Report on the research visit to Utrecht University

April 2010

- Purpose of the visit

I am a beamline scientist at the European Synchrotron Radiation Facility (ESRF) in Grenoble and I am working at the Dubble beamline, a Dutch-Belgian project (NWO-FWO). I have recently started my contract and my specialty is the EXAFS technique. As a beamline scientist I am not only in charge of setting up and operating the beamline, but also to provide assistance to users in interpreting the data and to actively participate in research projects.

The purpose of this trip was to obtain training in fitting of EXAFS spectra and to interpret data obtained from an experiment performed at the DUBBLE beamline. In this experiment the reductive dissolution of iron oxides by sulphide has been investigated. The collaboration also involves the Hydrology Department at the University of Bayreuth in Germany. Furthermore, the visit is intended as a starting point for a collaboration with Dr. Thilo Behrends at the Earth Science department at Utrecht University, The Netherlands.

Thilo Behrends has expertise in applying EXAFS for addressing environmentally relevant problems. On my turn, I have followed a course on EXAFS software from the Ifeffit package in Ghent in January 2010. This visit provided me the opportunity to deepen my knowledge on using these programs and to apply them to real data.

- Future collaboration with host institution

In order to establish collaborations with research groups in Belgium and The Netherlands involving the application of EXAFS I have visited Utrecht in the beginning of 2010. The research line of Thilo Behrends related to EXAFS application to environmental processes interests me very much and we agreed on establishing a long term collaboration. The exchange visit performed within FIMIN is considered the kick-off of this collaboration.

In the meantime, a proposal for another experiment at Dubble has been submitted. If this proposal is accepted, beamtime will be allocated before the end of 2010. This project requires another exchange visit to Utrecht in order to prepare this experiment.

Furthermore, the interpretation of all the previously collected spectra has not been finished yet and we are currently continuing on finishing the interpretation.

- Description of the work carried out during the visit and description of the main results obtained

The data that was analysed here, is from a successful experiment that took place at Dubble in October 2009.

The experiment consisted on collecting on-line XAFS and WAXS data from suspensions during the reaction of the iron oxide lepidocrocite (γ-FeOOH) with sulfide S(-II).

In a short description, the strategy used for the data analysis consisted on three steps. In step 1, the spectra of the initial material of known structure, lepidocrocite, was fitted. In the second step, the spectra collected from a sample considered as a likely end-product of complete reaction of lepidocrocite with S(-II) was fitted. A successful fit was obtained by assuming a Fe-coordination similar to that of iron the FeS mineral machinawite. The results are illustrated in figures 1 and 2, where you can see a good agreement between the data in blue and the fit in red for both steps, based on both structures mentioned above (only shown for the real part of the Fourier transformed EXAFS spectra).

The fitting was very time consuming because several approaches were used in the fitting procedure. This was done in order to test several hypotheses but also to familiarise myself in guiding the software in fitting EXAFS spectra.

The third step and the big question motivating this experiment is to identify the structure of lepidocrocite in intermediate steps during the reaction with S(-II) in order to learn more about the reaction mechanism. Our current approach is combining the models obtained from step1 and step 2 for the two end members of the reaction for reconstructing the Fe coordination in intermediate steps.

I have started working on this step but the result is still unknown as it requires more analysis time and turns out to be very complex.

