In situ AFM imaging of dissolution and growth of struvite surface

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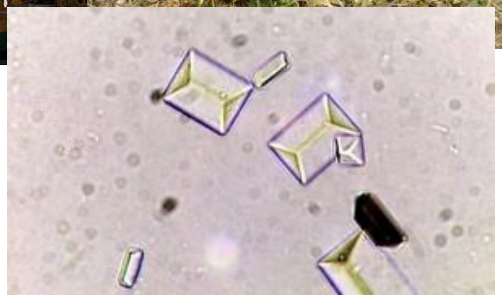
Outline

- Introduction
- Synthesis of struvite crystals-Characterization analysis
- In situ flow-through AFM experiments (dissolution & growth)

Introduction

Amsterdam West WWTP



The crystallization of struvite, **$MgNH_4PO_4 \cdot 6H_2O$** is relevant

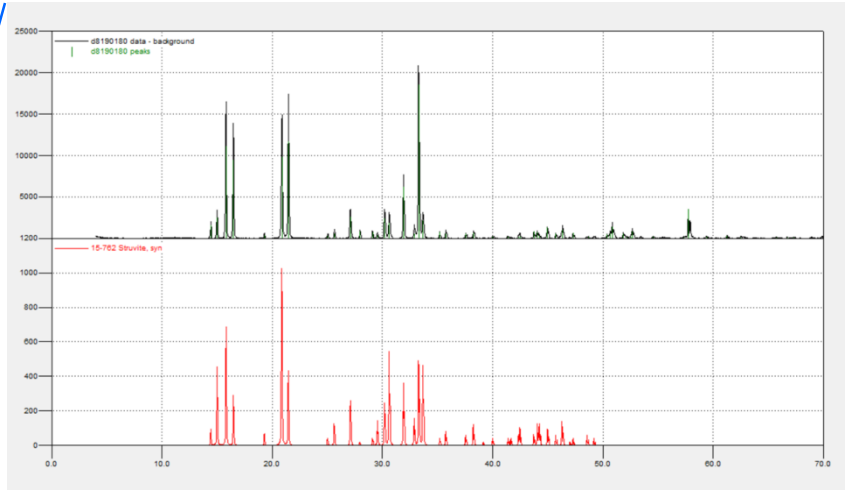
- for nutrient (nitrogen and phosphorus) recovery in water purification
- scale formation in pipelines in Wastewater treatment plants
- a primary component of so-called *infection stones* arising from urinary tract infections

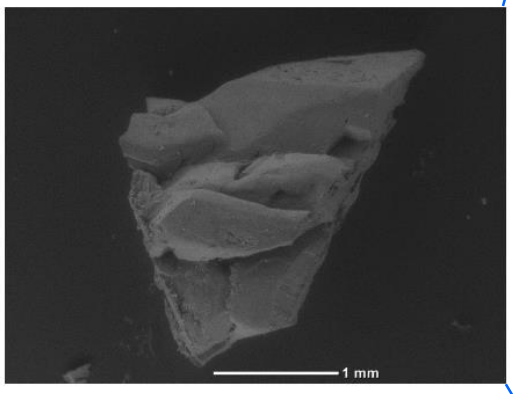
➢ Numerous studies focus on fundamental aspects of struvite crystal nucleation and growth at near molecular level → Knowledge of these processes can aid in the design of conditions that either promote or inhibit struvite formation, depending on the application

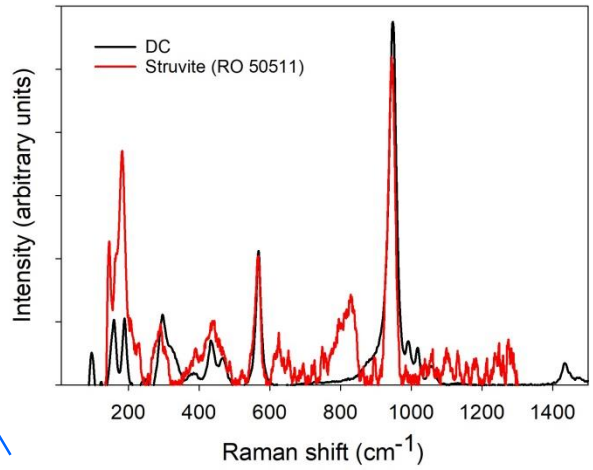
➢ The **aim of this study** is to investigate at nanoscale, dissolution and growth of struvite by in situ AFM experiments

