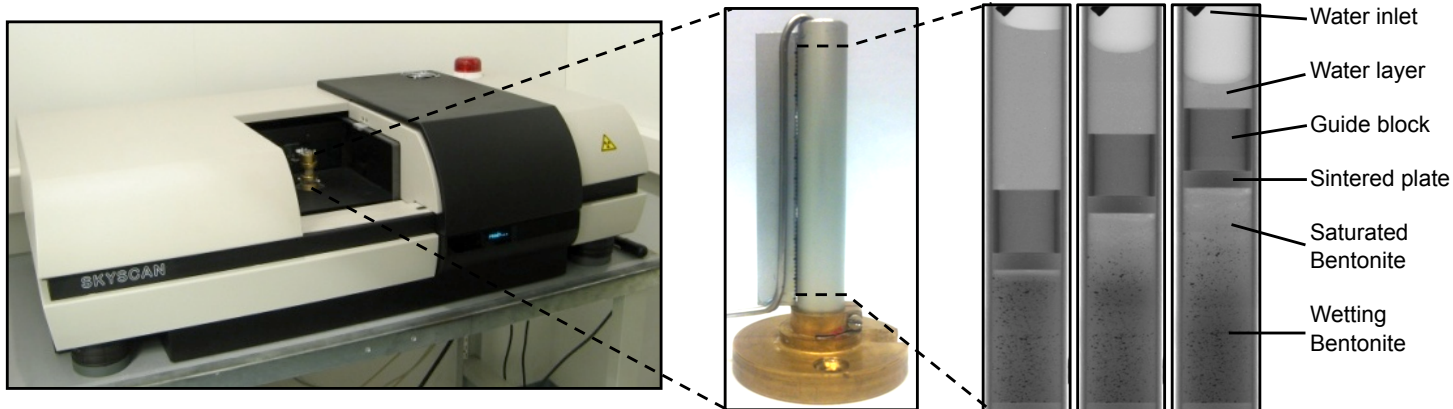


Measurement of water transport and swelling of bentonite clay using X-ray imaging

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Finland





Objectives:

1. To develop experimental methods based on X-ray imaging that can be used to gain detailed information on water transport and swelling dynamics of bentonite.
2. To produce detailed and well characterized data on wetting/ swelling processes and thereby to support modeling of bentonite buffer behaviour and erosion process.

Outline:

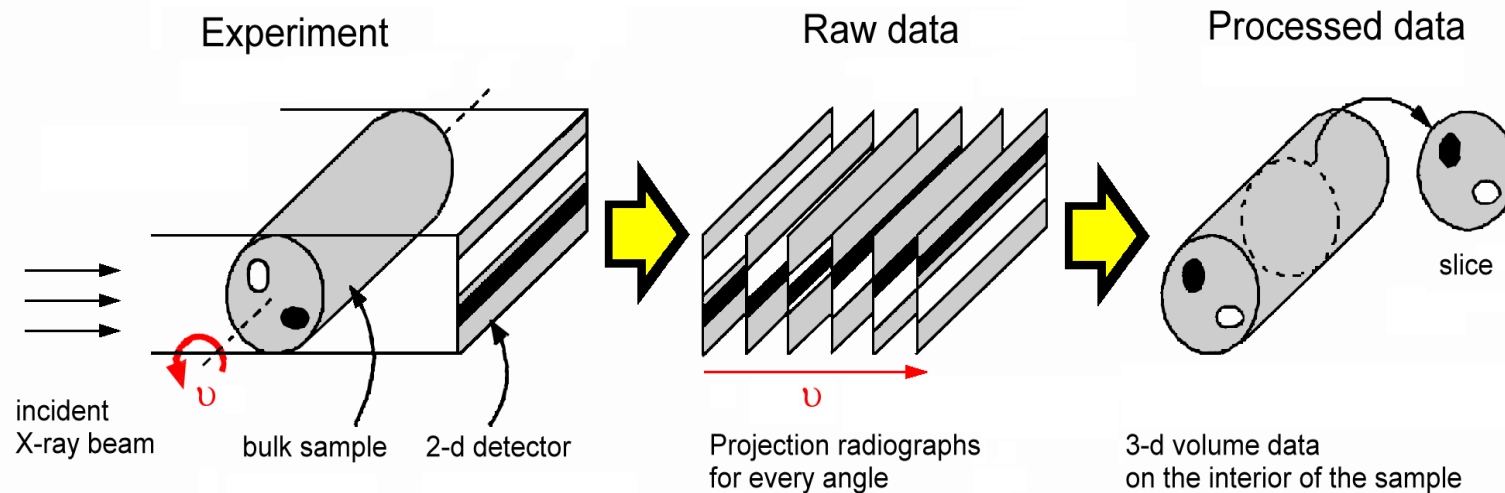
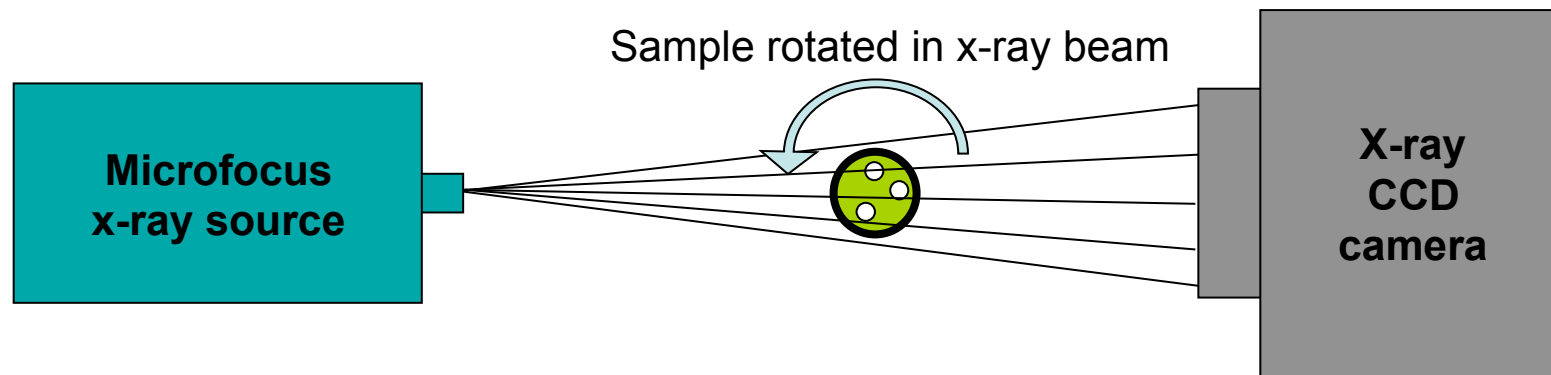
1. X-ray tomography and X-ray imaging
2. Image analysis
3. Results:
 - Wetting in a closed volume (X-ray tomography)
 - Wetting and free swelling in a narrow channel (X-ray imaging).
4. Conclusions

Background, motivation:

- Swelling of bentonite is an inherently complicated process.
->So will be any (physico-chemical) model that faithfully describes it.
- Need experimental data to support modelling
 - Identifying essential dynamical features/processes
 - Finding the values of unknown model/material parameters
 - Validation of models
- Many conventional methods rely on intrusive point measurements
- X-ray methods are non-intrusive and can provide very detailed 3D or even (3+1)D 'field' data. Sub-micron spatial resolution.

X-ray Tomography

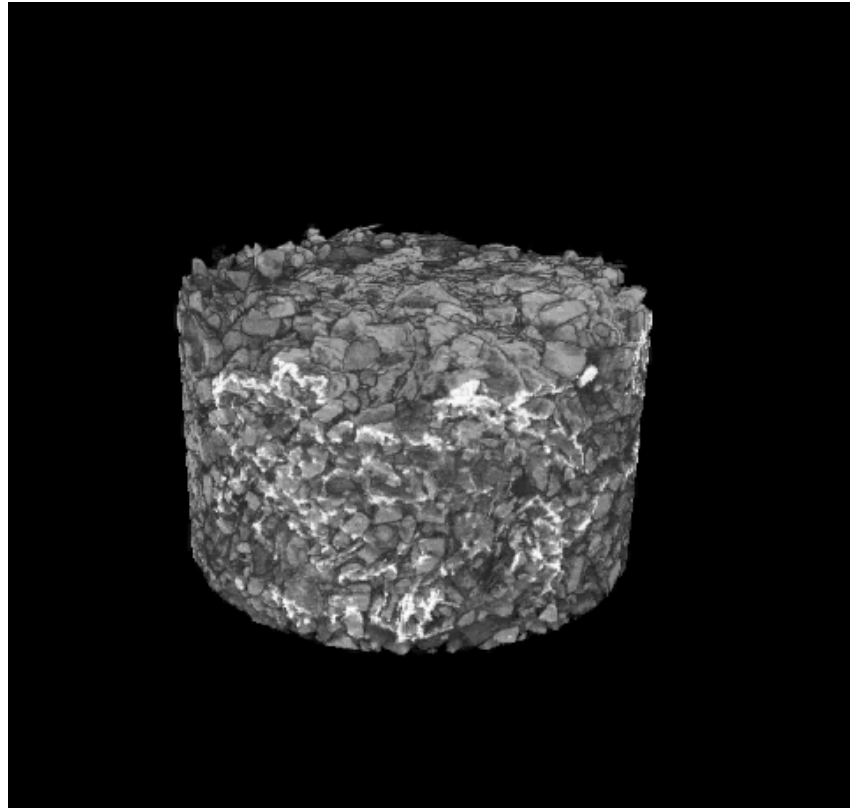
A non-intrusive method for measuring internal 3D structure of inhomogeneous materials.



