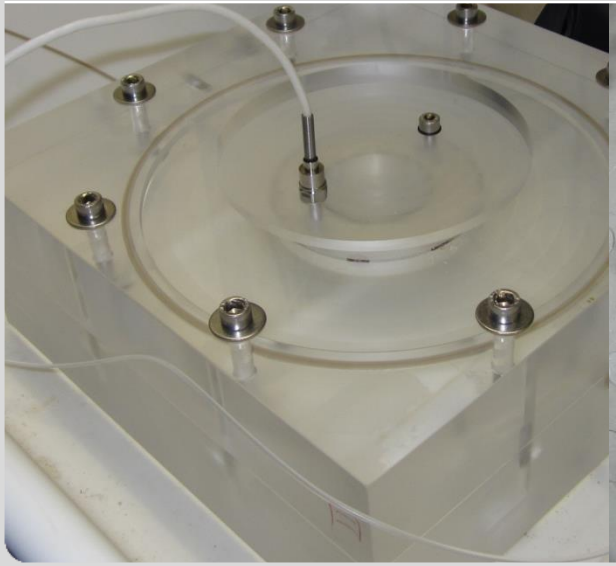
Karlsruher Institut für Technologie

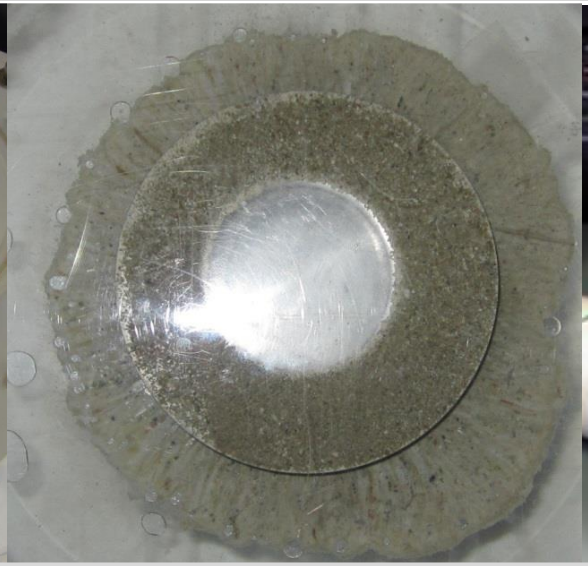
Bentonite erosion experiments

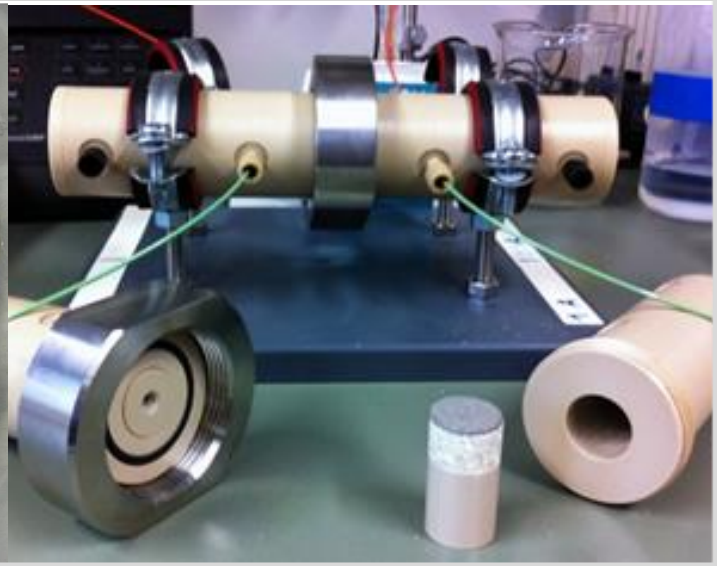
Rinderknecht F.*, Friedrich F., Bouby-Laliron M., Heck S., Götz R., Huber F., Hilpp S., Geyer F., Geckeis H., Schäfer T.

*corresponding author: Franz.Rinderknecht@kit.edu

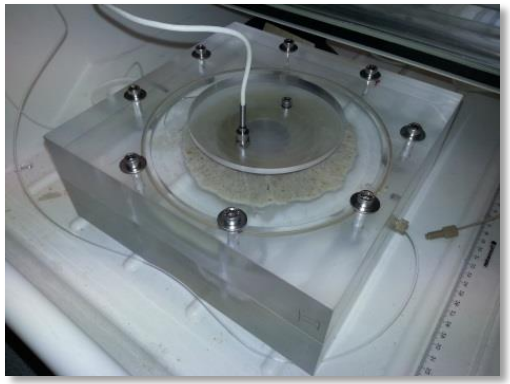
Institute for Nuclear Waste Disposal (INE), Geochemistry



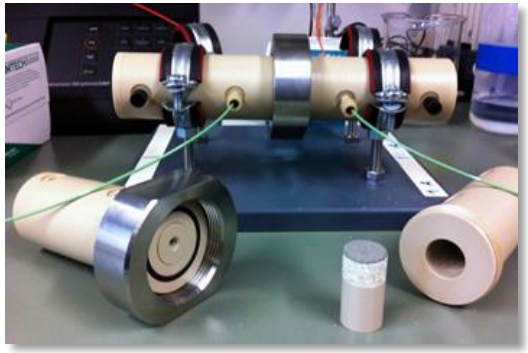




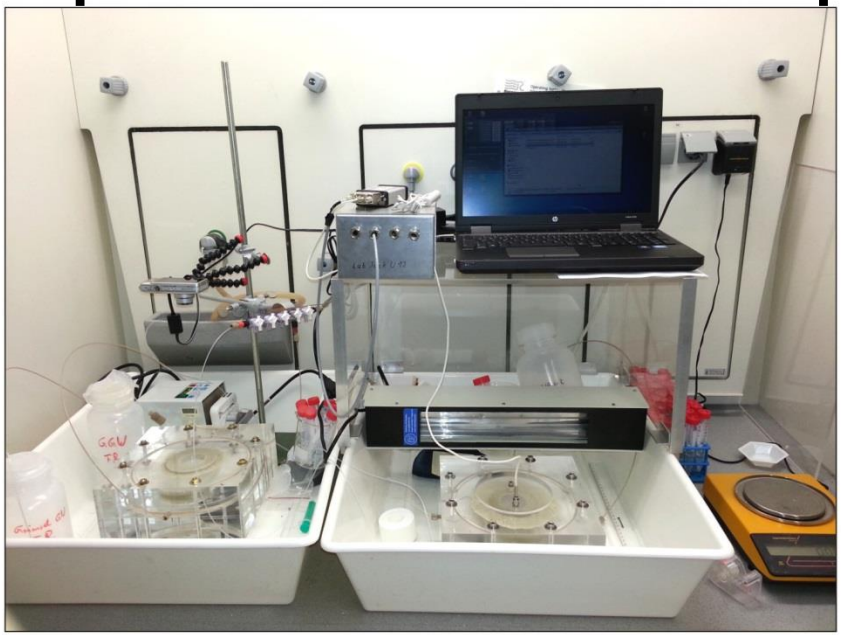
Outline

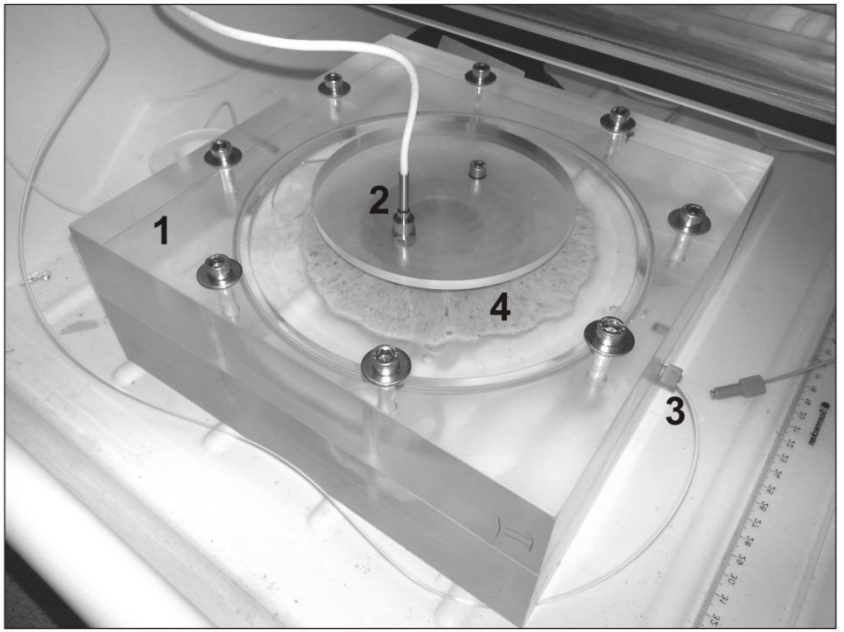


- Artificial fracture Set-up
  - Experimental set-up, bentonite samples and preparation
  - Swelling and characterization of eroded material
  - Sampling for mineralogical analysis
- Update on benchmark test - stagnant phase
- Double-side reactor
  - Experimental set-up, bentonite samples
  - Results
  - Outlook

