

## 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 using the **Electronic Report Submission Application**:

*<http://193.49.43.2:8080/smis/servlet/UserUtils?start>*

### ***Reports supporting requests for additional beam time***

Reports can now 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.



	<b>Experiment title:</b> Study of enhanced arsenic adsorption by nanoparticles	<b>Experiment number:</b> CH - 3099
<b>Beamline:</b> BM-25A	<b>Date of experiment:</b> from: 08/06/2010 to: 12/06/2010	<b>Date of report:</b>
<b>Shifts:</b> 12	<b>Local contact(s):</b> Jon Ander Gallastegui Mañaricua	<i>Received at ESRF:</i>
<b>Names and affiliations of applicants (* indicates experimentalists):</b> * <sup>1</sup> Diego Morillo (Main Proposer) <sup>1</sup> Francisco Blanco  <sup>1</sup> Centre GTS, Department of Chemistry, Universitat Autònoma de Barcelona, 08193 Bellaterra (Barcelona), Spain		

## Report:

Arsenic biogeochemistry has received public and scientific attention due to environmental and public health disasters around the world. Numerous remediation methodologies have been developed to remove As from drinking water (membranes, coagulation, anion exchange, etc.) although adsorption is the most commonly used due to its efficiency, capacity and relative low cost. Many different materials are being used for this purpose and the development of nanotechnology at the end of 20th century has widened the variety of adsorbents in several industrial and environmental applications. The nanosized iron oxide materials take advantage of the affinity of Fe(III) for aqueous inorganic arsenic species. The small size of nanoparticles provides several advantages, mainly related to the extremely high surface area which allows improving the removal capacity several orders of magnitude [1].

In this study, we carry out the contact of superparamagnetic iron oxide nanoparticles (SPION) with synthetic arsenic solutions in form of arsenate, arsenite or mixture of both. The study of the processes controlling arsenic adsorption expects to provide information to optimize the load of the cellulose sponge with SPION and get adsorbent system with high loading capacity which proves the potential application to water treatment purification. The objective of this study at ESRF synchrotron is to characterize the arsenic adsorbed in the SPION loaded over the sponge and it is expected to evaluate the arsenic speciation in order to develop a methodology to study arsenite and arsenate in the same sample. To achieve this objective we applied synchrotron radiation techniques at ESRF based on X-Ray Absorption Near Edge Structure (XANES).

The first step of this study was the preparation of the experiment and the samples. To study the arsenic loading capacity of the system, different concentration solutions of As(V) and As(III) were mixed with SPION loaded sponge in batch experiments. The sponge samples were dried, homogenised, mixed with polyethylene and converted into pellets by hydraulic pressure to be analysed at the experimental station of the synchrotron facility and encapsulated in kapton foils in order to avoid direct contact with the atmosphere.

The absorption of As was recorded at the edge energy for its K line at 7112eV. The selection of the detection mode depends upon the sample concentration and the matrix background [2]. Therefore, reference compounds were analysed by transmittance mode and the unknown samples were analysed by fluorescence detection mode (multi-element solid state detector). Speciation data was obtained by comparing the spectra from pure compounds, NaAsO<sub>2</sub>, NaHAsO<sub>7</sub>, with the spectra obtained in the sponge pellets. The reference compounds were prepared in the same way of the samples.

## Results

Results obtained for the analysis of the reference compounds diluted in polyethylene are presented in [Fig 1](#).

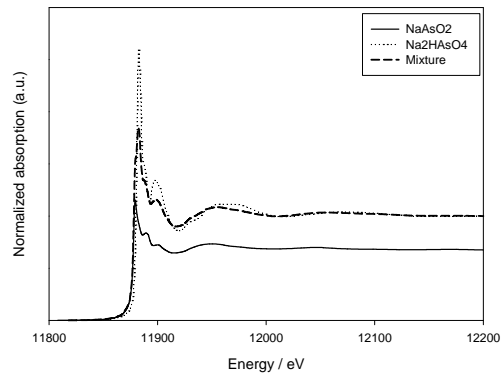


Figure 1: a) XANES spectra of three reference compounds.