A tomographic image: Spatial distribution of X-ray attenuation constant in the sample

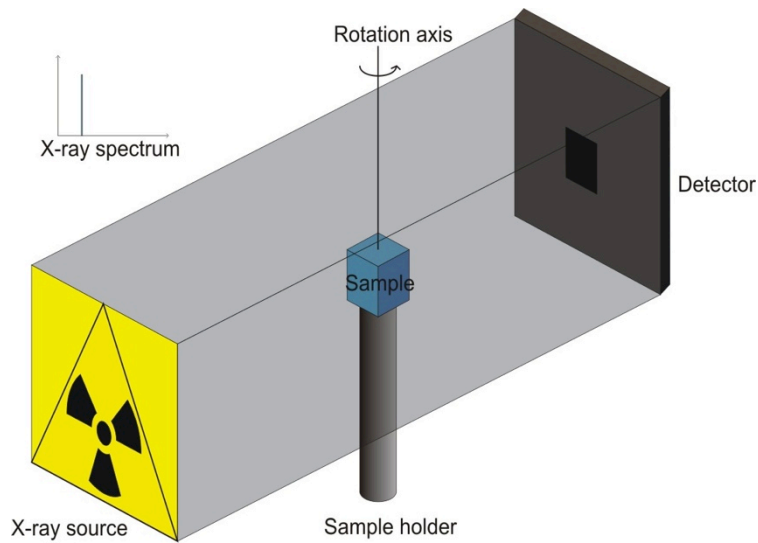
Example: Penetration of liquid in porous material.

Imbibition of liquid in porous sample (calcium carbonate).
Water with iodine salt used as a heavy tracer.



Synchrotron source

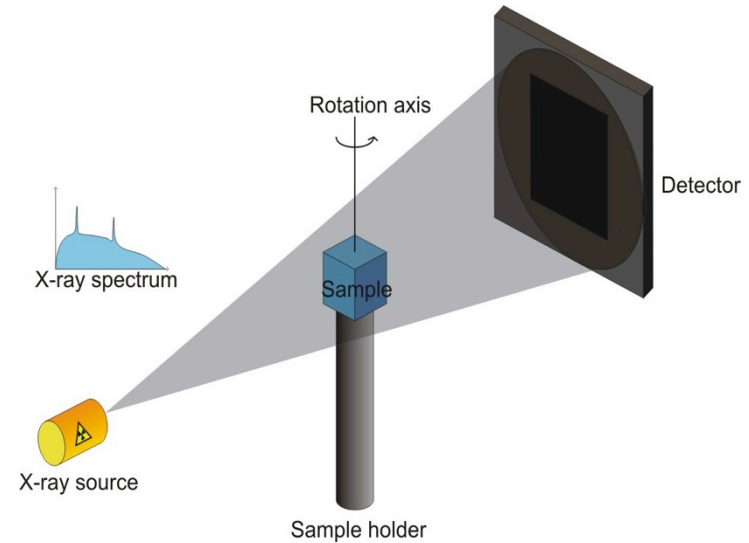
ESRF



- Parallel beam geometry
- Monochromatic x-ray beam
- High intensity fast imaging time)
- Resolution 0.7 mm.
- Very good image quality

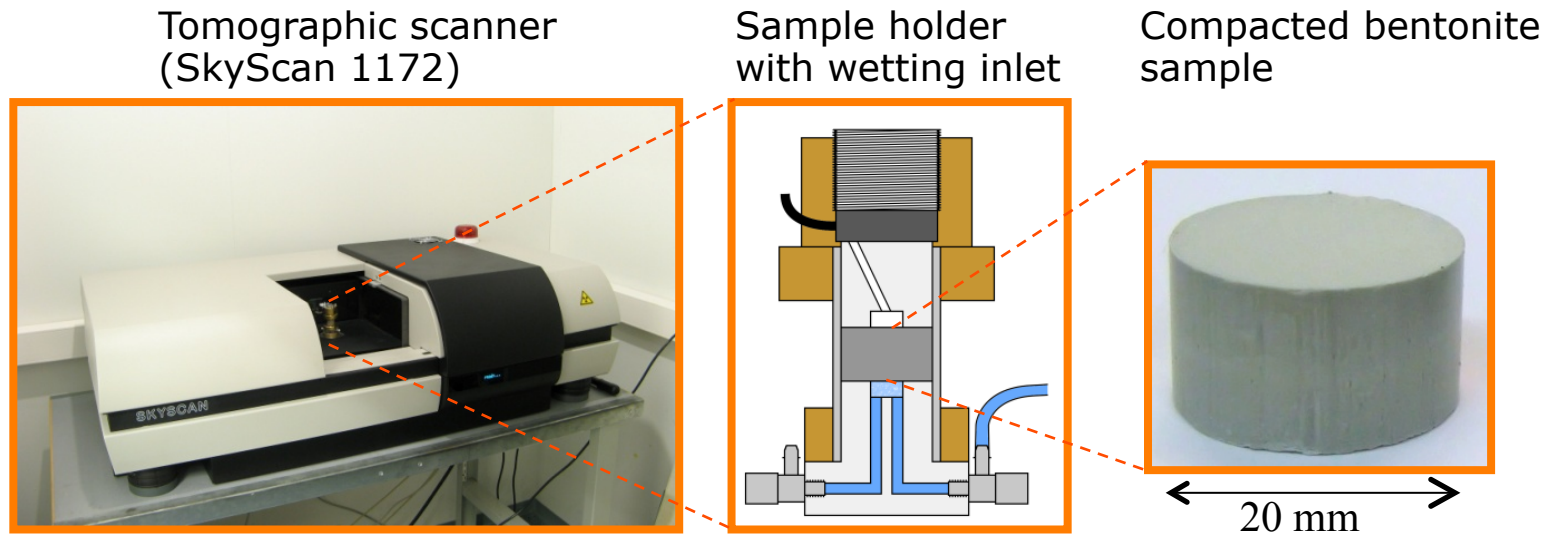
X-ray tube source

Table-top scanner



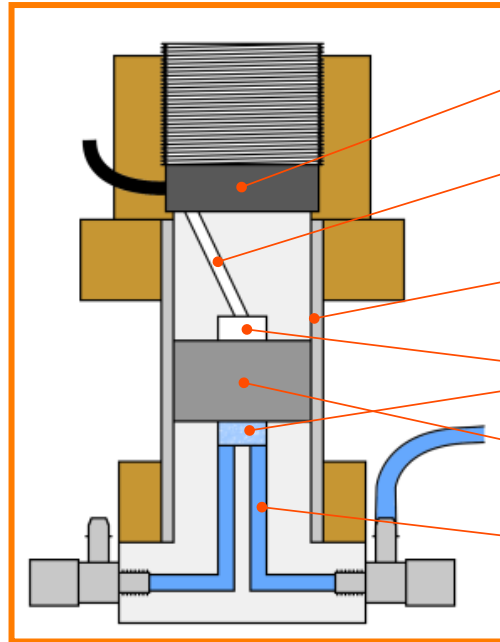
- Cone beam geometry
- Wide spectrum X-ray beam
- Moderate intensity and imaging time
- Variable resolution 0.9 – 35 mm.
- Good image quality

Application 1: Measuring wetting and swelling of bentonite in a closed volume (4D X-ray tomography)



- Compacted cylindrical purified bentonite sample doped with small marker particles (hollow glass spheres)
- Sample is held in a constant volume (2.3 cm^3) and wetted through one end

Sample holder



Force sensor

Venting channel

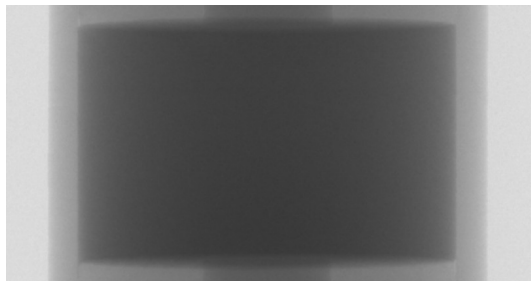
Plastic tube
(PEEK)

Sintered blocks

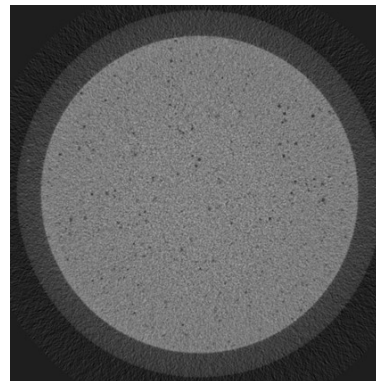
Bentonite sample

Wetting channel

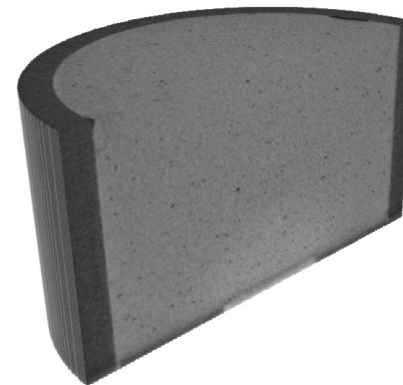
X-ray image



Reconstructed slice

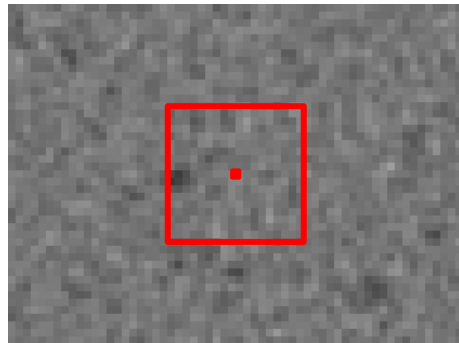


3D visualization
(half a sample)

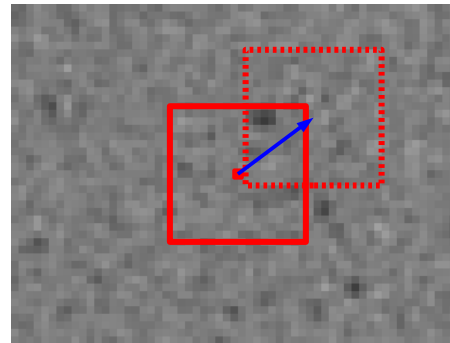


Deformation analysis

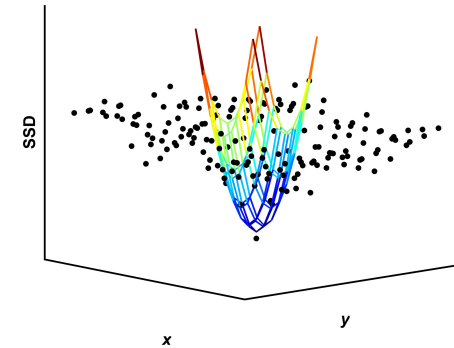
3D image correlation based on marker particles.



