

# **X-RAY DIFFRACTION AND COMPLEMENTARY ANALYTICAL TECHNIQUES INCLUDING MICROSCOPY, RAMAN, AND SMALL ANGLE X-RAY SCATTERING**

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Several case studies performed at the University of Minnesota Characterization Facility involving x-ray diffraction (XRD) in conjunction with other analytical techniques will be discussed including analysis of amorphous trehalose compacts, kidney stones, drug-embedded polymer coatings, and crystal growth in nanoporous matrices. Analytical techniques used to evaluate the materials include: atomic force microscopy (AFM), Raman spectroscopy, optical microscopy, and small angle x-ray scattering (SAXS).

Case 1: Amorphous trehalose compacts were stored at 43% RH (at room temperature) and evaluated by AFM, XRD, and Raman spectroscopy. Partial surface crystallization of trehalose dihydrate on the surface of the compacts was detected by AFM but not by XRD or Raman spectroscopy.

Case 2: Kidney stone disease occurs in over 10% of entire US population, but the understanding of its mechanism is not comprehensive enough to devise remedies for its prevention. A well-known hypothetical mechanism involves Randall's plaques in kidneys, which have been claimed as a nucleating point for the most common kidney stone, calcium oxalate. Micro-XRD was used to precisely determine the location, identity, and orientation of the biominerals in the Randall's plaques. Also, the growth of calcium oxalate on the plaques was characterized by AFM to evaluate the hypothesis of overgrowth.

Case 3: Drug-embedded polymer coatings were evaluated using XRD, Raman spectroscopy, and dynamic pulsed force mode AFM. Differences in viscoelasticity and tip-sample adhesion enabled the identification and distribution of components. Imaging under variable humidity and sample temperature was performed which may lead to a better understanding of drug distribution and mobility.

Case 4: Organic crystals were grown in nanoporous matrices to determine if the structure or crystal forms could be controlled. Micro-XRD, optical microscopy, and SAXS were used to study the crystals and the effect of functionalizing the walls of the nanopores.