

# TENSION AND TOMOGRAPHIC MEASUREMENTS WHILE DRAINING WATER FROM A GRANULAR SAMPLE

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*Summary* We report simultaneous measurements of capillary pressure and water distribution in an unsaturated bed of glass beads subject to evaporation-induced drainage using xray computed tomography (CT) and a tensiometer. The data reveal that drainage can occur with or without pressure jumps. In the statistical mechanics framework of Xu and Louge [PRE 92:062405, 2015], these observations suggest that the drainage phase transition involves reversible periods punctuated by irreversible avalanches.

## BACKGROUND

Unsaturated porous media are familiar features of industrial and geophysical systems such as fuel cells, oil deposits and soils. Recently, Xu and Louge [1] proposed a statistical mechanics that regards permeable solids as an ensemble of interconnected pores exhibiting geometrical “frozen disorder”, which they characterized by the distribution of two parameters  $\alpha$  and  $\lambda$  measuring, respectively, the specific areas of neck cross section and wettable pore surface relative to pore volume. In that framework, these authors attributed drainage and imbibition to irreversible collective first-order phase transitions occurring at capillary pressures  $\psi$  and degrees of saturation in wetting fluid  $S$  predictable from known distribution of  $\alpha$  and  $\lambda$  and surface energies. They showed how hysteresis in the “retention curves” plotting  $S$  vs  $\psi$  is an inevitable consequence of collective interactions among pores, like other systems where statistical mechanics applies, such as magnetic materials [2]. After calculating the “latent” energies released in the draining and wetting transitions, they attributed sudden “Haines” jumps in  $\psi$  and  $S$  to the viscous dissipation of those energies by rapid fluid motion. They then devised a simple model reproducing the time-history of fluid speed jumps observed in an ordered pore network [3].

In those experiments, Armstrong and Berg [3] noted the limited extent of domains where pores interact collectively. Xu and Louge [1] called them “mesoscopic domains”, and estimated their size in terms of  $S$ . Following Preisach [6], they showed that, if capillary pressure is applied on boundaries located farther than the mesoscopic size, then retention curves may no longer be unique. For example, upon reversing the rate of change of  $\psi$ , a new curve may invade the region in  $(\psi, S)$  between the two “main” hysteretic retention curve of drainage and imbibition [5]. This “return-point” hysteresis is also commonly observed in magnetization [2].

Recalling the numerical simulations of Ji and Robbins [4], Xu and Louge [1] also recognized that “avalanches” can follow the main drainage phase transition, particularly if the degree of geometrical pore disorder, measured as the standard deviation of  $\alpha$  relative to the mean  $\lambda$ , exceeds a critical value. Like the main drainage phase transition, these avalanches should be irreversible, perhaps contributing, once again, to “return-point” hysteresis.

Therefore, an important question arising in the theoretical framework of Xu and Louge [1] is whether a sample of mesoscopic size but relatively weak disorder also experiences irreversible avalanches as it loses wetting fluid following the draining transition and, conversely, whether any fluid drainage can ever occur reversibly.

## OBSERVATIONS

To inform this question, we carried out experiments on an open DELRIN plastic tube tightly fitting the 5 mm outer diameter of a T5x tensiometer with  $-160$  to  $+100$  kPa pressure range and  $\pm 0.5$  kPa accuracy manufactured by UMS GmbH (Munich), holding glass beads (65% SiO<sub>2</sub>, 25% Na<sub>2</sub>O, 8% CaO, 1% K<sub>2</sub>O, Potters MIL-PRF 9954-5) with diameter in the range 300 to 425  $\mu\text{m}$  to a height of about 20 mm, initially saturated with degassed, deionized water. This relatively small sample was on the order of the mesoscopic domain size [1]. A micro-CT scanner manufactured by Australian National University operating in the double-helix mode with a General Electric nano-focus x-ray source at 80 keV and 100  $\mu\text{A}$  produced 9000 counts on the detector in 1.3 s with a 0.8 mm-thick aluminum filter [7]. Three-dimensional tomographic reconstructions were achieved using the MANGO software package from 1440 projections acquired in approximately 30 min. Segmentation between air, water and glass was relatively straightforward (Fig. 1). To find the capillary pressure  $\psi_-$  where drainage transition occurs (a.k.a. the air-entry potential), we also evaporated the same beads over ten days in the much larger HYPROP kit of UMS GmbH with a cylindrical vessel of 80 mm diameter and 50 mm height. Air entry occurred after about 4 hours at  $\psi_- \simeq 1.2$  kPa (Fig. 1, bottom left).