(a)



(b)



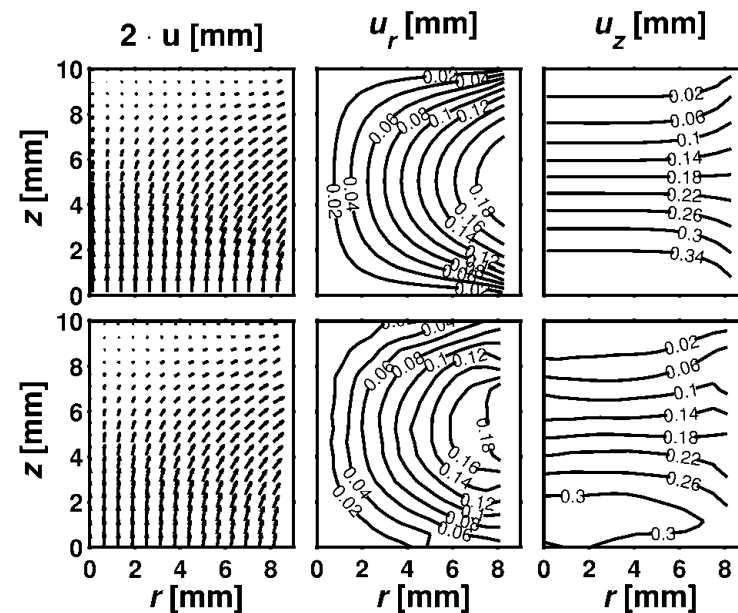
(c)

Compression test stage
with a rubber sample.



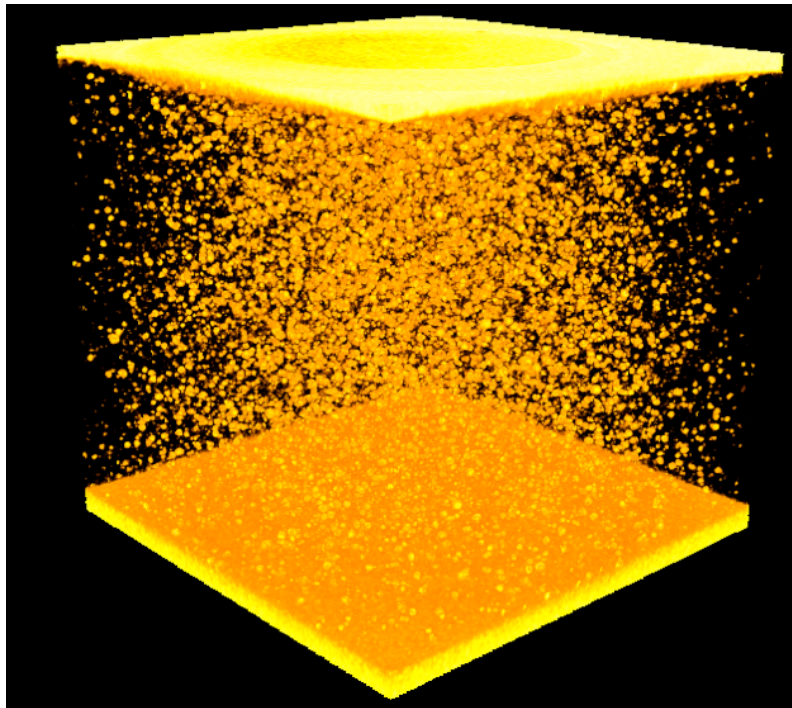
COMSOL

X-ray images



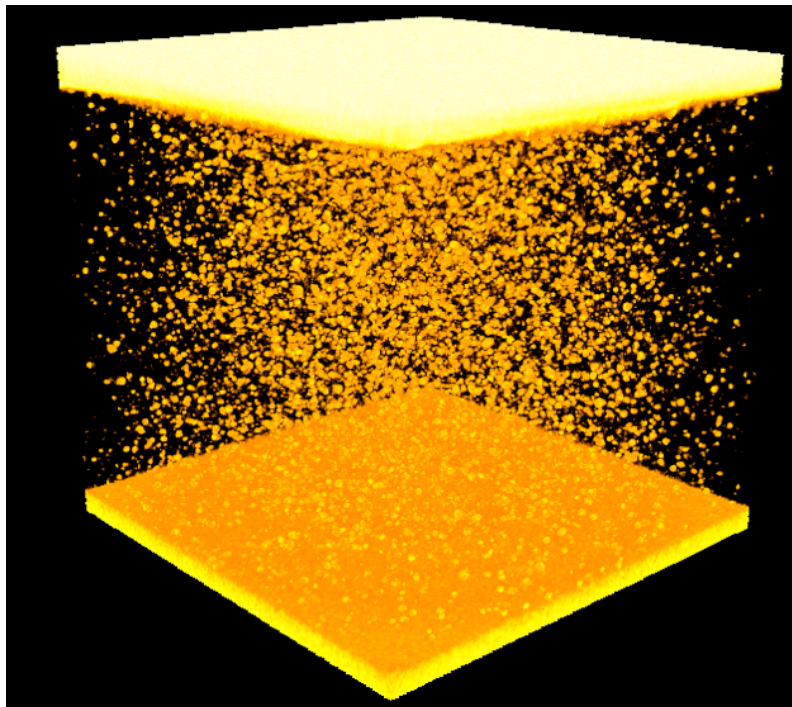
3D Displacement field computed by correlating two tomographic images

Thresholded image
(marker particles only)
Reference state



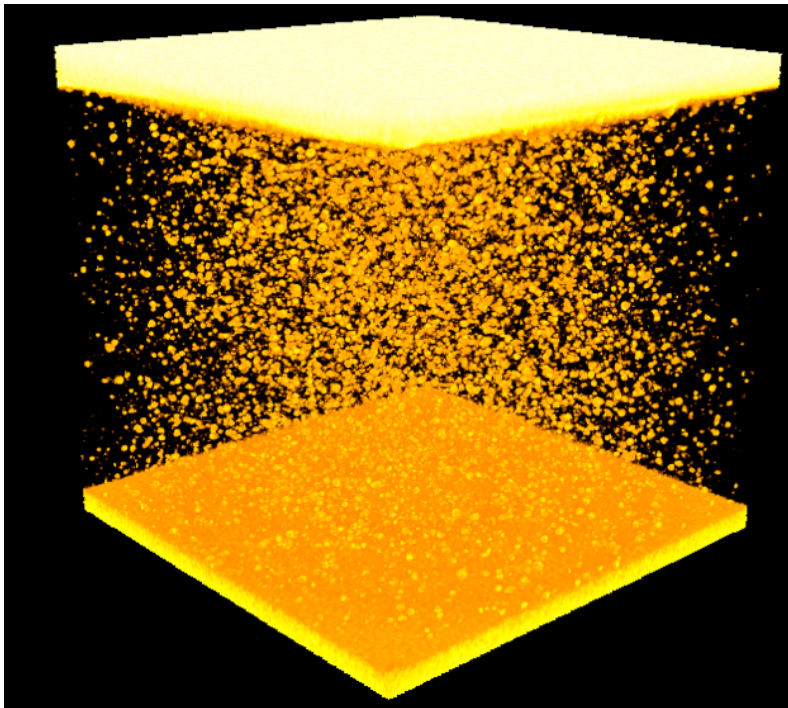
3D Displacement field computed by correlating two tomographic images