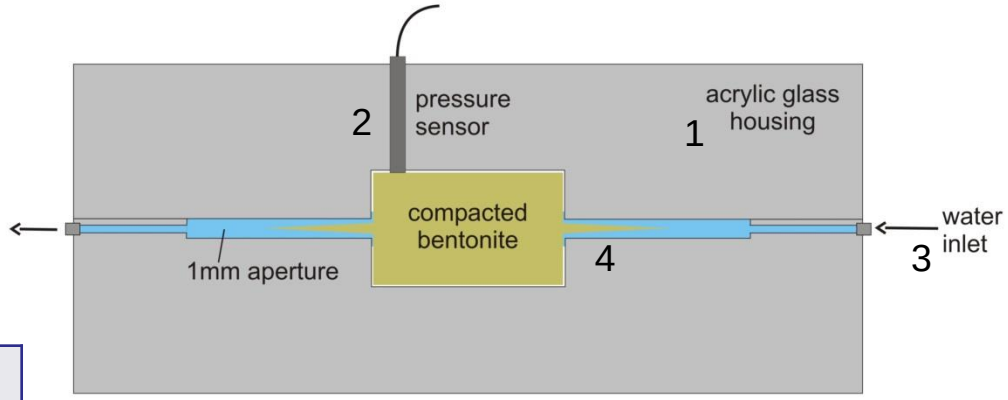


Experimental erosion set-up





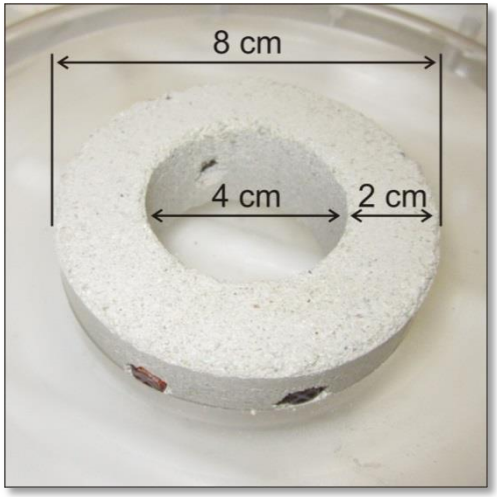
Left	Experiment for the mineralogical characterization of the erosion-“halo”. (running for 30-40 days)
Right	Set-up equipped with pressure sensor - for long-term erosion experiment. (running since October 2013)



side view

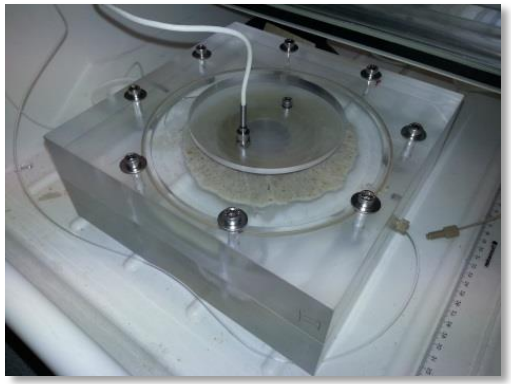
1	acrylic glass housing (with 1mm aperture)
2	the pressure sensor
3	water inlet
4	erosion halo

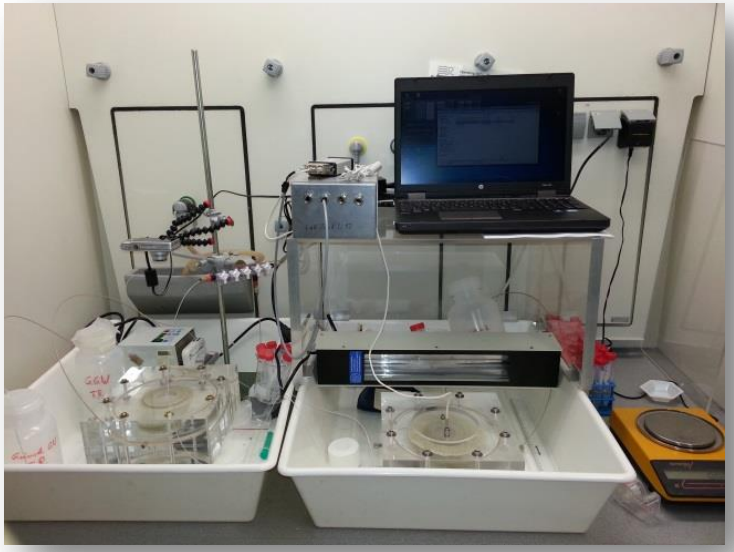
Bentonite sample



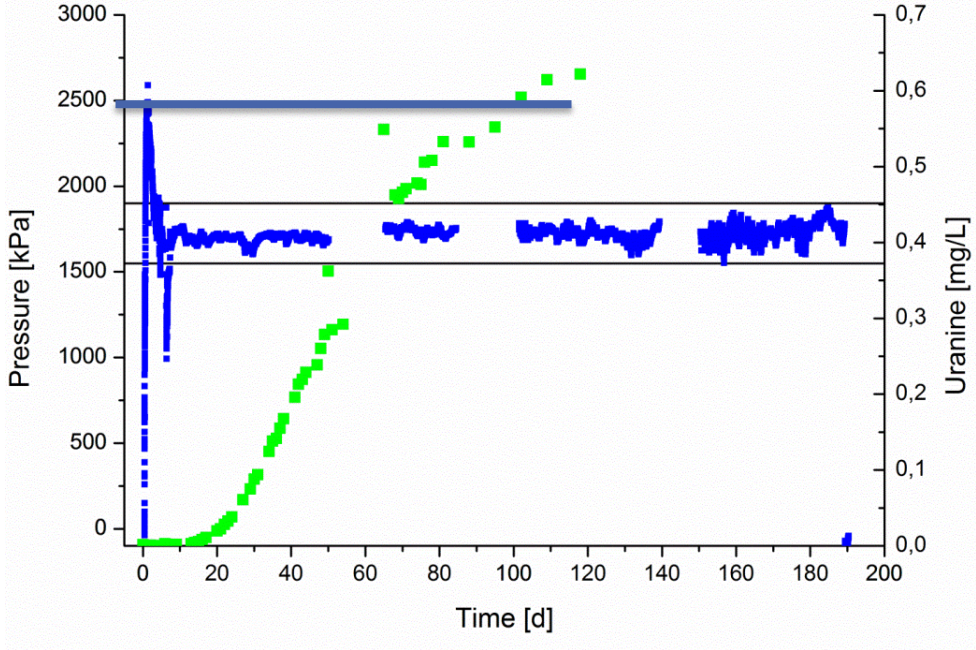
- Since 2013-10:
  - 2 erosion experiments to analyse the swelling zone
  - 1 experiment for effluent characterization and colloid generation
- Compacted Febex Bentonite (ρ = 1.65 Mg/m^3)
- Experiments are performed without presaturation
- 1 Benchmark test in addition
- Deviating in
  - Dry density
  - Bentonite sample
  - Aperture

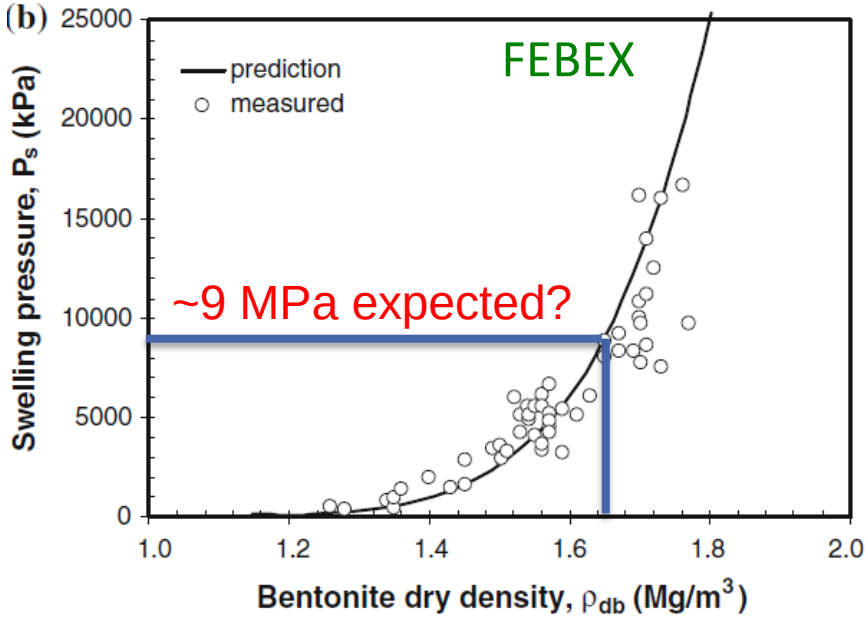
Swelling pressure and fluorescence measurements



