LABORATORY EXPERIMENTS TO UNDERSTAND COUPLED PROCESSES IN CLAY-BASED BARRIERS UNDER HIGH TEMPERATURE

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OUTLINE

- **BACKGROUND**: PROCESSES IN THE BUFFER, ITS CHARACTERISTICS AND WHAT WE KNOW ABOUT THE EFFECT OF TEMPERATURE
- THE EUROPEAN **HITEC PROJECT**
- DIFFERENT APPROACHES TO **TEST THE EFFECT OF TEMPERATURE ON THE BUFFER IN THE LABORATORY**

BUFFER CHARACTERISTICS

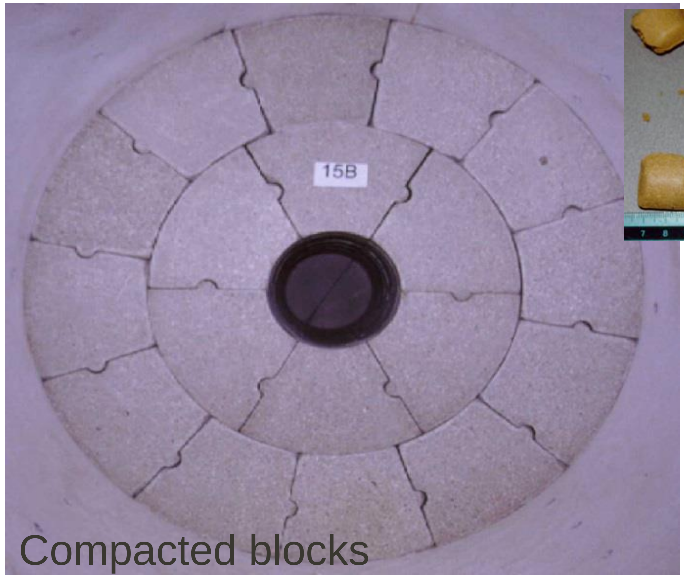
•Bentonite (expansive, smectite-rich material, can retain elements in its structure)

•Mixtures of bentonite and aggregates: crushed granite, basalt, quartz, zeolytes, graphite

• They can be prepared in the form of compacted blocks or as mixtures of pellets and powder or pellets of different sizes

•Blocks can be compacted from the material with its hygroscopic water content or previosly mixed with water: different initial degrees of saturation

•In the same barrier only blocks, pellets (vertical configuration) or combinations of both can be used



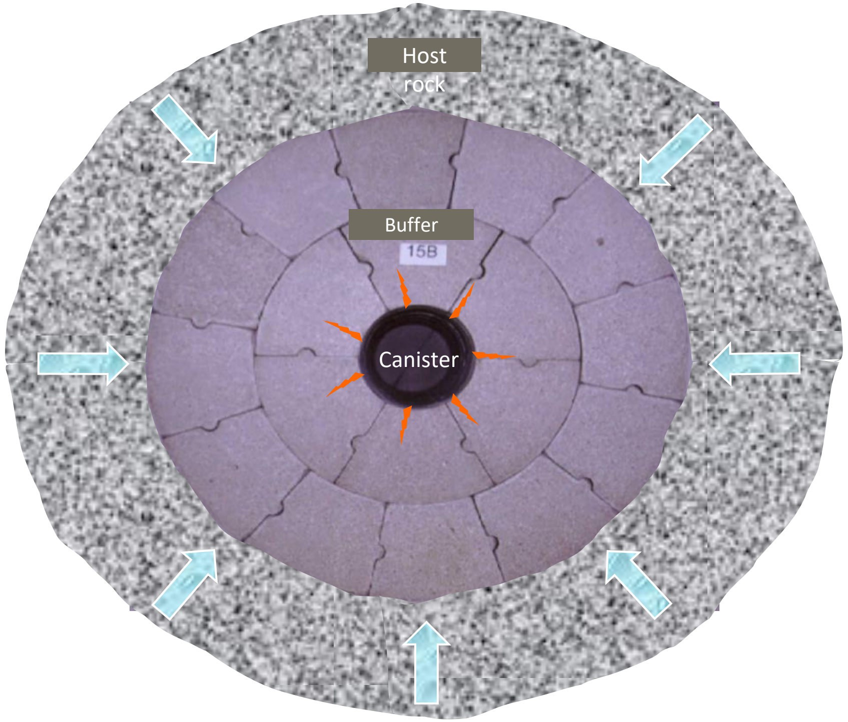
Compacted blocks

Pellets

Pellets/powder mixture

Different-size pellets

THE BARRIER DURING THE TRANSIENT STAGE



Hydration:

Development of swelling pressure

Sealing of voids, microstructural reorganisation

Compression of air in pores

Dissolution/advection of chemical species
→ changes in pore water composition

Exchange reactions

Heating:

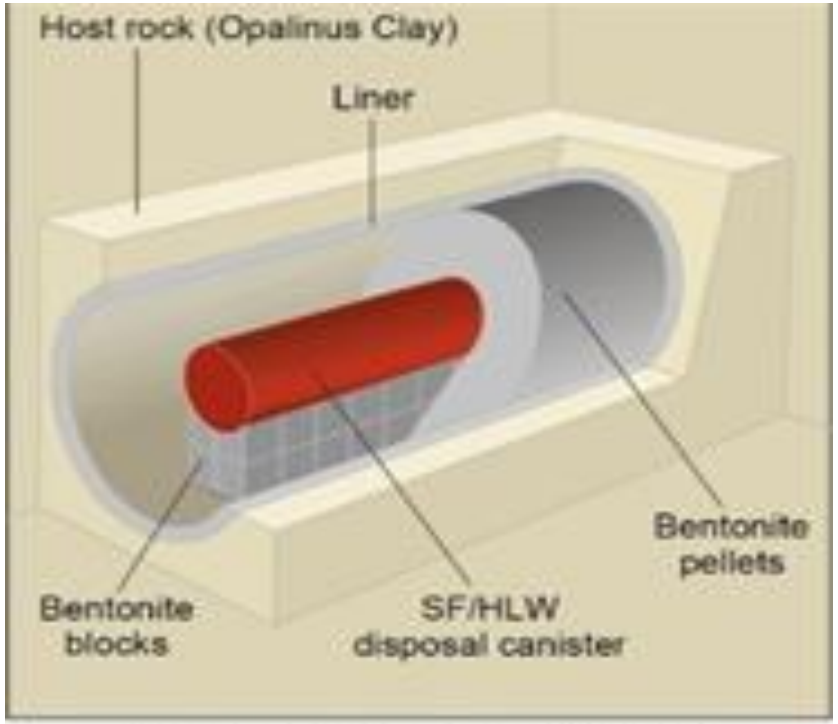
Drying near the container: cracking?

Vapour convection/advection

Precipitation of mineral species

Exchange reactions, mineral transformations

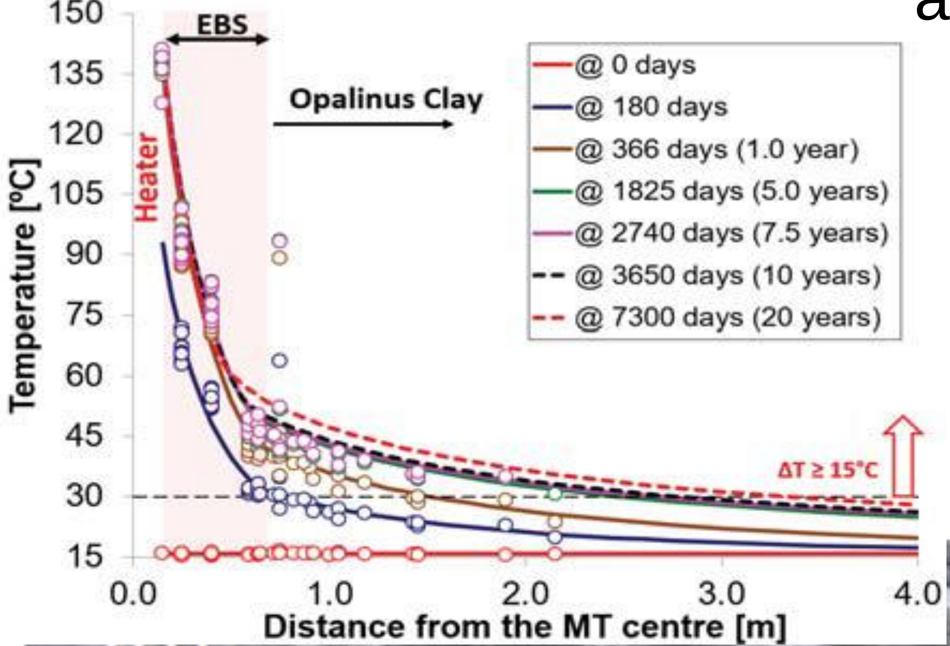
Gas generation

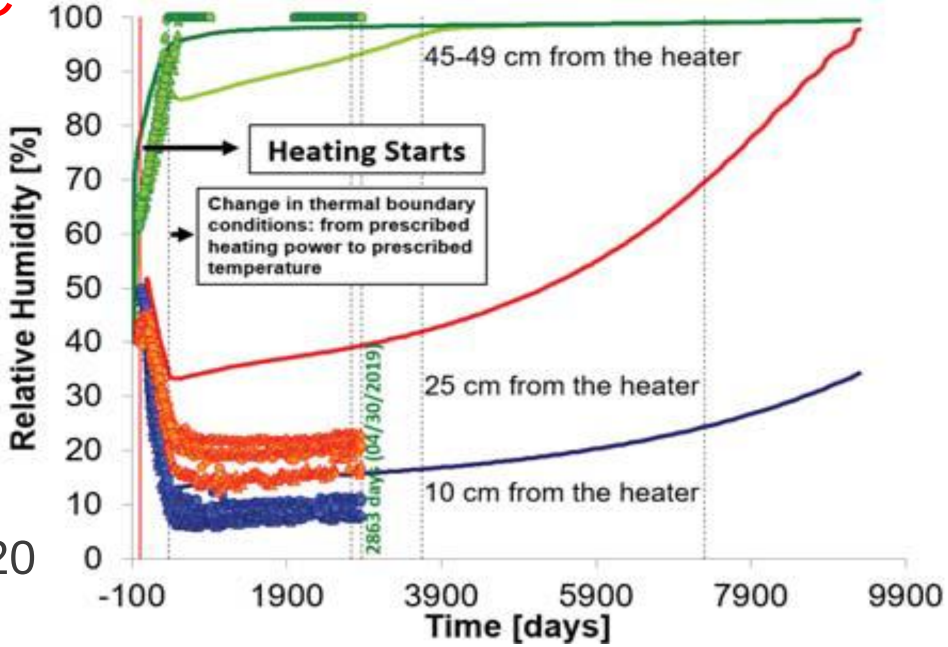


Emplacement drift for SF/HLW

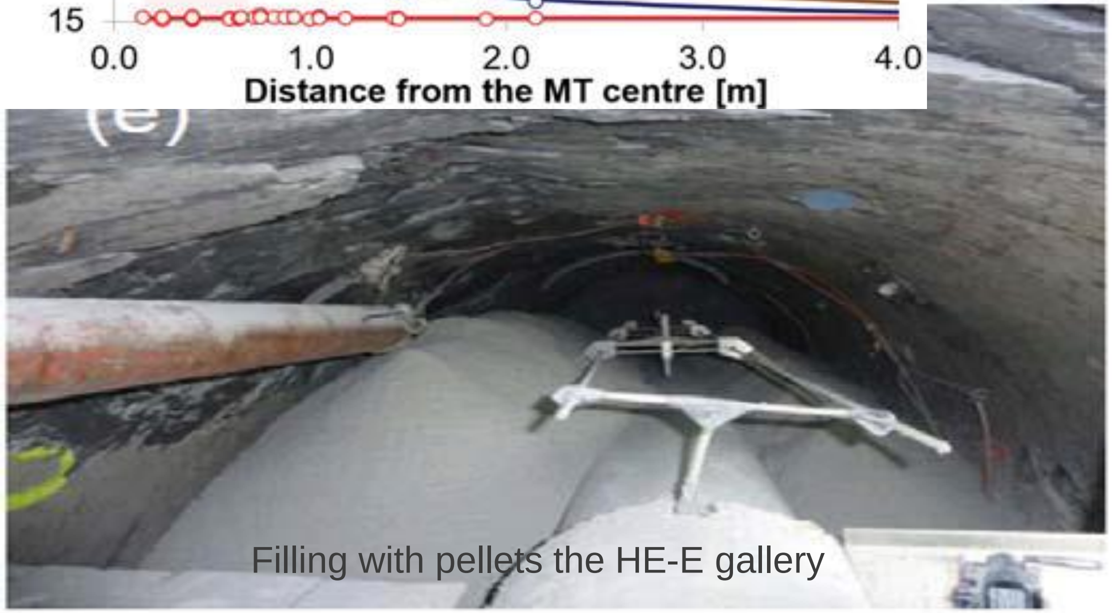
TEMPERATURE AND THERMAL GRADIENT AFFECT SATURATION (TH COUPLING)

Repository with limited water supply (Opalinus clay host rock, Mont Terri) and heater *T*=140°C

