INDUSTRIAL PILOT PROJECT

In-situ WAXS/SAXS analysis of formation and stability of amorphous calcium phosphate for dental applications

THE INDUSTRIAL CHALLENGE

Psilox AB develops and produces amorphous calcium phosphate (ACP) micro-particles for use in dental applications. The particles promote tooth mineralization and may be used to reduce sensitivity by occluding exposed dentin tubules. The synthesis occurs by precipitation from a heated salt solution, but the nucleation and growth processes are not yet completely understood, which motivated an attempt to analyze the precipitation of particles in-situ.

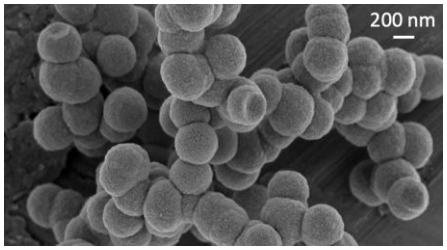

Figure 1. FEG SEM micrograph of amorphous calcium phosphate micro-particles.

WHY USING A LARGE SCALE FACILITY

At a high energy synchrotron source, a broad range of q-space can be probed using both wide and small angle X-ray scattering (WAXS/SAXS), and via Fourier transform of the diffraction pattern and pair distribution function (PDF) analysis, one could gain structural information, i.e. distribution of relative interatomic distances, even on amorphous materials. This overperforms the conventional X-ray diffraction technique (XRD) that is more suitable for crystalline materials. The high photon flux of the synchrotron also enables the fast scans required to capture nucleation, growth, or phase transformation of the particles in-situ.

HOW THE WORK WAS DONE

A small-scale reaction vessel with a viewing window for the X-ray beam was designed to enable in-situ analysis at beamline P21.2 at PETRA III in Hamburg. The set-up also enabled heating and stirring of the solution in order to track the phase development with increased temperature. Simultaneous and rapid acquisition of WAXS and SAXS data was performed during heating, and tests using varied ion concentrations in the reaction solution were performed.

The in-situ analysis was complemented by ex-situ analysis of particles in powder form. The on-site assistance and expertise of P21.2 beamline scientists Ulrich Lienert and Malte Blankenburg is greatly acknowledged.

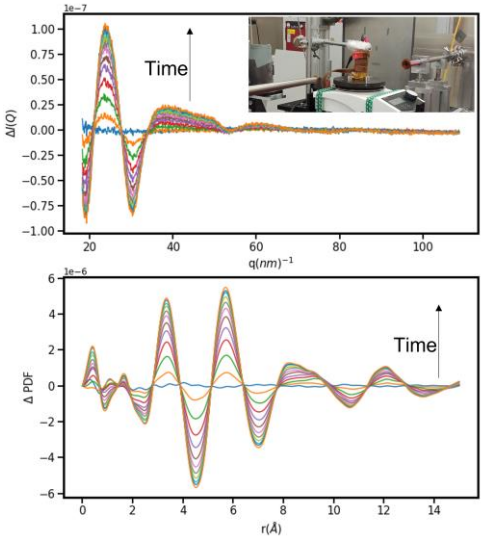

Figure 2. In situ experimental result of the WAXS of calcium phosphate with low magnesium doping: (top) differential scattering intensity and (bottom) differential PDF. Insert: set-up with the in-situ reaction vessel placed in the beam path of P21.2.

THE RESULTS AND EXPECTED IMPACT

From the in-situ experiment, we followed atomic scale development of the amorphous structure during precipitation. Compared to conventional analysis, we could observe the total scattering factor difference as a function of wavevector (q), i.e. scattering angle. After Fourier transformation of the scattering patterns via PDF analysis, we could demonstrate the evolution of interatomic spacing during nucleation and growth (see Figure 2). A positive intensity means an increased number of atoms separated at this distance, while negative values mean decreased numbers. The results will provide insight into the nucleation of ACP clusters and help refine the synthesis parameters in terms of optimal reaction temperature and time.

"The project has provided our small company with the rare opportunity of analyzing key process events with incredible detail." /Erik Unosson, Psilox AB

Contacts: Erik Unosson – Psilox AB, erik.unosson@psilox.com
Wei Xia – Uppsala university, wei.xia@angstrom.uu.se, Shun Yu – RISE, shun.yu@ri.se

Vinnova's project No: 2019-02565 **Duration:** August 2019 -- October 2022

Funded by Sweden's Innovation Agency, Vinnova, in order to build competence and capacity regarding industrial utilisation of large-scale research infrastructures such as MAX IV and ESS.