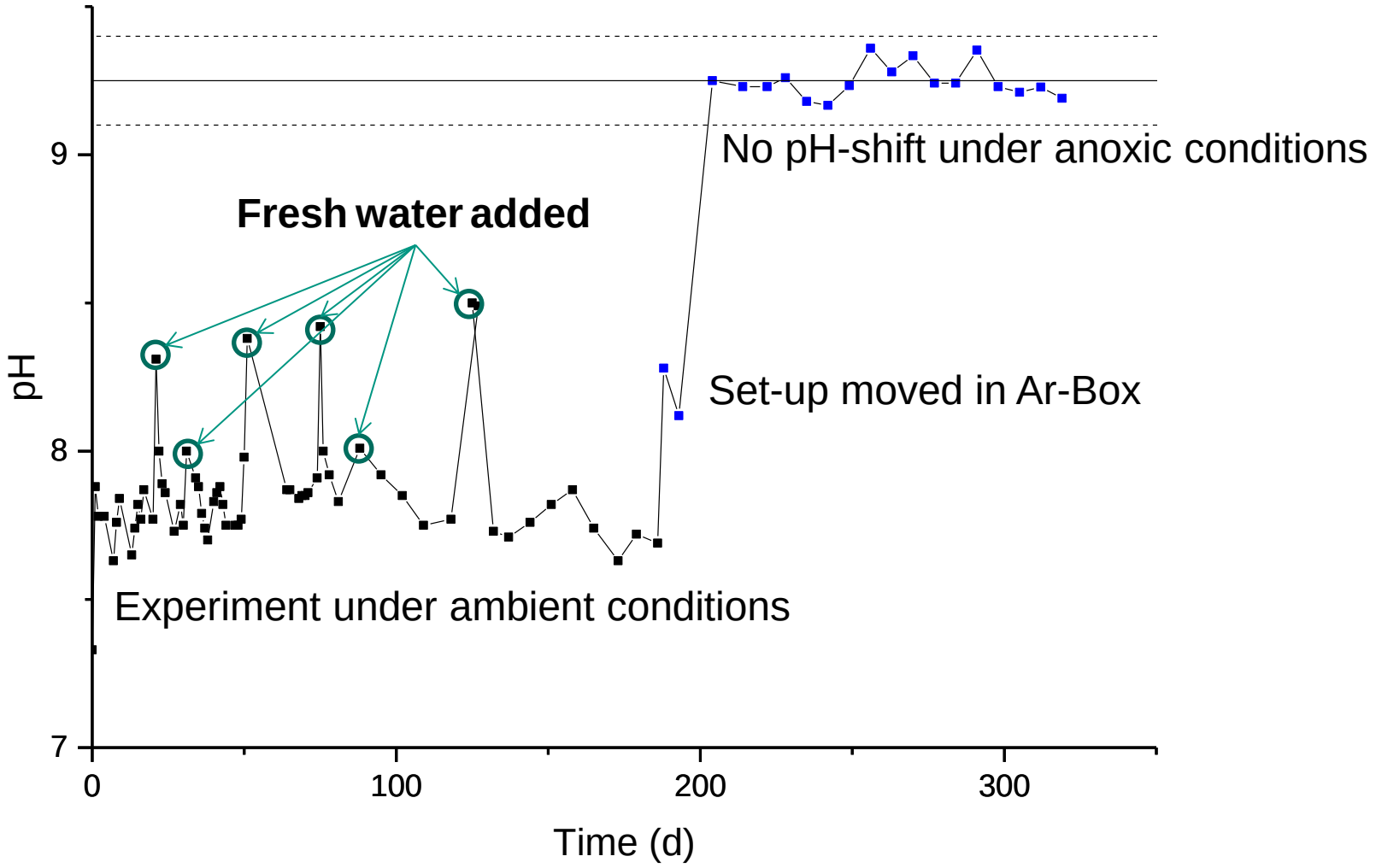


Agus & Schanz (2008) *Acta Geotechnica* 3:125–137

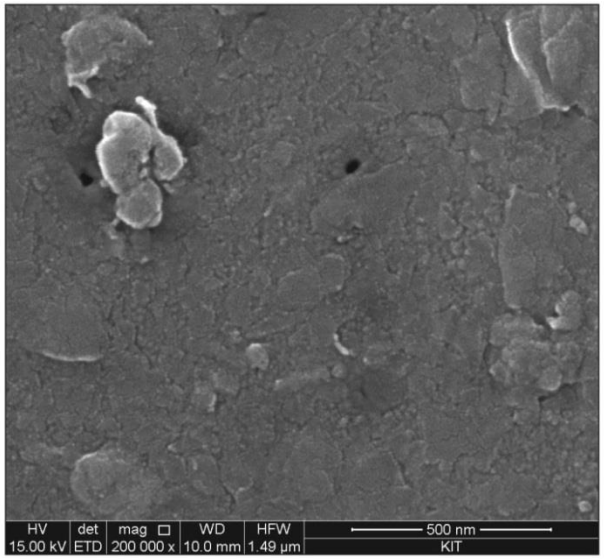


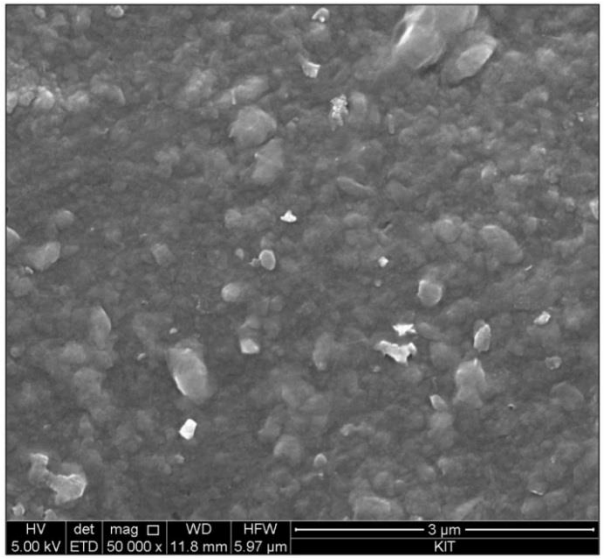


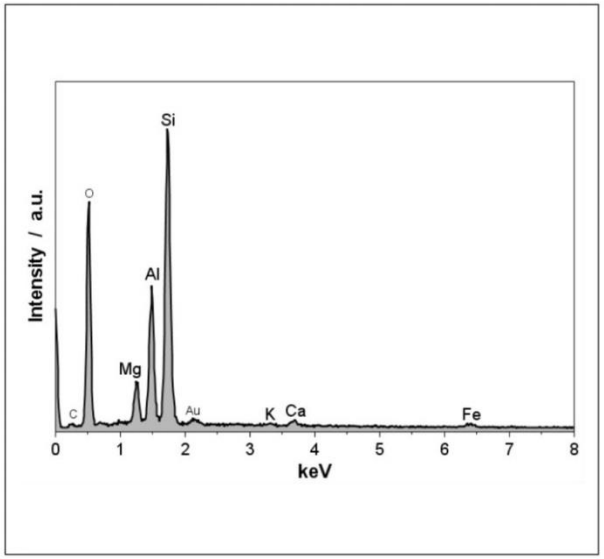
pH-monitoring of the effluent



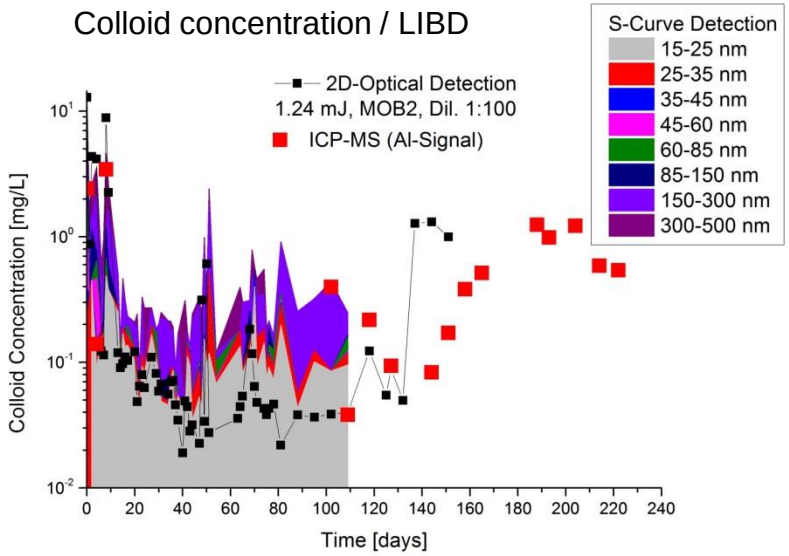
Colloids in the effluent







Colloid concentration / LIBD

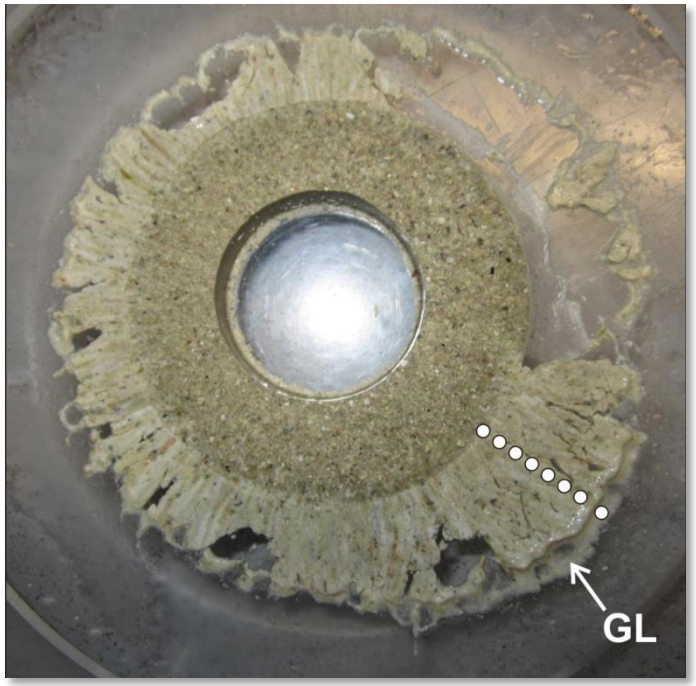


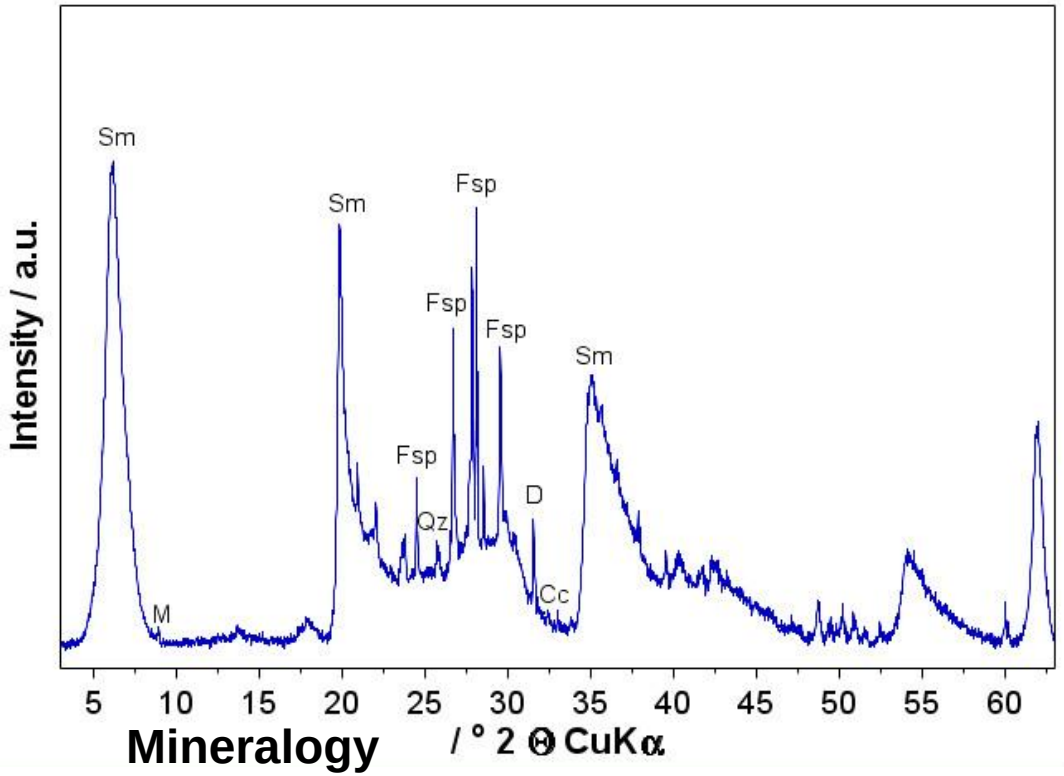
Colloidal concentration in water samples (filter)

Outlook

- Ongoing frequent water sampling
- Analysis of elemental composition, pH, colloid size and concentration
- Radionuclide sorption reversibility studies