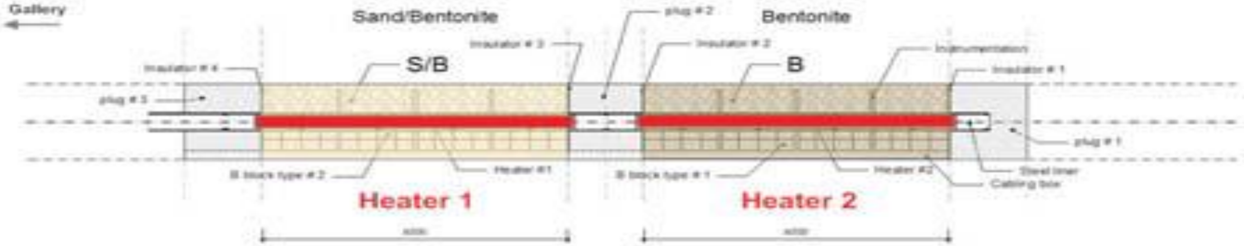




Gens et al. 2020



Filling with pellets the HE-E gallery



Longitudinal section of the HE-E Test

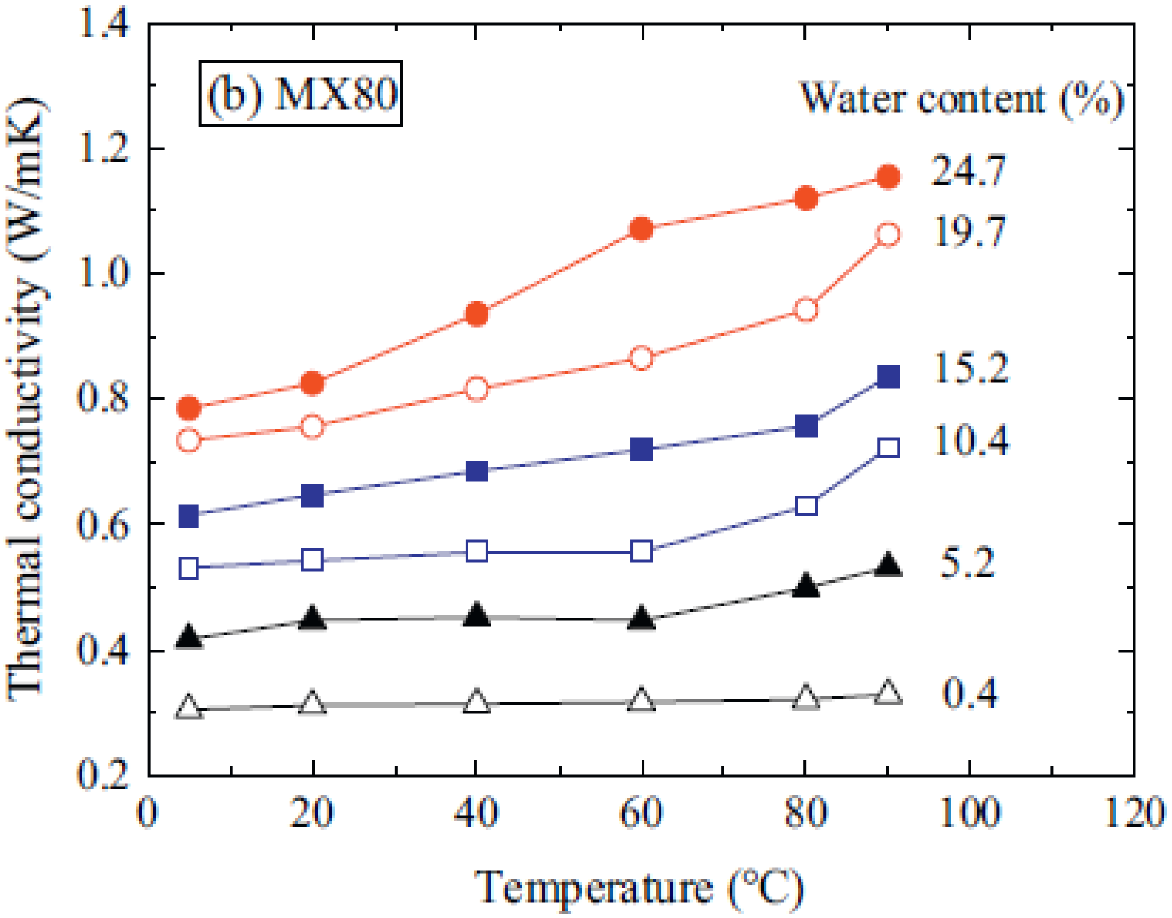
Thermal conductivity

Mineralogy

Water content ↑

Dry density ↑

Temperature ↑



Evolution of thermal conductivity with *T* for MX-80 bentonite compacted at 1.6 g/cm^3 with different water contents (Xu et al. 2019)

Permeability

Void ratio ↑

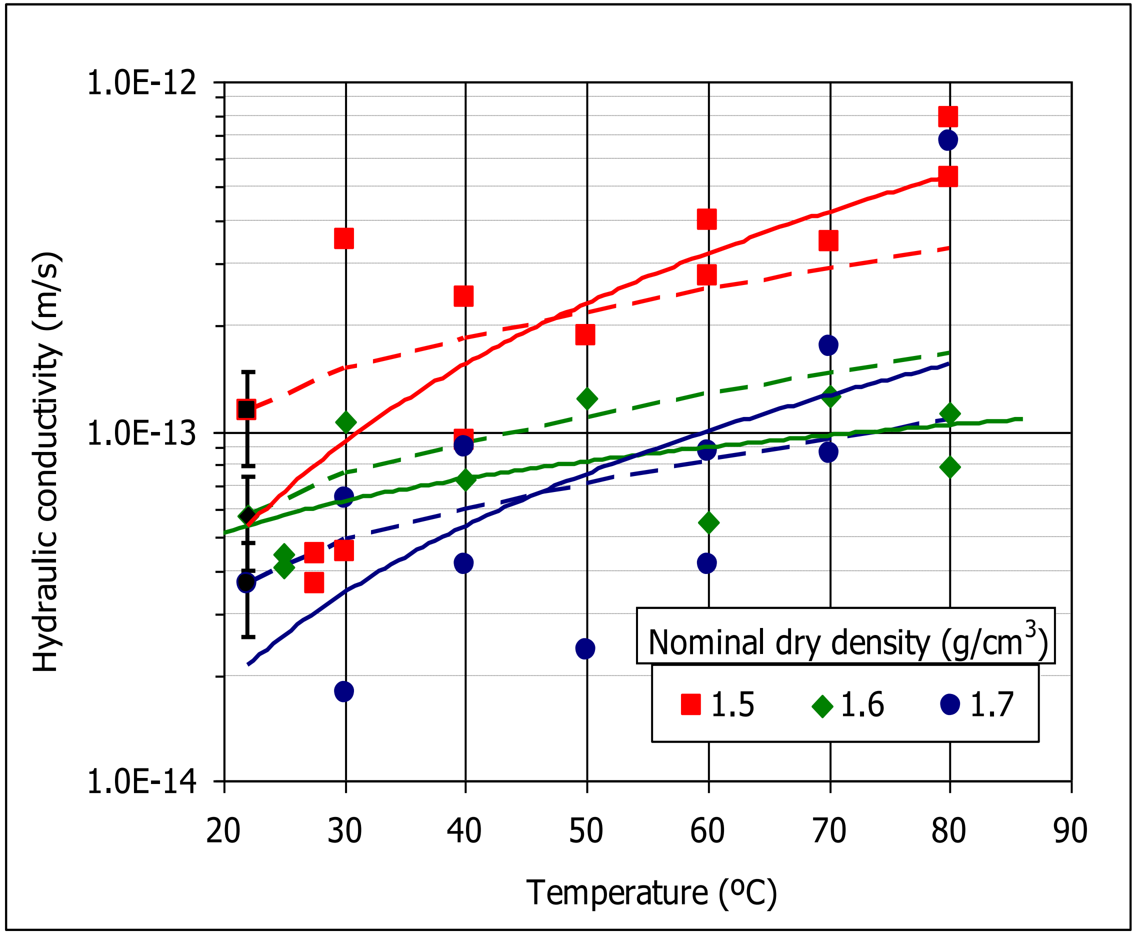
Degree of saturation ↓

Salinity of the permeant ↑

Threshold gradient

Temperature ↑

Expected increase with *T* because of the changes in water properties, particularly water kinematic viscosity, but other factors could affect (mineralogical, geochemical, microstructural changes



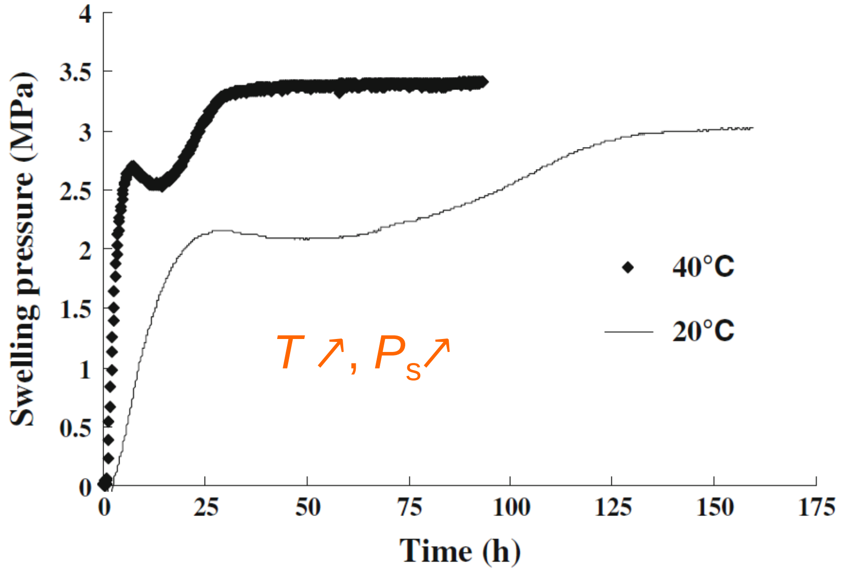
Change of hydraulic conductivity with *T* for FEBEX bentonite compacted at different dry densities. The dotted lines indicate the expected change on the basis of the water properties changes with T (Villar & Gómez-Espina 2009)

Swelling capacity

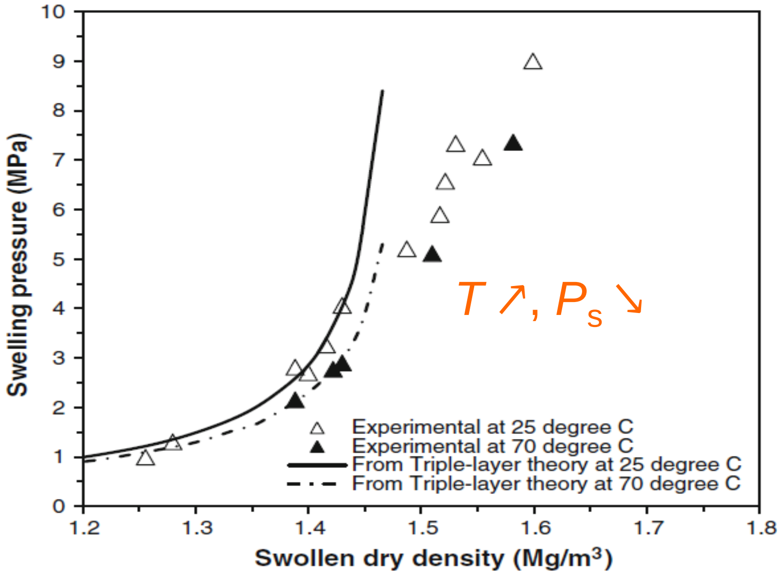
- Smectite content ↑
- Exchangeable cation
- Dry density ↑
- Availability of water (S_r) ↑
- Salinity of saturation water ↓
- Stress history
- **Temperature ?**

Effect of *T* on swelling pressure is believed to depend on the predominant exchangeable cation (Pusch et al. 1990). However, according to the results available in the literature, the trends are unclear (compilation by Chaaya et al. 2022):

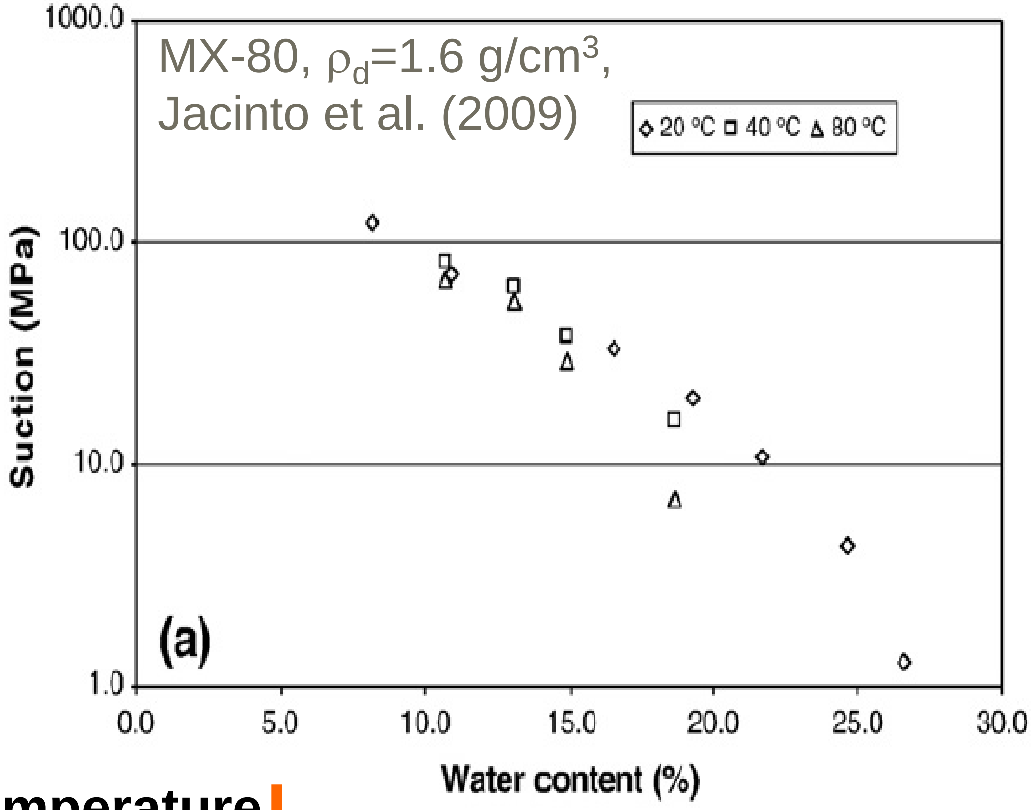
GMZ01 (Na) (W. M. Ye et al. 2012)



MX80 (Na) (Tripathy et al. 2015)



Water retention capacity (WRC)



