EUROPEAN SYNCHROTRON RADIATION FACILITY

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ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title: Deciphering mechanism of interaction between biologically produced selenium nanoparticles and zinc metal ion**	**Experiment number**: 26-01-991
Beamline: **BM26A**	**Date of experiment**: from: 3rd Apr, 2014 to: 7th Apr, 2014	**Date of report**: 6th June, 2014
Shifts: **12**	**Local contact(s)**: BANARJEE Dipanjan	*Received at ESRF:* 6th June, 2014

Names and affiliations of applicants (* indicates experimentalists):
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Report:

A) Overview:

Understanding the interaction of metals in general - Zn in this case - and biologically produced colloidal elemental selenium (BioSeNPs) is essential to determine the fate, mobility and toxicity of Se in the environment.[1] To this point, this interaction was studied under different pH, ionic strength and Zn ion concentrations. The difference in ζ-potential measurements for BioSeNPs and BioSeNPs + Zn (-36 mV to -15 mV) suggests the electrostatic nature or covalent bond formation (can be either inner or outer sphere complex or both) interaction. X-ray photoelectron spectroscopy (XPS) analysis of Bio Se + Zn samples suggests the formation of an unidentified covalent Zn-Se phase.[2] However, this interaction cannot be confirmed due to close proximity of binding energy of ZnO to ZnSe. Thus X-ray Absorption Spectroscopy (XAS) is required to fully understand this interaction. The EXAFS measurements carried out at Dubble beam line has allowed us, for the first time, to identify the first and second neighbours of Zn adsorbed on the surface of BioSe under different experimental conditions.

B) Data quality:

The measurements were successful and data recorded was of high quality. A variety of model compounds (those which were not measured during earlier experiments) and samples were measured at Zn-k edge. Even though the Zn concentration was relatively "low" (around 2000 ppm) in some samples, we were able to record the data successfully, thanks to the Ge-solid state detector. Also, we used a new graphical interface called as Generic Data Aquisition (GDA) installed at Dubble beamline for the first time and it worked quite well. To this end, we would like to

acknowledge the help from Dr. Dip Banerjee and Dr. Alessandro Longo for their valuable support through out the beam time.

C) Status and progress of evaluation:

Primary data reduction has already been carried out. More detailed analysis and Feff ab-initio calculations and modeling will be carried out in the following months. The primary data analysis suggests to some interesting findings (please see section D for more details) and excellent signal/noise ratio related to excellent beam stability and detector sensitivity.

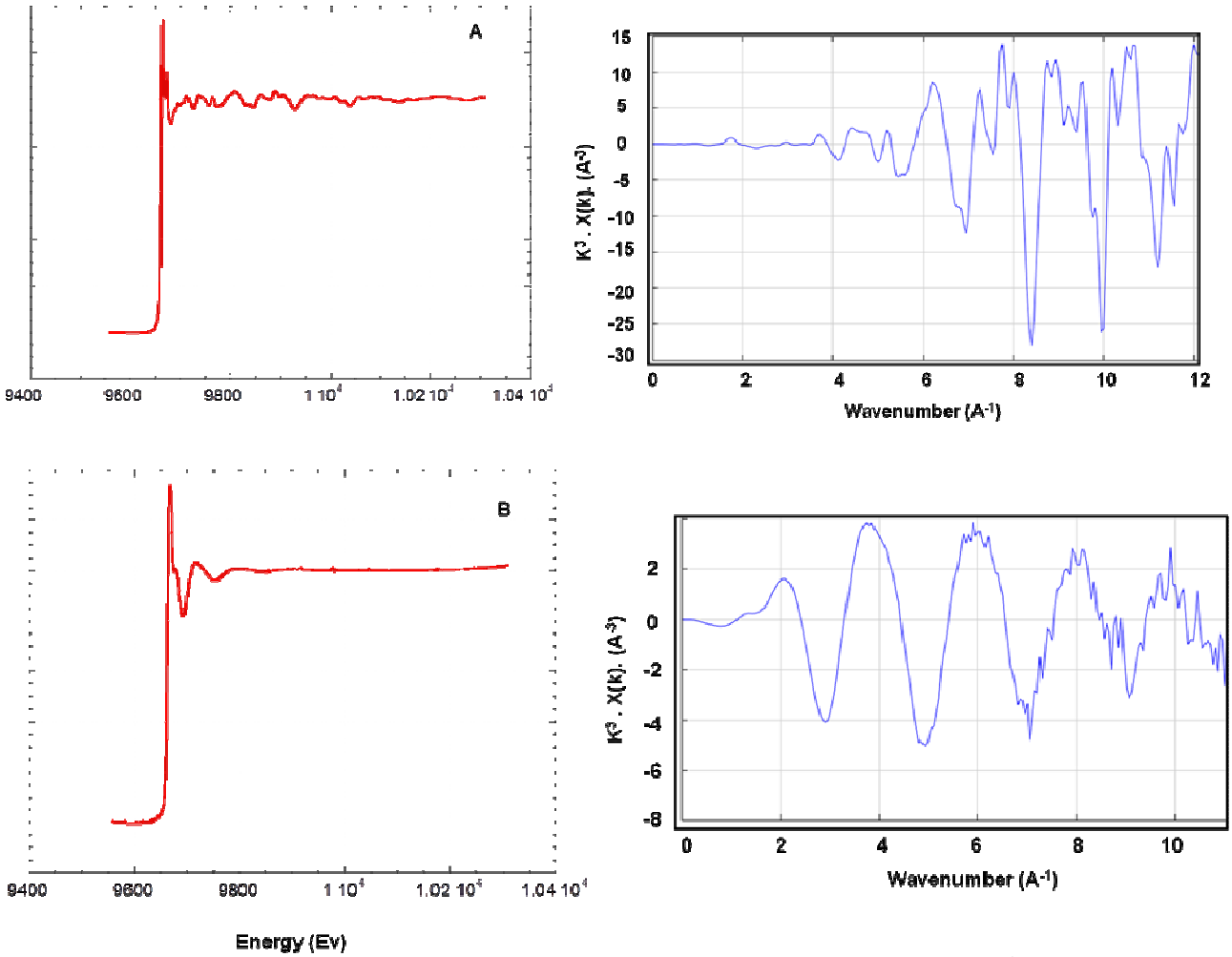

Figure 1. Zn K-edge data collected for (A) model compound with k^3 weighted EXAFS in transmission mode and (B) for a sample at high zinc loading along with k^3 weighted EXAFS in fluorescent mode at 50 K.

D) Results:

The primary data analysis already suggests intriguing results to be debvelopped incl. that Zn is adsorbed to BioSeNPs in different ways under different experimental conditions. In some cases, the $ZnCO_3$ precipitation is possible and in other cases, Zn is linked to oxygen like many organic compounds (Zn-acetate, Zn-lactate and so on). The results obtained at DUBBLE indicates the interaction of Zn is primarily to the polymeric substances attached to the surface of BioSeNPs. This understanding would help us to more closely predict the fate of BioSeNPs in the environment.

References:

1) Jain, R; Gonzalez-Gil, G; Singh, V; van Hullebusch, E; Farges, F; Lens, N. L. P; Biogenic Selenium Nanoparticles: Production, Characterization and Challenges. In *Nanotechnology*; Kumar, A., Govil, J.N.; Studium Press LLC, USA, 2014; pp 361:390 (in press)

2) Jain, R; Jordan, N; Schild, D; van Hullebusch, E; Weiss, S; Franzen, C; Farges, F; Hübner, R; Lens, P; Adsorption of zinc by biogenic elemental selenium nanoparticles. Resubmitted to *Environmental Science & Technology*