Sampling and X-ray Diffractometry





Sampling

XRD: Sampling line with 7 spots from gel-layer (GL) close to the ring

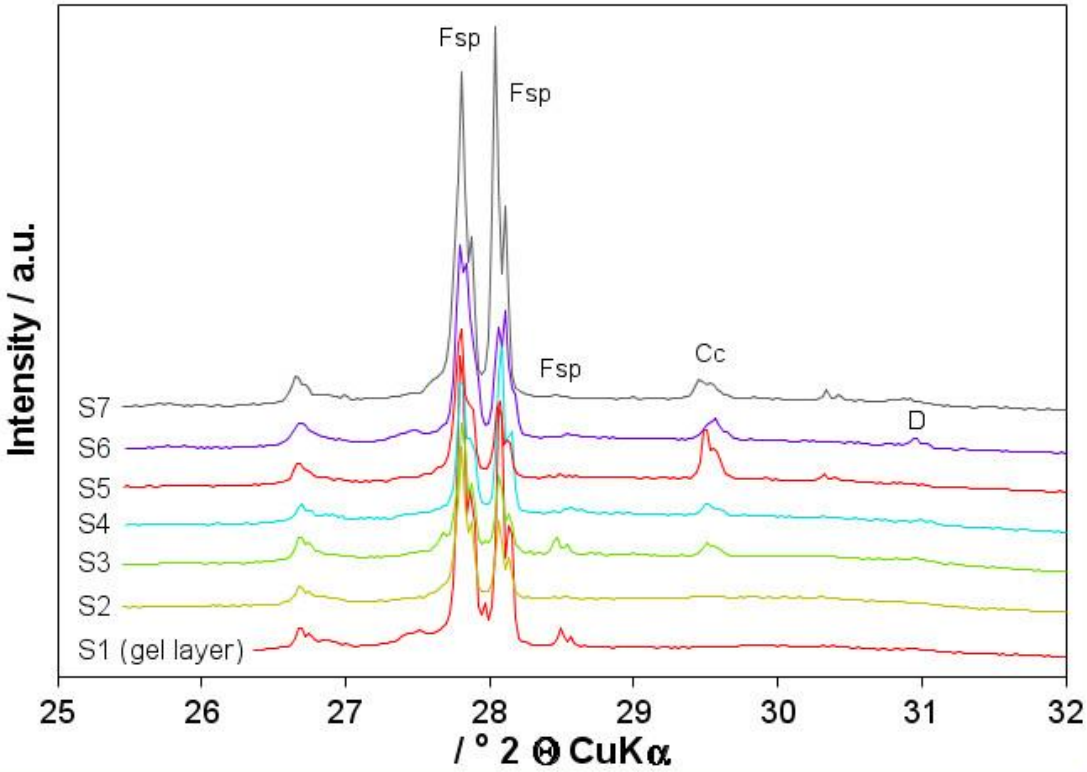
SEM: Samples of gel-layer, halo and ring

Mineralogy

Smectite (Sm)	80
Mica (Biotite) (M)	2
Quartz (Q)	3
2 Feldspars (Plagioclase, Anorthite) (Fsp)	9
Calcite (Cc)	2
Dolomite (D)	3

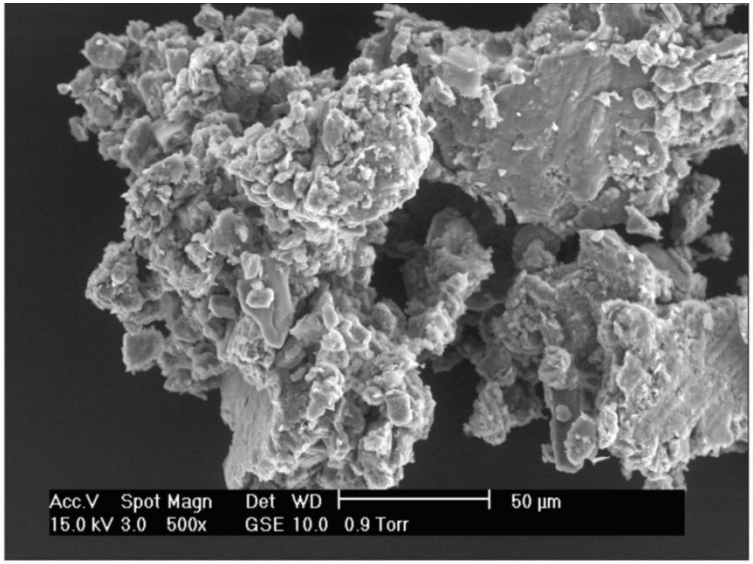
e.g. Caballero et al. (2005) Clay Min., 463.

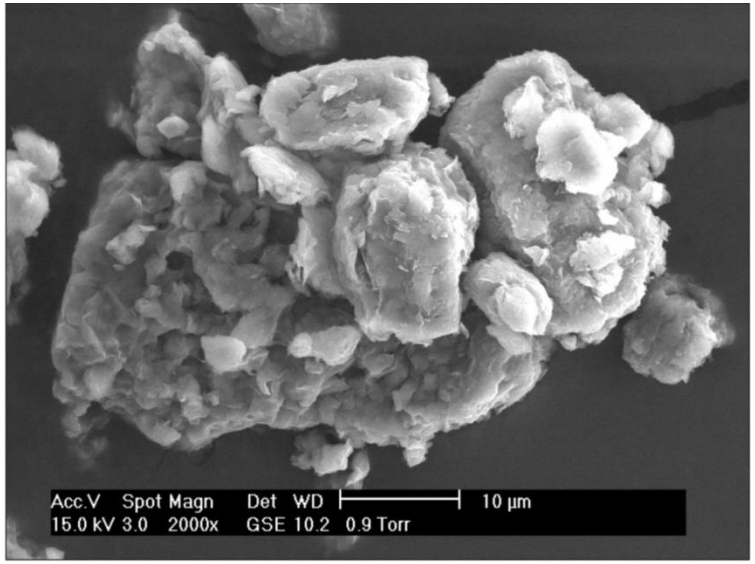
X-ray Diffractometry

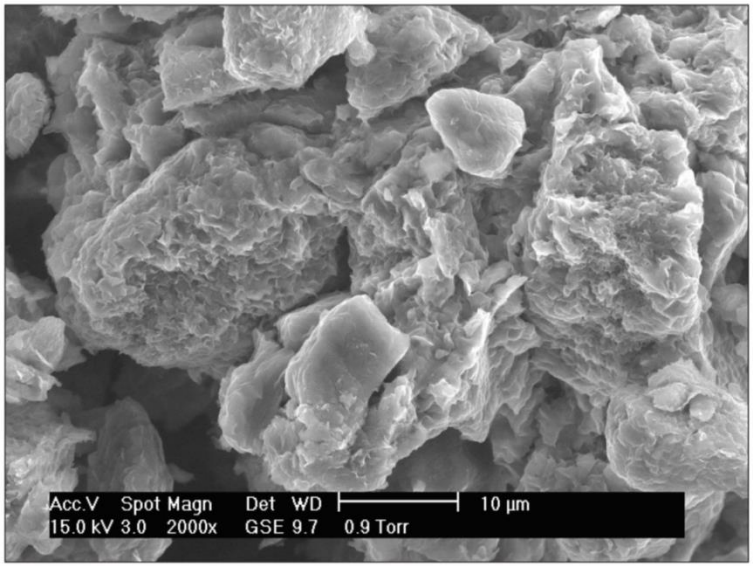


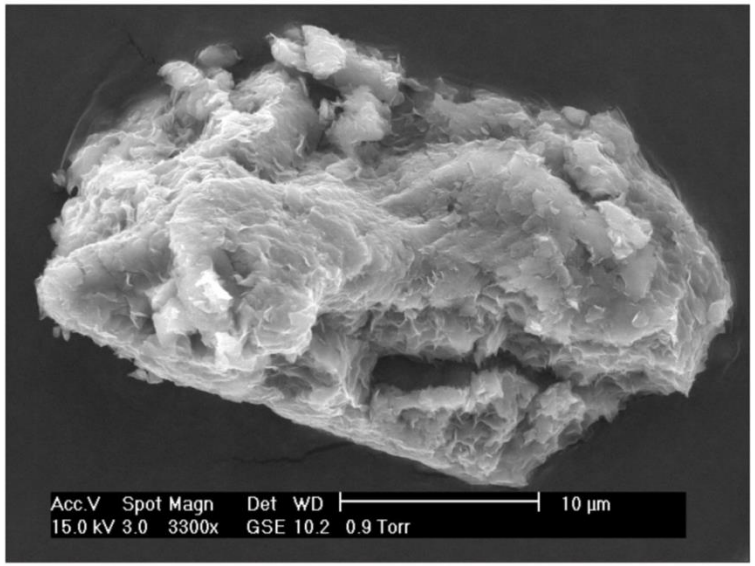
Experimental set-up, bentonite samples	Smectite	Mica	Dolomite	Ca-Feldspars	Calcite
S7 (near ring)	++	-	+	+	+
S6	++	-	-	+	+
S5	++	-	-	+	+
S4	++	-	-	+	+
S3	++	-	-	+	-
S2	++	+	-	+	-
S1 (gel-layer)	++	+	-	+	-