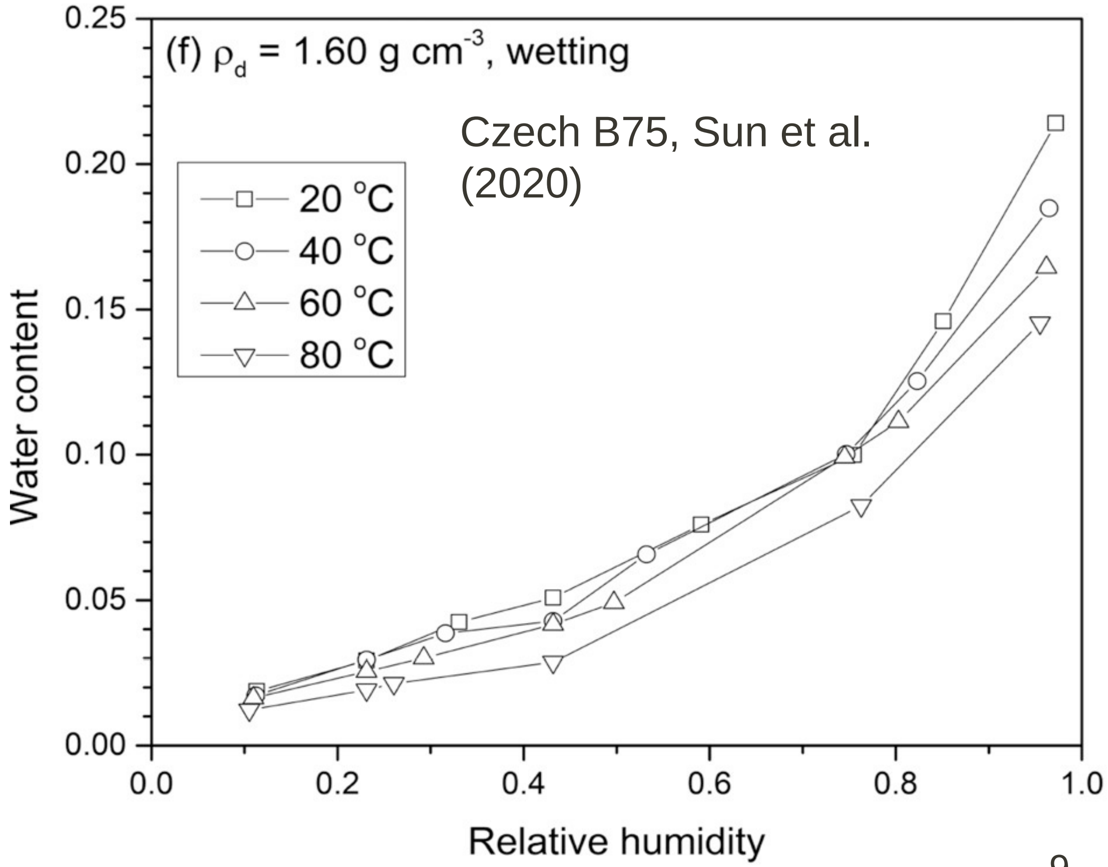
Strain state ↓

Void ratio ↑

Salinity of the permeant

Temperature ↓

- Changes with *T* are mainly due to changes in water surface tension (but also thermodynamics of adsorption play a role)
- There are not many results for *T*>80°C



HITEC (**Influence of temperature on clay-based material behaviour**) is one of the WPs of the EC-financed Joint Programme EURAD.

HITEC aims to improve Thermo-Hydro-Mechanical (THM) description of clay-based materials at elevated temperatures, providing results useful for different national waste management programmes. The host rock clays will be studied under saturated and partially saturated conditions under 100°C, while buffer bentonites will be studied both in saturated and unsaturated conditions under 150°C

HITEC: Why?

- **Clay host rock**

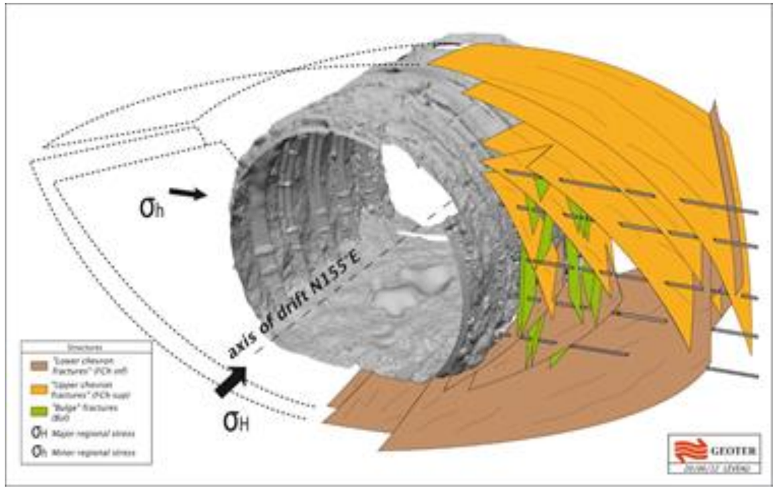
– The overpressure generated by the difference between thermal expansion coefficient of pore water and the solid rock skeleton may have deleterious consequences:

  - In far field, this could induce rock damage and reactivate fractures/faults.
  - In near field characterized by a fractured zone, this could induce fracture opening or propagation in this fractured zone, altering the permeability.

- **Buffer bentonite**

– Proving that higher temperatures than presently accepted are suitable is very relevant even for current concepts.

– It increases safety margin and gives greater credibility to the design (e.g. if it is proven to work for 130°C then for 100°C it is definitely safe).



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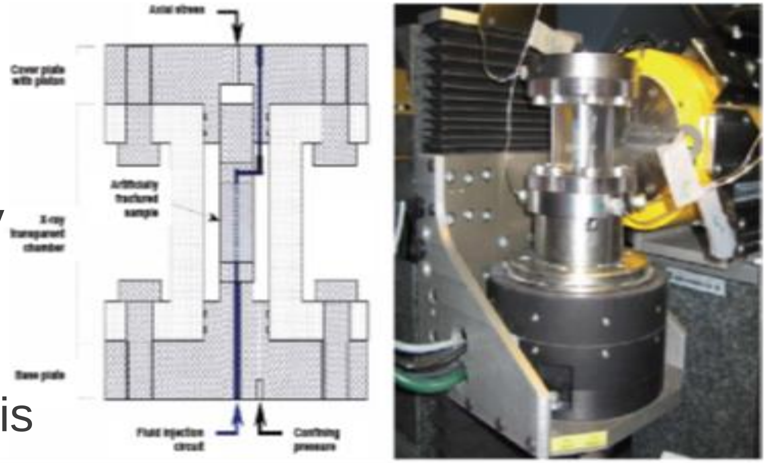


Olin, Svodoba, Grgic 2019

LABORATORY APPROACHES TO ASSESS THE EFFECT OF HIGH TEMPERATURE

- DETERMINE PROPERTIES AT HIGH TEMPERATURE (**Task 2 of HITEC**)
- PREHEAT SAMPLES AND CHARACTERISE THEM AT ROOM TEMPERATURE (**Task 1 of HITEC**)
  - Preheat the samples allowing evaporation (dry state)
  - Preheat samples in a closed system (wet state)
  - Steam heating
  - Preheat samples in conditions simulating the repository ones (**Task 3 of HITEC**)
  - Use samples coming from dismantling of large-scale tests

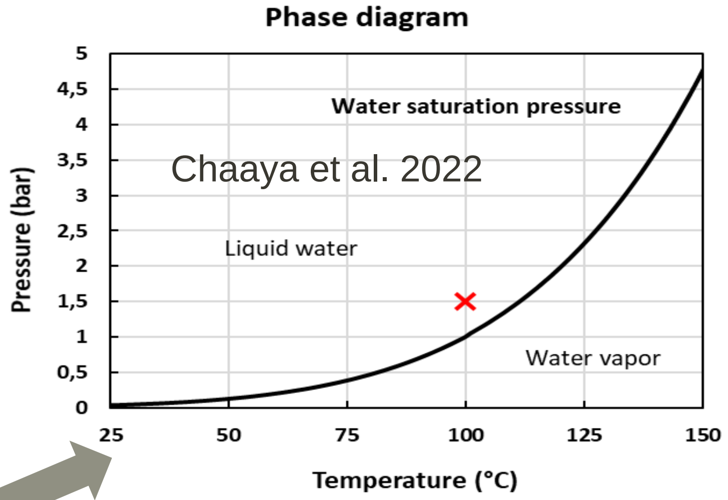
LABORATORY APPROACHES TO ASSESS THE EFFECT OF HIGH TEMPERATURE

- **DETERMINE PROPERTIES AT HIGH TEMPERATURE (Task 2 of HITEC):**
  - ✓ usually in samples compacted at relevant dry densities
  - ✓ mainly to assess THM properties (that we saw before)
  - ✓ mostly reproduce saturated conditions
  - ✓ provide representative parameters for models

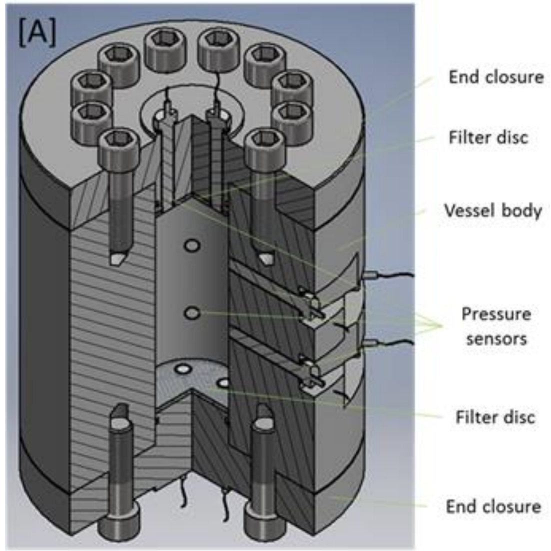
- PREHEAT SAMPLES AND CHARACTERISE THEM AT ROOM TEMPERATURE (**Task 1 of HITEC**)

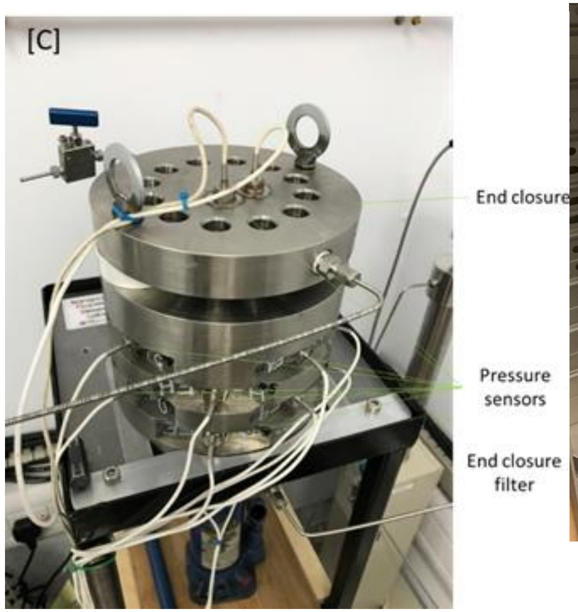
DETERMINATION OF SWELLING AND PERMEABILITY AT HIGH TEMPERATURE

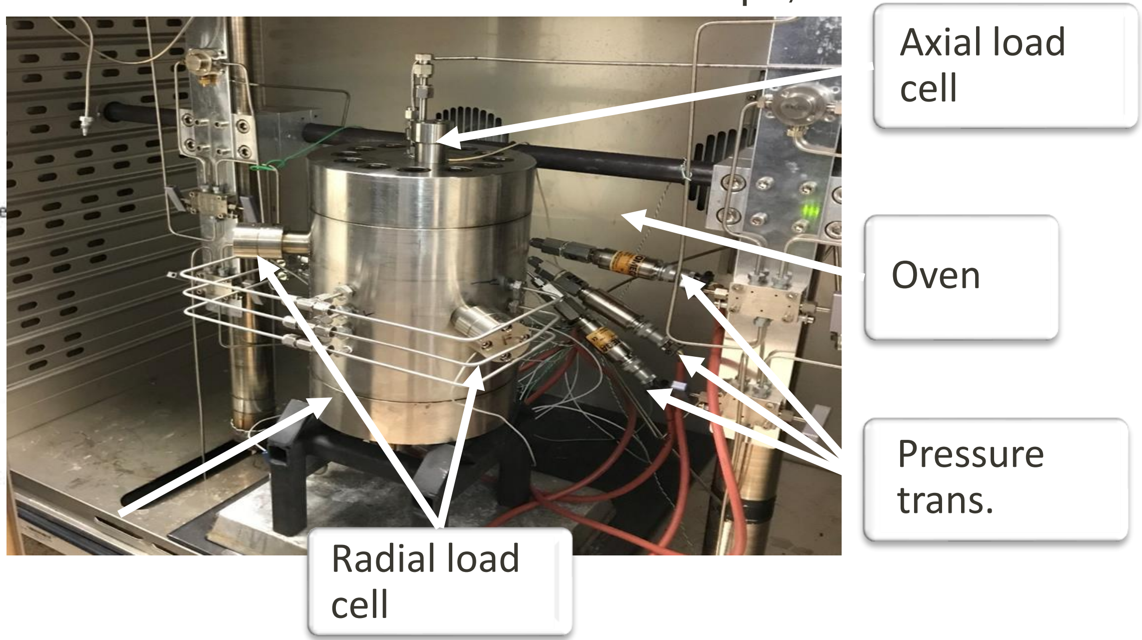
- It is possible to determine swelling pressure and hydraulic conductivity in the same set-up (also at room *T*)
- Thick-walled customed-designed stainless steel cells (pressure vessels). Steel with lower thermal expansion coefficient (e.g., INVAR) may be used
- Cells are wrapped with thermal mats or put in ovens or thermal baths. In the first case external insulation is required
- Injection and backpressures are applied (*P* should be selected considering the phase diagram). Both inflow and outflow are usually measured, but outflow should be used to compute permeability



