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Use of 3D X-ray Computed Microtomography to Observe the Structure of Colloidal Zirconia Deposits in Porous Media

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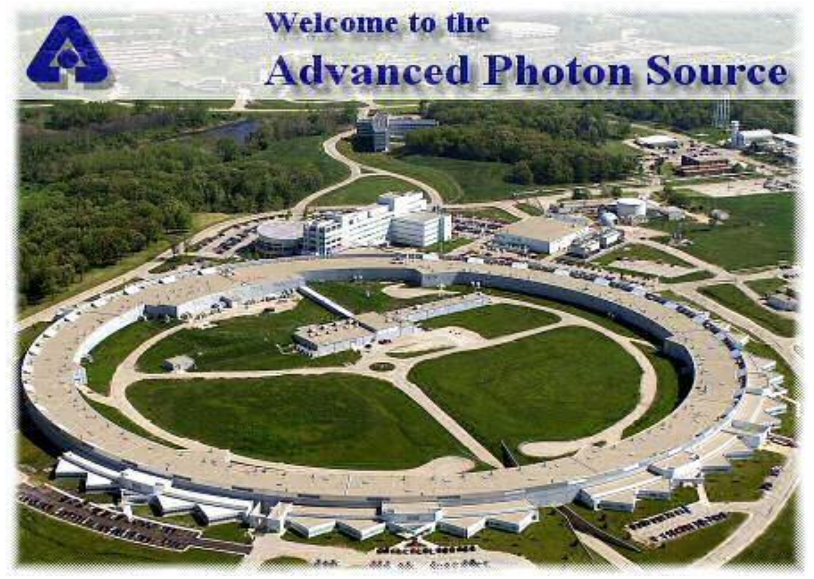
Overview

We are utilizing the tomography capability of the DuPont-Northwestern-Dow Collaborative Access Team (DND-CAT) at the Advanced Photon Source (APS), Argonne National Laboratory for studies of *in situ* sediment structure. Images of a sediment sample are taken at a number of different angles as the incident x-ray beam passes through it, and a three-dimensional view of the interior of the sample is then reconstructed from these maps using computed tomography. These images allow examination of individual sediment grains and the pore structure with a spatial resolution of as little as a few microns. In addition, the distribution of a particular element can be determined by difference tomography, i.e., by obtaining a series of images both above and below the x-ray absorption edge of the element of interest. We used this approach to resolve the distribution of deposited zirconia particles in a matrix of glass beads or silica gel particles. Zirconia particles were deposited in small columns under steady upflow conditions, and the columns were rinsed by several pore volumes of particle-free water. Column experiments were conducted with a range of influent zirconia concentrations and pore water flow rates, and the complete columns were analyzed at the APS. Additional experiments were conducted in a recirculating flume to observe the deposition of zirconia colloids in a naturally formed sand bed. In these experiments, small cores were obtained and transported to the APS for analysis.

