

Project name: **Study of atomic composition of waterwork sludge from Sydsvatten treatment plant Ringsjöverket with x-ray absorption spectroscopy**

Beamtime Report

dd.mm.yyyy - dd.mm.yyyy (Date of the report to be added)

General information

Name of the rapporteur	Name of the rapporteur's organisation
Kenneth M Persson	Sydvatten AB
Type of research (nanotechnology/health care/chemistry etc.)	Name of the research facility
Chemistry	MAX IV LAB
Date of the measurement, duration	Location of the event
17 Sept 2013	MAX IV LAB, Lund University, Lund, Sweden
Facility personnel participating in the measurement	
<i>Kajsa Sigfridsson and Stefan Carlson</i>	

Description of the project

Research description (short summary as written in the application)
The specific aim has been to increase knowledge about how water works sludge is composed at the atomic level. To be able to do so, an x-ray absorption spectroscopy method has been applied to thickened waterworks sludge from the drinking water production during the months August and September 2013.
Summary of activities (experiments performed, beamtime used, preliminary overview of results, next steps and other relevant information)
<p>The primary method was the Beamline I811 of MAX-II, which is used for materials science research in the photon energy range 2.4 - 21 keV. This corresponds to a wavelength interval of 0.6 - 5 Å. The method allowed to measure the bonding distances for iron-iron, iron – oxide and iron- hydroxide in the sludge. The speciation of iron in the sludge samples could also be investigated.</p> <p>XANES The Fe K-edge from the filter compound (black) was compared to ferrihydrite (red).</p> <p>The edge was similar in shape and position to ferrihydrite (Fe₂O₃ x 0.5H₂O), a common hydrous ferric (Fe³⁺) oxide mineral that can be expected to be found in iron-rich aqueous solutions. The source of ferrihydrite can be inorganic as well as biological (used for example in cellular iron storage in most</p>

living organisms). Nanocrystals of ferrihydrite adsorbs for example humic acid, expected to be present in the sludge sample. A humus sample would have been an interesting reference sample. In the literature, Fe^{3+} was mixed with humic acid (PPHA) to different concentrations (high and low) and pH and the Fe coordination was studied with XAS (Karlsson & Persson (2010) *Geochimica et Cosmochimica Acta* 74, 30-40). The PPHA samples with high $[\text{Fe}^{3+}]$ (49 200 $\mu\text{g/g}$) and pH 6.9-5.6 had indeed XANES and EXAFS spectra similar to the filter sample. In the paper these samples were described as >50% in organic complexes with a 5-membered chelate ring structure (similar to desferrioxamine B) and the rest was some ferric hydroxide. This description could fit very well also here.

EXAFS

The EXAFS of the filter sample was best fitted with a model (fit c) mixing characteristics of ferrihydrite (Fe-Fe, 3.07 Å and 3.49 Å) and humic acid bound Fe^{3+} (Fe-C, 2.95 Å and 3.77 Å). The chi data was fitted to $k=13 \text{ \AA}^{-1}$ (see annex 1). The coordination numbers in fit c are uncertain, but the found distances and proportions are very similar to what was found in the above mentioned PPHA samples with high $[\text{Fe}^{3+}]$ (49 200 $\mu\text{g/g}$) and pH 6.9-5.6. Fe-C shells and a Fe-Fe shell could be observed.

To summarize, in the first shell Fe is coordinated by oxygen at 1.98 Å distance. The $N \sim 3$ is low, when adopting the amplitude reduction factor 0.96 from the Karlsson & Persson paper. The O-shell in our sample might be ill defined, e.g. combination of many similar distances, which would reduce the amplitude of the FT peak. Fe is probably coordinated by 5 or 6 oxygen ligands anyway.

How would you describe cooperation and assistance from industrial liaison officers and national contact points while preparing and carrying out the research at large scale facilities?

The facilitators have facilitated the work substantially. Particularly Andreas Lassesson has helped Sydsvatten finding the right way forward for Sydsvatten.

Other personal remarks

Annexes

Annexes

(list of annexes; meeting minutes, graphical illustrations, tables and other supplementary data)

Annex 1: Fe K-edge XAS on waterworks sludge