Electron Microscopy / Gel Layer

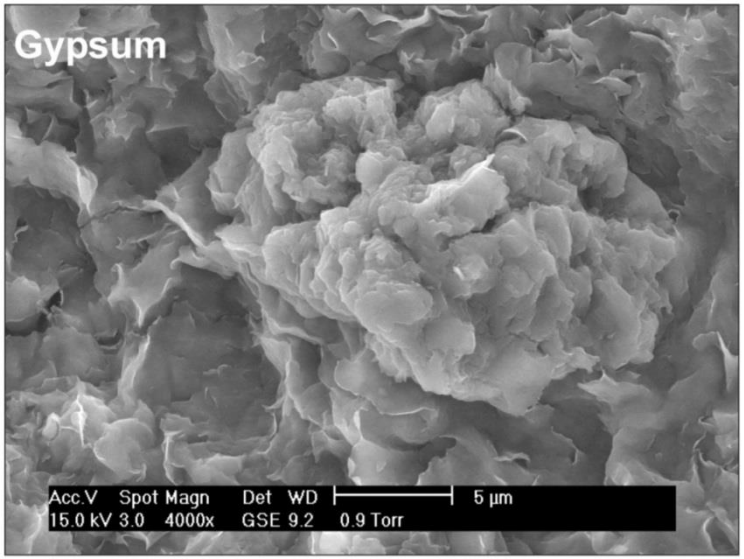


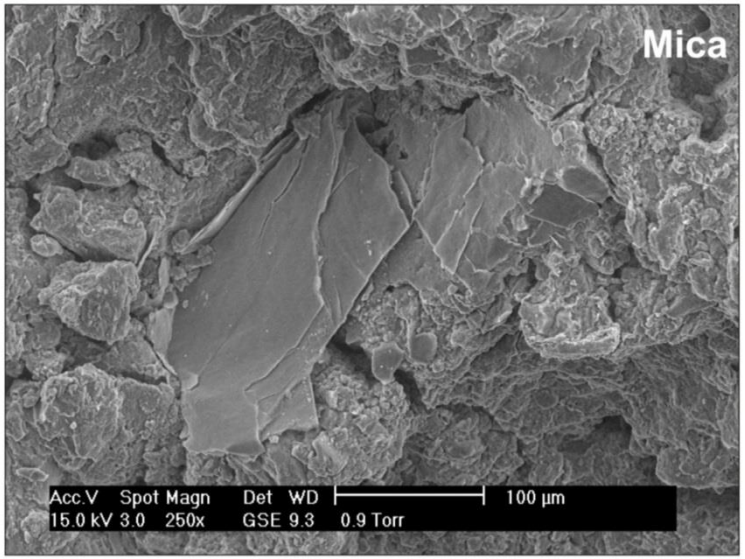


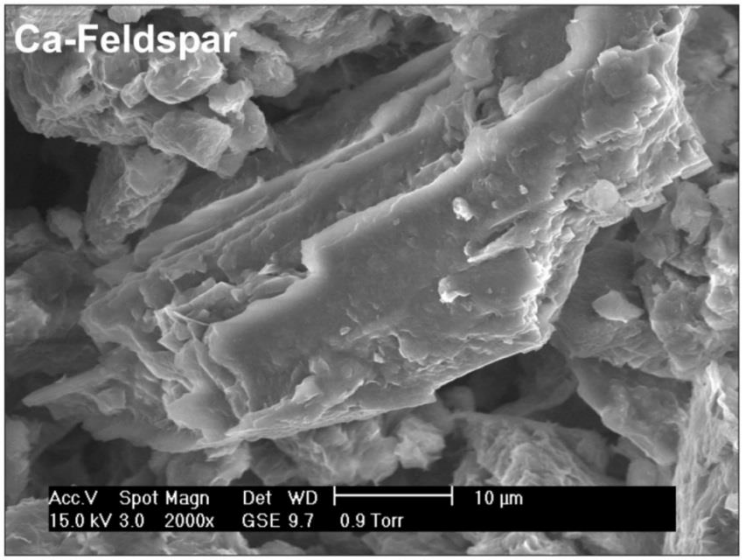


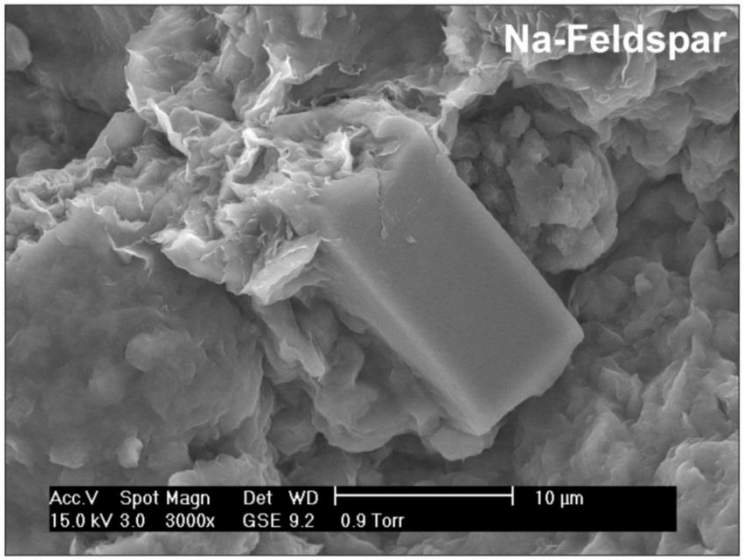


Electron Microscopy / “Halo“



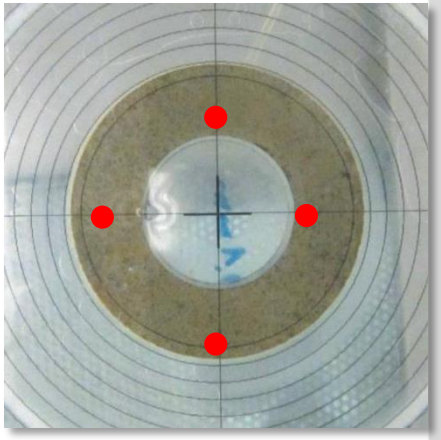






Update on the benchmark test

- Artificial fracture set-up is modified (aperture 1mm → 0.1 mm)
- Pressure monitoring is possible at 4 different positions
- Presaturated (first run) and not presaturated bentonite rings were provided by CIEMAT

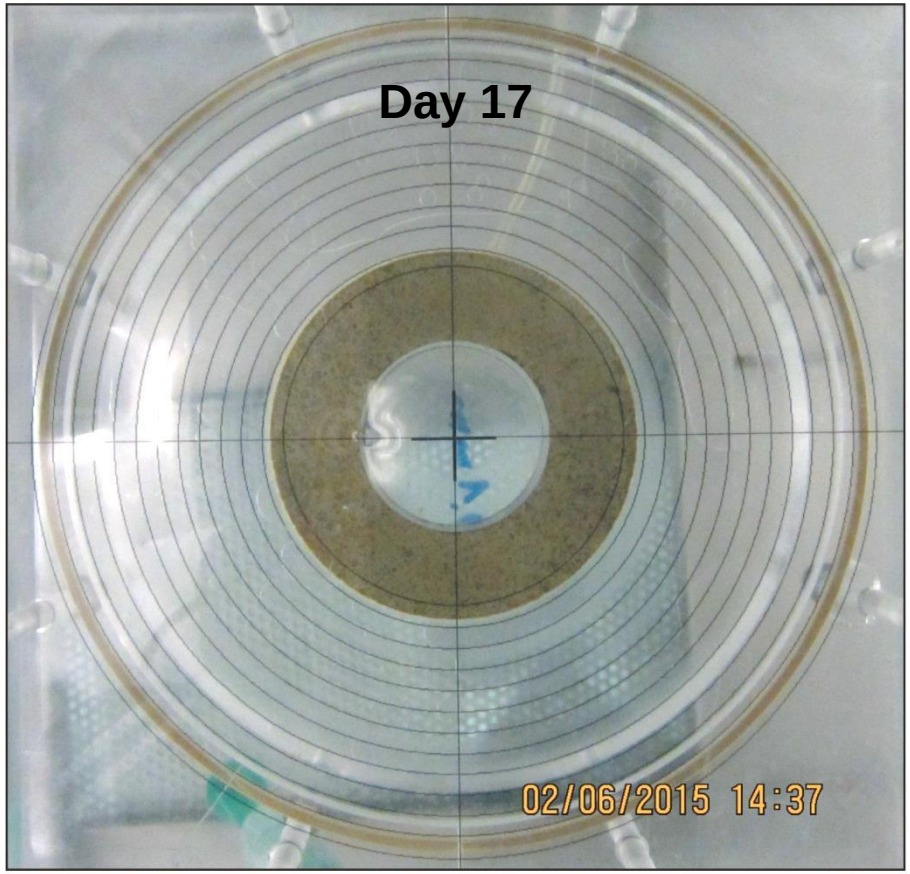


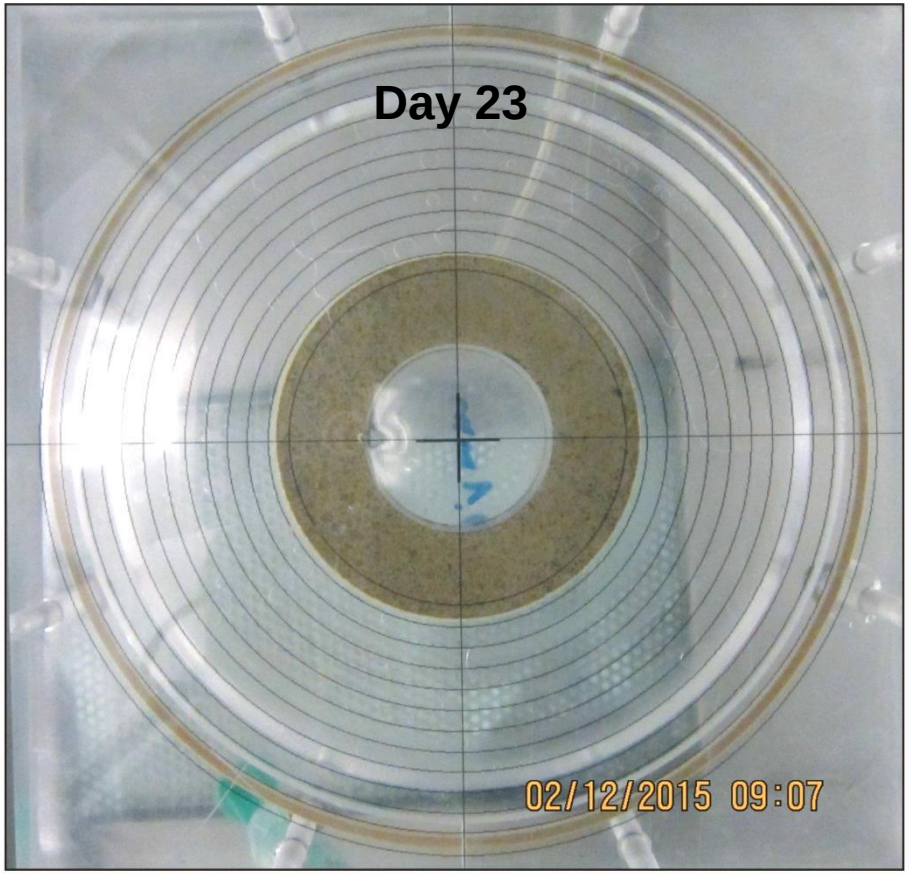
Timescale	Material and parameters
30 days: no flow ✓ 28 days; flow conditions • 14 days: 10^{-6} m/s • 14 days: 10^{-4} m/s	Sample: Na-montmorillonite (PGN, Nanocor Inc.) Dry density: 1.4 Mg/m^3 Test solution: 1mM NaCl in deionized water Presaturation with test solution prior to test (CIEMAT)