Thresholded image
(marker particles only)
Compressed state 1

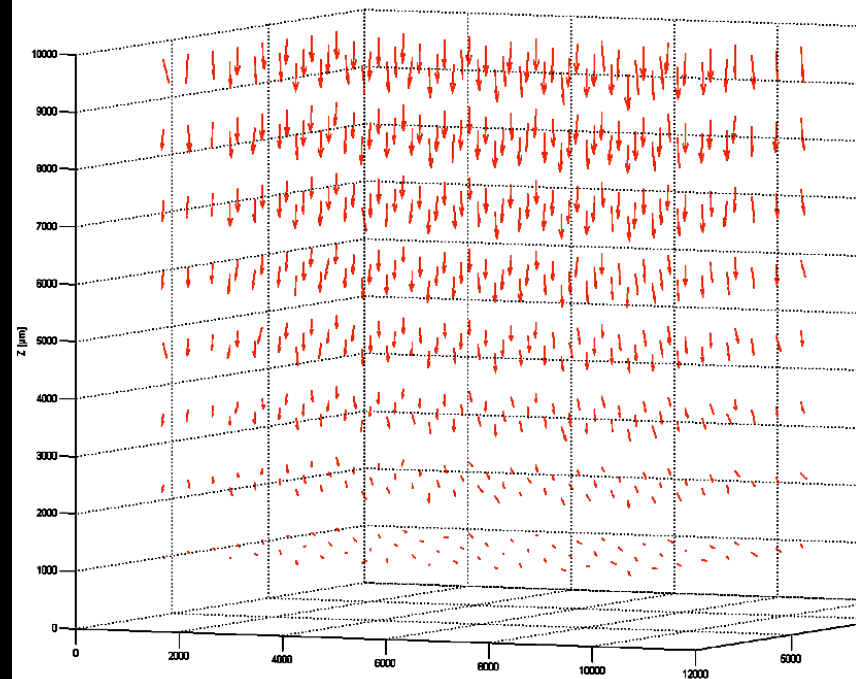


3D Displacement field computed by correlating two tomographic images

Thresholded image
(marker particles only)
Compressed state 1



Displacement field



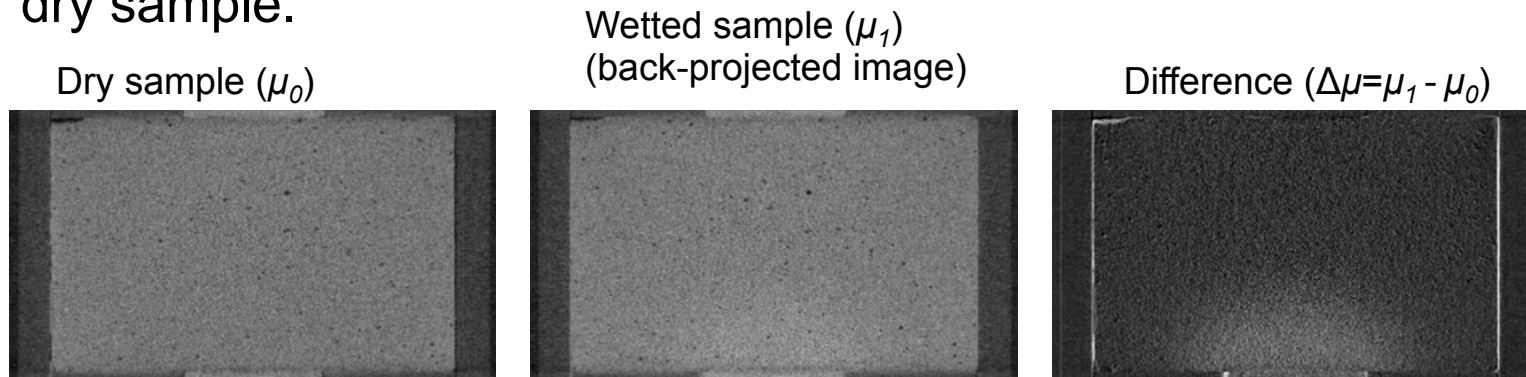
Measured 3D displacement field + initial density of bentonite



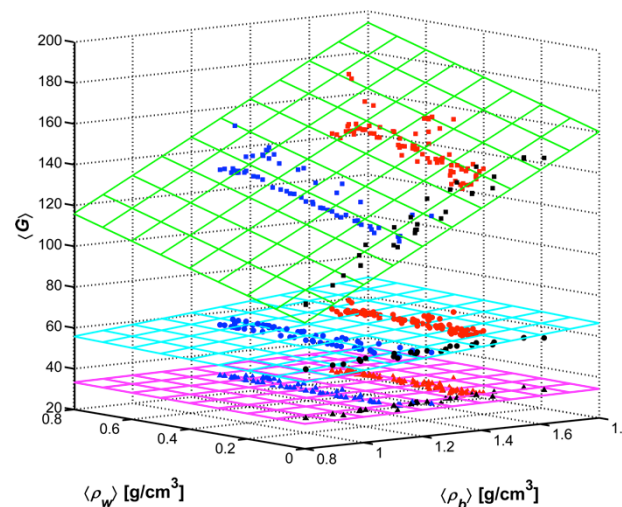
Local (dry) density of bentonite $\rho_s = \rho_s(\vec{r})$ in deformed state.

Water content analysis

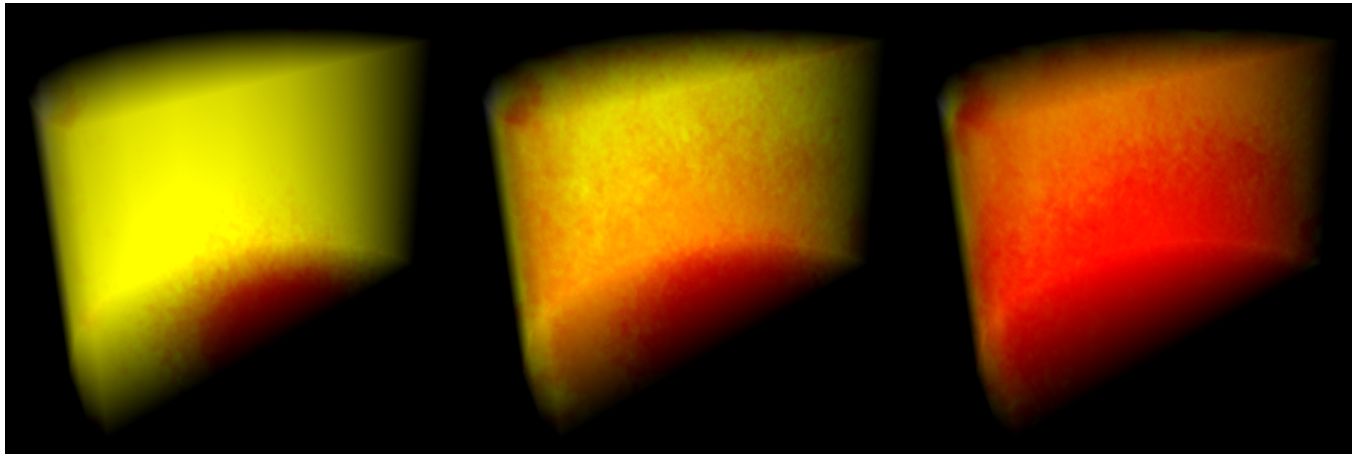
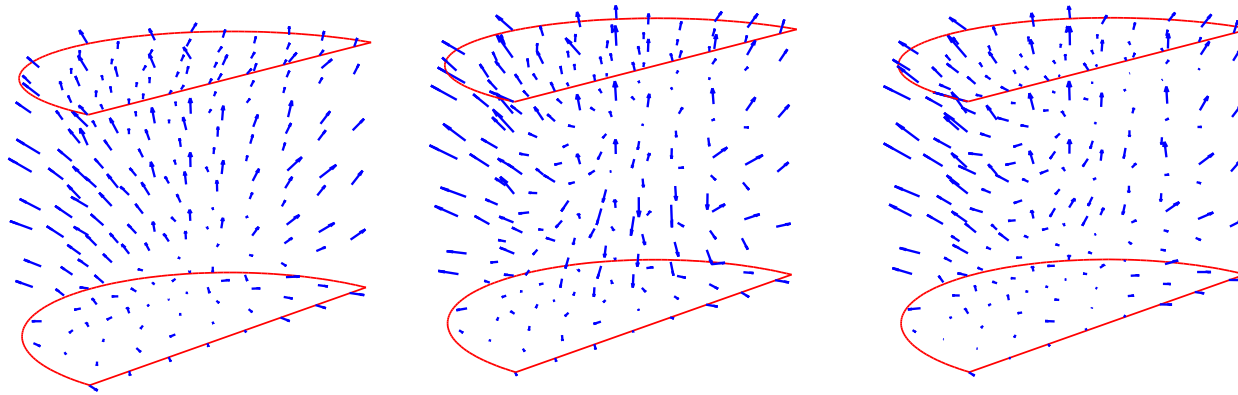
- Water content found based on difference images between wet and dry sample.



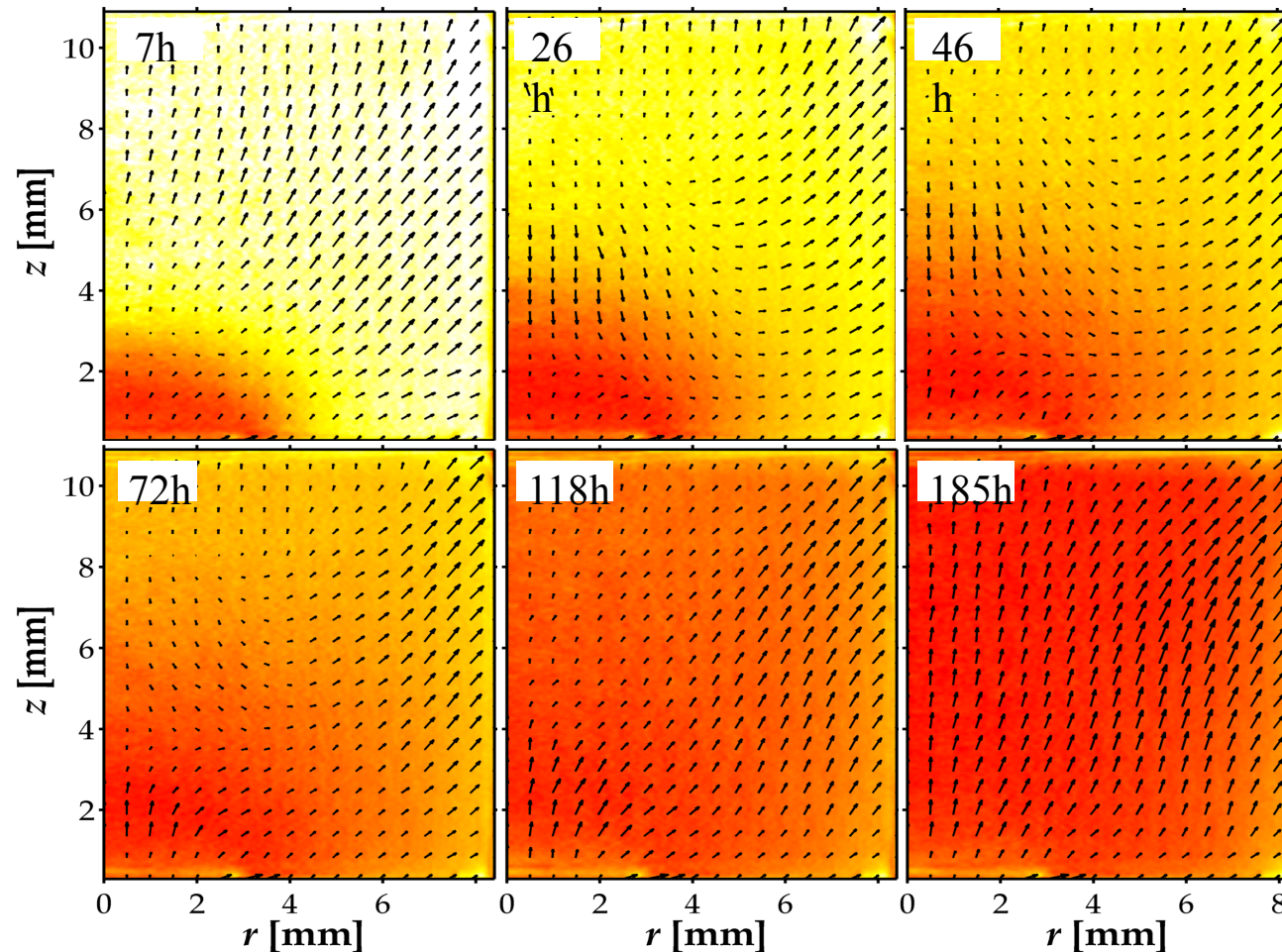
- Experimental correlation between difference image gray scale value and partial densities of water and bentonite