Synthesis & Characterization analysis

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



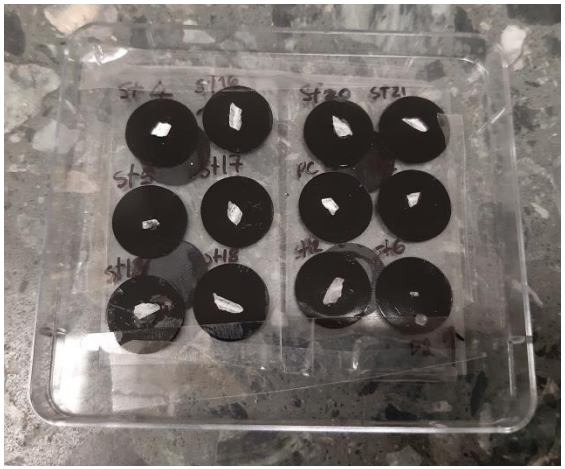


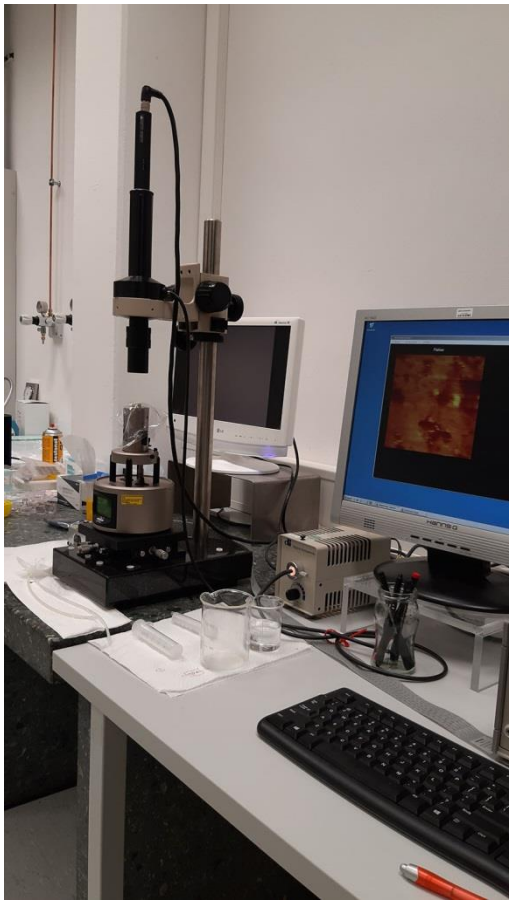


- Na_2SiO_3xH2O (25% w/w) in drops in $NH_4H_2PO_4$ 0.5 M
- $Mg(CH_3COO)_2 \cdot 4H_2O$ 1M

