

Pore Water Chemistry Lab and Physico-chemistry of materials

PORE WATER CHEMISTRY LABORATORY



PHYSICAL CHARACTERIZATION

- Grain Density
- Dry Bulk Density
- Gravimetric Water Content
- Physical Porosity



MINERALOGICAL CHARACTERIZATION

- X-Ray Diffraction
- Thermal Analysis, DSC
- SEM, TEM, XPS, Mössbauer
- FTIR
- RAMAN



SURFACE PROPERTIES CHARACTERIZATION

- Total surface area
- Characterization of the dry state:
 - ▶ N₂ adsorption isotherms (BET)
 - ▶ Porosimetry (Hg Porosimetry)
- Characterization of the saturated state:
 - ▶ H₂O adsorption isotherm
 - ▶ d(001) basal spacings

PHYSICO-CHEMICAL PROPERTIES

- Cation exchange population
- Total CEC
- Variable Charges
- Potentiometric titrations
- Soluble salts by aqueous leaching
- Pore water extracted by *Squeezing*

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Centrifuge HETTICH Rotina 380R (11000 rpm)



Jacomex anoxic glove box (< 1 ppm O₂)

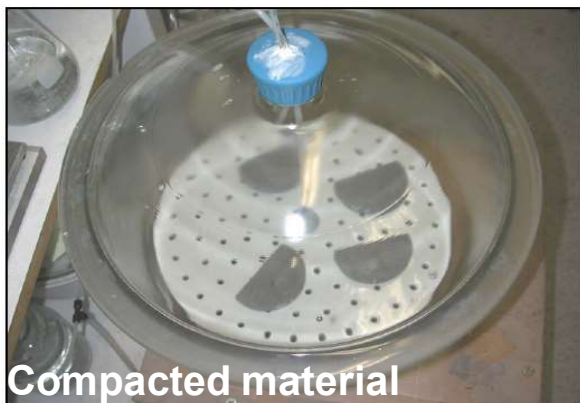


AquaMate 8000 UV-Vis Spectrophotometer (190-1100 nm)

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Mineralogical, Surface and FQ Characterization

Water adsorption/desorption isotherms



Vacuum desiccators with sulphuric acid solutions to apply different total suction



Vacuum desiccators with saturated salts solutions to apply different water pressures



Nicolet iS50 FTIR spectrometer:

Spectral range: 11000 – 50 cm^{-1} (NIR, MIR, FAR)
 Diffuse Reflectance Tecnique (DRIFT)
 Attenuated Total Reflection (ATR, diamond)
 Coupled to WETSYS water vapour generator



Purge gas generator:

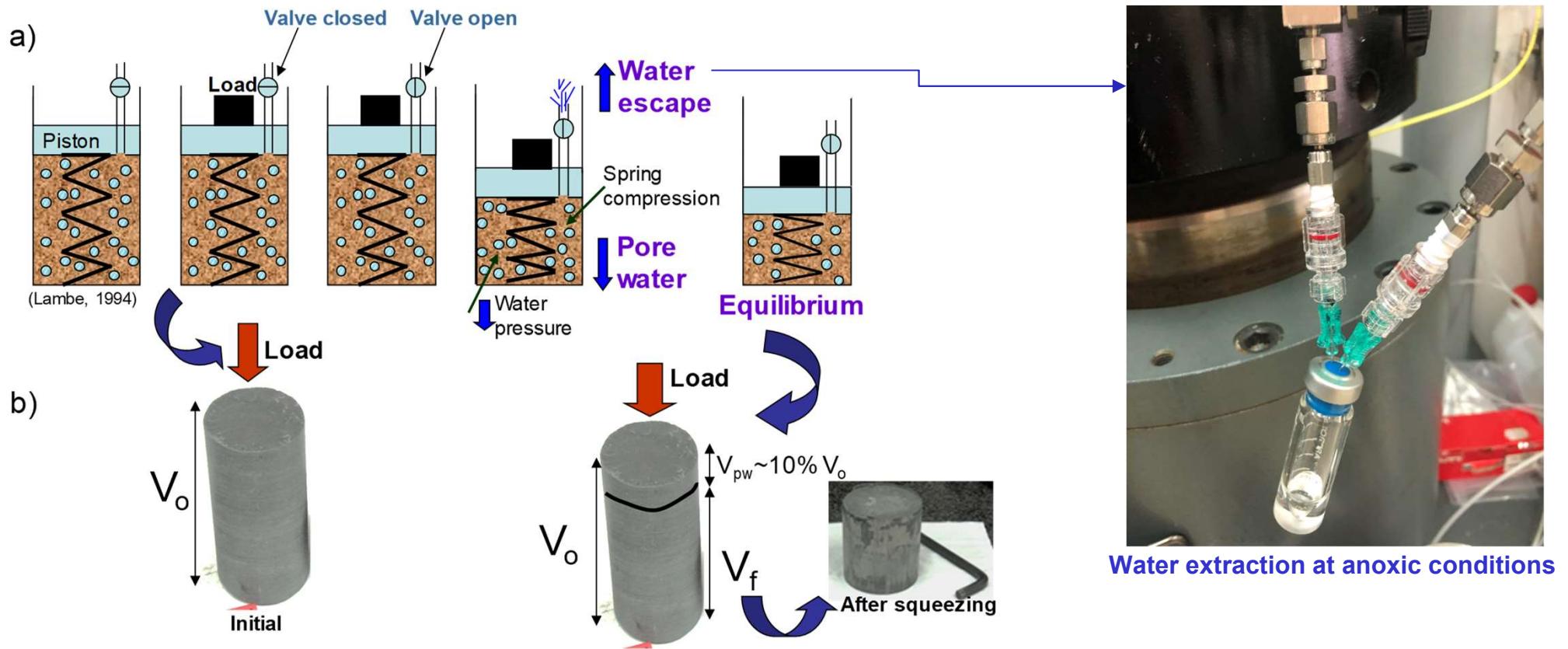
- CO_2 -free
- -73°C dew point
- No particles $> 0.01 \mu\text{m}$