As Fig. 1 suggests, drainage from the smaller CT sample exhibited no jump in  $\psi$  above noise for the first 4 hours. Then, near (likely after) the phase transition, significant pressure jumps occurred between long periods of gradual adjustment. During the latter,  $B$  and  $C$  had visible changes in water and air conformation from the previous scan, but without jump. This suggests that some changes in saturation can occur reversibly, as Berg, et al [8] implied. Conversely, irreversible

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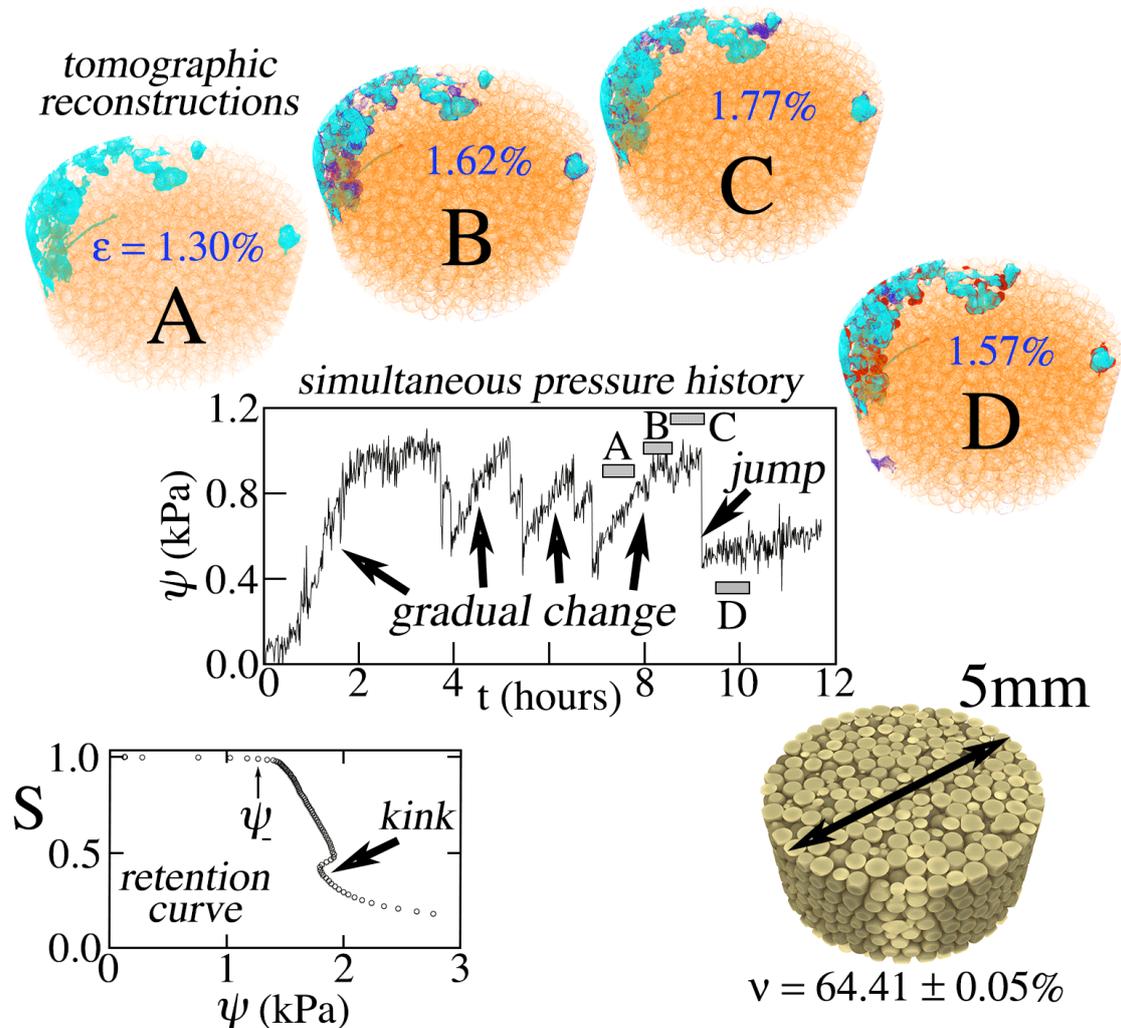


Figure 1: Top: tomographic reconstruction of scans A to D; light blue is air without discernible motion from the previous scan. Deep blue and red are, respectively, advancing and receding air;  $\epsilon$  is air volume fraction. Middle: capillary pressure  $\psi$  vs time; segments show scan duration. Bottom right: glass beads shown without surrounding air or water;  $\nu$  is solid volume fraction. Bottom left: drainage retention curve  $S \equiv 1 - \epsilon/(1 - \nu)$  vs  $\psi$  measured in the larger UMS HYPROP.

$\psi$ -jumps occurred around the drainage transition, again with detectable changes in water and air conformation. (However, it is unclear what role the cylindrical wall played). Meanwhile, because the retention curve had an obvious “kink” in  $\psi$  (Fig. 1, bottom left), we suspect that the sample holder of the HYPROP was much larger than the mesoscopic domain size, thereby allowing the  $(\psi, S)$  region between the main curves of drainage and imbibition to be invaded [1, 6], possibly leading to irreproducible return-point hysteresis, albeit yielding a smoother retention curve.

In short, our simultaneous tomographic reconstructions and tension measurements suggest that the drainage phase transition predicted by Xu and Louge [1] is accompanied by irreversible “avalanches” [2, 4] interspersed with reversible periods of gradual changes in saturation. In future, faster synchrotron CT-scans should be conducted to observe these avalanches as they redistribute liquid on msec timescales [1, 3], while simultaneously recording their pressure jumps.

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