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Final Laboratory Report

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Deliverables D3.34 and D3.36

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KEYWORDS
FORGE, sand-bentonit, index properties, two-phase flow,
suction, swelling, strain, PET, MIP, mock-up
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1. Nagra contribution to deliverables D3.34 and D3.36

1.1 Research objectives

The proposed concept for laboratory experiments (WP 3.2.5 / WP3.3.5) aim at providing a better understanding of the impact of different boundary conditions (e.g. hydrochemical interactions between cement porewater and sand/bentonite) on the long-term performance of the engineered barrier systems of a L/ILW disposal system. The objectives of the investigation programme can be broken down into the following categories:

- Develop a solid and reproducible protocol for column experiments with cement materials and S/B mixtures aiming at the determination of two-phase flow parameters (QA)
- Collect phenomenological evidence and develop conceptual models of gas transport in S/B with special focus on the associated hydro-chemical interactions
- Collect experimental data for a laboratory database for the determination of representative two-phase flow parameters of S/B. The parameters are input data for modelling gas transport processes in the S/B buffer of a L/ILW disposal system
- Collect laboratory data to support parametric models for the simulation of hydro-chemical sealing processes at the interface concrete – S/B
- Demonstrate process understanding in the framework of an intermediate-scale validation experiment.
Tab. 1: laboratory experiments listed against the main project objectives.

<table>
<thead>
<tr>
<th>Objectives</th>
<th>Reproducible protocol for column experiments</th>
<th>Phenomenological evidence &amp; conceptual understanding</th>
<th>Laboratory database &amp; representative two-phase flow parameters of S/B under different boundary conditions</th>
<th>Demonstrate process understanding</th>
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Experiment setup

- **Oedometer cells:**
  - Distilled water: X
  - Formation water: X
  - High-pH water: X

- **Perm. cells:**
  - Distilled water: X
  - Formation water: X
  - High-pH water: X

- **Triaxial cells:**
  - Distilled water: X
  - Formation water: X
  - High-pH water: X

- **Modular columns:**
  - Dynamic sat. exp. Tracer tests: X
  - Gas tracer tests: X

- **Micro cage (MIP):**
  - Distilled water: X
  - Formation water: X
  - High-pH water: X

- **PET experiment:**
  - Distilled water: X
  - Formation water: X
  - High-pH water: X

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1.2 Relevant gas paths in the L/ILW repository concept

The Swiss low- and intermediate level waste (L/ILW) repository concept foresees cavern plugs and a repository seal to prevent excess gas pressures in the repository. The main aim of the plugs and the seal are to increase the gas transport capacity of the backfilled underground structures without compromising the radionuclide retention capacity of the engineered barrier system (Nagra, 2008). The design option is called "engineered gas transport system (EGTS)" (Fig. 1). It involves specially designed backfill and sealing materials such as high porosity mortars as backfill materials for the emplacement caverns and sand/bentonite (S/B) mixtures with a bentonite content of 20 – 30 % for backfilling other underground structures and for the seals.
Fig. 1: Schematic picture of repository layout (top figure) and layout of repository sealing (bottom left) and sealing of emplacement caverns (bottom right). Red arrows indicate gas release pathways.

As seen in Fig. 1, there are different potential gas pathways that may or may not occur due to gas production in the repository after closure, namely:

- Gas flow into the host rock
- Gas flow along the EDZ
- Gas flow through the plugs and the seal

Preliminary experimental studies confirmed the high gas transport capacity of the S/B mixtures (Romero et al., 2003). Numerous experiments have shown as well the ability to design S/B mixtures with specific target permeabilities for water and gas flow (Agus et al., 2010; Colmenares Montanez, 2002). First in-situ experiences were gained through the Gas Migration Test (GMT) at Grimsel Test Site (GTS), but with in-silo emplacement (Shimura et al., 2008).

