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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:** Health effects of crystalline silica. XAS study of Fe in artificial stone and in lung tissues	**Experiment number:** CH4476
Beamline:	**Date of experiment:** from: 04/11/2015 to: 09/11/2015	**Date of report:** 30/02/16
Shifts: 18	**Local contact(s):** Giovanni Lepore	*Received at ESRF:*

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Report:

Aims

The proposal was aimed at gaining insight on the Fe associated to crystalline silica materials in relation to its possible health effects. The proposal experimentally focussed on two different topics. Two kind of silica-bearing materials have been investigated: artificial stones and autoptic lung tissues collected from subject died with silicosis. The results are expected to provide a fundamental insight in the understanding of the role played by Fe speciation, namely concerning its presence in unsaturated coordination sites and in the modulation of ROS generation during inflammation of lung cells.

Methods (1): artificial stone

Artificial stone samples brought to the experiment consisted of three different samples per commercial material (raw, dusts from industrial dry polishing, dusts from industrial wet polishing). All considered samples were investigated by XAS spectroscopy in the pre-edge, XANES and EXAFS regions, using the Fluorescence set up.

Methods (2): XAS set up

XAS spectra have been collected at the Fe K-edge (7.112 keV) using Si(311) crystals in the monochromator, at room temperature and in vacuum, to minimise the contribution from air scattering. The Fluorescence signal was collected by a high-purity Ge solid-state detector. Calibration of the energy was obtained in situ through a metal Fe foil measured in a second chamber, at the same time of the samples.

Methods (3): lung tissues

This part of the experiment was highly exploratory, as the best conditions to register a XAS signal coming from the tissue or from the dusts contained in it were not a priori known. For this reasons, three different kind of samples were considered:

1) A human lung tissue in the form of a bulk fragment, analysed as it was;
2) An hystological section of the same tissue, put over a polycarbonate support, to minimise interference at the X-rays between the support and the sample; also this tissue was analysed as it was;

3) Dusts recovered from the tissue after chemical digestion, and put over a cellulose mixed ester filter (as it is usually done for the airborne dust samples). In this case, the sample was put into a kapton bag to preserve it during the investigation.

All samples were investigated in the Fluorescence mode, at room temperature but not in vacuum, because of the presence of biological matter into the vacuum chamber.

Preliminary Results

We got successful spectra of 9 artificial stone samples, which included a so called "ici white" sample, i.e. a sample with peculiarly low Fe content. In all the cases, spectra reveal, at a first consideration, a very complex Fe speciation. Due to the heterogeneous nature of these materials, only a general picture coming out from the comparison of the fits of the 3 sets of samples investigated in this experiment and those obtained on the 4 sets of samples investigated in CH3929 will allow to unravel the complex Fe speciation and to determine the presence and the amount of surface Fe sites with unsaturated coordination able to influence the adverse effects of SiO_2.

Concerning the lung tissues, we got excellent spectra (with very good signal-to-noise ratio) for the dusts and for the massive lung tissue. The spectra of the hystological sections were a little more noisy, but still clearly interpretable. Most of the spectra appear dominated by the feature of a a Fe oxide very similar to ferrihydrite (Figure 1), presumably contained in ferritin units. This behaviour is well illustrated by the Figure here below. Further studies, aimed at localising the Fe speciation with respect to the distribution of the silica particles in the hystological section, are planned.

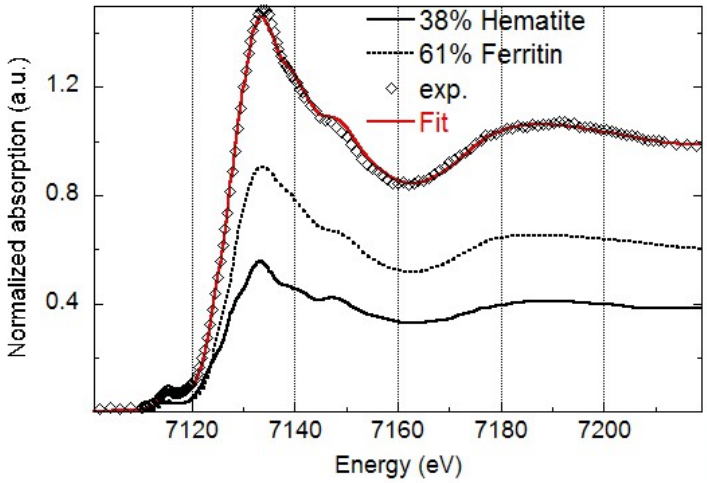

Figure 1.

Linear combination fitting performed on a Fe XANES spectrum acquired on a histological section during a bulk XAS experiment at BM8. Good match with the experimental spectrum of the sample was obtained using a linear combination of the reference spectra of ferritin (61%) and hematite (38%). The spectra of ferritin and hematite are reported scaled by their weight in the linear combination.