Phase 1: Swelling under Stagnant Conditions

- Nearly no swelling within the first 20 days!
- Problems with air bubbles

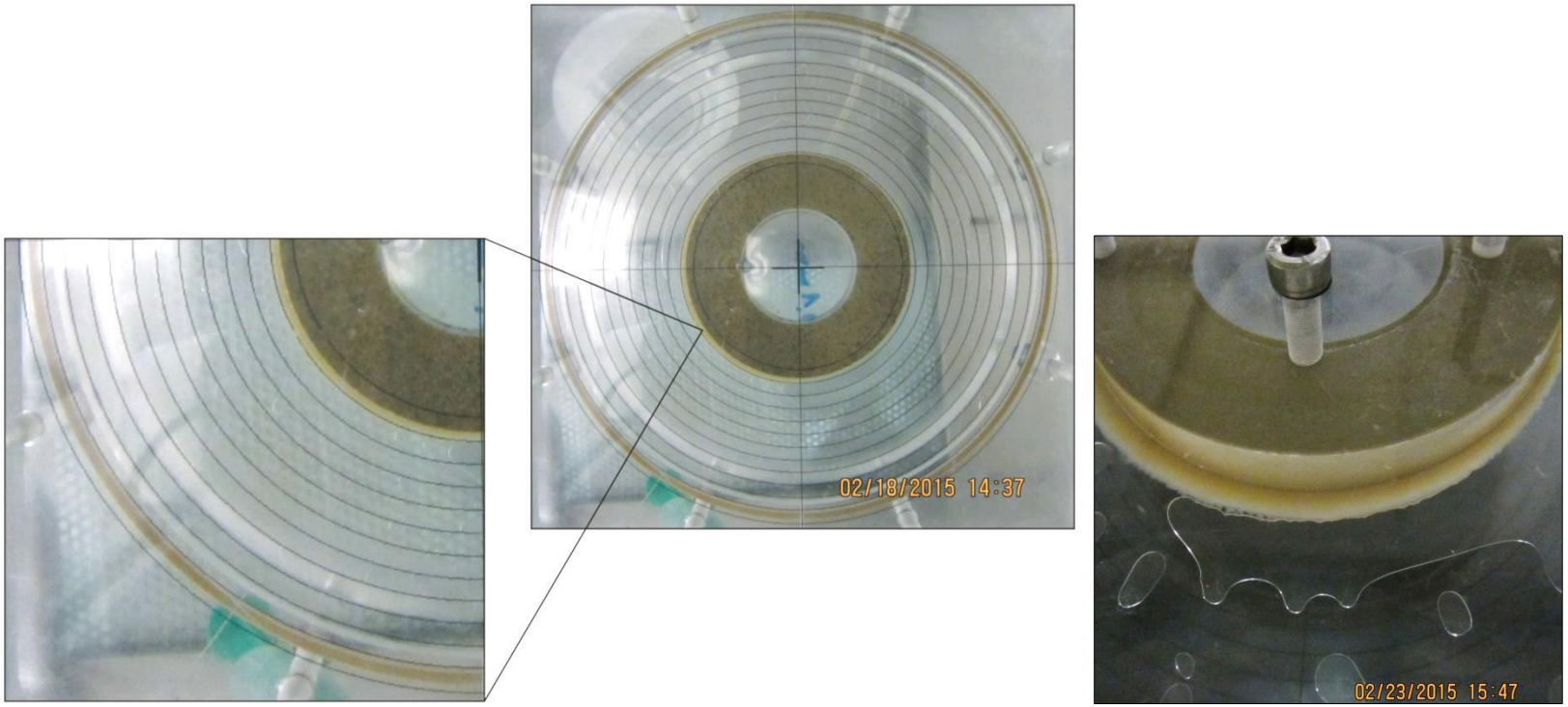
- Refill of solution was necessary nearly every day!





Phase 1: Swelling under Stagnant Conditions

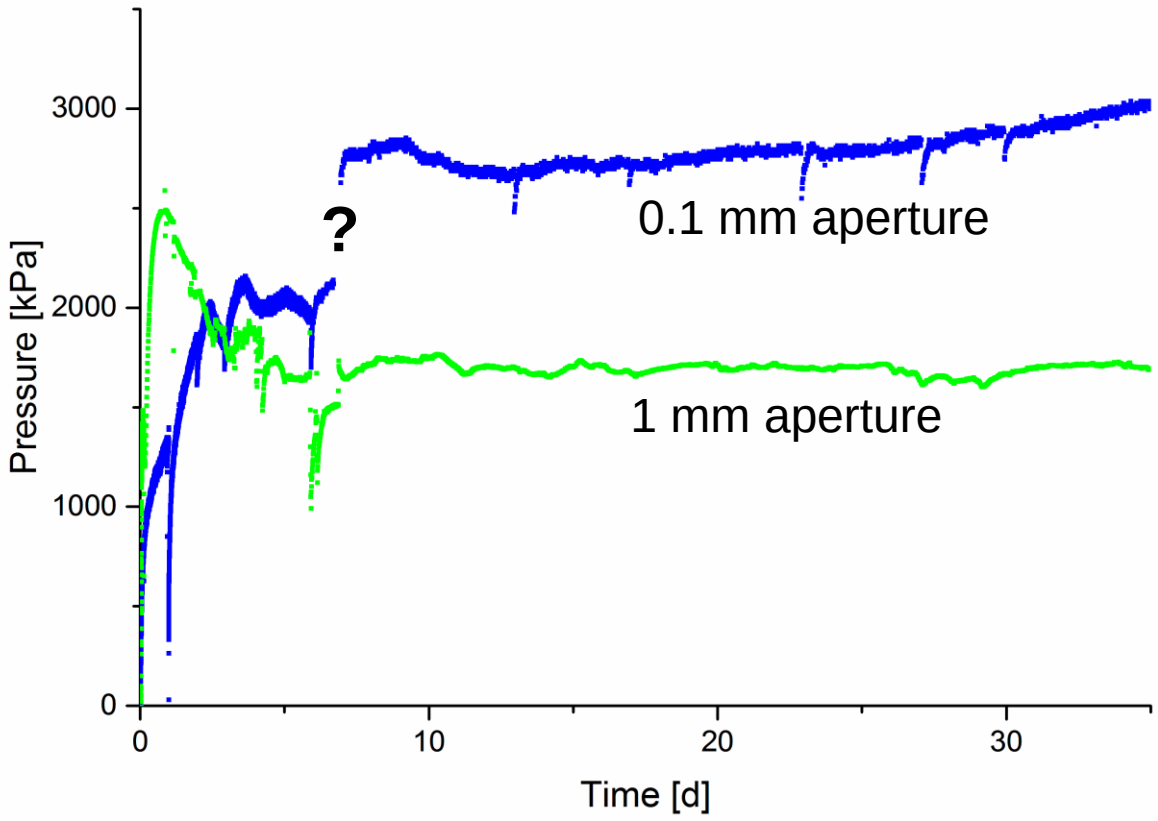
Thin halo visible after ~ 25 days



Phase 1: Swelling under Stagnant Conditions

Pressure

1 mm Aperture vs. 0.1 mm aperture

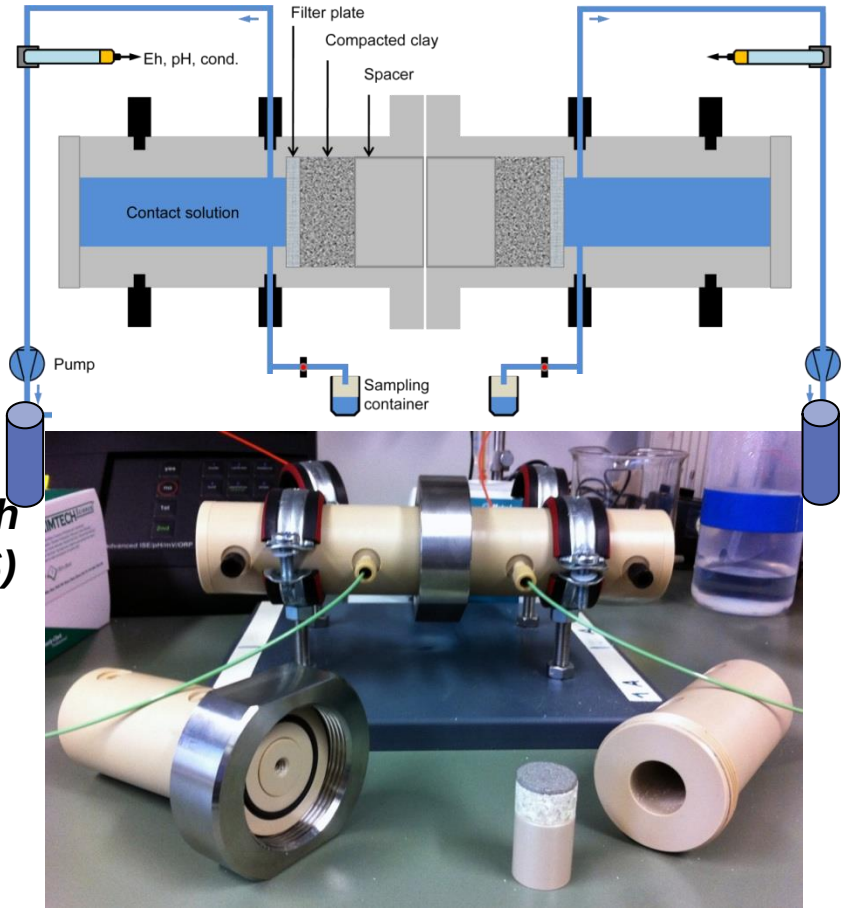


Bentonite erosion experiments under quasi-stagnant flow conditions: evolution of the physico-chemical parameters

Design: double-side reactor

M. Bouby, S. Heck, S. Hilpp, F. Geyer, T. Schäfer

- *Compacted MX80 (Volclay); 1.6 Mg/m³*
- *Erosion of one side of the pellet*
- *2 Na-exch., 2 Ca-exch., 2 Na/Ca-exch. (50/50), 2 raw … (duplicate expts)*

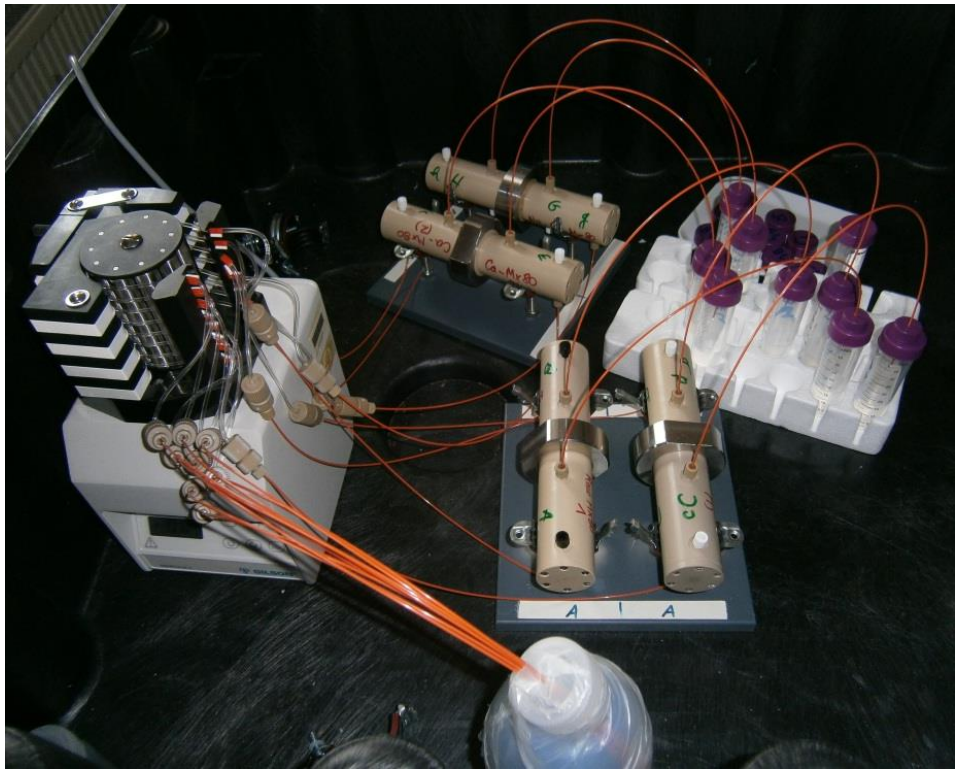


- *Synthetic ground water simulating a fresh age ice melt water (high pH, carbonated, low IS)*