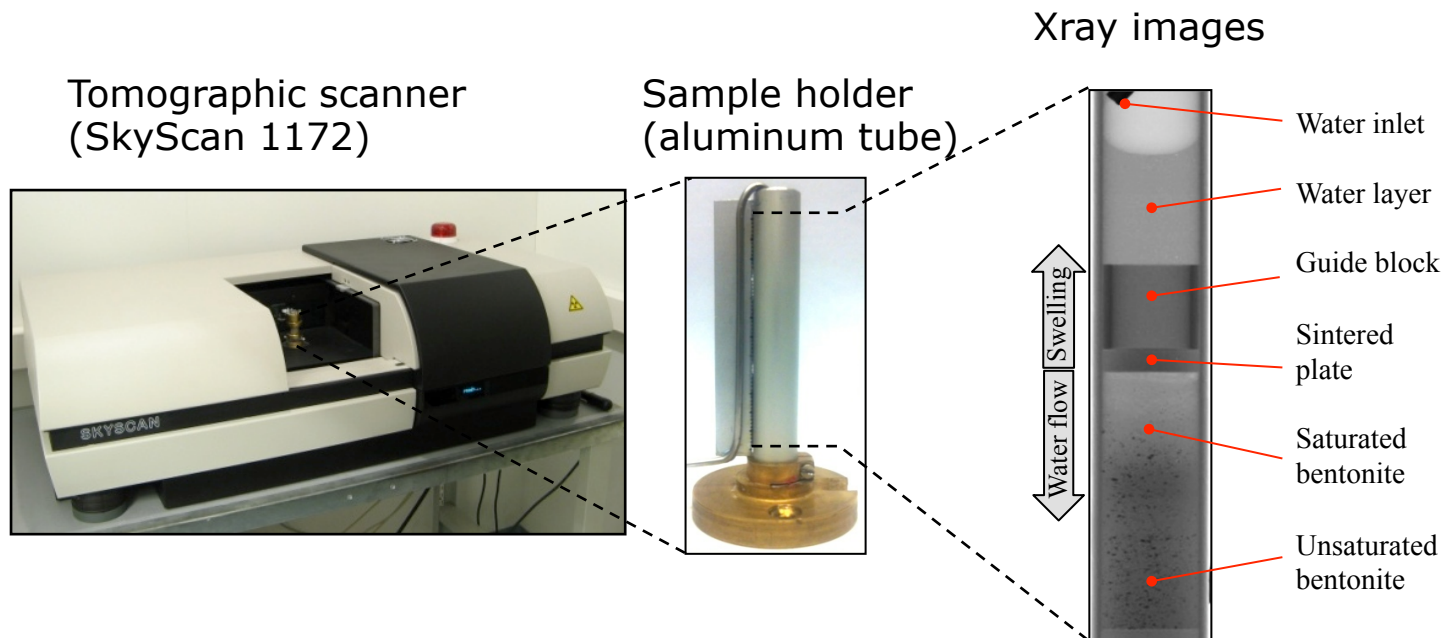
Results: Measured evolution of water content and displacement field in a wetting bentonite sample



Results: Measured evolution of water content and displacement field in a wetting bentonite sample. Averaged over azimuthal angle



Application 2: Free swelling in a channel (2+1D X-ray imaging)

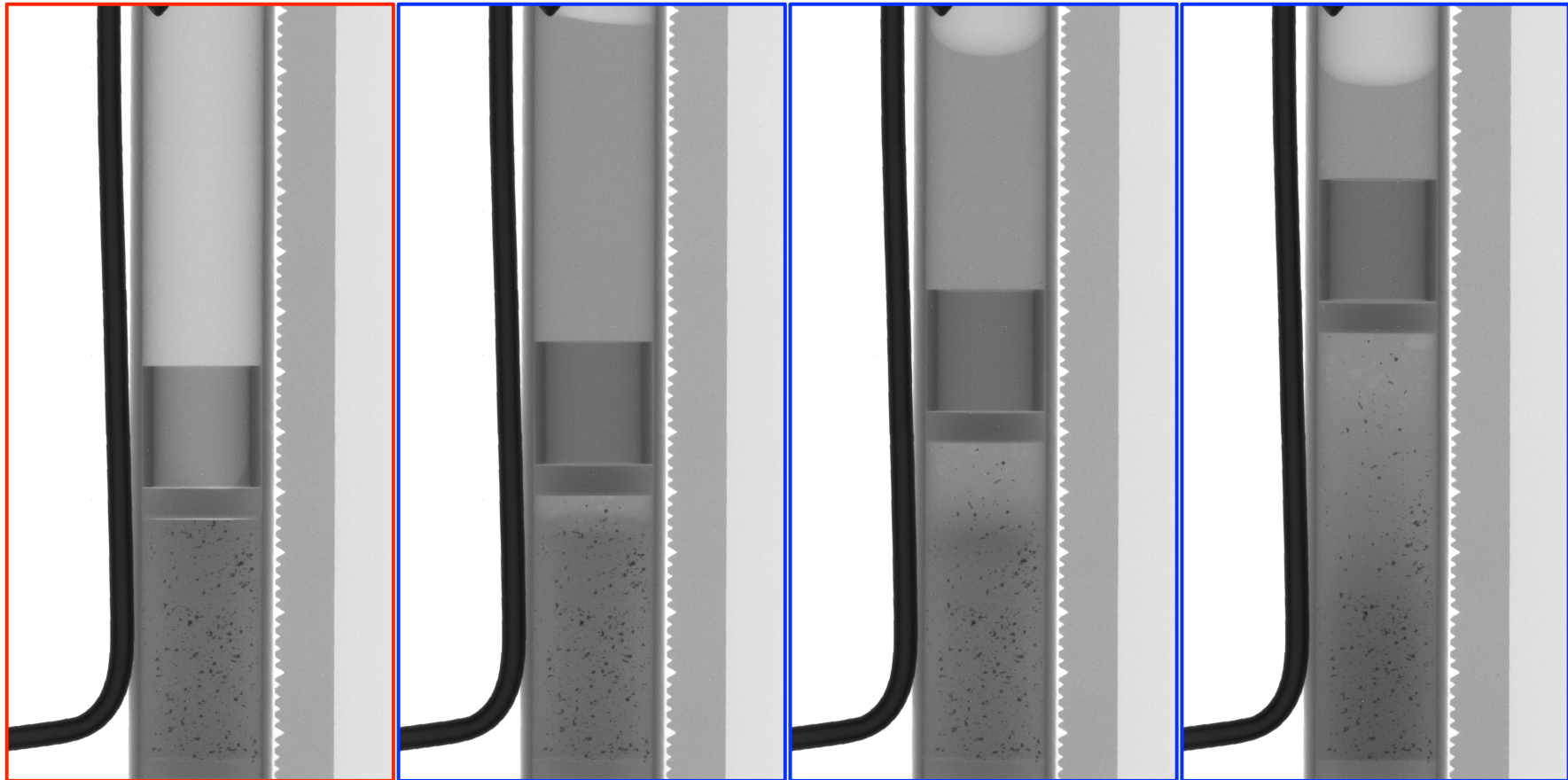


- Bentonite sample doped with small marker particles compacted in an aluminum tube
- Water added on top of the sample which is allowed to swell freely upwards.
- Wetting/swelling monitored by X-ray imaging.

