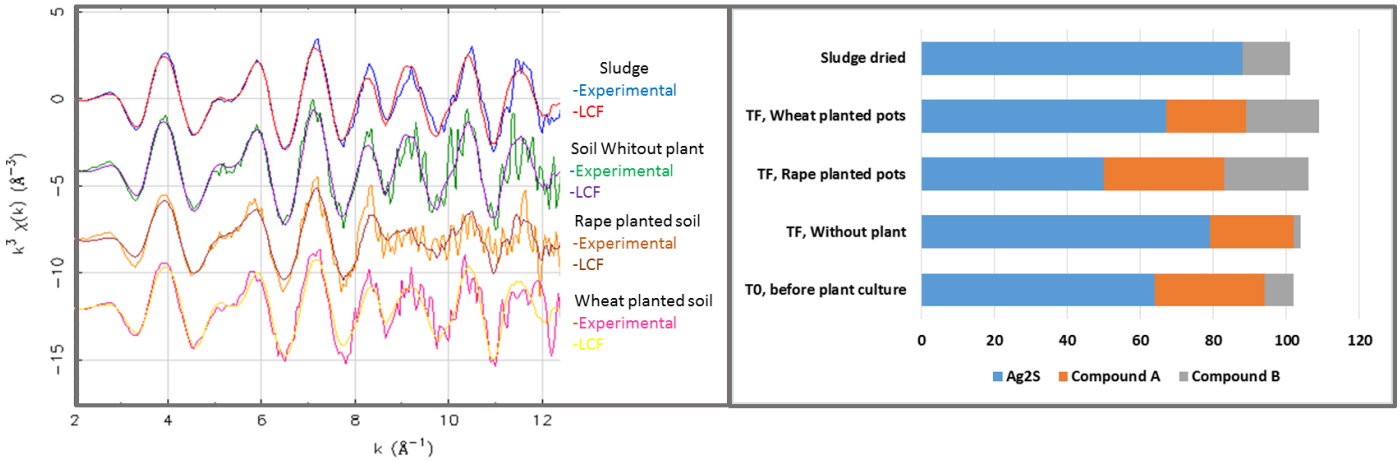

Fig.2. Ag k-edge EXAFS spectra and linear combination fits (LCF) a) and % distribution of Ag species from LCF b) for the sludge and the soils fine fraction (<2µm) at the end of plant culture

Justification and comments about the use of beam time (5 lines max.):

Despite the very low Ag content (a few tens of ppm), very good quality spectra up to 12 A-1 were recorded. The beamline run very smoothly during the experiment, we observed no radiation damage during data acquisition, the change in energy and cryostat loading were done with no problem. We recorded between 5 and 10 spectra for each sample depending on the Ag concentration.

Publication(s):

- *Fate of AgNPs in sewage sludge after application on agricultural soils*. Pradas Ana, Kaegi Ralf, Fernandez-Martinez Alejandro, Carriere Marie, Santaella Catherine, Sarret Geraldine. RST Conference, Pau (France) October 2014. Oral presentation.
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