- **pH = 8.4, IS ~ 1. 3 10^{-3}M**

- **Na^{+} 1.2 mM – 28.4 mg/L**
- **Ca^{2+} 0.05 mM – 1.5 mg/L**
- **F^{-} 0.1 mM – 2.8 mg/L**
- **Cl^{-} 0.074 mM – 2.64 mg /L**
- **SO_4^{2-} 0.04 mM – 4.13 mg/L**
- **Si 0.5 µM – 14 µg/L**
- **HCO_3^{-} 1mM – 84 mg/L**

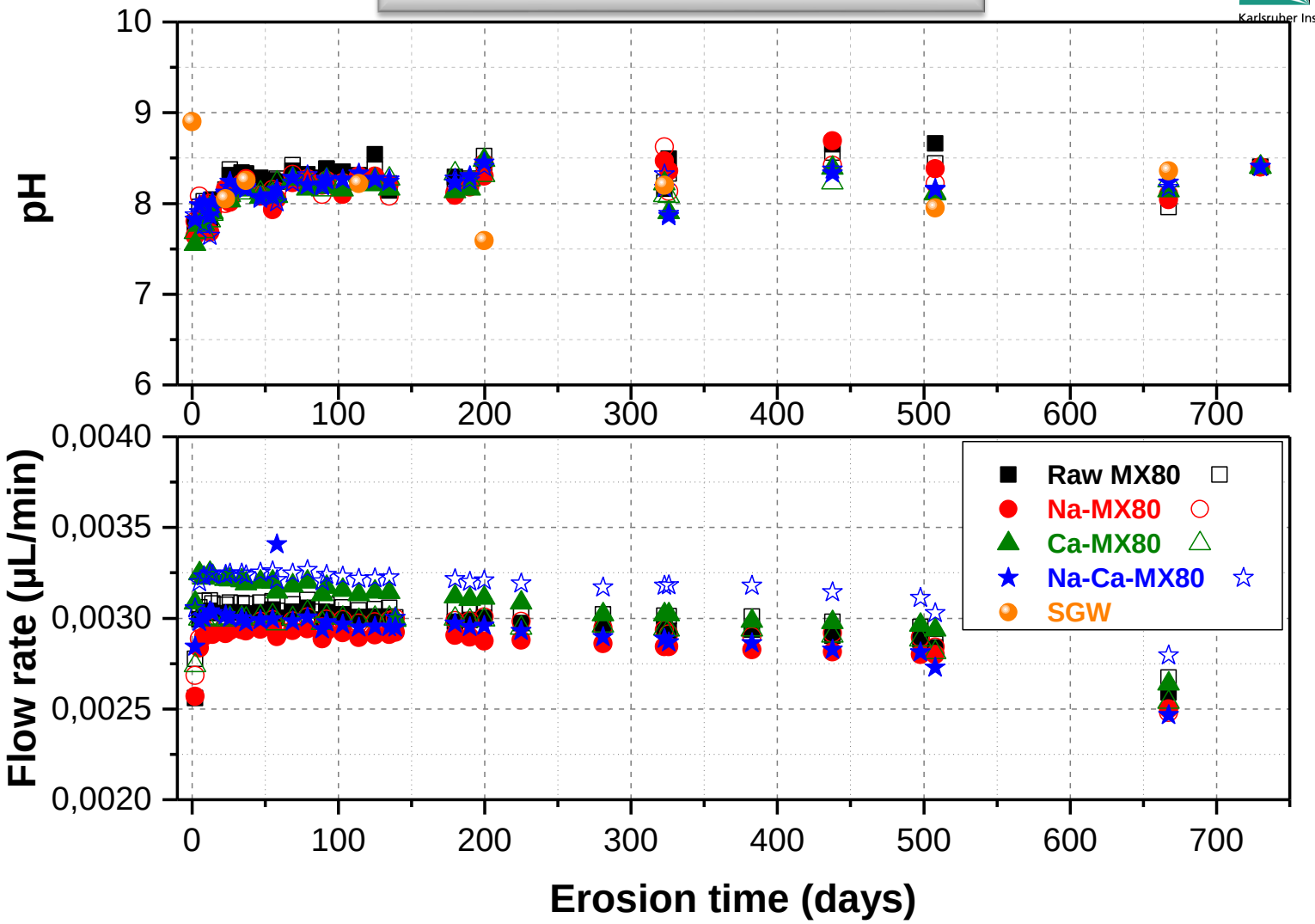
- *3 µL/min = 2.3 10^{-4} m/s or 7261 m/y*
- *Continuous elution (no recirculation)*
- ***Started 13/03/13 (2 year ago)***
- *Regular samplings*



Analysis

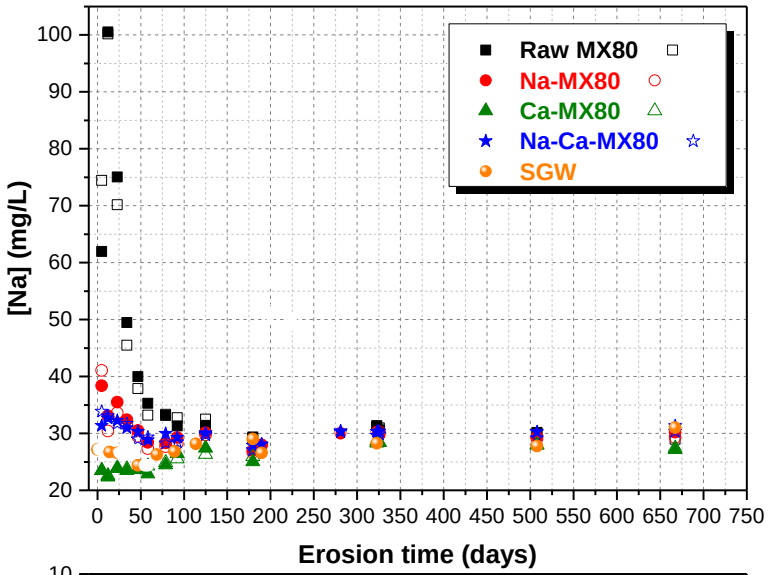
- **pH**
- **flow rates control**
- **χ composition evolution (IC, ICP/OES-MS)**
- **Colloid size determination and concentration**
- **RNs sorption behavior test with the eroded material**

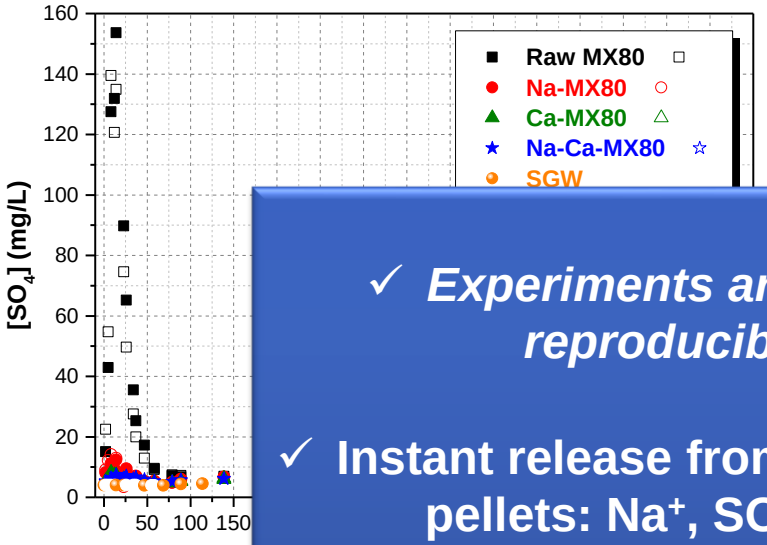
pH, flow rate evolution

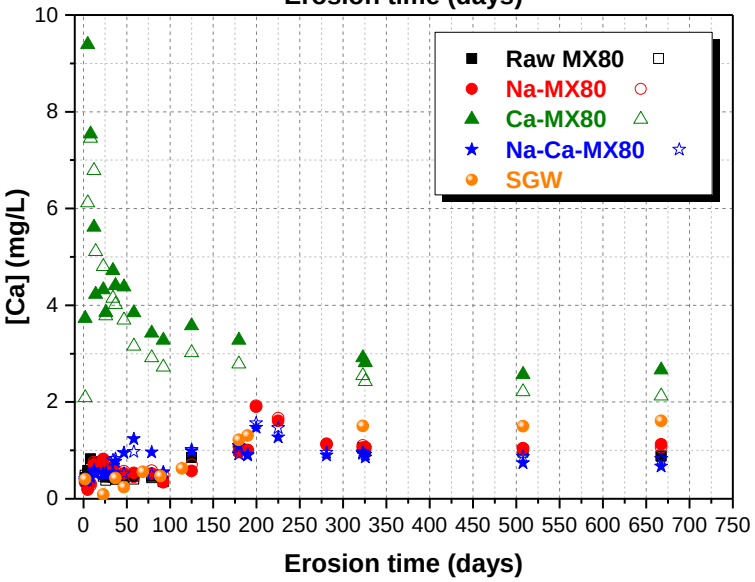


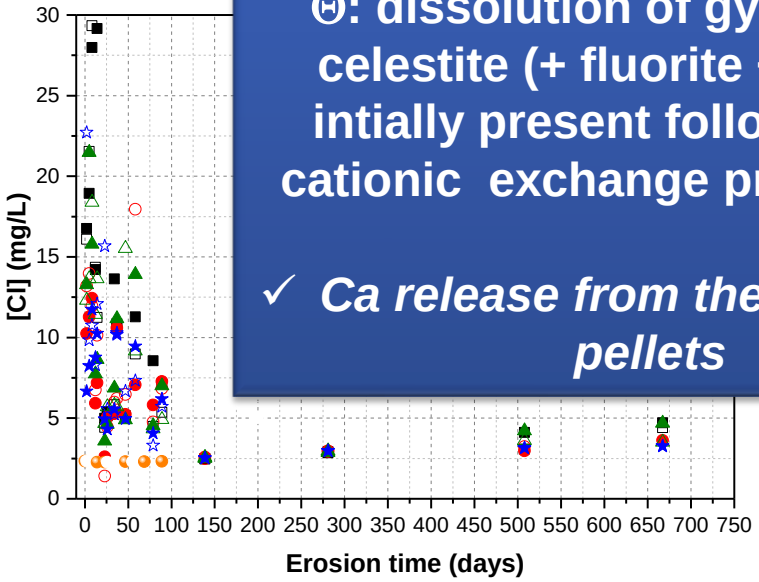
Constant pH and flow rates!