Numerical simulations of the entire repository were conducted which demonstrate the effective functioning of the EGTS concept for a range of repository configurations and parameter variants. The model calculations show clearly an engineered gas transport system can release
the gas that is produced in emplacement caverns very efficiently from the underground structures to the backfilled access tunnel into the adjacent rock formations. The model calculations show as well that both the length and the average gas/water permeability of the repository seal V4 are critical design parameters to avoid excess gas pressures in the repository. It is thus important, but yet to be demonstrated, that the two-phase flow properties from small-scale material samples are appropriate for the simulation of combined gas/water transport processes on the repository scale. Real scale validation experiments are indispensible for building confidence in the functioning of the EGTS.

More recent higher resolution modelling studies of the repository seal V4 have revealed a rather complex gas and water flow pattern in the repository seal in the first 10’000 years. In the early stage after sealing the repository water from the adjacent formations will be pushed into the S/B seal under hydrostatic pressure (few 10s of bars). After about 1000 years, the gas pressure in the repository is expected to become higher than the water pressure and gas begins to be expelled from the repository through the S/B seal into the neighbouring rock formation. Provided the modelling concept and the model assumptions underlying these results are correct, this would mean that the V5 plugs would, at no time, fully saturate during the gas production phase (about 100 ky) and thus would always remain highly conductive during the gas production phase.

Based on the evolution scenarios described above it is expected that a) boundary conditions differ depending on the location within the repository; and b) boundary conditions may change over time, both influencing the two-phase flow properties of the plugs and the seal. Two types of boundary conditions may influence the two-phase flow properties, namely a) the hydrochemical composition of the inflowing water; and b) the stress conditions exerted from the surrounding rock mass.

1.3 Expected boundary conditions

1.3.1 Hydrochemical boundary conditions

While standard laboratory experiments on bentonite and sand-bentonite mixtures are performed with distilled water, three types of water are expected at or near the repository plugs and the seal:

1. Water infiltrating into the tunnel system from the Opalinus clay will have high salinity (close to sea water) and a pH around 7. Due to the very low permeability of the Opalinus clay inflow rates into the repository are expected to be rather small.
2. Water saturating the repository plug from the overlying formations will have relatively high salinity (about 20-50% sea water) and a pH around 7. Inflow rates into the repository primarily depends on the pressure distribution around the plug and hydraulic permeability of the construction (S/B) material.
3. Water that is in direct contact with the concrete inside the emplacement caverns will equilibrate to reach a high-pH water.

Interaction with saline water

Past studies consistently show that water with higher salinity lead to a decrease in swelling pressure and an increase in hydraulic permeability (Studds et al., 1998; Karnland et al., 2006; Wilson et al., 2011). This effect has been attributed to one or more factors, such as exchange of Ca$^{2+}$ for Na$^+$ in the montmorillonite cation exchange positions or osmotic effects. Karnland et al. (2006) have performed swelling and permeability tests with a number of different bentonites. The results of the tests made with MX80 Na-bentonite are shown in Fig. 2 below. The figure...
shows that the impact on both parameters is stronger for lower dry densities such as those expected for 80/20 S/B mixtures. For salinities as expected in Opalinus Clay (i.e. about 0.3M) a reduction in swelling pressure of about a factor of three would be expected, whereas the reduction in hydraulic permeability should be about two, at the most.

![Swelling pressure and hydraulic permeability](image.png)

**Fig. 2:** Swelling pressure (left plot) and hydraulic permeability of Wyoming MX-80 reference material as a function of dry density and salinity of the saturation solution (from Karnland et al., 2006).

**High-pH interaction**

Chemical and mineralogical interfaces develop between cement and bentonite due to contrasting chemical properties of the two materials, with chemical reactions proceeding through a number of processes, such as (e.g. Savage, 1997; Gaucher and Blanc, 2006):