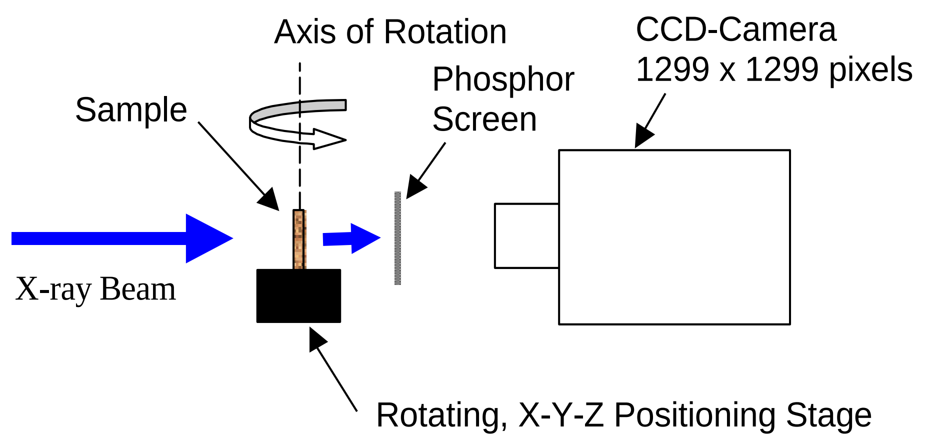


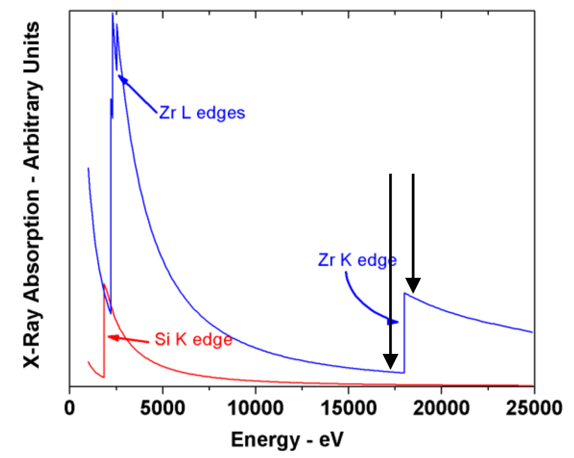
The advanced Photon Source provides a source of hard x-rays that we use to perform computed microtomography (CMT) experiments.

A specific x-ray wavelength is selected by Bragg diffraction off a two crystal Si(1,1,1) mono-chromator. This high flux of monochromatic light allows us to:

- Reach μm-scale resolution.
- Collect data on a short time period.
- Avoid beam hardening effects.

Principle of Z-Contrast X-ray tomography

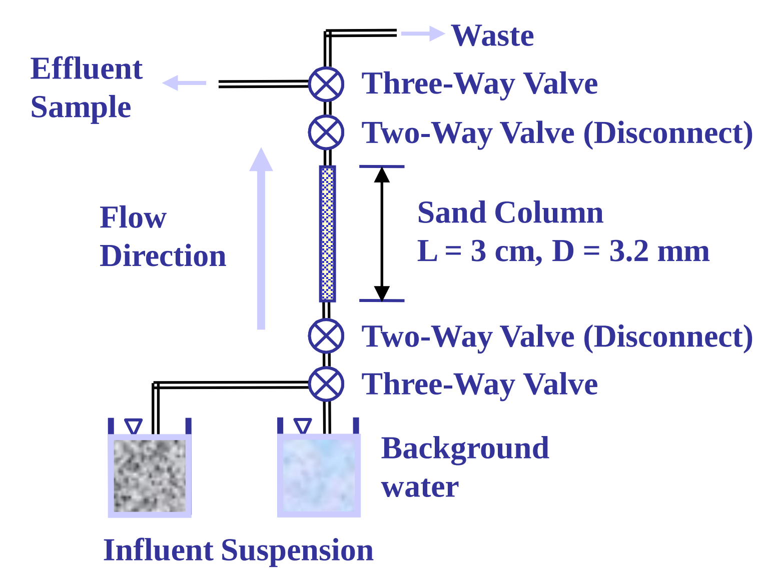




- 2D X-ray absorbance maps of a sample are obtained at different angles
- Tomographic reconstruction converts sequence of 2D maps into a 3D image.
- X-rays of different energy are absorbed to a different extent by different elements
- Use monochromatic beam at two different energies to map specific element. Here we used energies above and below the Zr K edge.

