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 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 background water. 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(111) monochromator. This high flux of monochromatic light allows us to:  
- Reach μm-scale resolution
- Collect data on a short time period.
- Avoid beam hardening effects.

Column Experiments to Observe Colloid Filtration

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. Further analysis on the APS 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 background water. 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(111) monochromator. This high flux of monochromatic light allows us to:  
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Principle of Z-Contrast X-ray tomography

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

Flume Experiments to Observe Bedform Structure

Flume experiments were 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. Further analysis on the APS 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 background water. 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(111) monochromator. This high flux of monochromatic light allows us to:  
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Conclusions

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- Analyses were performed at the DuPont-Northwestern-Dow Collaborative Access Team (DND-CAT) Synchrotron Research Center, which is supported by the E.I. DuPont de Nemours & Co., The Dow Chemical Company, the U.S. National Science Foundation through Grant DMR-9304725, and the State of Illinois. The Advanced Photon Source is supported by the U.S. Department of Energy.

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