In situ AFM experimental procedure

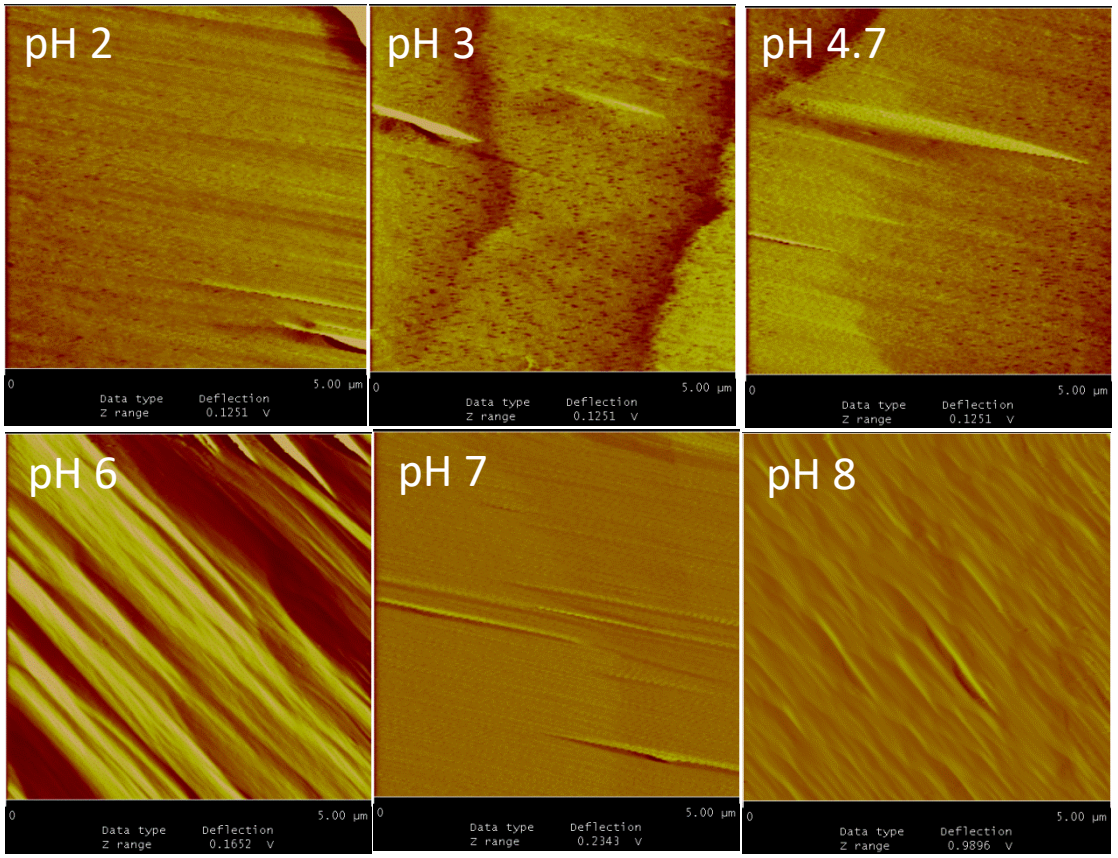
- Cleaved struvite crystals along (100) plane to expose a fresh surface
- Flow through experiments were performed in a fluid cell at (22 ±1 °C)
- Effective flow rate of fresh solution (22µL•s^{-1})