Column Experiments to Observe Colloid Filtration

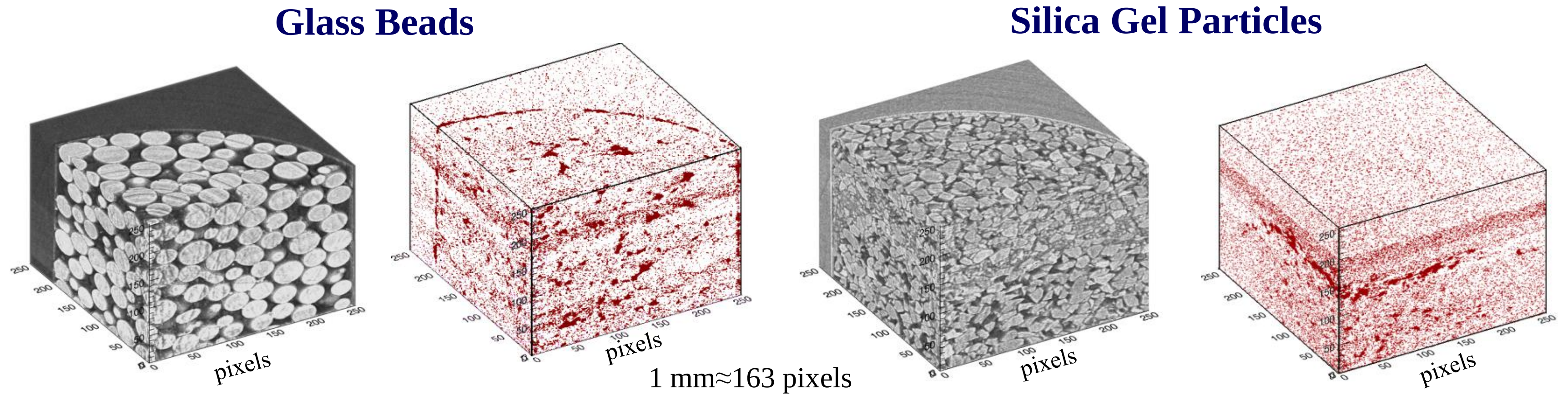
SETUP



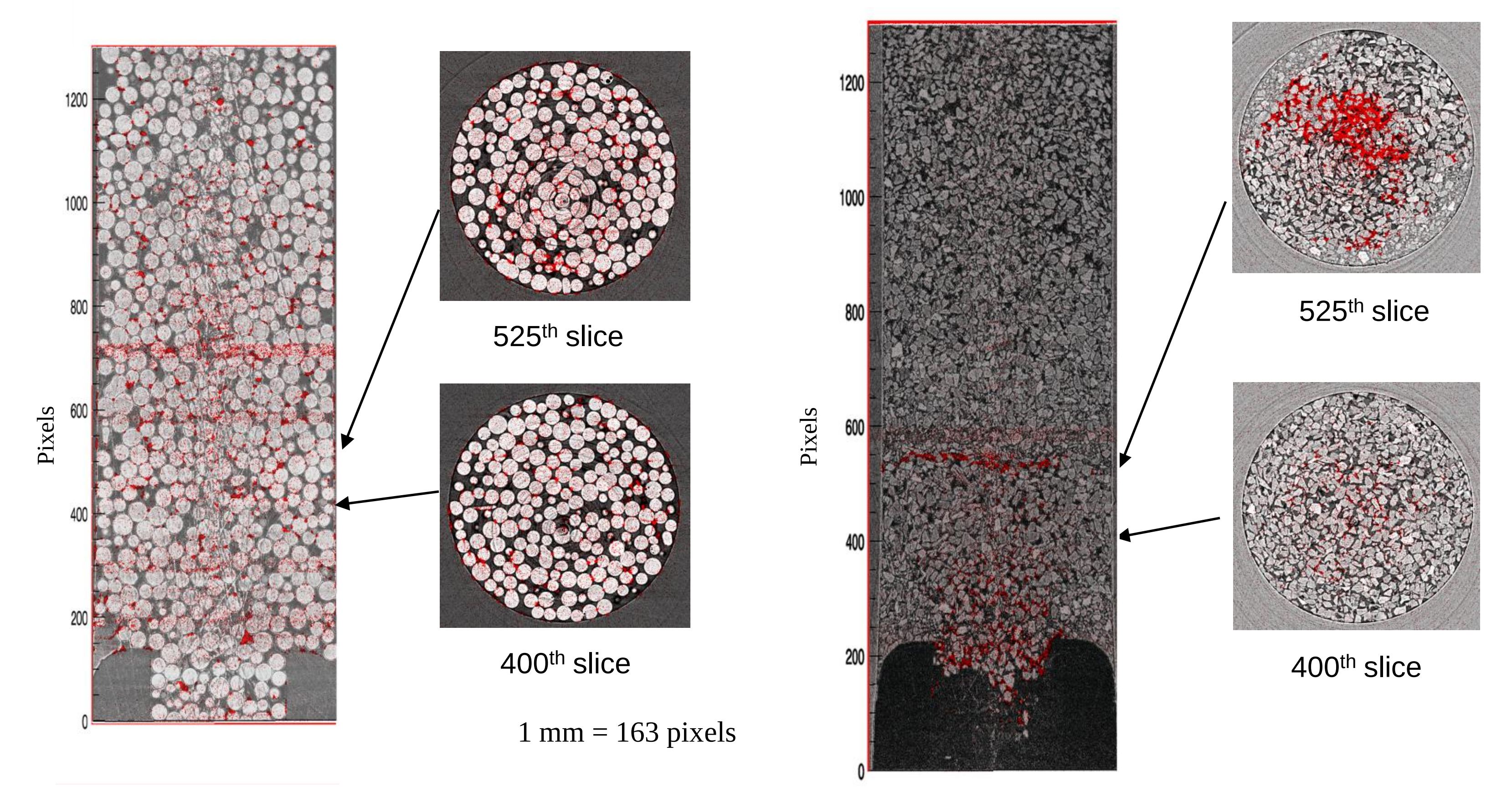
Column experiments were conducted to observe the deposition of zirconia colloids in a porous medium composed of glass beads or silica gel particles. Columns were equilibrated with background water, 20-60 pore volumes of zirconia suspension were pumped through, and then the columns were rinsed with 15 pore volumes of background water. Columns were then transported to the APS for analysis.

- Glass Beads: d = 100 μm, Silica Gel: d = 70-115 μm
- Zirconia Colloids: dg ≈ 1.5 μm
- Darcy Velocity: v = 0.01 - 0.5 cm/s
- Inlet Colloid: C_0 = 10, 125, 500 mg/L