The direct comparison of XANES spectra for arsenic standards and arsenic adsorbed in the static adsorption mode studies (shown in Figure 2) was made. The first study was realised with the different species alone. The results show that the As(V) spectra at pH=3.6 were similar than reference compound (Fig 2a) but As(III) spectra were different (Fig 2b). This difference was due to the interaction between Fe(III) of SPION and As(III) [3].

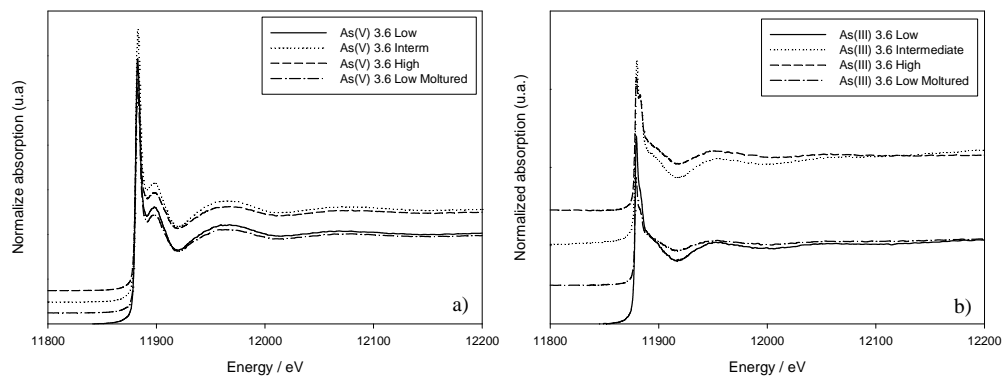


Figure 2: XANES spectra of the As(V) (a) and As(III) (b) adsorption over SPION loaded sponge.

The effect in the adsorption process of presence of the two species of arsenic was studied for the speciation study as show the Figure 3a. Then, by applying a PCA a linear combination fit will be carried out to study the contribution of each As reference species (Fig 3b). The results confirm that Arsenate specie is predominant (97%) which is easily removed of the contaminated water with this adsorbent system.

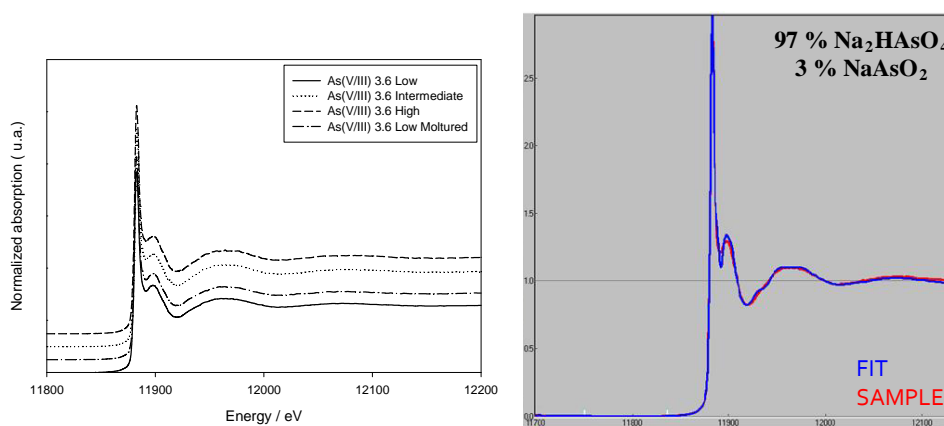


Figure 3: XANES spectra of the As(V/III) adsorption over SPION loaded sponge (a) and PCA analysis for the As(V/III) adsorption (b).

## References

- [1] Muñoz, M.; Gonzalo, A.; Valiente, M. *Arsenic(V) removal from aqueous solutions by iron(III) loaded chelating resin*. *Environ, Sci. Technol.* **36** (2002).
- [2] D.C. Koningsberger, R. Prins, *X-ray absorption*. Wiley, New York (1988).
- [3] Manning, B. A., Fendorf S. E., *Goldberg S. Surface Structures and Stability of Arsenic(III) on Goethite: Spectroscopic Evidence for Inner-Sphere Complexes*. *Environ. Sci. Technol.* **32** (16), 2383-2388 (1998).