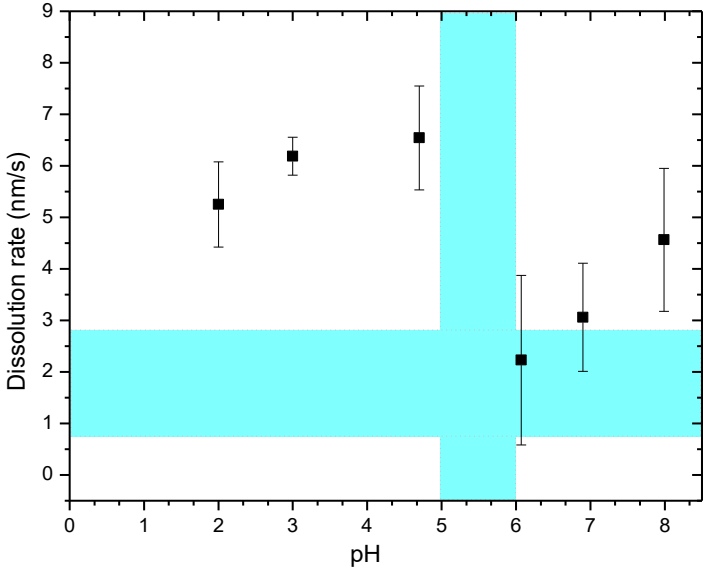




In situ AFM dissolution experiments

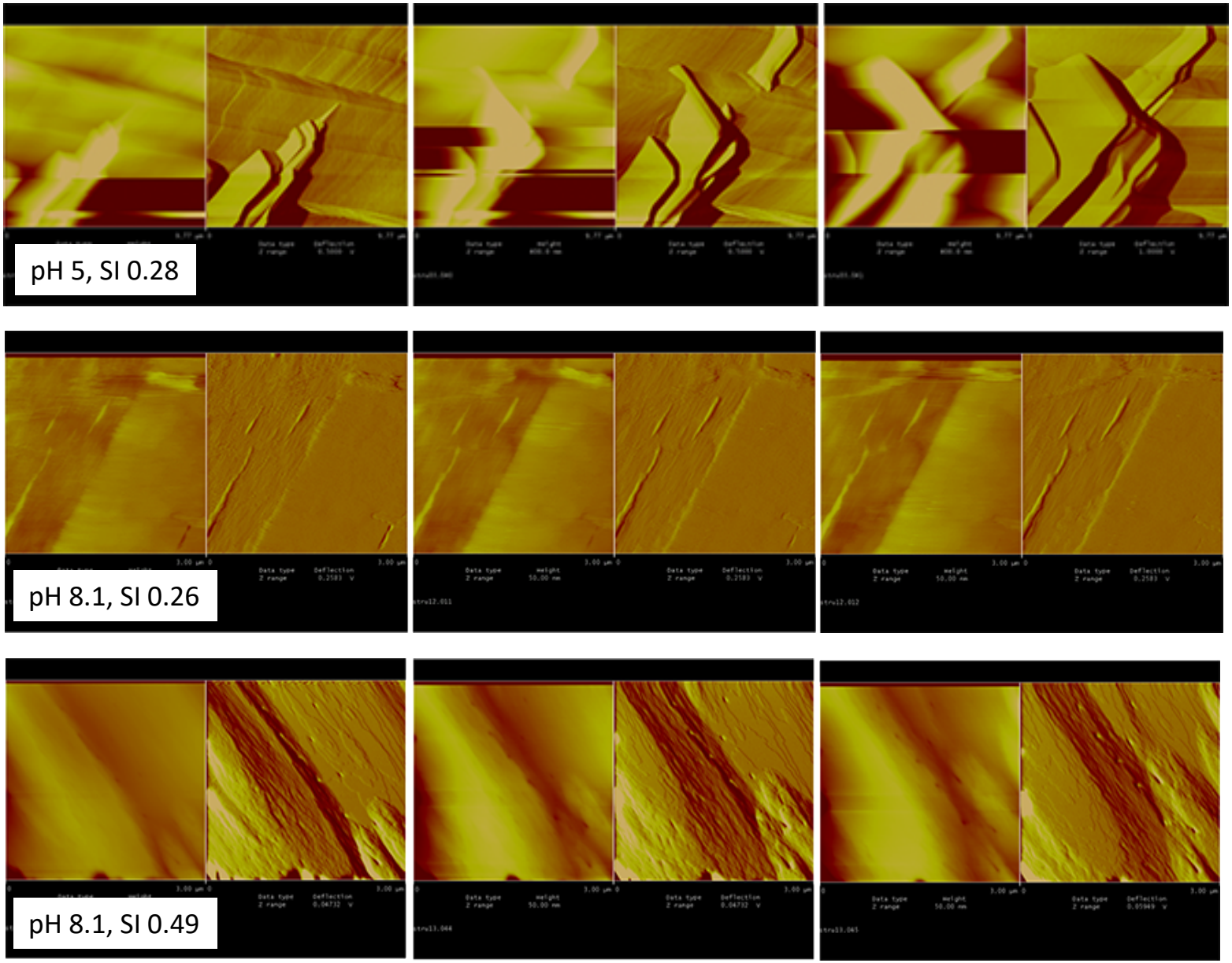
- Deflection images of struvite surface in deionized water at different pH values (dissolution)
- At low pH values: deep etch-pits formation
- At high pH values: low dissolution rates