- Background Conditions: pH = 7, 50 mM $CaCl_2$

RESULTS



3D tomographic reconstructions. The black and white images show both the structure of the pore spaces and grains. The distribution of deposited zirconia colloids is shown in red.



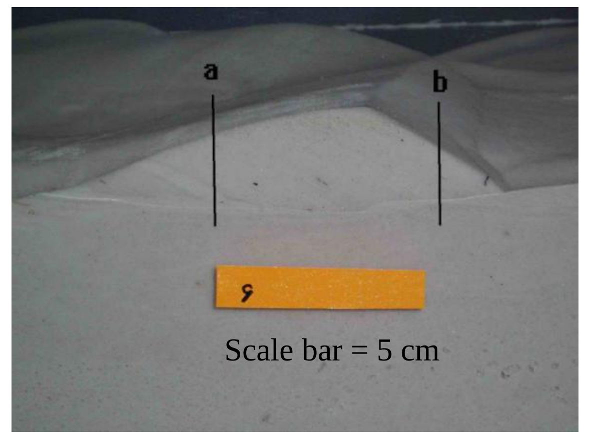
These two-dimensional slices of the tomographic reconstructions show the zirconia deposits (red) overlaid onto the black and white images of the grains and pore space. The rectangles show vertical slices and the circles show horizontal slices. Significantly more deposited zirconia around the 525th slice in the silica gel column shows, which apparently results from higher pore density at this location.

