In situ, Time Resolved Small Angle Solution X-ray Scattering: Nucleation and growth of iron based colloids

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Introduction:

The migration of toxic contaminants (cations, anions, and organics) in many near-earth surface environments is controlled by their interaction with poorly ordered iron sulphide (mackinawite) and iron oxide (ferrihydrite) phases. However, the mechanisms and kinetics of the nucleation and growth of colloidal Fe-S and Fe-OH particles from aqueous solution are poorly known.

Goals and means:

Here we present results from synchrotron-based, time-resolved Small Angle X-ray Scattering (SAXS) experiments that followed the nucleation and growth kinetics of Fe-based colloids from aqueous solution.

Experimental methods:

stopped flow system

Experimental conditions

light source: undulator source (flux ~ 8x10¹⁰ ph/s); low divergence;

stopped flow system

fast and accurate mixing,

10 ms deadtime,

T - 25 - 80°C

axial or anaxial

Experimental conditions

Fe⁴⁺, Fe⁵⁺; H₂S, KOH; ratios 1:1 to 1:10

20 - 500 milliseconds / scan

2 sec - 60 sec total time / run

Results:

On the left, a 3-dimensional plot of the results from an experiment where a Fe⁴⁺ and a KOH solution were mixed at a ratio of 1:5 and where SAXS patterns were collected every 20 milliseconds is shown.

The nucleation of FeOOH particles occurs very fast (~120msec) while the growth can be followed for the whole length of the experiment.

In the Fe-S system at a mixing ratio of 1:5 the nucleation (~2sec), growth or aggregation (~25sec) and the possible re-dissolution is shown.

Kinetic rates and mechanisms were derived from experiments (and from the calculated Rg and Io) in various mixing ratios via an Avrami type equation with α normalized to 1.

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Summary

In environments where the growth of such colloids affect the behaviour of toxic trace elements (e.g., acid mine drainage, waste water treatment), this kinetic data can be used in planning the nature of the remediation procedures.