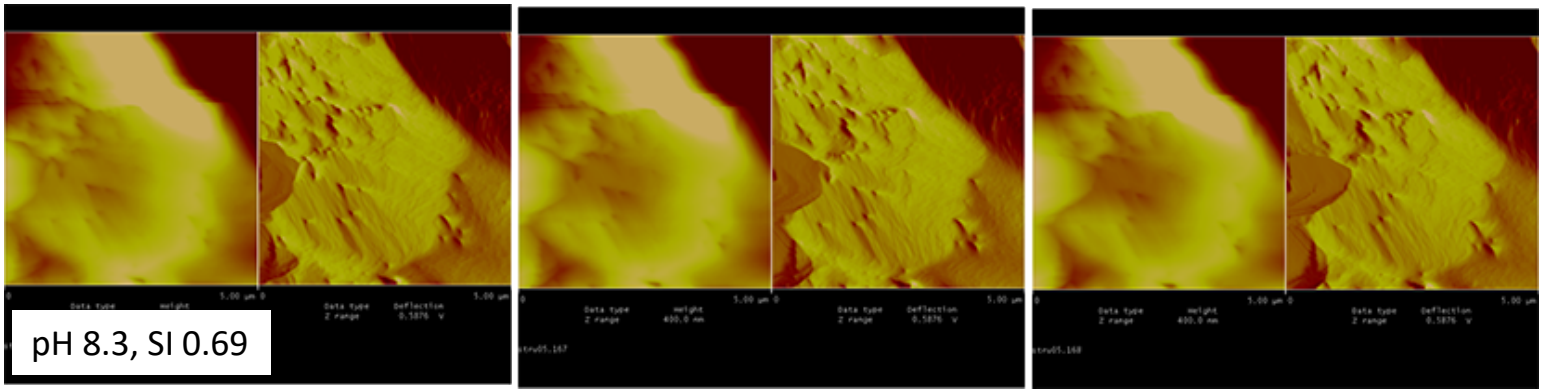


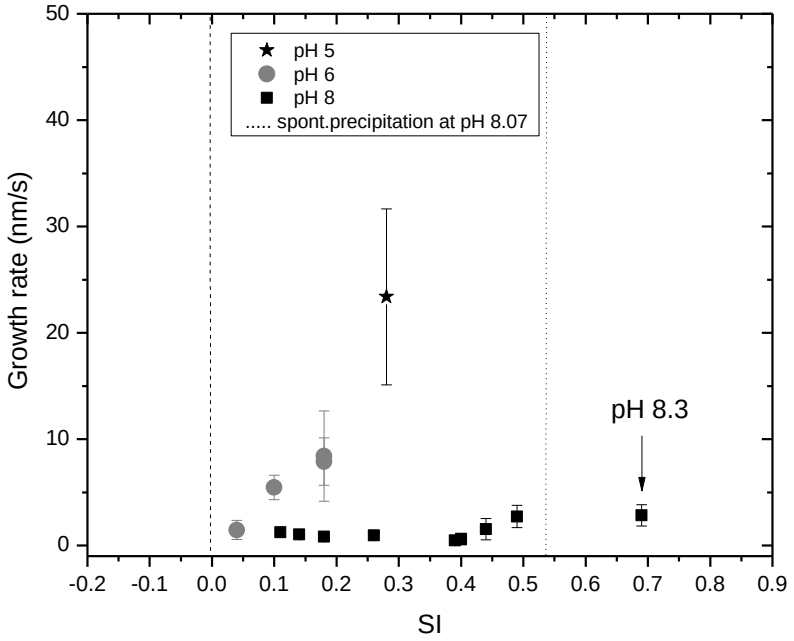
In situ AFM growth experiments. I

Height-Deflection images of struvite surface in supersaturated solutions, $NH_4H_2PO_4/MgCl_2$ (growth)

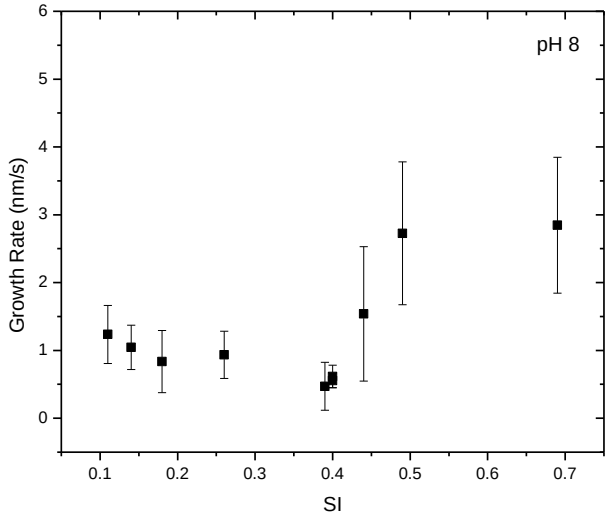


In situ AFM growth experiments. II





- 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

Acknowledgements

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