# REFINING PINK BEAM FAST MICROTOMOGRAPHY TO STUDY TIME RESOLVED MULTI-PHASE FLOW IN 3D POROUS MEDIA

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**Summary:** Pink beam fast x-ray microtomography was optimized for two-phase flow experiments. The most ideal configuration utilizes a 1 mm copper filter and 1.5 mrad platinum mirror to filter out the low and high energy x-rays, respectively. This configuration, along with minimizing x-ray exposure when not scanning, limits bubble formation in the wetting phase while providing adequate image quality. A case study looking at the production of interfacial area in a transient and quasi-static two-phase flow experiment shows that transient flow produces more interfacial area.

## 1. INTRODUCTION

In recent decades, much insight into multi-phase flow in porous media has been gained using x-ray microtomography ( $\mu$ CT) facilities at synchrotron beamlines around the world [1].  $\mu$ CT imaging provides a non-destructive means to acquire high-resolution images of in-situ multi-phase flow processes in three dimensions. Standard monochromatic  $\mu$ CT imaging has been successful for many quasi-static studies, but time resolved studies of dynamic processes is lacking. Many subsurface mechanisms, such as geologic CO<sub>2</sub> sequestration and groundwater remediation, involve multi-phase flow under dynamic conditions. There have been few sub-second studies [2,3] at specific ultra-fast beamlines. Utilizing pink beam, beamlines not specifically set up for ultra-fast studies can also collect time-resolved multi-phase flow data. Initial studies [4] have utilized this technique to generate data with a time-resolution of 1-2 minutes. Our goal was to refine the pink beam methodology to achieve improved time-resolution, and study the effect of flow condition (quasi-static vs. transient) on fluid-fluid interfacial area development in two-phase fluid flow. Interfacial area is an important parameter in mass transfer during groundwater remediation efforts as well as a measurement of interest as a validation tool for thermodynamic theory development and numerical model efforts that seek to model two-phase flow.

#### 2. EXPERIMENTAL METHOD

The experiments were performed at the GSECARS 13-BMD beamline at the Advanced Photon Source located at Argonne National Laboratory. The full white x-ray beam is converted to pink beam by removing low energy x-rays with an in-line filter and reflecting the filtered beam vertically down from a platinum mirror towards the experimental setup, removing the high energy x-rays. Initial two-phase flow experiments resulted in excessive bubble formation in the wetting phase due to radiation exposure from the pink beam. X-ray interaction with materials occurs to a higher degree at low energies, therefore it was hypothesized that changing the in-line filter to something that would remove more of the low energy x-rays would alleviate bubble formation. Initially a 1 mm titanium filter was utilized. Additional aluminum filters were tried, as well as a 1 mm copper filter. The production of bubbles and resulting air phase saturation were measured from  $\mu$ CT image. We collected images for each selected filter with the pink beam on a glass bead column saturated with a 1:6 potassium iodide solution (for contrast between the wetting and bubble phases). Accordingly, the angle of the platinum mirror was optimized to try and achieve peak x-ray intensity near the iodine K-edge (Figure 1, left) for optimal contrast while keeping x-ray flux high enough to achieve high temporal-resolution scanning.

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For the two-phase flow experiments, the column was saturated with wetting phase then drained at a flow rate of 0.2 mL/hr, pulling non-wetting phase (air) into the system. After residual wetting saturation was reached, the pump was reversed and main imbibition (MI) was performed until residual non-wetting saturation was reached. Main drainage (MD) and three scanning curves (two starting on the MI curve and one on the MD curve) were also performed. For the transient experiment, the pump was only stopped to reverse flow, while during the quasistatic experiment, flow was stopped 6-10 times for each cycle and interfaces were allowed 15 minutes of relaxation. Isosurfaces between fluid phases were generated using a marching-cubes algorithm in Avizo<sup>TM</sup>, and specific interfacial area (a<sub>nw</sub>) was calculated by measuring total surface area of the triangulated isosurfaces over the total volume of the sample. The interfaces were split into those connected and disconnected to bulk phase.

#### 3. RESULTS

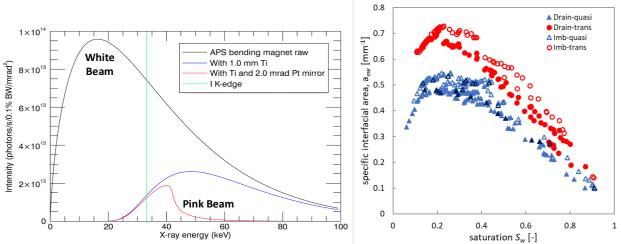
Optimization of the pink beam configuration led to the following results and a 12 second acquisition time:

- A 1 mm Cu filter is most beneficial for preventing bubble formation in the wetting phase along with reducing unnecessary x-ray exposure of the sample
- A 1.5 mrad mirror angle and a 15 ms exposure time produces optimal image contrast and quality for samples with iodine contrast agents

For the first time, transient and quasi-static two-phase flow data were collected in 3D. Transient flow tends to produce more total interfacial area than quasi-static flow (Figure 1, right). The transient experiment generates significantly more disconnected interfaces than in the quasi-static experiment too. It is concluded that when interfaces are not allowed to relax during transient flow, the system is forced further away from equilibrium and the surface energy of the system does not reduce to its minimum value at a specific saturation.

### References

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**Figure 1:** (Left) X-ray intensity distributions of APS 13-BMD for different beam configurations. (Right) Specific interfacial area  $(a_{nw})$  as a function of wetting saturation  $(S_w)$  for the transient and quasi-static flow experiments.