Free swelling in a channel

Initial state (dry)

Wetting/swelling during ~ 4 days



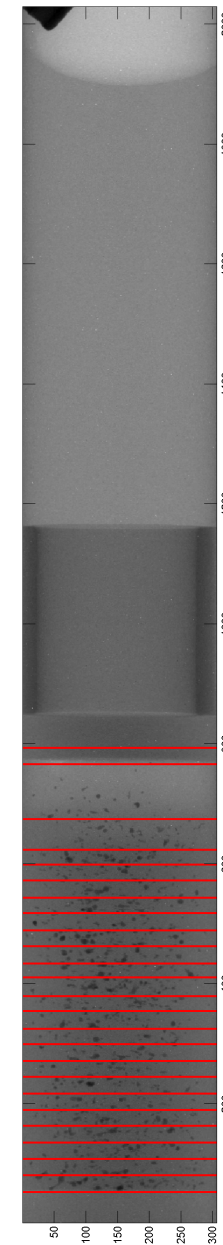
Free swelling motion analysis and solid phase density

- Bentonite material (solid phase) motion tracked by image correlation based on marker particles
- Bentonite dry density $\rho_s = \rho_s(z, t)$ calculated from known initial density and displacement field
- Gray scale value carries information on water content



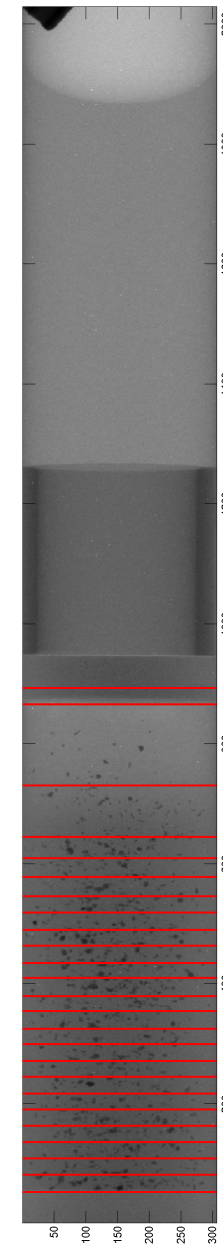
Free swelling motion analysis and solid phase density

- Bentonite material (solid phase) motion tracked by image correlation based on marker particles
- Bentonite dry density $\rho_s = \rho_s(z, t)$ calculated from known initial density and displacement field
- Gray scale value carries information on water content



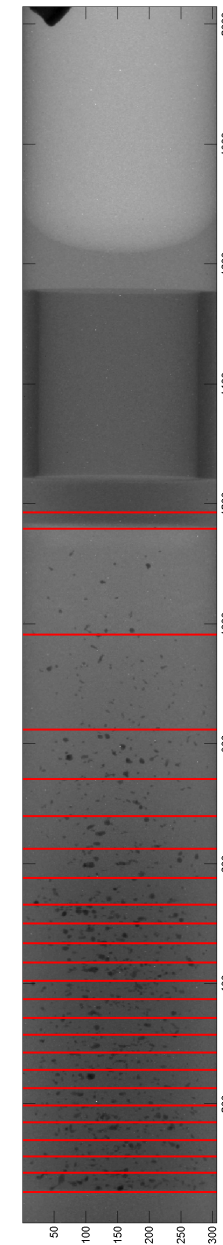
Free swelling motion analysis and solid phase density

- Bentonite material (solid phase) motion tracked by image correlation based on marker particles
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- Gray scale value carries information on water content



Free swelling motion analysis and solid phase density

- Bentonite material (solid phase) motion tracked by image correlation based on marker particles
- Bentonite dry density $\rho_S = \rho_S(z, t)$ calculated from known initial density and displacement field
- Gray scale value carries information on water content



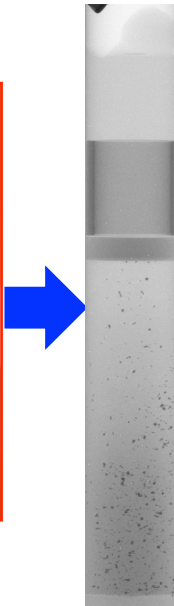
Calibration of X-ray image grey scale (attenuation coefficient)

Measurement of local water content distribution in the final state
(partially wetted sample)

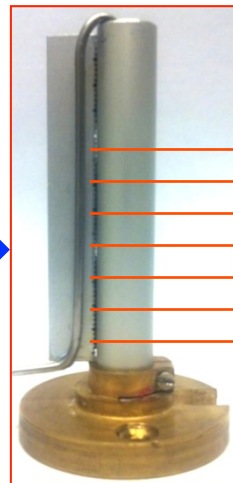
Sample and sample holder
frozed in a liquid nitrogen



X-ray image of
frozen sample



Slicing



Weighing

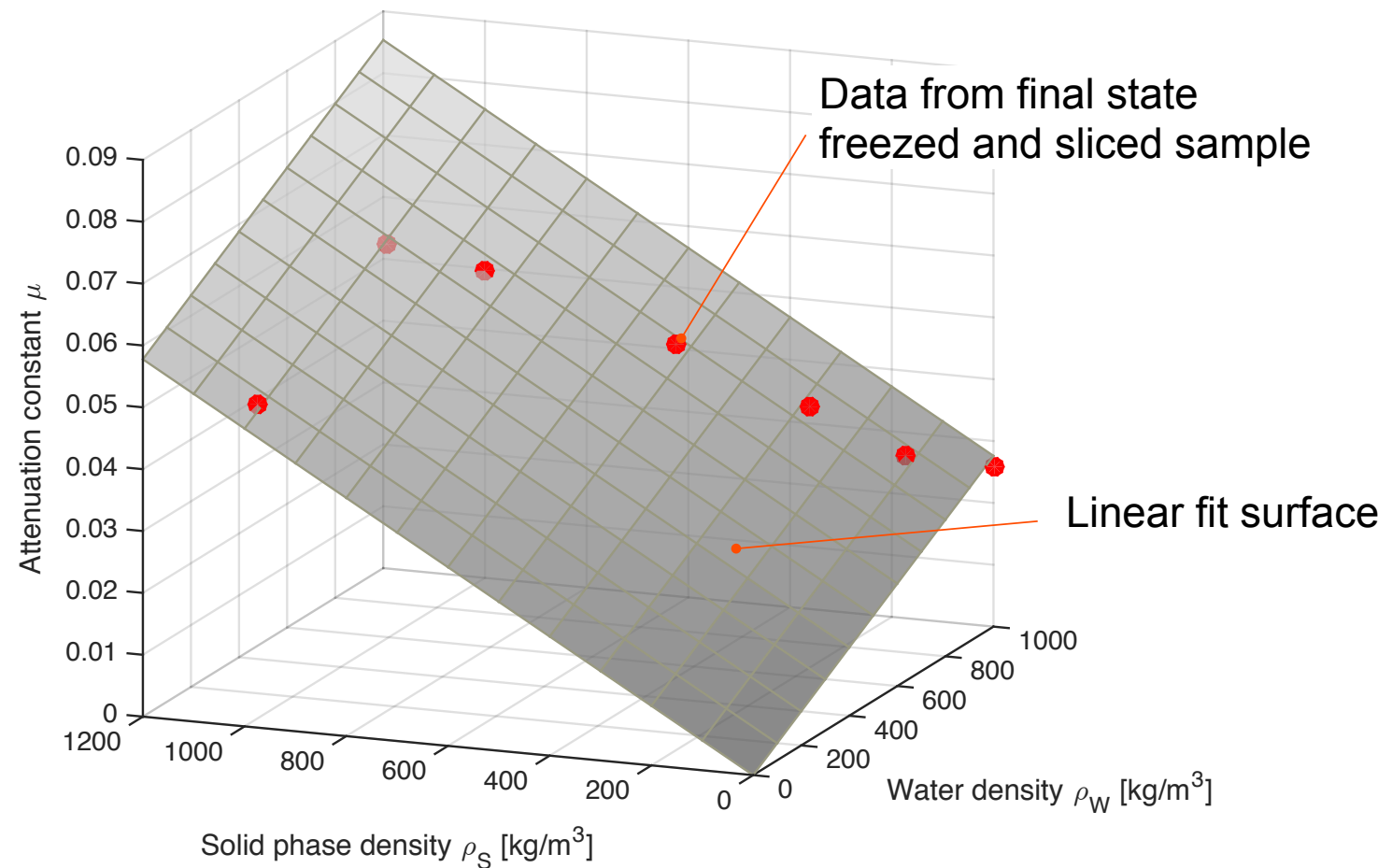


Drying ...



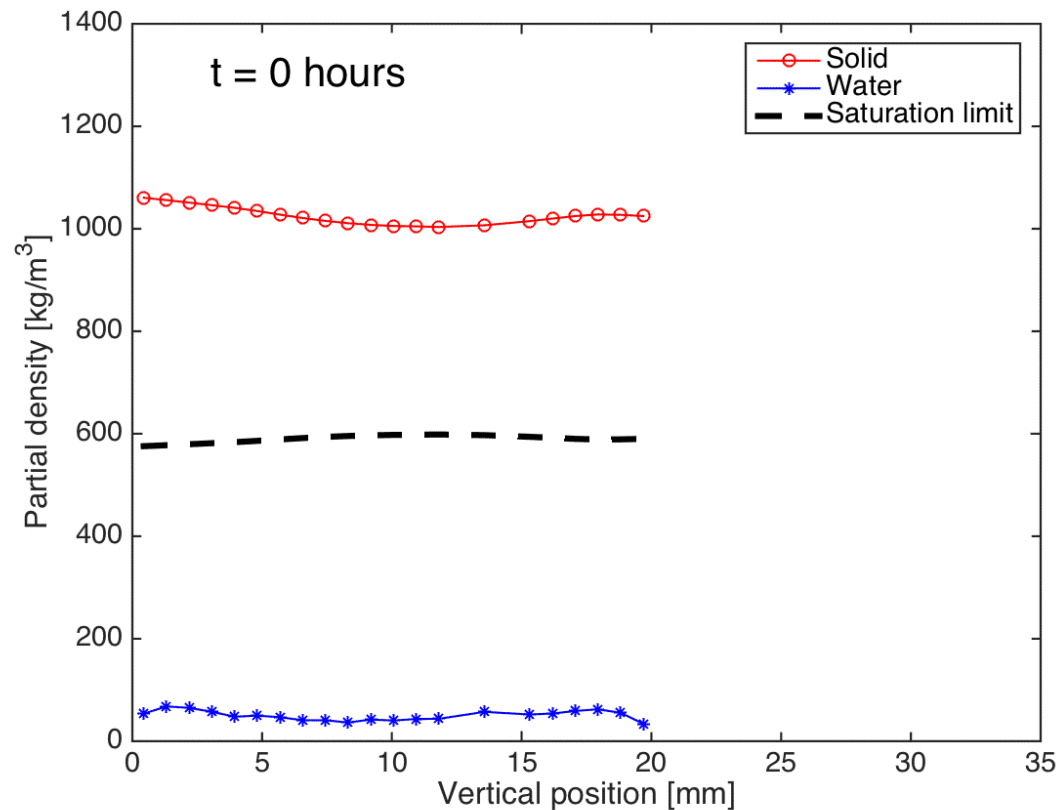
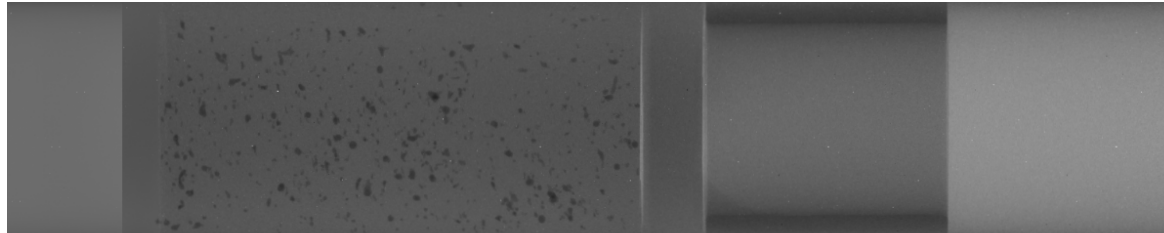
Combined information from motion tracking and post mortem water content measurement allows calibration of x-ray image grey scale to local solid and water content during swelling.