[Na^+, Ca^{2+}, SO_4^{2-}, Cl^-] evolution



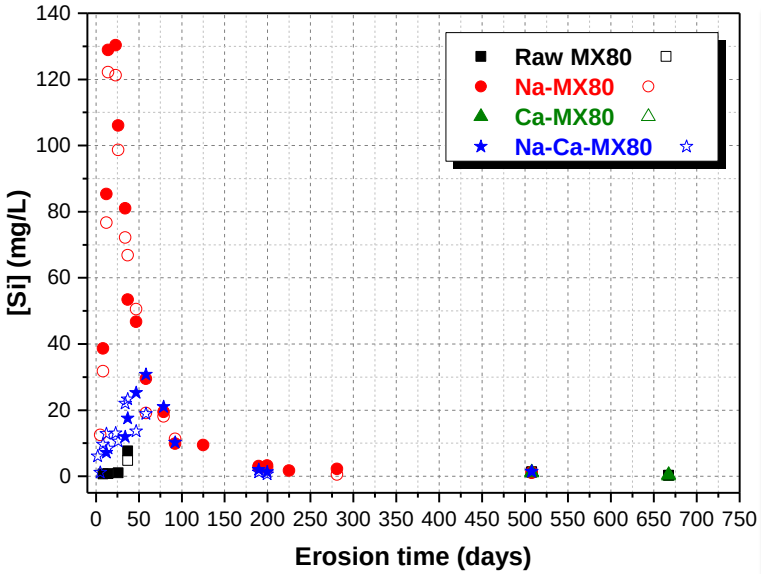


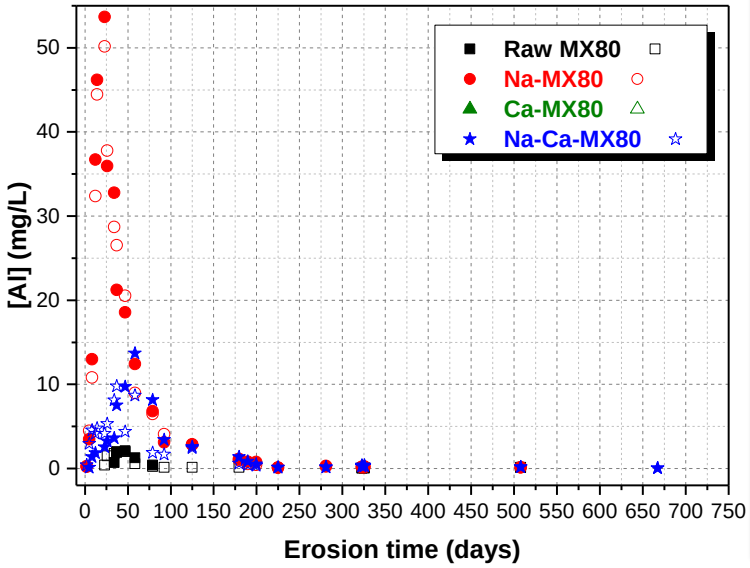




- ✓ *Experiments are very reproducible*
- ✓ **Instant release from the RAW pellets: Na^+, SO_4^{2-}, Cl^-**
- **Θ: dissolution of gypsum + celestite (+ fluorite + NaCl) intially present followed by cationic exchange processes**
- ✓ *Ca release from the Ca-MX80 pellets*

[Si, Al, Fe, Mg] evolution

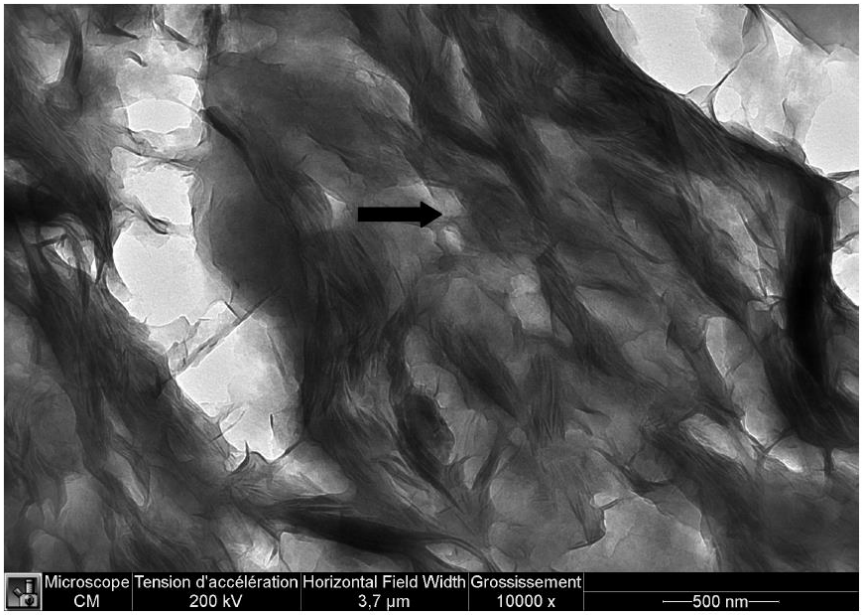




- ✓ *Experiments are very reproducible*
- ✓ *Eroded material detected and*
- ✓ *Identified as MX80 colloids from the [Si]/[Al] and [Al/Mg] ratios*
- ✓ *Pronounced effect of the cation-exchange process:*
  Ca-MX80 no erosion or <<<< raw << Na-Ca-MX80 <<<Na-Mx80
- ✓ *When effective, colloids production maximum after ~ 25 days up to ~ 500 mg/L*
- ✓ *Decrease of production and level off after 6 months*

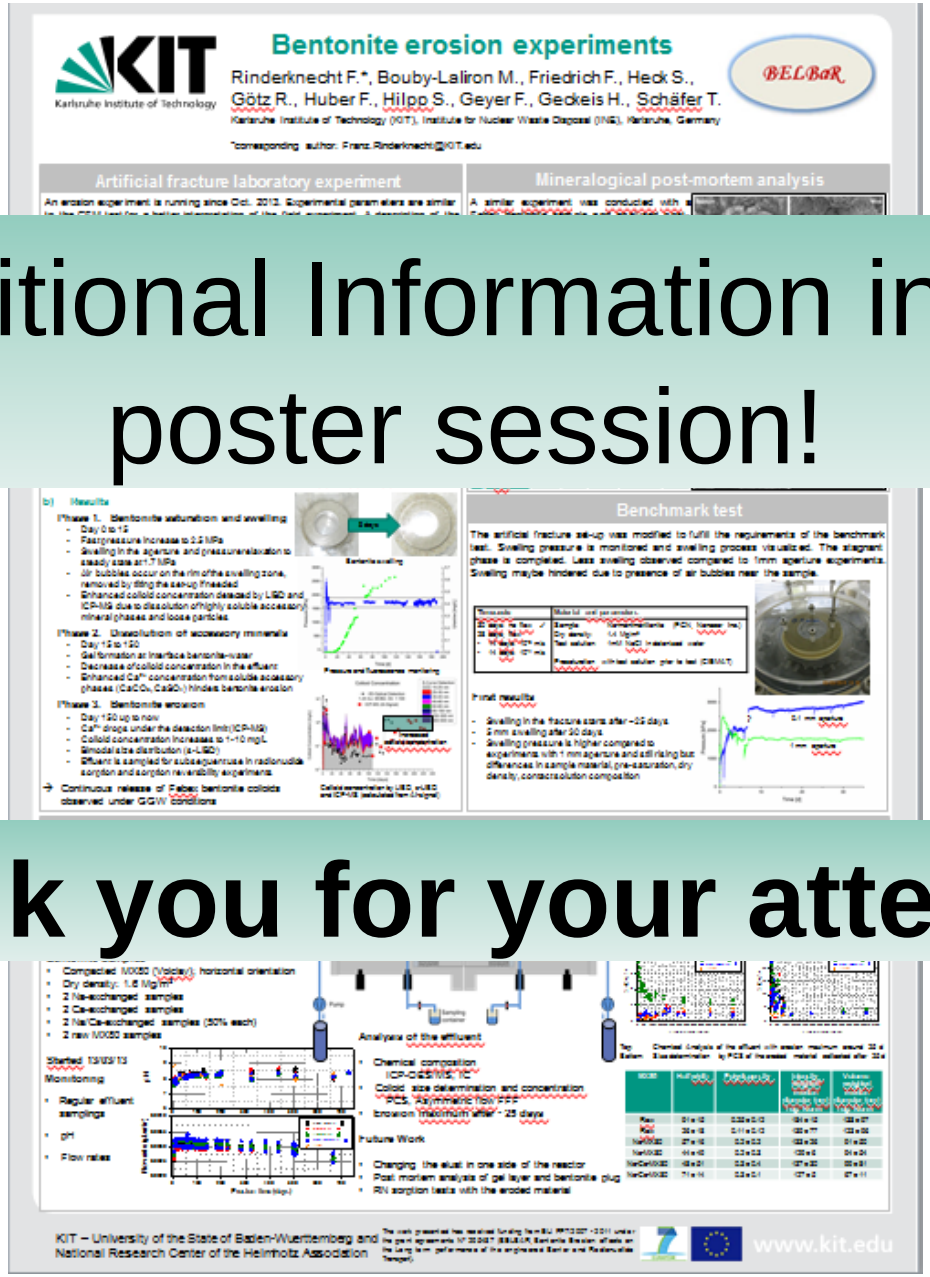
Perspectives 2015 and beyond…

- **New (RNs) sorption test with eroded material**
- **Changing the erosion/elution in one side keeping the other as witness**
- **Post-mortem analysis (gel layer and bentonite plug characterization) as previously tested in past experiments**



Water exchange to 1) methanol and 2) to an epoxy resin followed by hardening

- Cutting (FIB, microtome)
- Further characterization (LA-ICPMS, XRD, STEM-HAADF)



Additional Information in the poster session!

Thank you for your attention!