BGS set ups, Graham et al. 2022

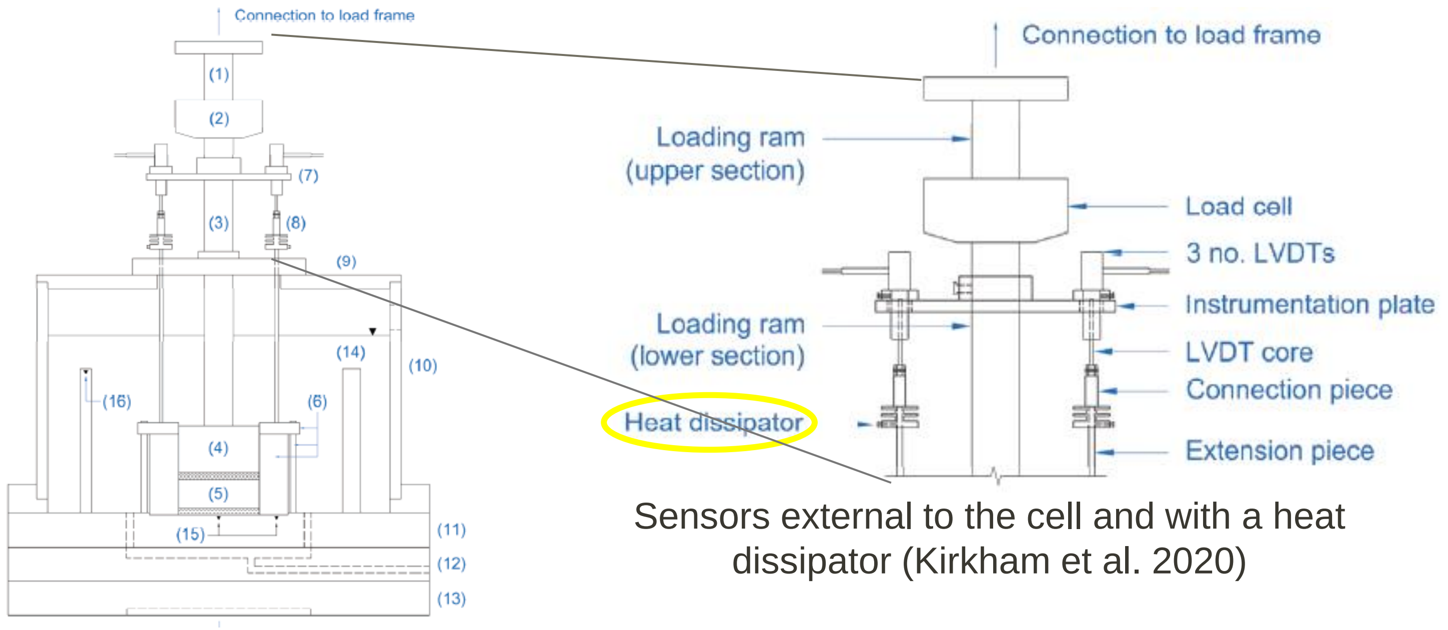






SOME TECHNICAL CONSIDERATIONS OF TESTING AT HIGH TEMPERATURE

- Measurement of axial and radial stresses: sensors (preferably made of the same material as cells) should withstand high *T* (temperature-compensated) (and *P*, and corrosion) in the long term or be external:



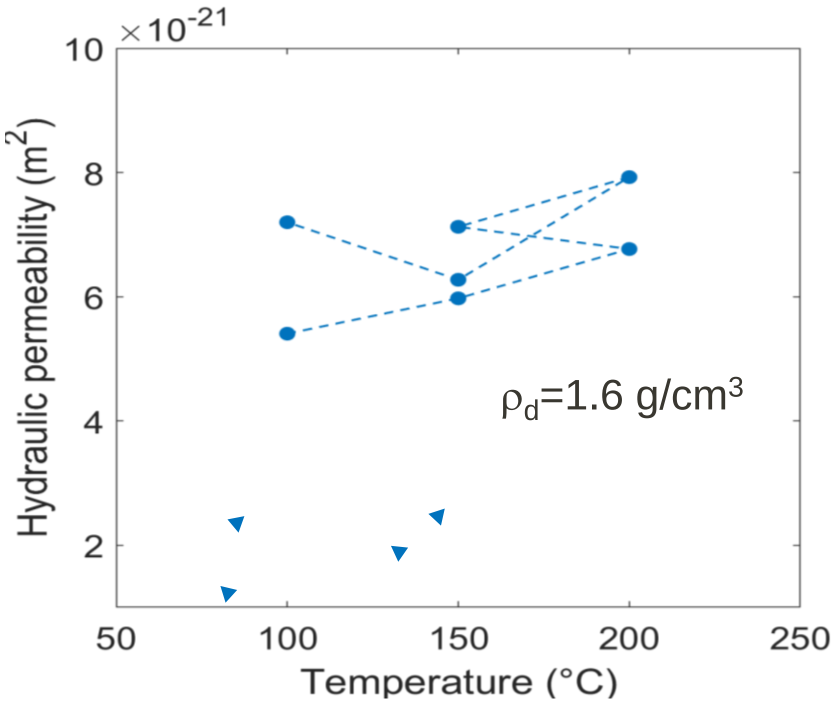
Sensors external to the cell and with a heat dissipator (Kirkham et al. 2020)

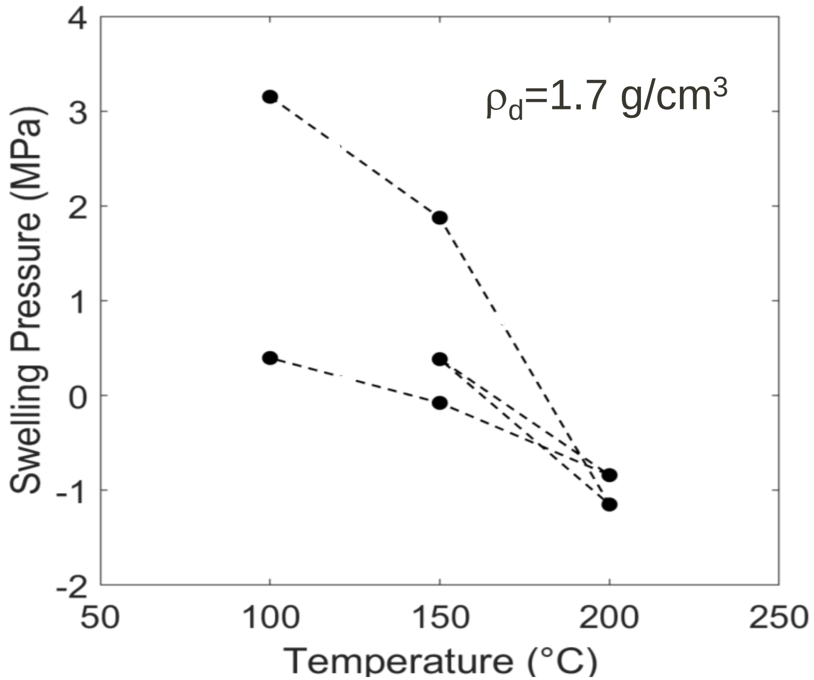
Sealing of sensors' inlets with high-T-resistant silicone

- Connections with cables should also withstand *T* to avoid vapour leaks through them (HP-HT hermetic connectors)
- Inlets for sensors insertion are potential vapour leaks: they should be sealed (HP-HT sealing/thread compounds, HT cable glands)
- Importance of calibration of strains and stresses of the equipment (e.g., Kirkham et al. 2020, Daniels et al. 2021)

British Geological Survey

MX-80

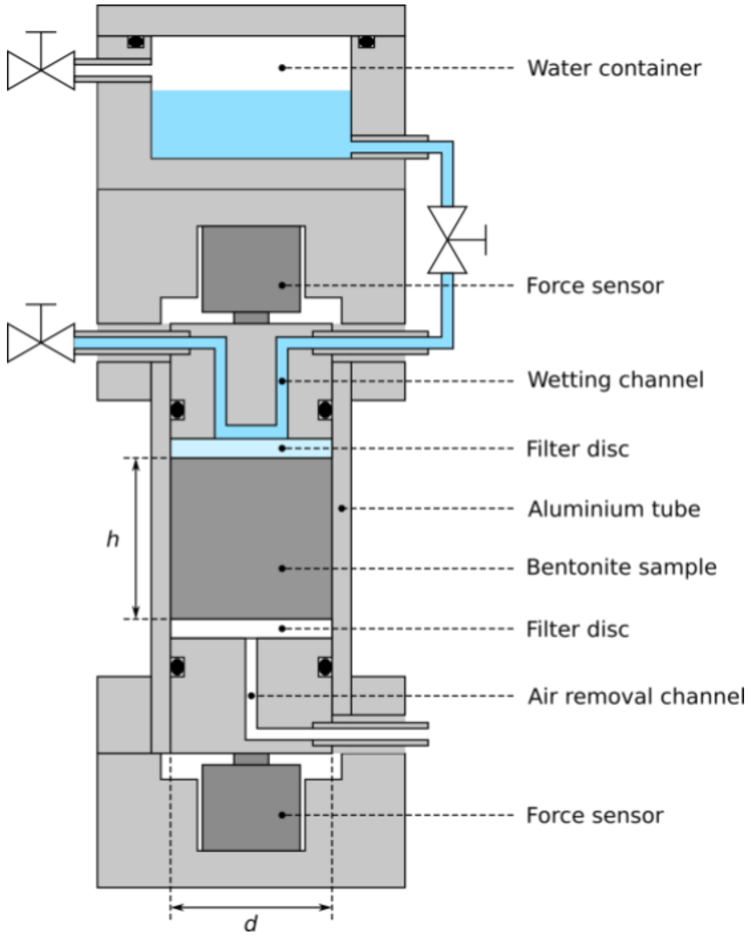




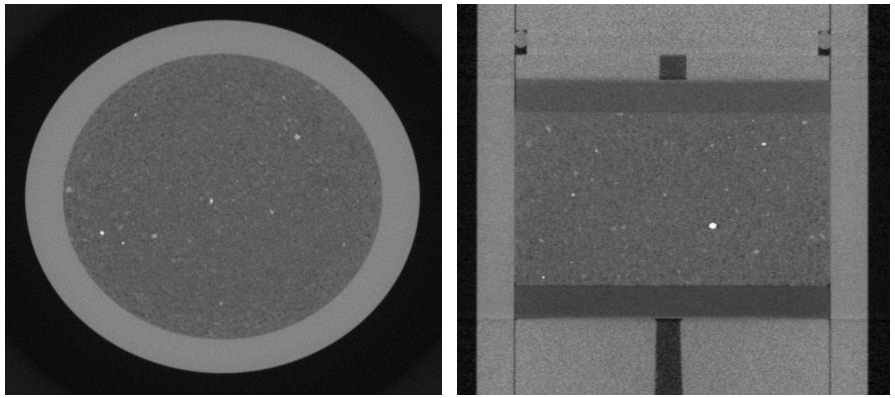
- The extent of the influence of temperature may depend on the dry density

MEASUREMENT OF WATER TRANSPORT AND SWELLING USING X-RAY

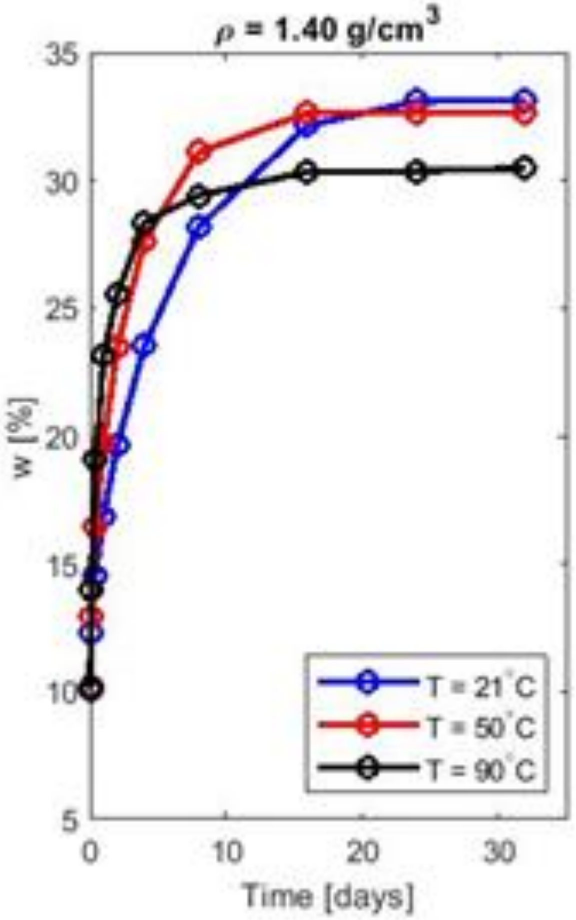
X-RAY IMAGING OR TOMOGRAPHIC METHOD



- Aluminium cells
- Smaller samples to get higher resolution
- Possibility of isothermal tests and thermal gradient tests
- For the imaging (which takes ~20 min) heating is stopped and an insulation box is used
- Complex calibration



2D slides of a 3D tomographic image of the sample holder and the sample



Wyoming-type Na bentonite

Yliharju et al. (2022)