Calibration based on final state data:

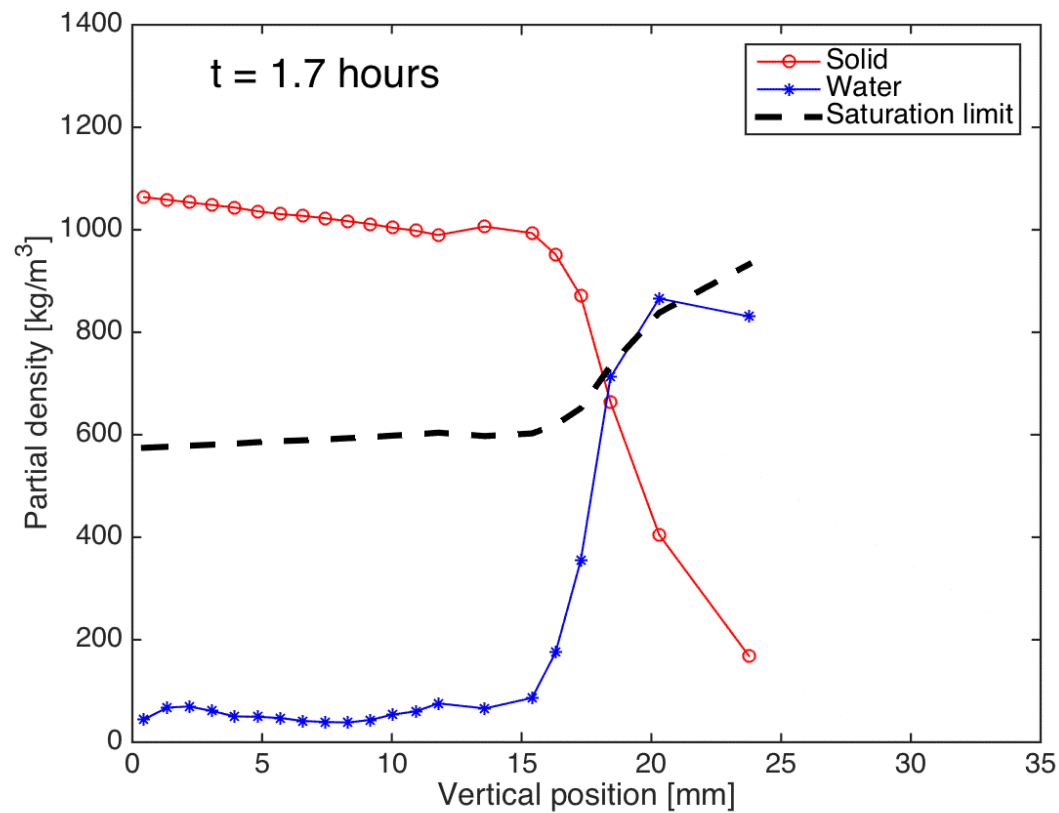
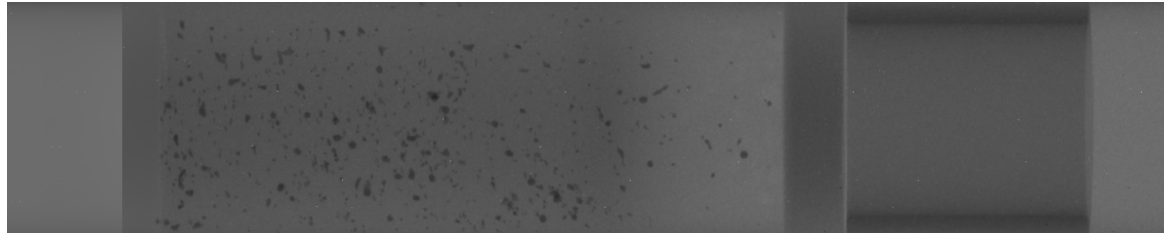


$$\rho_w = \rho_w(\rho_s, \mu)$$

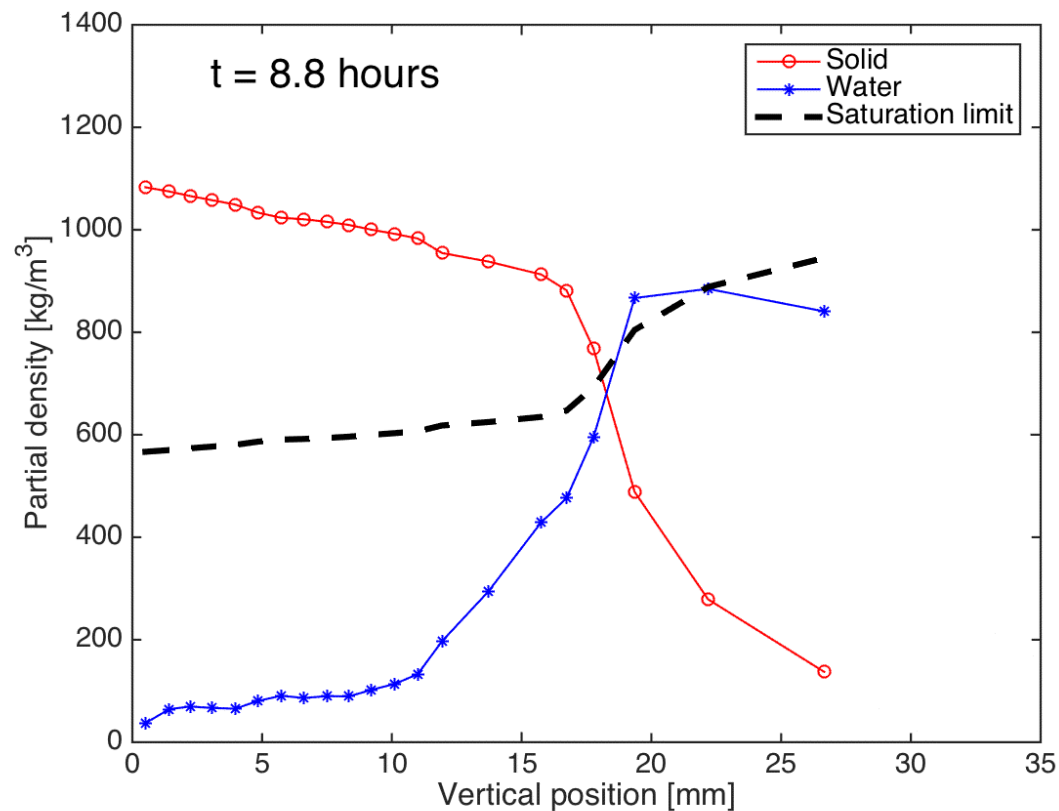
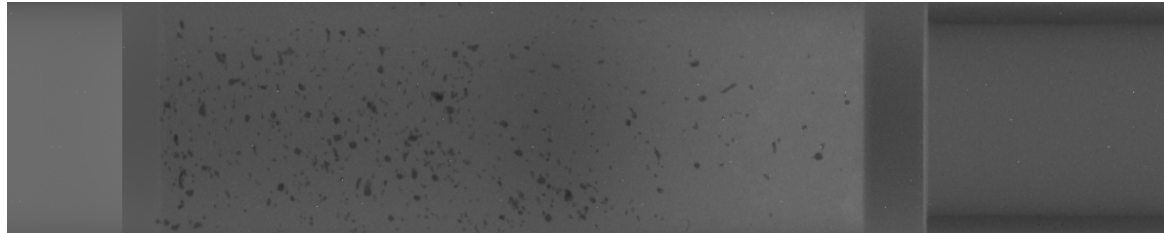
Final result: Evolution of bentonite (dry) density and water content in the channel.