TG-DSC-ATD / Water adsorption:

THEMYS 1750 +
WETSYS (Water vapour generator):
 5-95% RH, 20-70 $^\circ\text{C}$
 Heat capacity (C_p , $\text{J}\cdot\text{mol}^{-1}\cdot\text{K}^{-1}$)



Applying Squeezing Technique



Water extraction at anoxic conditions

Figure 1. a) Hydromechanical analogy for load changes during squeezing and consolidation (Terzaghi, 1925; Lambe and Whitman, 1969): The resistance of the internal phase compression is represented by a spring and the rate at which the pore water flows is dependent on the size of the valve aperture. First the valve is closed and in equilibrium. When a load is applied, the piston load is apportioned by the water and the spring in relation to the stiffness of each. All the applied load is resisted by an increase in the fluid pressure. If the valve is opened, the excess of pore pressure will dissipate by water escaping through the valve. The piston drops and the volume chamber decreases until there is a new equilibrium when the load is carried by the spring and the water pressure has returned to the original hydrostatic condition; b) Illustration of the reduction of volume, V_o to V_f , of a saturated core sample from Opalinus Clay (BHT-1 m. 12.42) after a squeezing test. The volume of expelled pore water is represented by V_{pw} . (Fernández et al., 2014)

Types of Squeezing cells



up to 100 MPa

L = 250 mm
20 mm wall, $\phi_{\text{inner}} = 70$ mm



up to 200 MPa

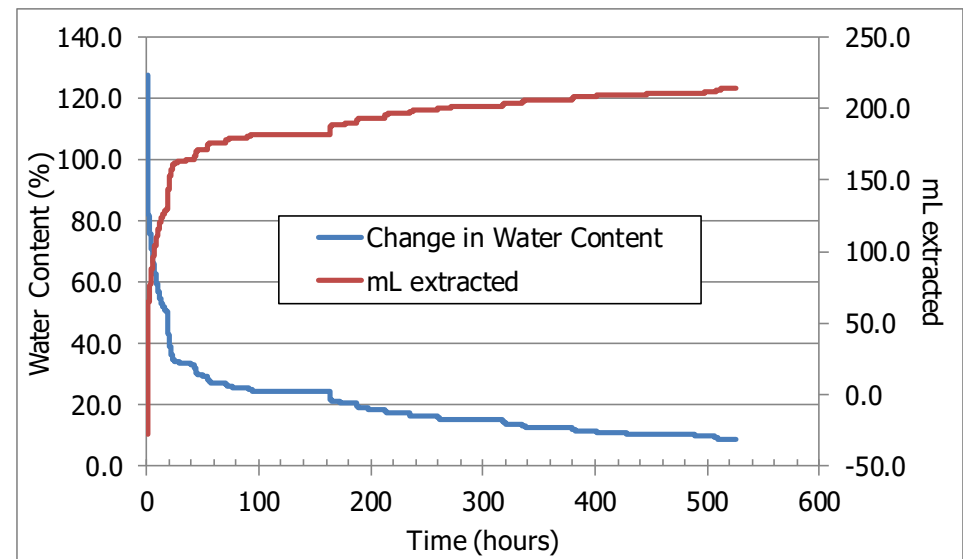
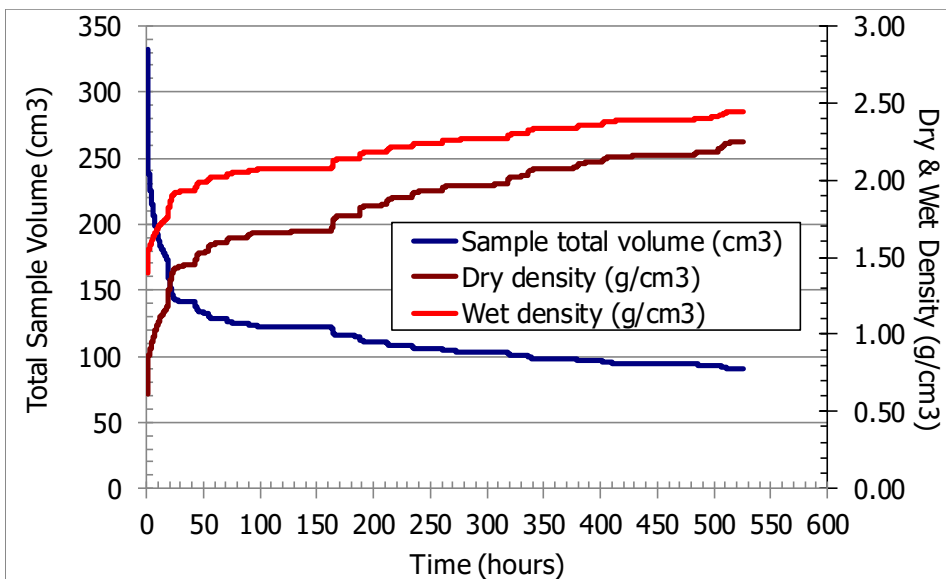
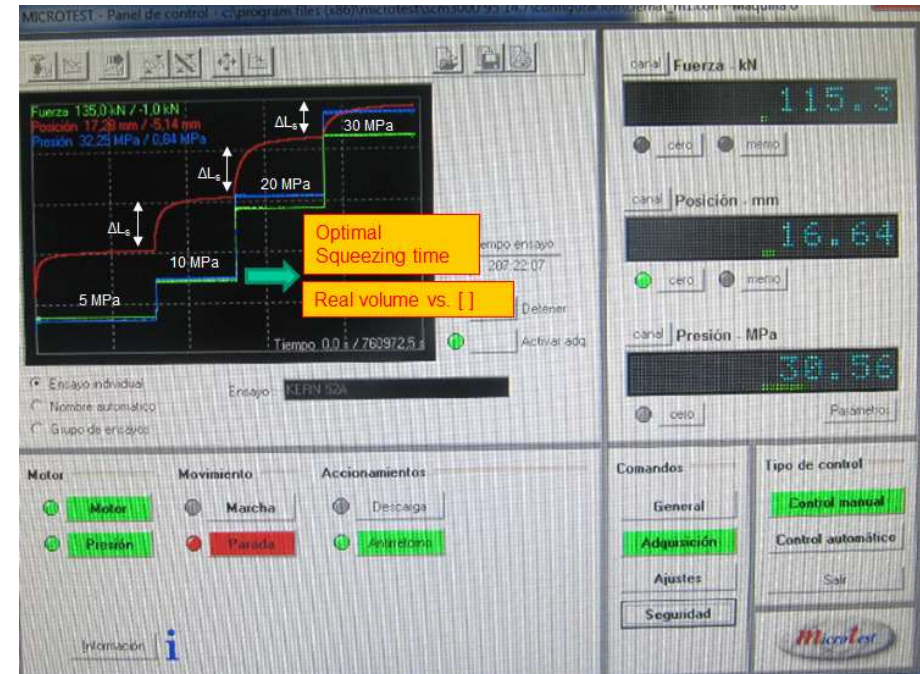
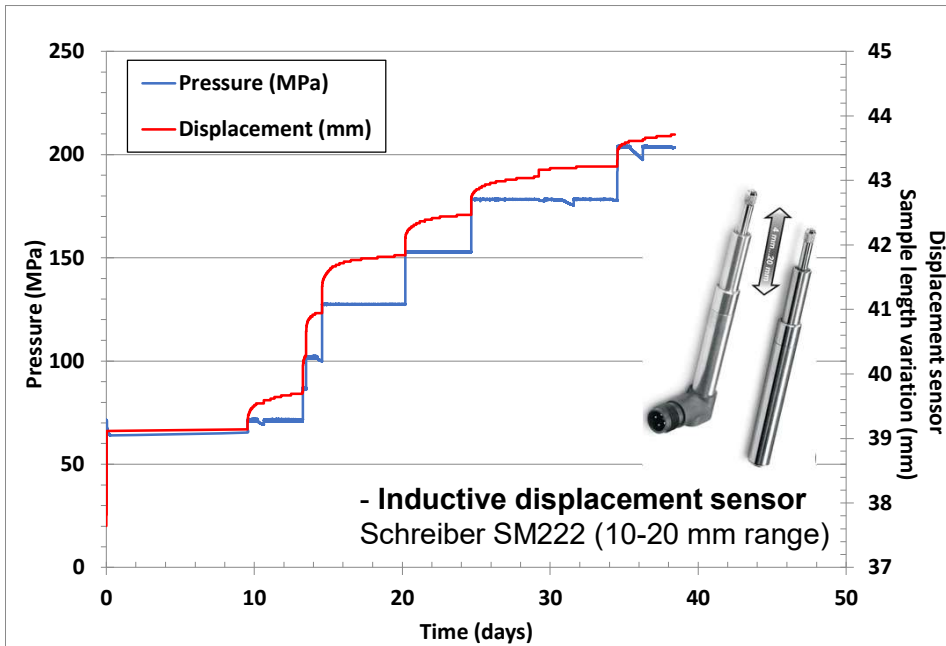
L = 500 mm
45 mm wall, $\phi_{\text{inner}} = 70$ mm



