SAXS/WAXS studies of the precipitation and crystallisation of iron and aluminium (oxy)hydroxides

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Introduction and methods

The precipitation and crystallisation of ferric and aluminium (oxy)hydroxide colloids occurs in many natural environments. The sorption of pollutant by these particles often controls their transport and bioavailability in groundwater.

The formation mechanisms and kinetics of both poorly ordered (e.g. ferrihydrite and psuedo-boehmite) and crystalline (e.g. goethite and bayerite) ferric and aluminium (oxy)hydroxide have been studied using time-resolved *in situ* synchrotron based Small and Wide Angle X-ray Scattering (SAXS/WAXS) techniques. The effects of pH, ionic strength and the addition of contaminants (e.g. phosphate) have been studied. The main aim being to examine the reaction processes on various length scales from Å's (WAXS) to 100's nm (SAXS).

Results

SAXS data for the crystallisation of aluminium (oxy)hydroxide from solution at 70ºC under alkaline (7.5-11.2) conditions indicates a two stage crystallisation process (fig.1). The initial stage is marked by rapid growth followed by a slower growth/crystallisation step.

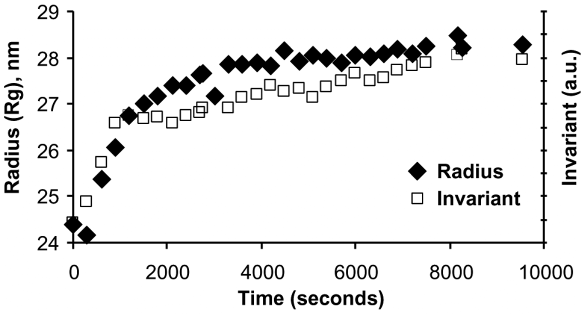

Figure 1. Particle size (Rg) and SAXS invariant data for the crystallisation of Al-(oxyhydr)oxide at 70ºC and pH 10.8.

In situ studies of the precipitation of ferrihydrite using a rapid injection stopped-flow cell have provided quantitative kinetics for reactions occurring on time scales of <1 to 4 seconds. In general the reaction rate decreases with increasing pH. SAXS/WAXS studies of the crystallisation of hematite and goethite from ferrihydrite will also be presented.

Mineralogy and Paragenesis of Sulfide Chimneys on the southern East Pacific Rise between 7 to 21 South

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The southern East Pacific Rise axis is known as one of the fastest spreading plate boundary. Neo-volcanism in form of pillow - and sheet lava culminate in hydrothermal plum activity. A cyclic 4-stage development has been suggested for the hydrothermal activity (Holler, Marchig et al 1990). Sulfide chimneys in form of "black smokers" from 11 locations have been investigated. They represent single chimney to a group of smokers, to an assembly of collapsed chimneys. The material was collected during Geometep 4 and 5 on board R V Sonne. The investigated chimneys show variable height and thickness. They comprise weathered basaltic material and massive sulfide assemblage of iron, copper and zinc., and represent friable to hard material. Major sulfide minerals present in them are pyrite, chalcopyrite and sphalerite. Intermediate mineral product includes marcasite, pyrrhotite, chalcopyrrhotite,covellite, isocubanite. Most common textural pattern of these sulfides is colloidal or gel material which is represented by a rhythmic banding of pyrite in alternation with gangue material. Concentric alternation layering of pyrite and gangue material is also seen where the core consists of either pyrite or gangue. Pressure solution effects during layering of pyrite suggests solution effects even after the main phase of mineralization. 3-generations of sulfide minerals could be observed which represent both a high and a low temperature association of them. Pyrite of first generation (Py I) is euhedral with numerous inclusions often linear arrangement is a typical of them. Blebs of rounded chalcopyrite in pyrite, replacement of Py 1 by chalcopyrite, pyrite of either second or even third generation (Py 2, and Py 3), and later by marcasite are a common feature. Sphalerite frequently shows skeleton texture in which previous generation of sphalerite(Sp 1) has been replaced by them., allowing only remnant sphalerite to occur in isolated pockets. Subsequent low temperature emanations result in the precipitation of colloidal and gel material and amorphous silica producing alternation bands of pyrite(Py 2) and gangue of silica.. The grain margins in low temperature pyrite grains(Py 2) is rounded. Inclusions in Py 2 have random pattern. Even Py 2 cross cuts Cp 1 suggesting its later phase. Pyrite of 3 generation(Py 3) is characterized by cross relationship with rhythemic and concentric layering, often filling the cracks and parbally replacing Py 2. Generally a fewer inclusions are present in Py 3 grains. Marcasite replaces Py 2 both along the boundary as well as in a gel pyrite - gangue mosaic. Chalcopyrite of 1 generation (Cp 1) is euhedral to granular Cp 1 is also inter grown with Py 1 , and present as included grains in Py I and Py 2. In high temperature assemblage.Covellite along the Cp 1 margin shows conversion from Cp 1 at lower temperatures.