Flume Experiments to Observe Bedform Structure

SETUP

A flume experiment was conducted to observe the deposition of zirconia colloids in a sand bed. The bedforms were naturally formed and then a zirconia suspension was added into the flume. After zirconia deposition occurred, cores were obtained from the bed at different locations and transported to the APS for analysis.

- Stream Velocity = 9 cm/s, Depth = 14.5 cm
- Sand dg = 100 μm, Zirconia C_0 = 100 mg/L
- Bedform Height = 1.9 cm, Wavelength = 11.9 cm
- Background Water Conditions: pH = 6.5, I = 5 mM NaCl

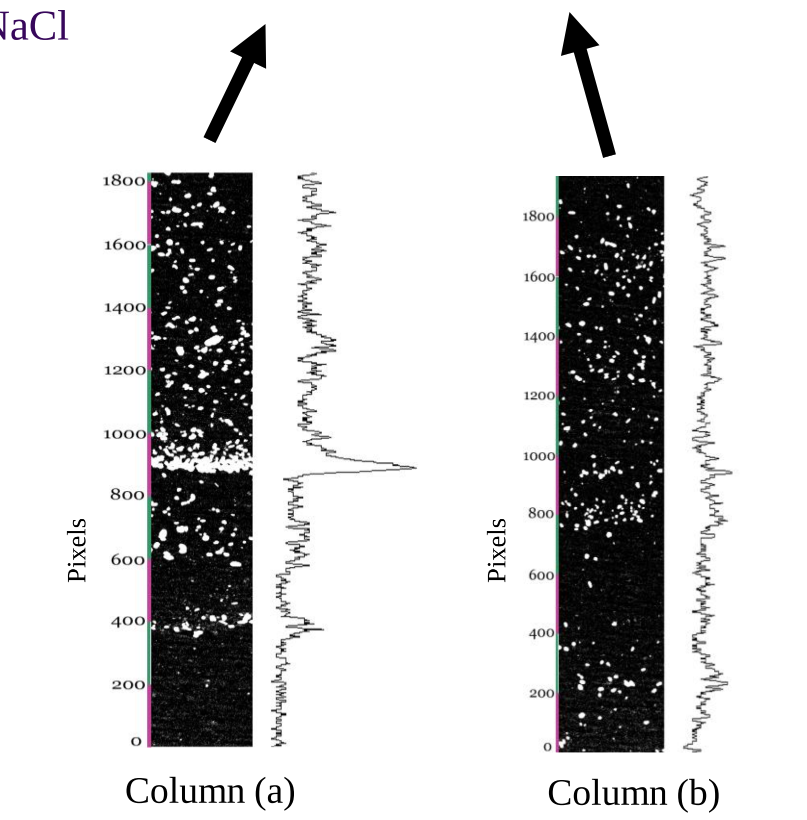


RESULTS

The images to the right show maps of the attenuation of the x-ray beam as it passes through the column. This can be thought of as the projection of the 3D distribution of zirconia concentration onto a plane.

The amount of zirconia present in a single slice is shown in the histograms next to the columns.

The sand contained a subpopulation of zirconia-rich grains, which were denser and accumulated in specific layers because of bed sediment transport.



1 mm = 163 pixels

Conclusions

X-ray microtomography performed at a synchrotron radiation source was used to resolve *in situ* sediment structure in 3D with micron-scale resolution. Difference tomography was also used to determine distributions of deposited zirconia colloids within various porous media. Heterogeneous deposition structures were observed in all cases. In column experiments, these structures appeared to result from preferential colloid deposition at grain intersections, regions of high grain packing, and regions where colloid accumulation clogged pore spaces. In flume experiments, depositional structures were clearly related to packing heterogeneities formed during bedform migration. Subsequent analysis will explore quantitative correlations between the structure of the porous medium and colloidal deposits.

Acknowledgements

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