In situ AFM imaging of dissolution and growth of struvite surface

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Outline

- Introduction
- Synthesis of struvite crystals-Characterization analysis
- In situ flow-through AFM experiments (dissolution & growth)
The crystallization of struvite, $\text{MgNH}_4\text{PO}_4\cdot6\text{H}_2\text{O}$ is relevant

• for nutrient (nitrogen and phosphorus) recovery in water purification
• scale formation in pipelines in Wastewater treatment plants
• a primary component of so-called *infection stones* arising from urinary tract infections

- Numerous studies focus on fundamental aspects of struvite crystal nucleation and growth at near molecular level → Knowledge of these processes can aid in the design of conditions that either promote or inhibit struvite formation, depending on the application

- The **aim of this study** is to investigate at nanoscale, dissolution and growth of struvite by in situ AFM experiments
Synthesis & Characterization analysis

- Obtained struvite-type crystals by single diffusion gel growth technique
- SEM, XRD, Raman

- \( \text{Na}_2\text{SiO}_3\times\text{H}_2\text{O} \) (25\% w/w) in drops in \( \text{NH}_4\text{H}_2\text{PO}_4 \) 0.5 M
- \( \text{Mg(CH}_3\text{COO)}_2\cdot4\text{H}_2\text{O} \) 1M
In situ AFM experimental procedure

- Cleaved struvite crystals along (100) plane to expose a fresh surface
- Flow through experiments were performed in a fluid cell at (22 ±1 °C)
- Effective flow rate of fresh solution (22µL•s⁻¹)
In situ AFM dissolution experiments

- Deflection images of struvite surface in deionized water at different pH values (dissolution)
- At low pH values: deep etch-pits formation
- At high pH values: low dissolution rates
Height-Deflection images of struvite surface in supersaturated solutions, $\text{NH}_4\text{H}_2\text{PO}_4/\text{MgCl}_2$ (growth)

In situ AFM growth experiments. I

pH 5, SI 0.28

pH 8.1, SI 0.26

pH 8.1, SI 0.49
At low pH values: increased growth rates

At pH 8: differences in rates imply differences in mechanism
Conclusions

Nanoscale observations reveal..

- Dissolution experiments: Elongated etch pits at low pH values, whereas at equilibrium pH 8 evolution of etch pits were retarded.

- Growth experiments: Step growth at low supersaturation to 2-dimensional layer generation and spreading at high supersaturation.
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