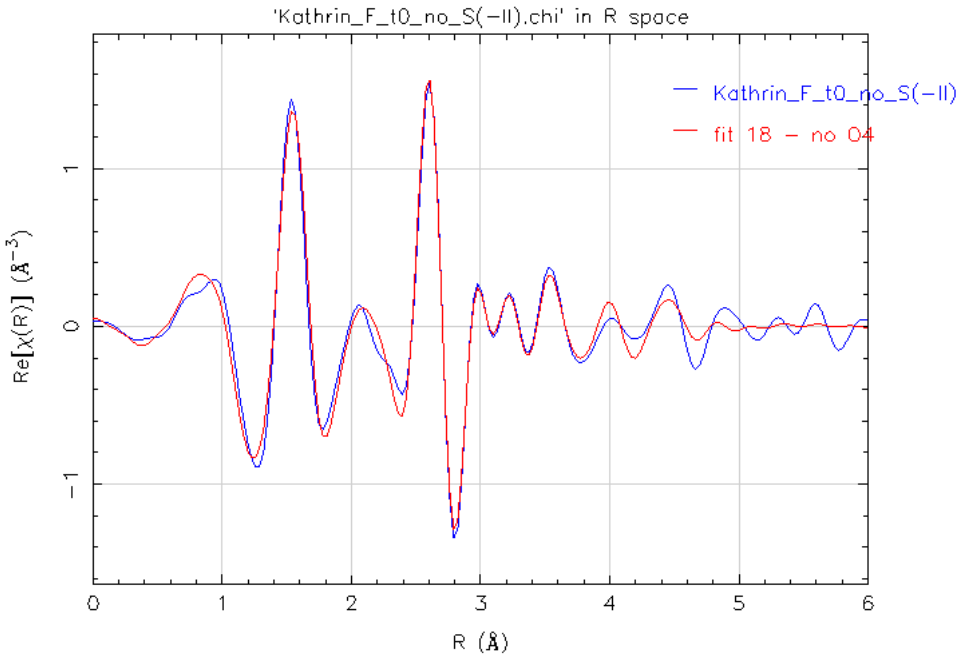

Figure 1 – Data and fitting curve – beginning of reaction

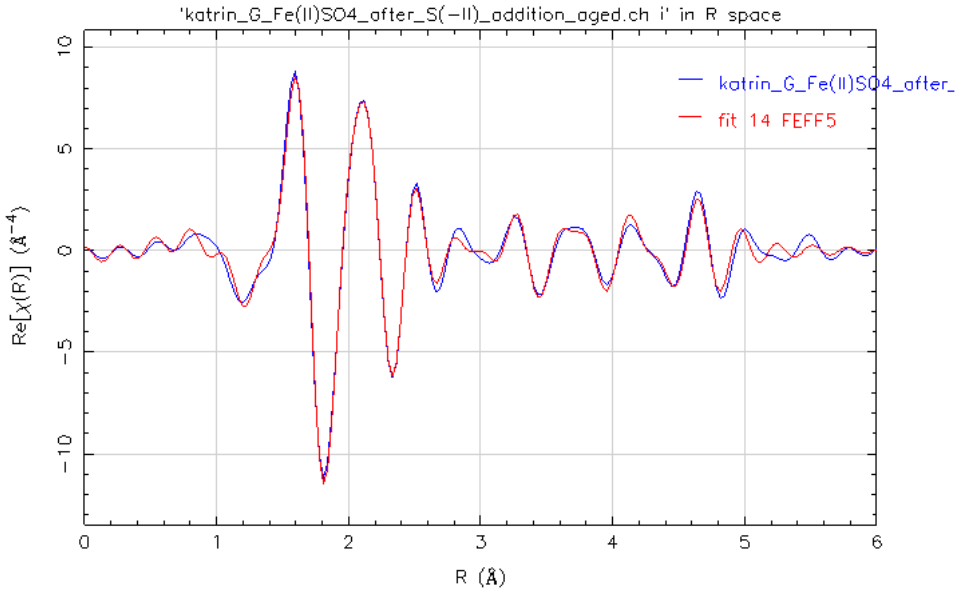

Figure 2 – Data and fitting curve – end of the reaction

- Projected publications/articles resulting or to result from the grant.

The results obtained from the exchange visit were included in a poster presented by Thilo Behrends during the EGU conference in Vienna. After finalizing the fitting of the spectra we plan to publish the results together with the group in Bayreuth in a peer-reviewed international journal. In particular, qExafs data collected during the same experiment, can bring relevant or detailed information regarding intermediate stages of the reaction.

- Other comments (if any)

One particularity of the EXFAS technique, is that it requires knowledge about the system you are studying, in order to be able to analyse data. Therefore long-term collaborations are more fruitful and this is the ultimate aim of this project.

To conclude, in my opinion the research visit in Utrecht was very useful. I have significantly improved my skills in fitting EXAFS spectra and I have learned a lot about the background of the research project and the relevance of the research questions.

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