Related work at LBNL, Wu et al. 2022

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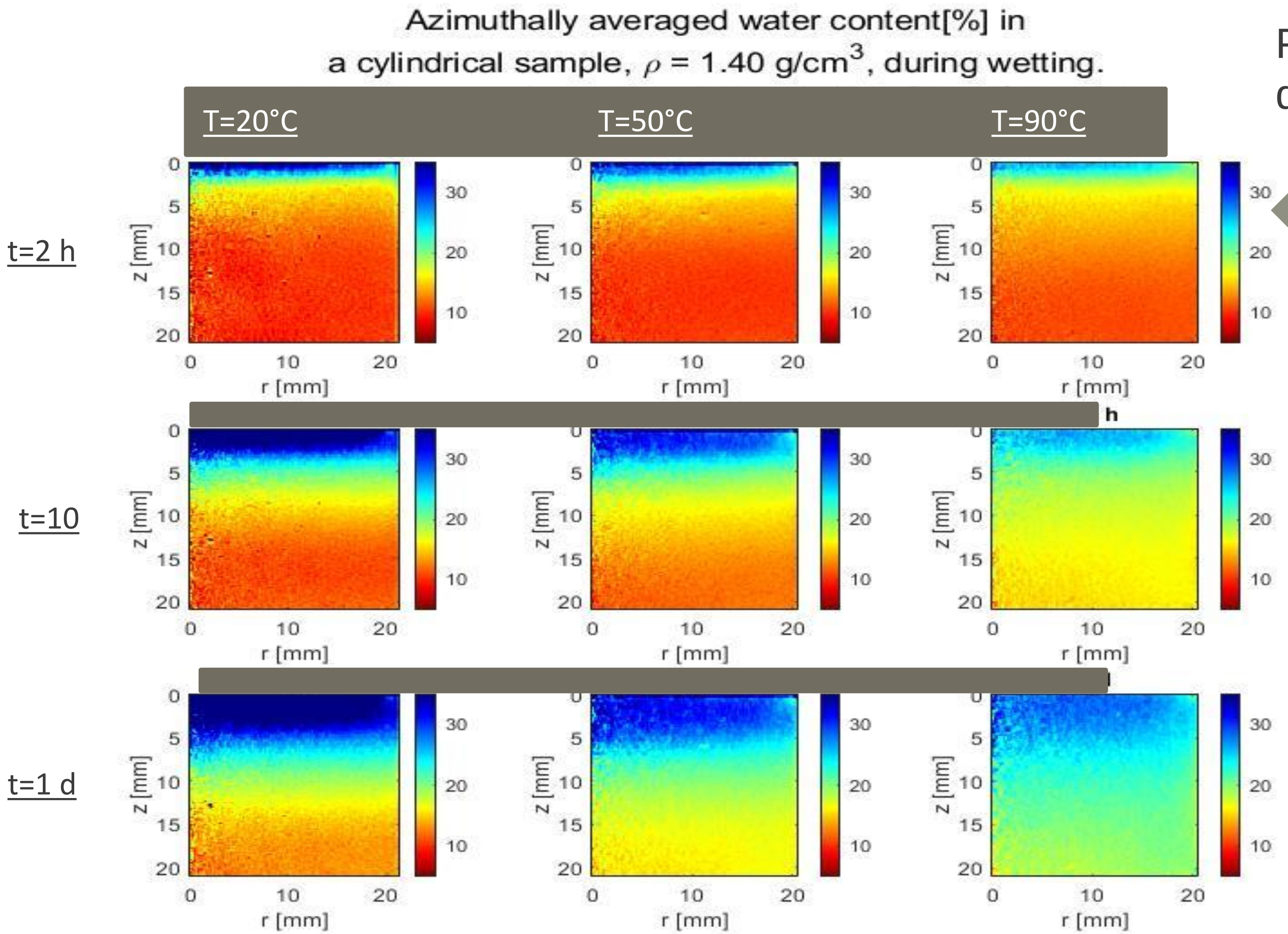
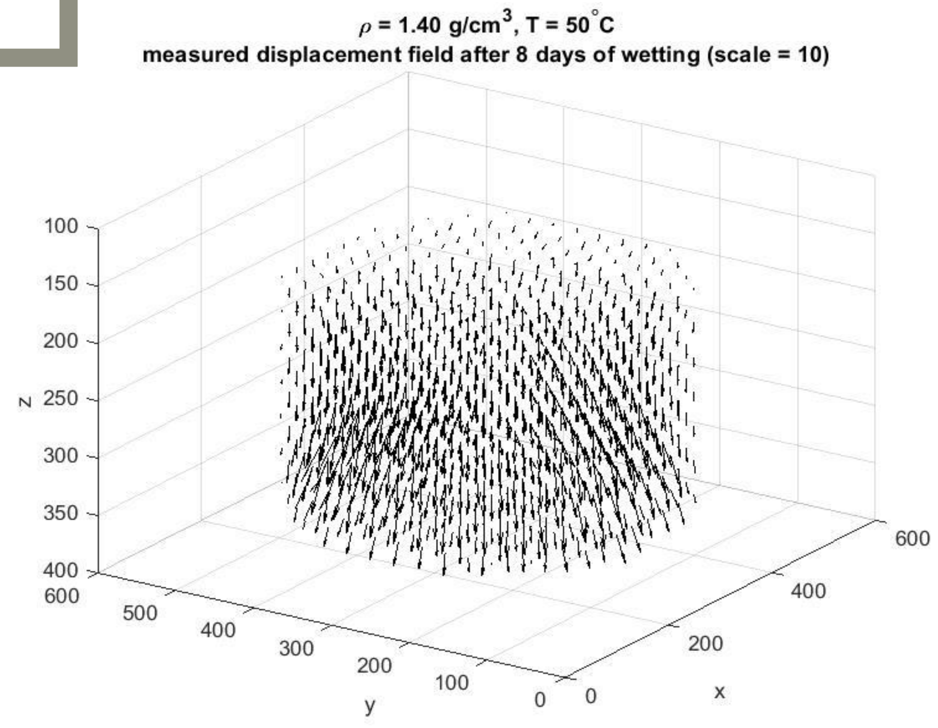
X-RAY IMAGING OR TOMOGRAPHIC METHOD



Possibility of knowing how the water is distributed inside the samples



Measured displacement field

Results rely on the accuracy of the calibration (e.g., local beam hardening correction, stitching, normalisation, thresholding, etc. (Harjupatana et al. 2022))

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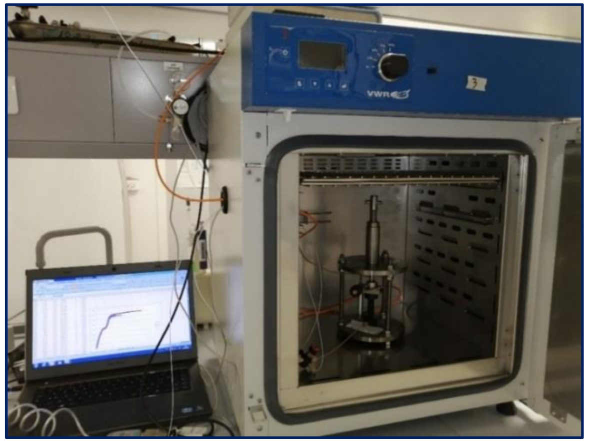
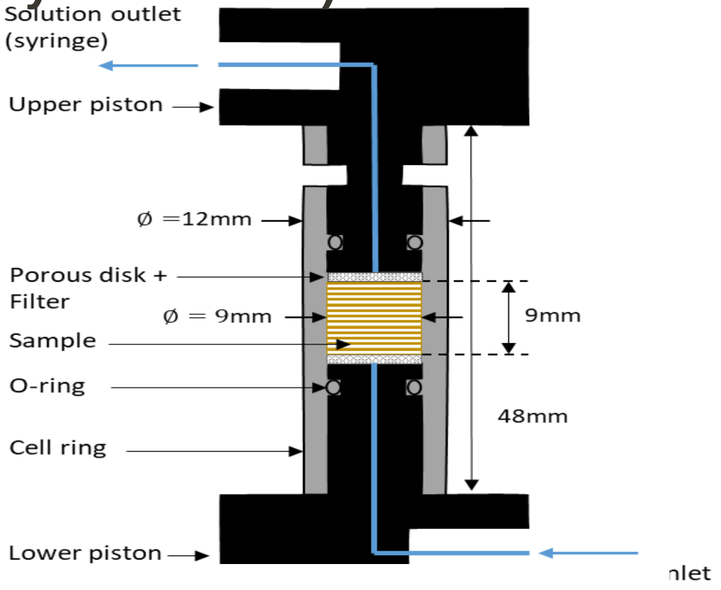
DETERMINATION OF SWELLING AND PERMEABILITY AT HIGH TEMPERATURE

TEST FOCUSED ON PROCESSES UNDERSTANDING (BRGM, Chaaya et al. 2022)

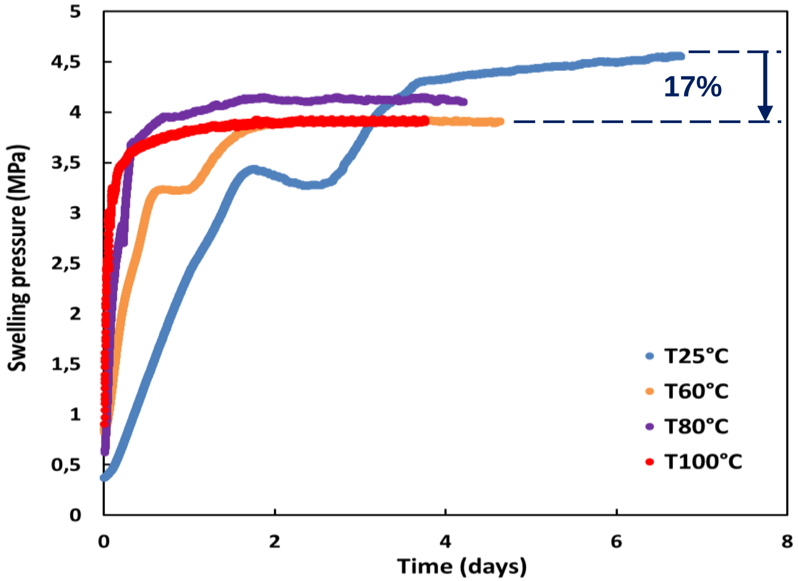
- Homoionized smectite: understand the actual effect of exchangeable cation
- Miniature oedometer placed in an oven

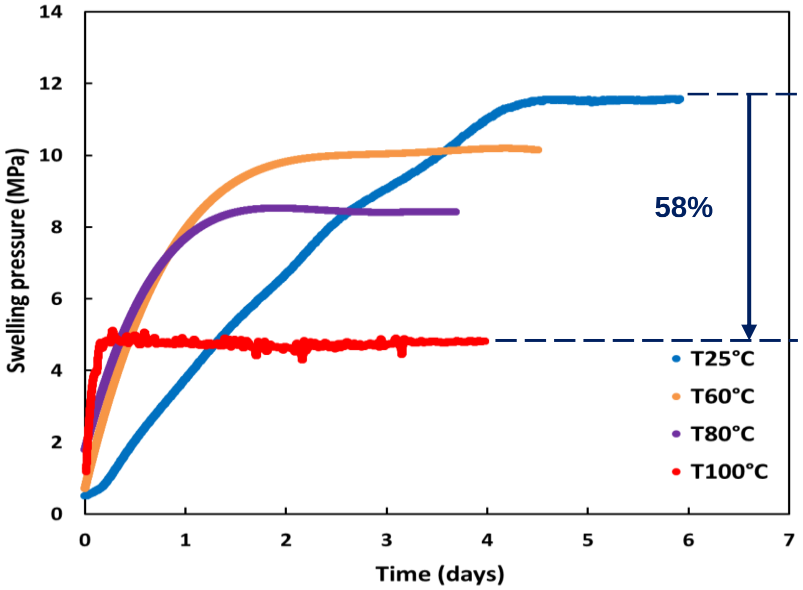


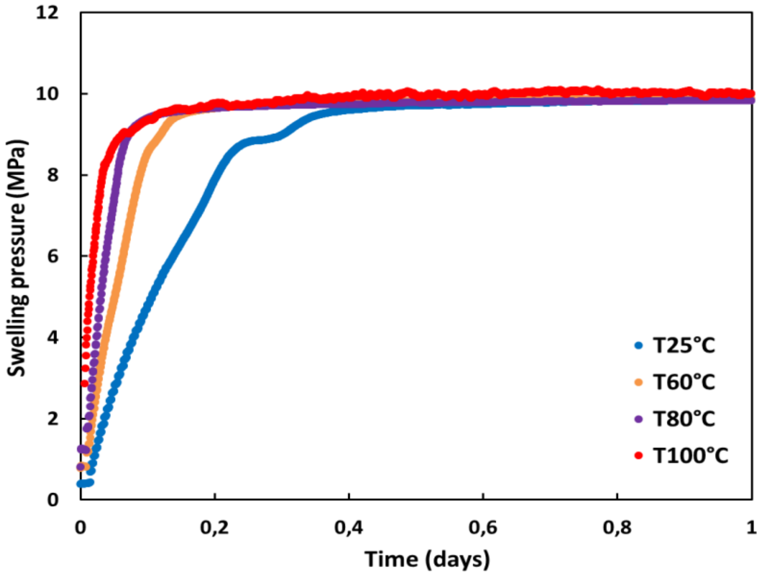


brgm — Geoscience for a sustainable Earth

The extent of T influence may depend on dry density:







Evolution of swelling pressure under different temperatures in homoionic bentonite compacted at different dry densities

LABORATORY APPROACHES TO ASSESS THE EFFECT OF HIGH TEMPERATURE

- DETERMINE PROPERTIES AT HIGH TEMPERATURE (Task 2 of HITEC)
- **PREHEAT SAMPLES AND CHARACTERISE THEM AT ROOM TEMPERATURE (Task 1 of HITEC)**
  - **Preheat the samples allowing evaporation (dry state):**
    - Representative for material close to the heater in repositories where it will remain dry for very long, because of high *T* or limited water availability and possible vapour escape
  - Preheat samples in a closed system (wet state)
  - Steam heating
  - Preheat samples in conditions simulating the repository o … C)
  - Use samples coming from large-scale tests dismantling