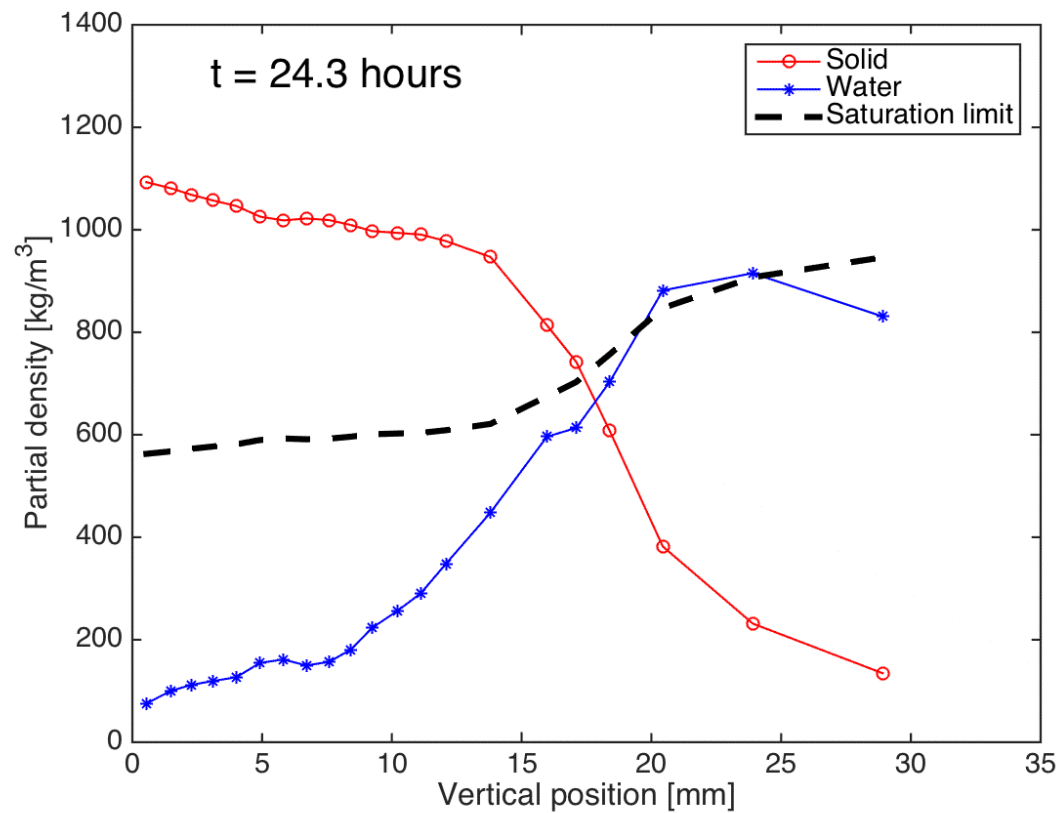
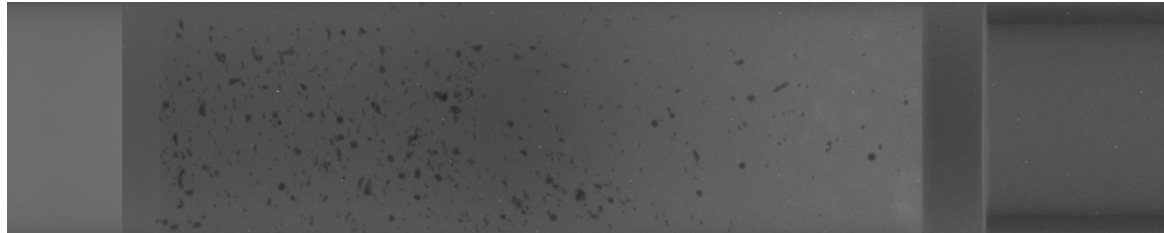
Final result: Evolution of bentonite (dry) density and water content in the channel.



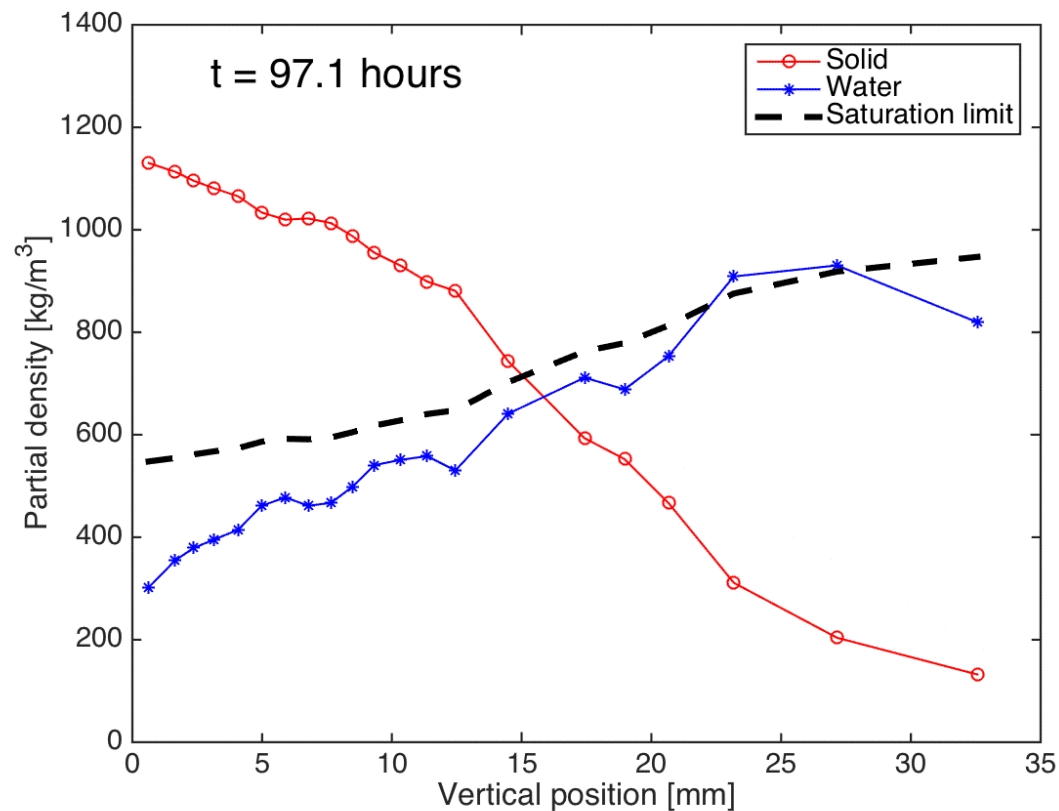
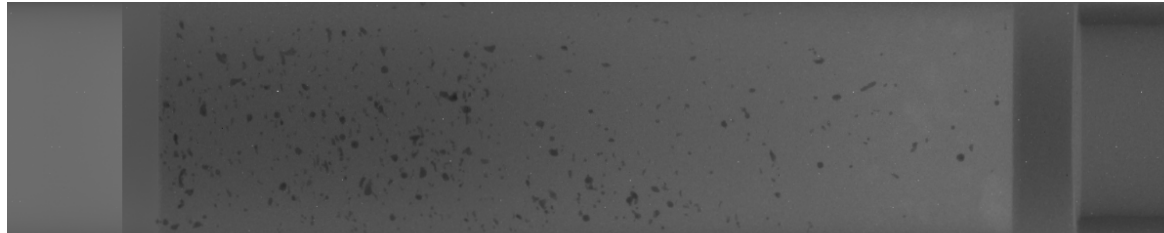
Final result: Evolution of bentonite (dry) density and water content in the channel.



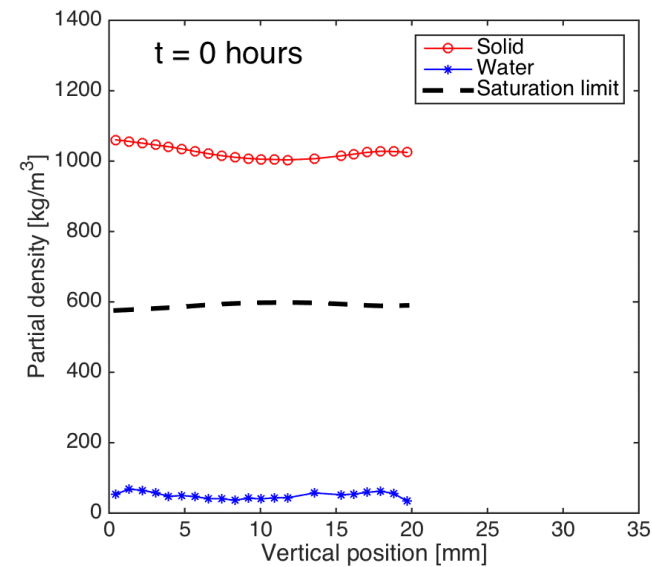
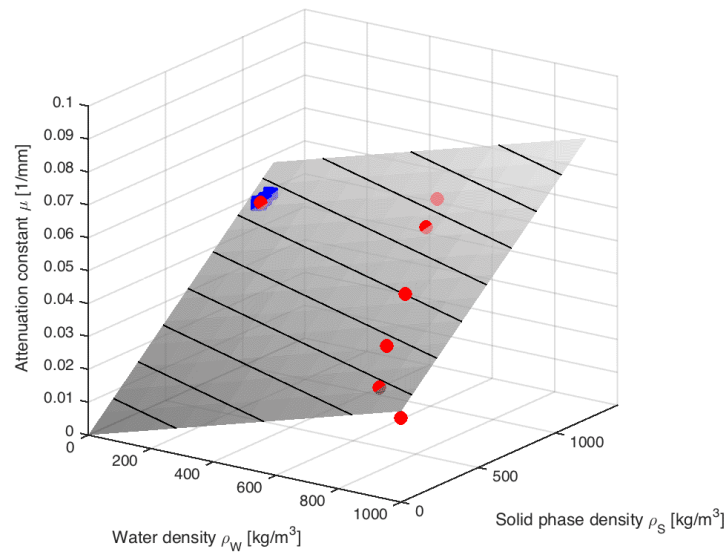
Final result: Evolution of bentonite (dry) density and water content in the channel.



Final result: Evolution of bentonite (dry) density and water content in the channel.



Calibration test: Evolution of bentonite (dry) density, water content and attenuation coefficient.



Conclusions:

- Non-intrusive methods based on X-ray imaging and X-ray tomography for measuring water transport and swelling of bentonite have been developed.
- The method have been used to monitor wetting and swelling in constrained volume and free swelling in a narrow channel.
- The method yields detailed time-series data on:

$\vec{u} = \vec{u}(\vec{r}, t)$ Solid phase displacement field (deformation)

$\rho_s = \rho_s(\vec{r}, t)$ Solid phase (partial) density field

$\rho_w = \rho_w(\vec{r}, t)$ Liquid phase (partial) density field

- The results are useful especially in validation of models
- The data is available on request