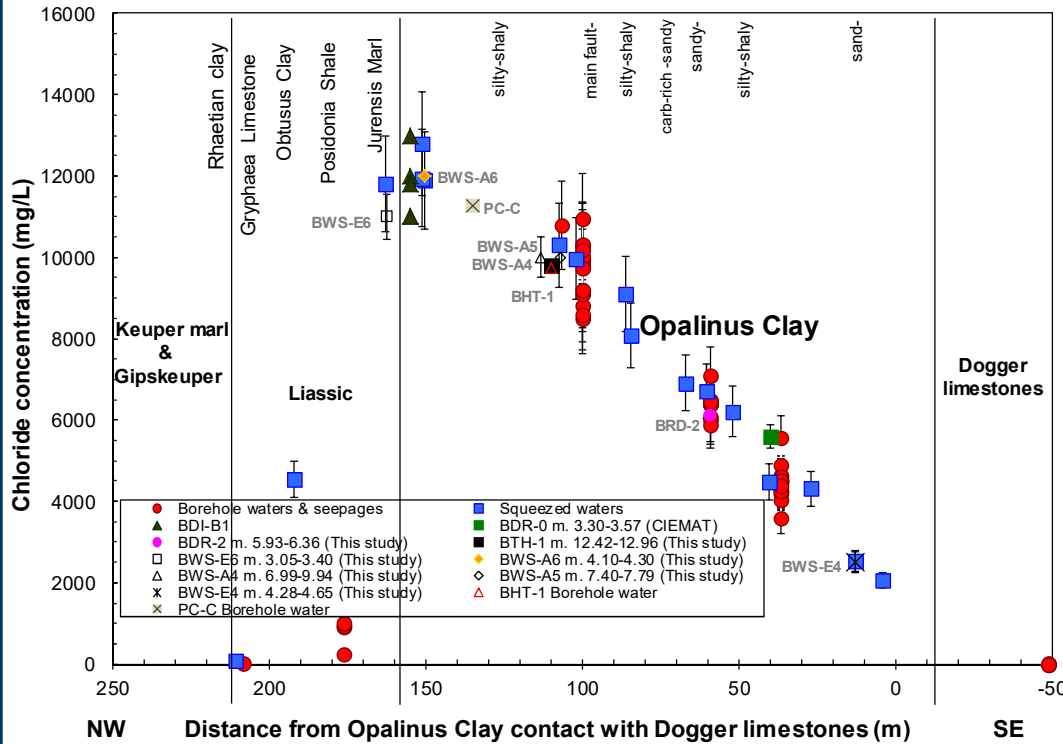
up to 800 MPa

L = 250 mm
90 mm wall, $\phi_{\text{inner}} = 60$ mm

Applying Squeezing Technique

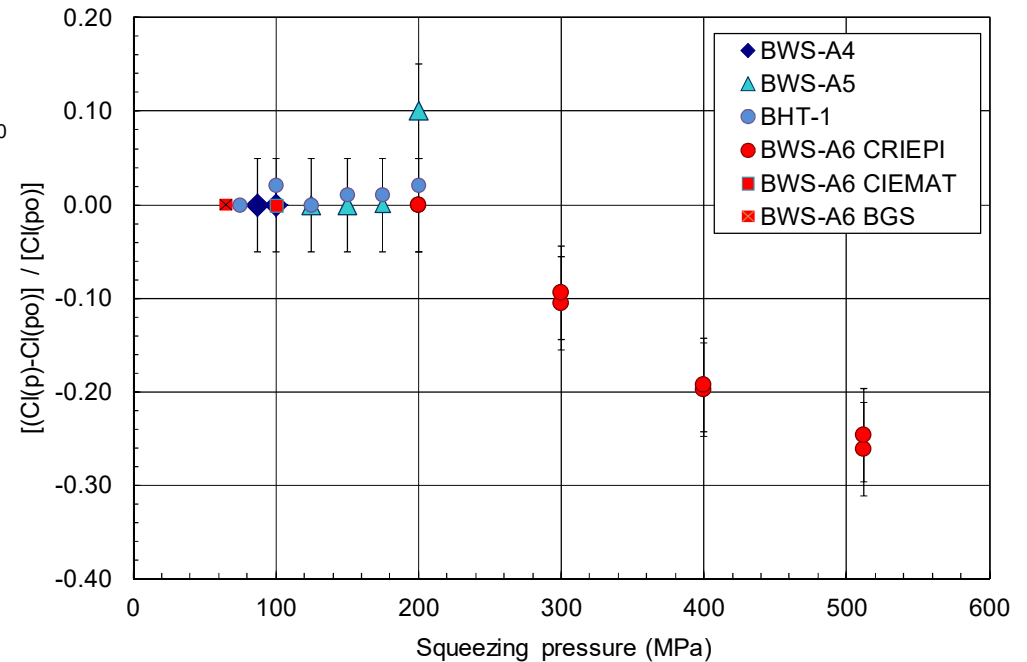


Applying Squeezing Technique



NW-SE cross-section of chloride concentration in waters from Mont Terri: Comparison of borehole waters and squeezing waters (Fernández et al., 2014)

Deviation of chloride concentrations at high squeezing pressures (Cl(p)) from those measured at lowest pressure yielding water (Cl(po)) for squeezing tests (Fernández et al., 2014)



Indirect Methodology

PHYSICAL PARAMETERS

MINERALOGY

- Non-Clay Minerals
- % Clay Minerals
- Main, accessory and trace minerals

- Grain size analysis
- Specific surface
- Dry Density
- Water content
- Pore Size Distribution
- Total porosity
- Geochemical Porosity

CHEMICAL PARAMETERS

- Ion exchange capacity
- Exchangeable Cations
- Selectivity coefficients
- Aqueous Extracts

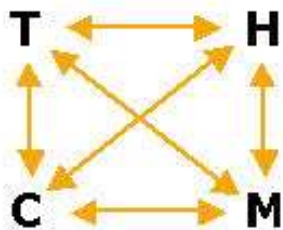
GEOCHEMICAL MODELLING FOR PORE WATER CALCULATION

Global Methodology

GEOCHEMICAL AND MINERALOGICAL CHARACTERIZATION

SURFACE PROPERTIES CHARACTERIZATION

PHYSICO-CHEMICAL CHARACTERIZATION



THMC BEHAVIOUR AND MODELLING