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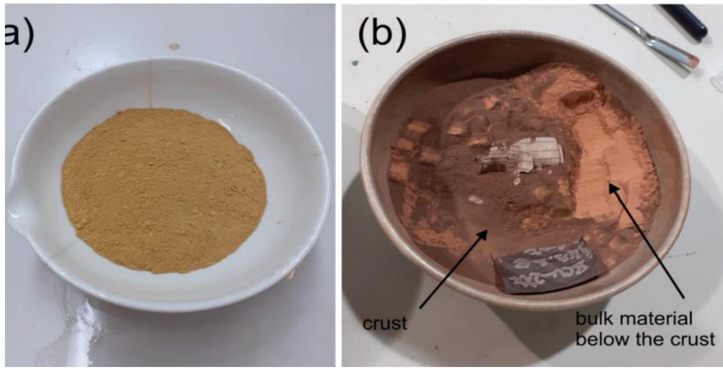
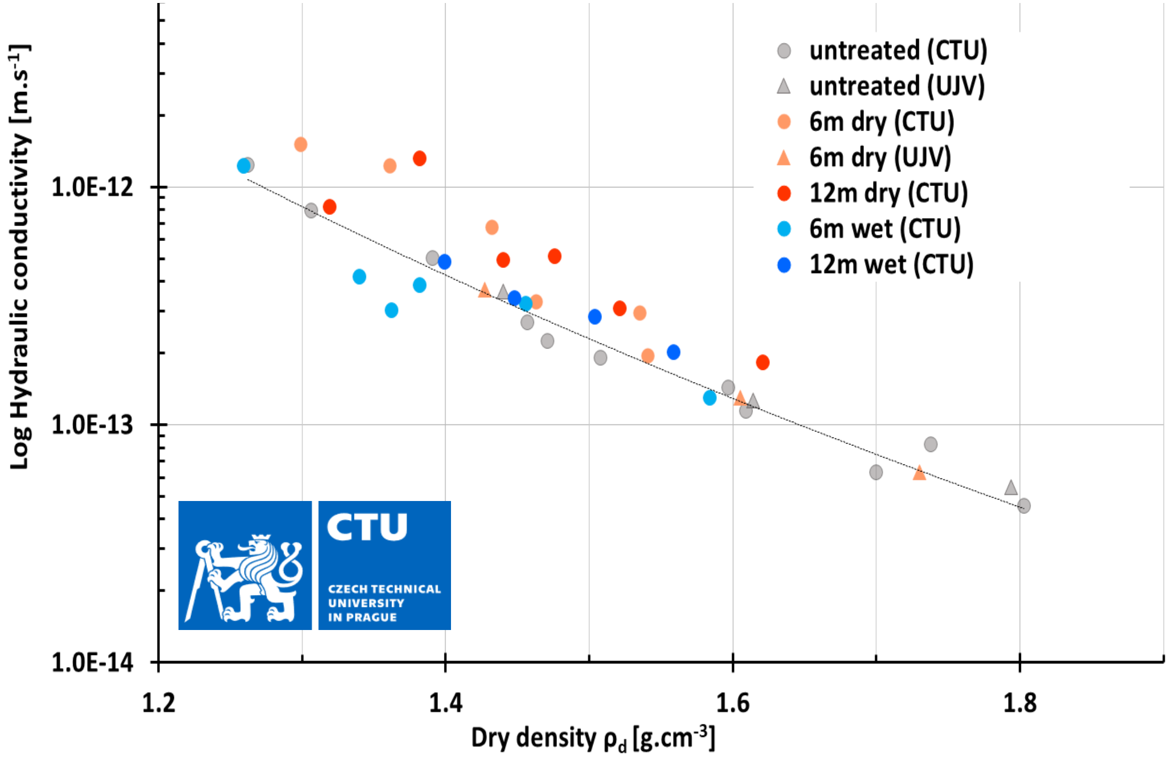
  - Preheat the samples allowing evaporation (dry state)
  - **Preheat samples in a closed system (wet state):**
    - special vessels to withstand vapour pressure (no boiling) and avoid leaks
    - relevant if the buffer is installed close to saturation, vapour cannot escape or water availability is high
  - Steam heating
  - Preheat samples in conditions simulating the repositor
  - Use samples coming from large-scale tests dismantlin

CTU CZECH TECHNICAL UNIVERSITY IN PRAGUE

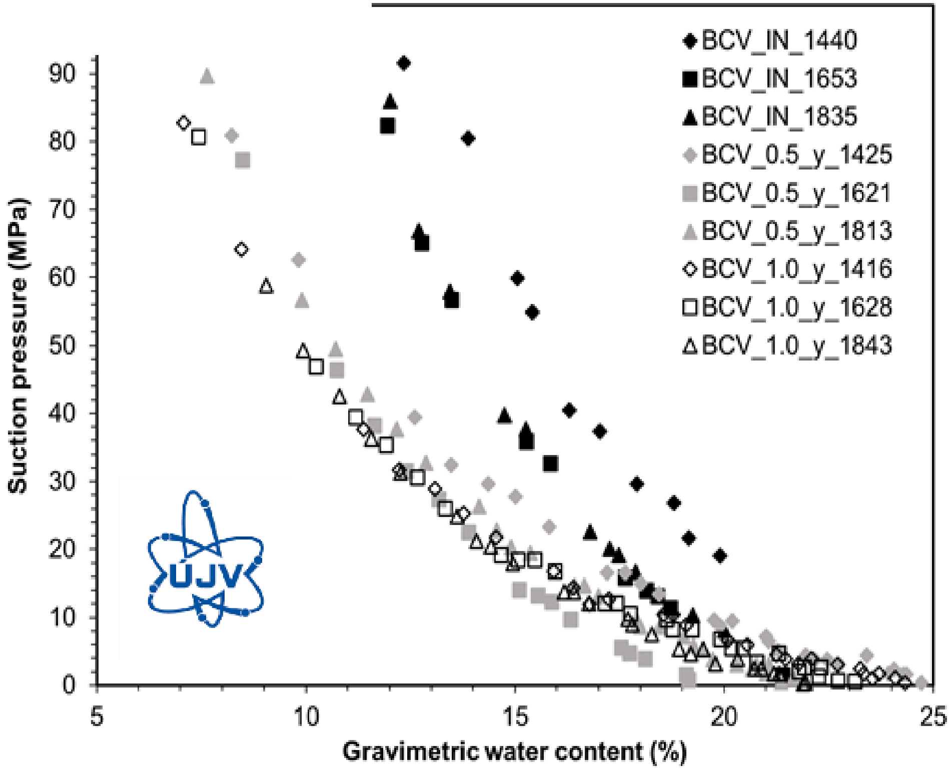
PROPERTIES OF SAMPLES PREHEATED IN DRY/WET CONDITIONS

- After treatment samples are allowed to cool down, ground and stabilised (or not) at given RH conditions. The material can then be used for mineralogical, geochemical or THM determinations.
- For THM determinations the samples have to be remoulded and compacted
- Tested in standard equipments





Hydraulic conductivity of BCV bentonite heated at 150°C in dry and wet state for 6 and 12 months (HITEC Deliverable D7.8)



Water retention curve of BCV bentonite heated at 150°C in dry state for 6 and 12 months (Kašpar et al. 2021)

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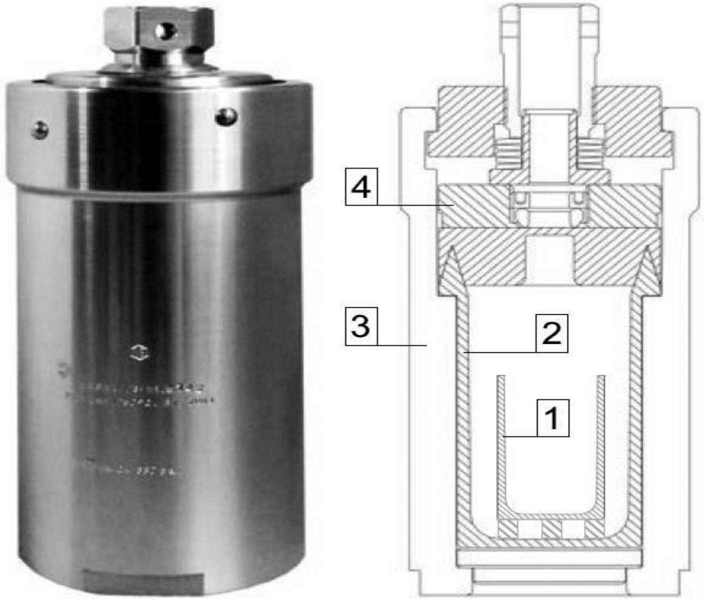
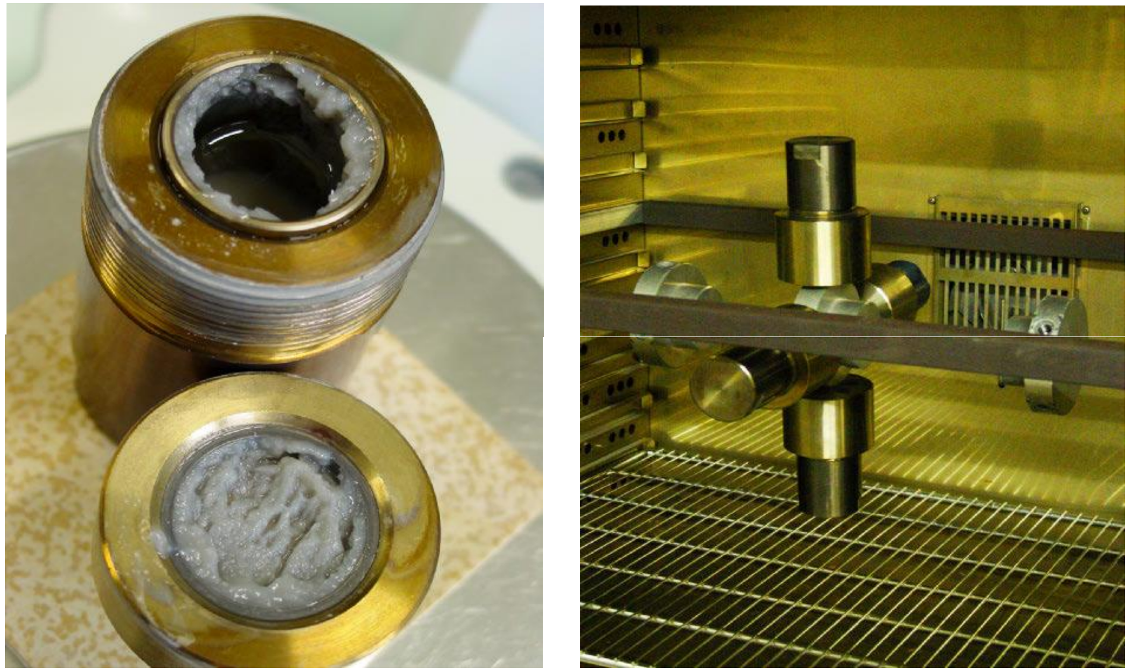
- **PREHEAT SAMPLES AND CHARACTERISE THEM AT ROOM TEMPERATURE (Task 1 of HITEC)**
  - Preheat the samples allowing evaporation (dry state)
  - Preheat samples in a closed system (wet state)
  - **Steam heating**
    - At low solid:liquid ratio (batch experiments)
    - Well-known studies (Couture 1985…) but under extreme conditions
    - Representative of long-term stage of the repository or worst-case scenario (?)
  - Preheat samples in conditions simulating the repository ones (Task 3 of HITEC)
  - Use samples coming from large-scale tests dismantling

STEAM HEATING

- High pressure vessels (autoclaves) in stainless steel, titanium, PEEK
- Temperatures are much higher (>>150°C) than those currently considered in most repository concepts
- Studies designed to analyse illitisation process (needs high *T* and K content in the system), cementation (silica precipitation) and loss of swelling capacity (in terms of water uptake, interlayer changes)
- The materials tested are usually previously purified (<2 μm fraction) and homogenised (Na, Ca…)

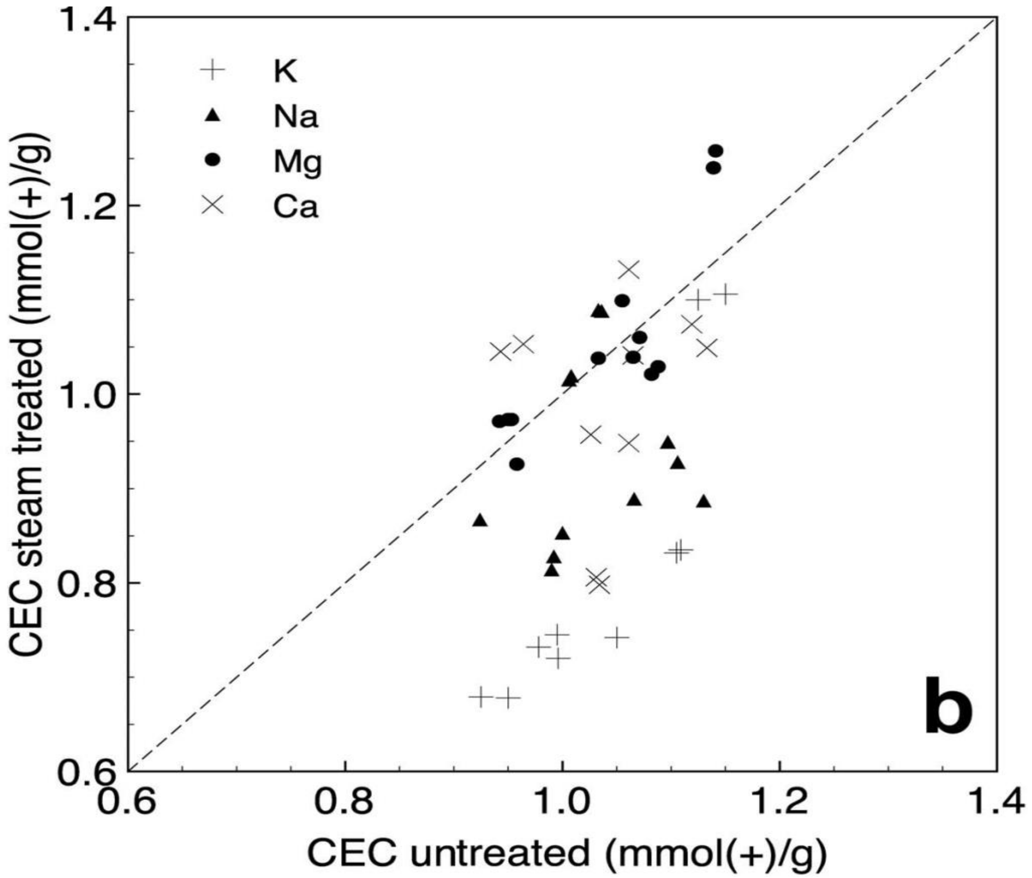


Pressure digestion vessel (1) 50 mL PTFE insert for the sample; (2) 250 mL PTFE insert for the water with lid; (3) high-alloy stainless steel body with bayonet lock; and (4) rupture disc to limit maximum pressure reliably (Heuser et al. 2014)



Details of titanium autoclaves and mounting arrangement of autoclaves in the oven (Leupin et al. 2014)

STEAM HEATING



Changes of Cationic Exchange Capacity for homoionic smectite after 6 days treatment with water vapour 200°C (Heuser et al. 2014)

- Results are dependent on solid:liquid ratio, time, temperature, potassium concentration in pore water, layer charge of smectite, interlayer cation
- Maybe for these reasons there is no general agreement on the effect of steam on bentonite properties, although none of the “new” studies point to drastic changes, but to slow changes in the smectite character (from montmorillonite to beidellite) or layer charge
- Attention should be paid to what is meant by terms such as “swelling capacity”

Wersin 2007, Leupin et al. 2014

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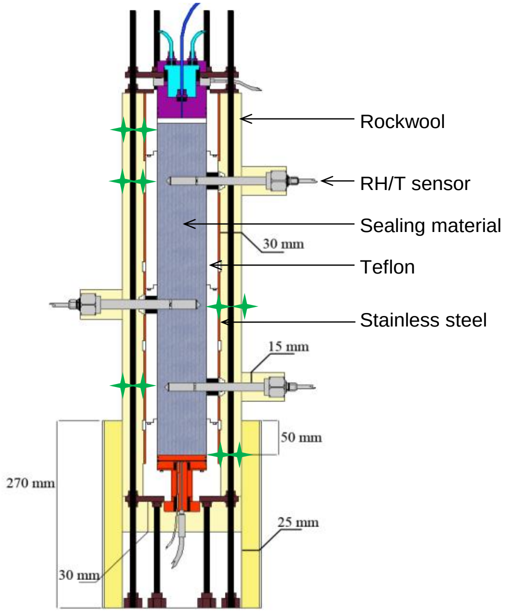
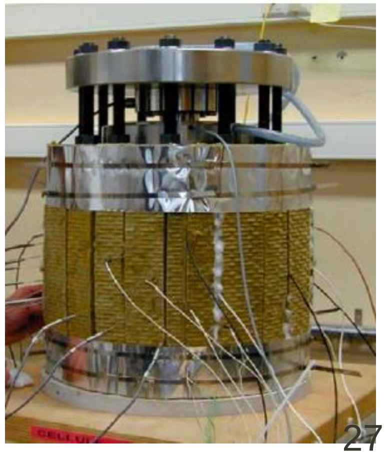
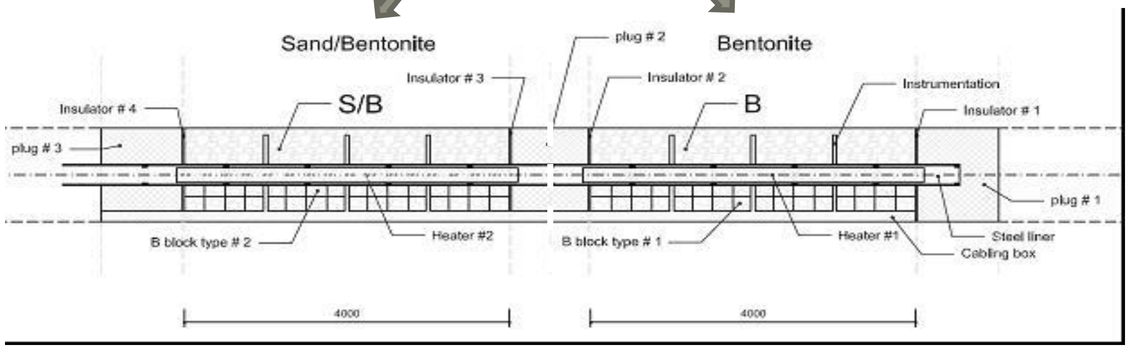
  - Preheat the samples allowing evaporation (dry state)
  - Preheat samples in a closed system (wet state)
  - Steam heating
  - **Preheat samples in conditions simulating the repository ones (Task 3 of HITEC)**
    - Tests in TH cells, with thermal and hydraulic gradients
    - Bentonite prepared as in the barrier (density, w.c.)
    - Provide online and postmortem results
    - Useful to validate models
  - Use samples coming from large-scale tests dismantling

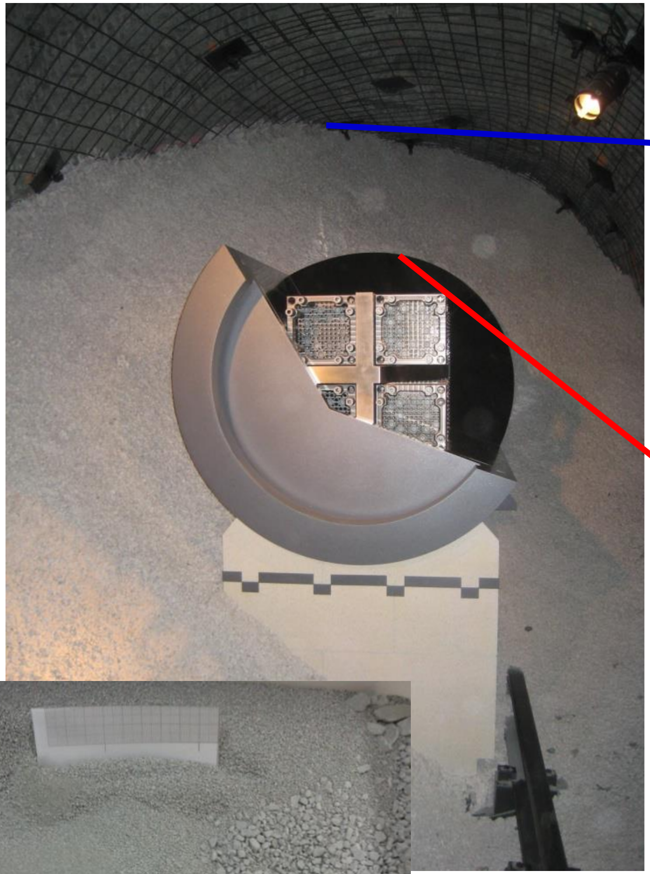


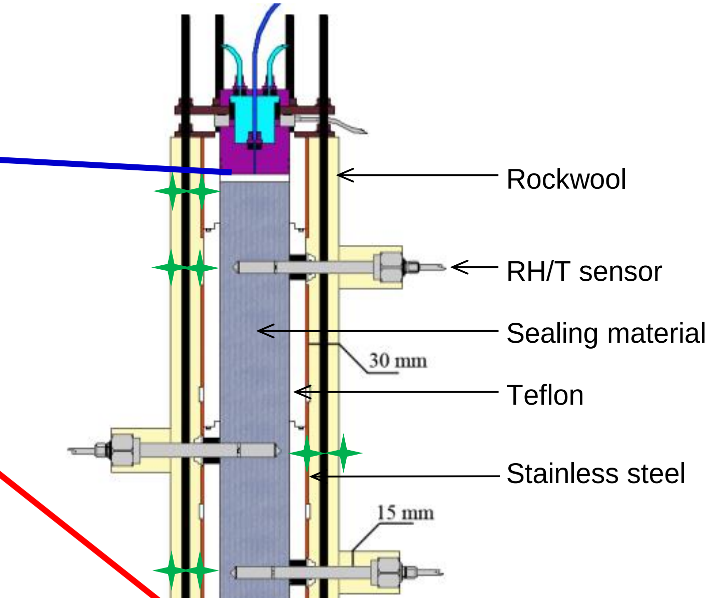


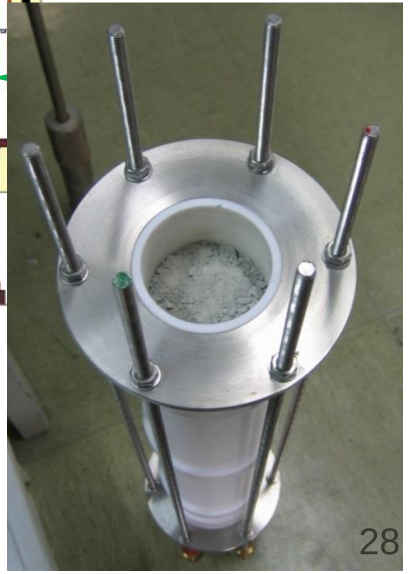
THE HE-E IN SITU EXPERIMENT AT MONT TERRI URL AND A COUNTERPART COLUMN AT CIEMAT

MX-80 bentonite pellets (B) (ρ_d=1.5 g/cm^3, *w*=6%)









Maximum heater surface temperature of 140°C, increased almost linearly to its maximum value in a period of 1 year

Natural hydration (Opalinus clay formation)

Pearson water (mg/L)

Cl^-	SO_4^{2-}	HCO_3^-	Mg^{2+}	Ca^{2+}	Na^+	K^+	Sr^+	pH
10636	1354	26	413	1034	5550	63	47	7.6

Injected at 0.06 bar in the column cell

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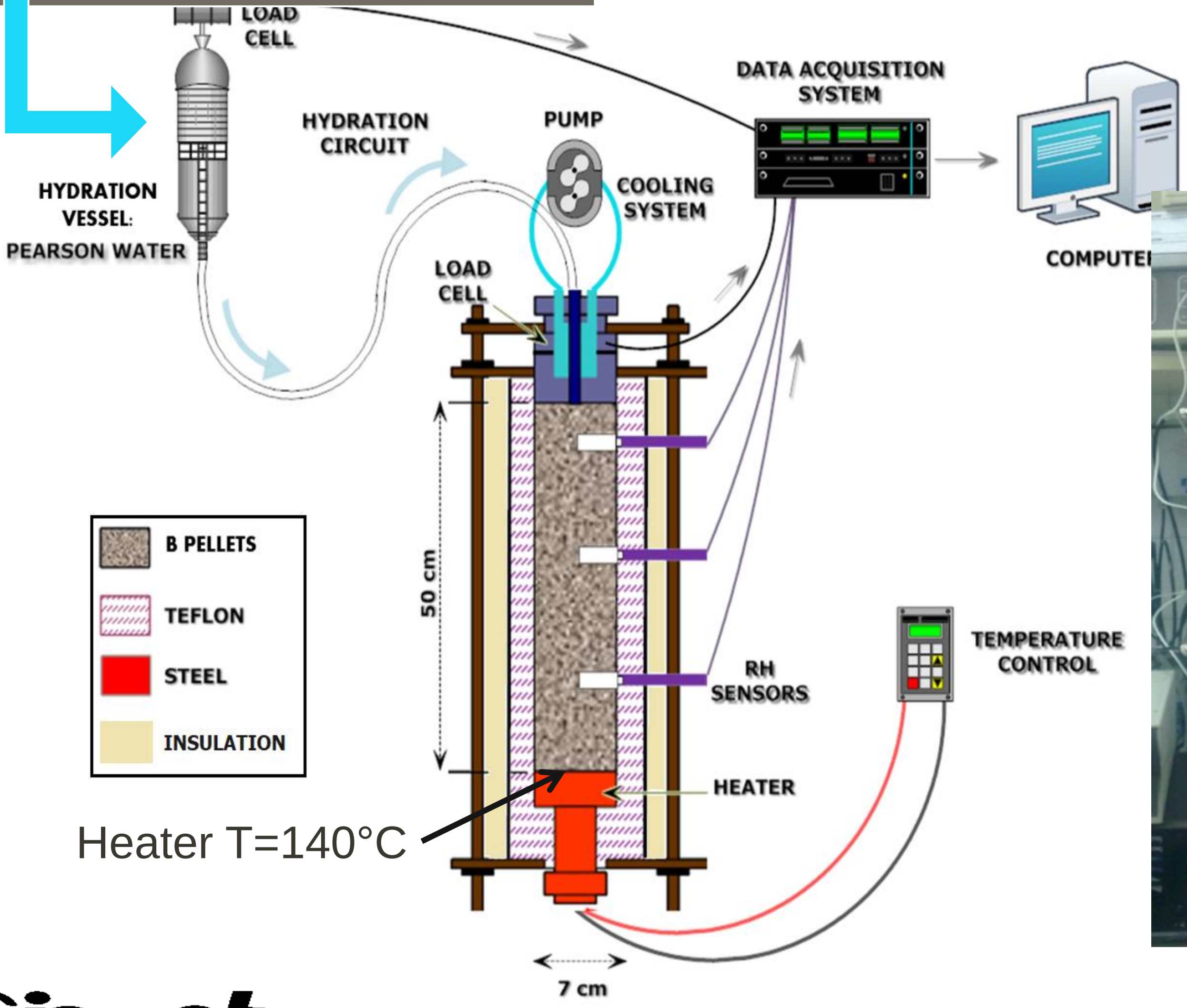
LABORATORY TESTS TO REPRODUCE COUPLED BARRIER PROCESSES

This is very different to the actual hydraulic boundary conditions in Mont Terri, where the flow rate is very low

THE HE-E COLUMN: EXPERIMENTAL SET-UP



Heater T=140°C

Villar et al. 2022

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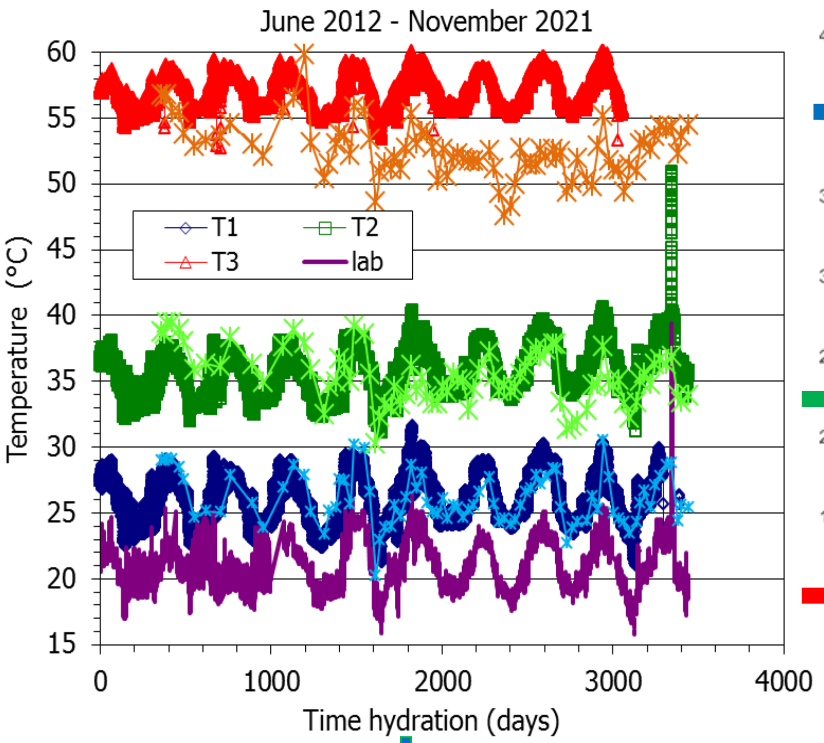
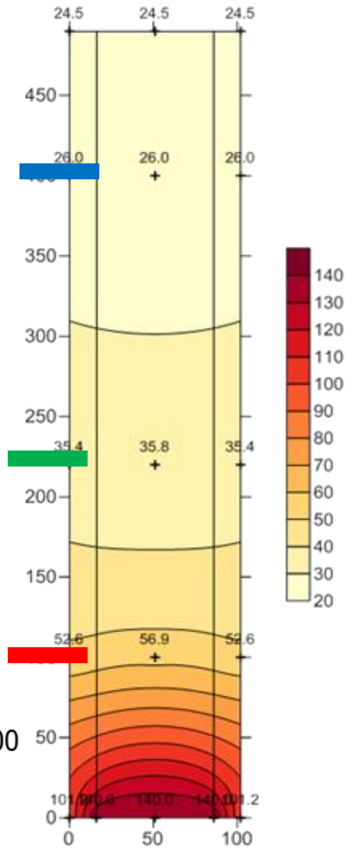
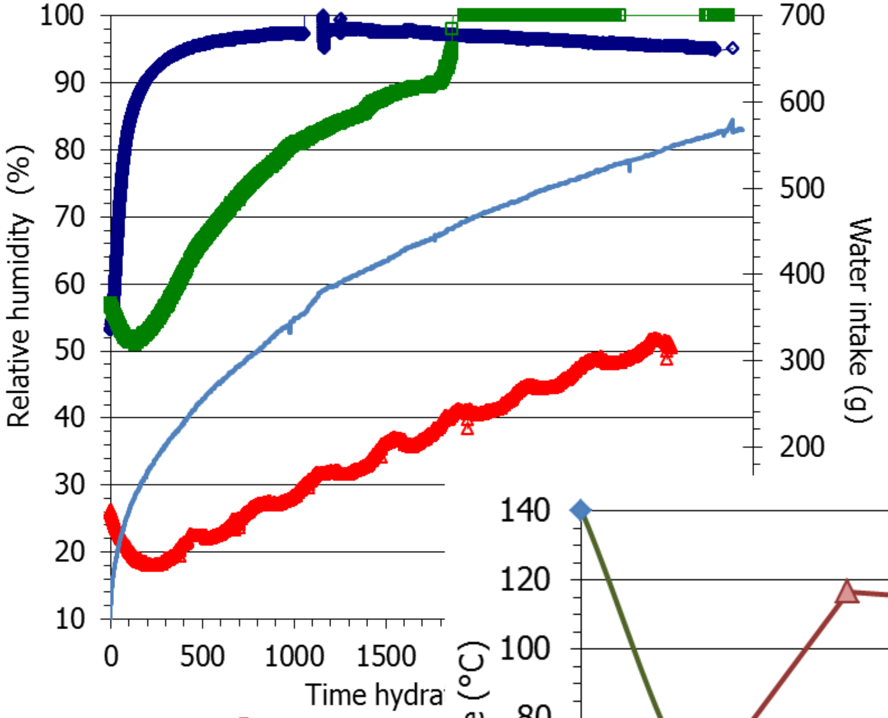
THE HE-E COLUMN: ONLINE RESULTS, HEATING+HYDRATION PHASE

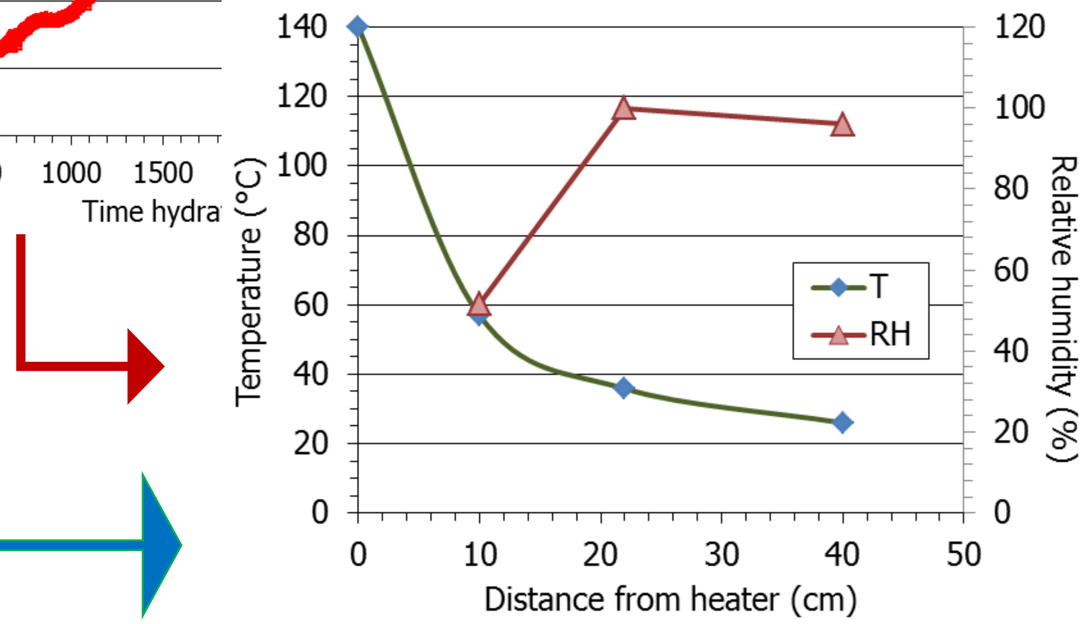
Thermal conditions not easy to reproduce





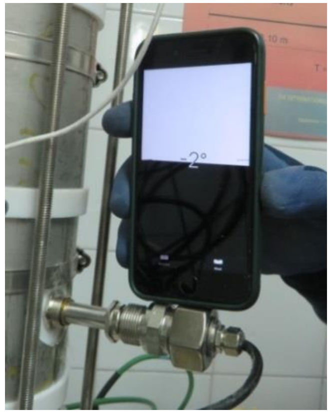


FINAL STATE



T >100°C just at less than 4 cm from the heater

THE HE-E COLUMN: CELL DISMANTLING AFTER TEN YEARS OF OPERATION



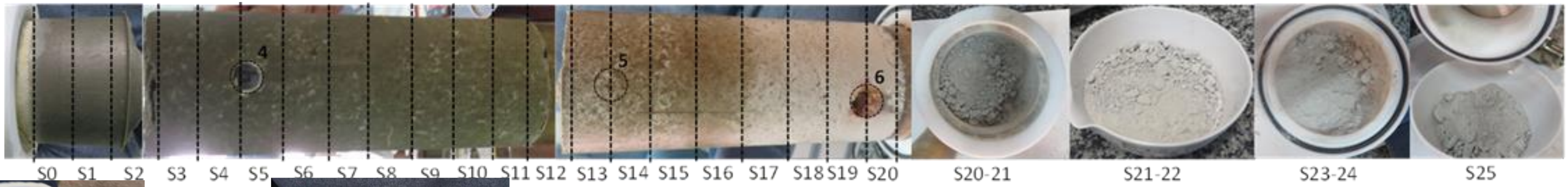
Extraction of sensors from the cell

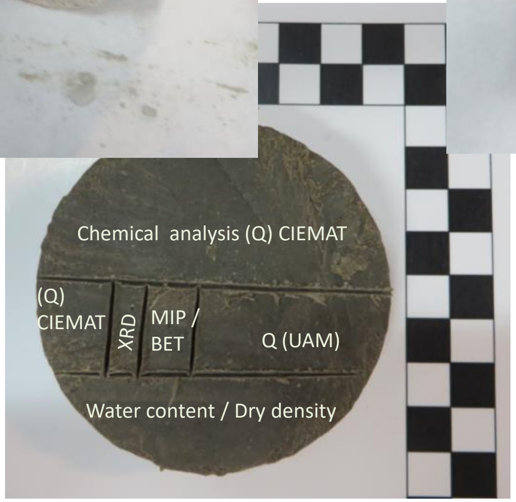
SENSORS TILTED AND CORRODED, ESPECIALLY TOWARDS THE BOTTOM: THE SALINITY OF THE PORE WATER INCREASES TOWARDS THE HEATER. THIS IS ENHANCED BY TEMPERATURE

THE HE-E COLUMN: CELL DISMANTLING AFTER TEN YEARS OF OPERATION

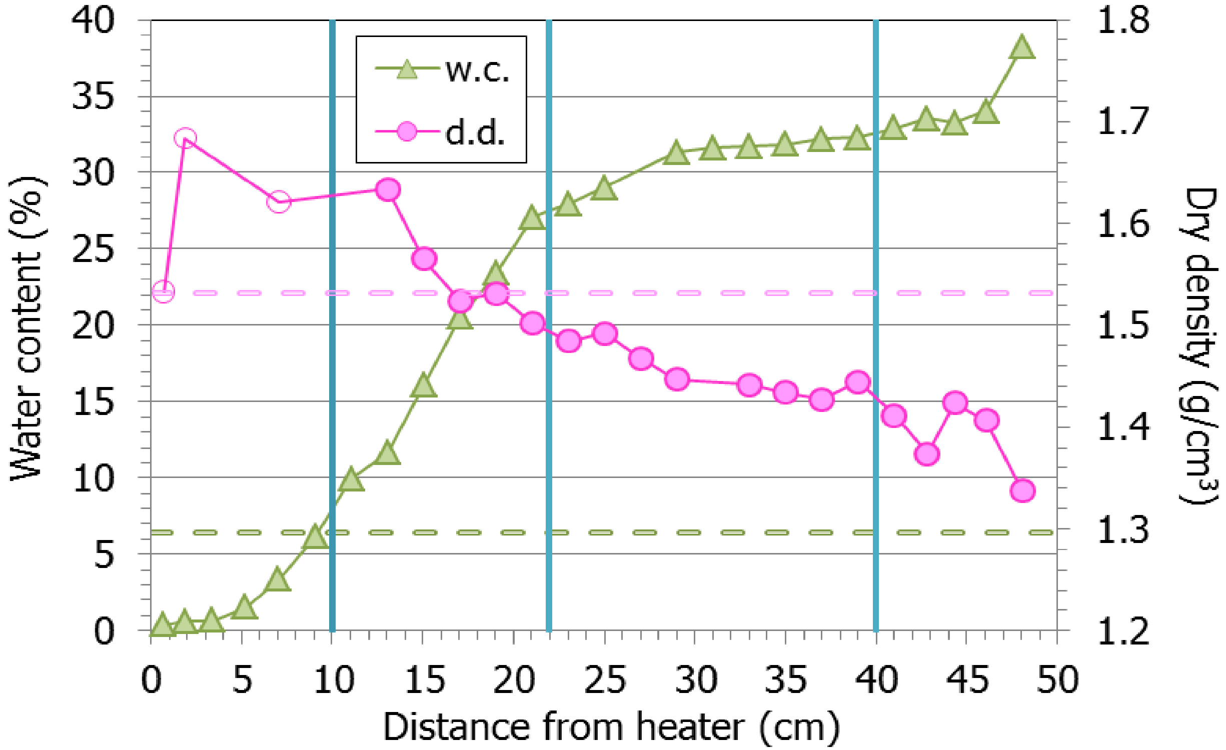
This could be an indication of which the state of the bentonite in the in situ test could be

THE HE-E COLUMN: CELL SAMPLING





These densities had to be estimated from the weight of the material contained in a certain column volume



Final w = 22.0%, ρ_d = 1.52 g/cm^3, S_r = 75%

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LABORATORY APPROACHES TO ASSESS THE EFFECT OF HIGH TEMPERATURE

- DETERMINE PROPERTIES AT HIGH TEMPERATURE (Task 2 of HITEC)
- PREHEAT SAMPLES AND CHARACTERISE THEM AT ROOM TEMPERATURE (Task 1 of HITEC)
  - Preheat the samples allowing evaporation (dry state)
  - Preheat samples in a closed system (wet state)
  - Steam heating
  - Preheat samples in conditions simulating the repository ones (TH cells, Task 3 of HITEC)
  - **Use samples coming from large-scale tests dismantling**

    LOT (Karnland et al. 2009, Prototype (Olsson et al. 2013), ABM (Svensson 2015), Ophelie (Van Humbeeck et al. 2009)

CONCLUSIONS

- ✓ The effect of temperature on hydro-mechanical properties of bentonite has been systematically studied for **temperatures of up to 100°C** and is quite well established with respect to safety functions: temperature modifies some properties but they keep in values acceptable for complying with the safety functions:
  - The increase of hydraulic conductivity with temperature cannot be explained in most cases solely by the increase in water kinematic viscosity: other factors may play a role (microstructure, mineralogy or pore fluid chemistry changes).
  - The effect on swelling capacity seems to depend on the predominant exchangeable cations, but literature results are not conclusive
  - Less work has been done on the effect of temperature on the water retention curve and thermal conductivity. It does not seem HITEC will fill this gap

CONCLUSIONS

- The extent of the temperature effect on some of the properties may depend on the density range, but the experimental evidence does not show consistent trends
- Most laboratory studies have focused on compacted bentonite, therefore it cannot be stated if the effect of temperature on some properties is affected by the initial fabric (compacted powder, grains, pellets) or not.

CONCLUSIONS

- ✓ Although the effect of **temperatures higher than 100°C** has been considerably studied concerning mineralogical transformations (not always in clearly representative conditions), less is known with respect to HM properties for this range of temperatures:
  - mainly because of the testing experimental issues, since testing at high temperatures involves additional challenges: sensors suitability, vapour leaks, equipment calibration
  - in particular, there are less studies on unsaturated materials

CONCLUSIONS

- ✓ Different testing approaches may reproduce different phases/concepts of repository
- ✓ The testing approach would depend on what we are looking for: process understanding, parameters for models?

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EURAD-WP7 HITEC

Roy Chaaya, Jiri Svodoba and other HITEC participants provided results for this talk

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