- Interaction of cement/concrete with groundwater will lead to leaching of cement in accord with a sequence of decreasing solubility of cement minerals with time, i.e. potassium/sodium hydroxide, followed by portlandite dissolution, followed by calcium silicate hydrate gel.
- Diffusive transport of cement pore fluids into compacted bentonite. Diffusion of bentonite pore fluids into the cement/concrete will lead to precipitation of carbonates (aragonite, calcite) at the interface and a decrease in porosity.
- Mixing and reaction of cement pore fluids with the entrained pore fluid in compacted bentonite. Sharp gradients in pH (and partial pressure of carbon dioxide) across the interface encourage the rapid precipitation of solid carbonates, such as aragonite and calcite, and hydroxides such as brucite (Mg(OH)$_2$), leading to a decrease in porosity.
- Fast exchange of cations in cement pore fluids (principally K, Na, and Ca) for cations (principally Na$^+$ in MX-80 bentonite) in interlayer sites in montmorillonite, leading to a decrease of swelling pressure. This process is diffusion-controlled and time-dependent in the sense that cement leachates become increasingly calcic with time due to incongruent dissolution processes (e.g. Berner, 1992), such that early exchange of Na by K will be replaced by Ca with increasing time.
- Decrease of swelling pressure of bentonite in contact with cement or cement pore fluids has been documented by a number of authors (e.g. Karnland, 1997; Karnland et al., 2007; Sugiyama and Tsuji, 2008) and has been attributed to one or more factors, such as exchange
of Ca\(^{2+}\) for Na\(^{+}\) in the montmorillonite cation exchange positions, osmotic effects, and/or mass loss due to mineral dissolution (Karnland, 1997).

- Slow hydrolysis of montmorillonite and other minerals present, either as additives (e.g. quartz sand), or as accessory minerals, such as quartz, feldspars, pyrite, and gypsum. At elevated pH, such reactions consume hydroxyl ions, thus chemically neutralising the advancing cement pore fluids. These reactions lead to an increase in porosity and may decrease clay swelling pressure due to mass loss.

- Precipitation of secondary minerals such as clays, hydroxides, carbonates, calcium silicate hydrates, and aluminosilicates such as zeolites and feldspars (e.g. Savage et al., 2007). These reactions may be relatively fast (e.g. hydroxides, carbonates, calcium silicate hydrates), or relatively slow (e.g. clays, zeolites, feldspars). For example, rate constants of dissolution/growth of aluminosilicates are typically less than 1E-10 s\(^{-1}\) in compacted bentonite at 25 °C, with growth rates being further inhibited by slow rates of nucleation and consequent development of reactive surface area (Steefel and van Cappellen, 1990).

- Mineral dissolution, especially silica and clay can lead to mass loss, decrease of swelling pressure, and an increase in porosity of the bentonite. The dissolution rate of quartz is greater than that for montmorillonite at high pH (e.g. Knauss and Wolery, 1988; Sato et al., 2004) and will thus tend to suppress dissolution of the latter.

- The replacement of montmorillonite by carbonates, hydroxides, and framework silicates (feldspars, zeolites) may lead to rheological changes at the interface.

Physical properties potentially changed by the development of the cement-bentonite interface can therefore be defined as: swelling pressure; porosity; diffusivity; permeability; and rheology. These issues will be considered in turn, below.

### 1.3.2 Confining stress conditions

After emplacement of the plugs and the seal the surrounding host rock is expected to exert an increasing confining stress that may influence the two-phase flow properties of the gas-permeable seals.

In the past 20 years more and more researches aimed at improving the understand of the main mechanisms of gas transport through compacted clays in saturated and unsaturated conditions. In the case of fully saturated clays two kinds of gas transport threshold pressures are generally detected (Tanai et al., 1997, Gallé and Tanai, 1998; Gallé, 2000; Arnedo et al. 2008). The first one is called gas entry pressure or critical pressure, and corresponds to the pressure below which the gas can migrate through the water phase by diffusion only. When the injection pressure is higher than the maximum capillary pressure that can be sustained by the largest pervious clay pores (air entry value), gas migrates by advection and the two phase flow is established. From this moment on, if the injection pressure is maintained, the gas can migrate through the specimen mainly by advection. This gas intake is generally associated with a water output on the other side of the specimen.

The breakthrough is observed when the gas injection pressure is increased and it is in general related to the formation or development of preferential migration pathways, which may enlarge and propagate in the clay (Volkaert et al. 1995; Horseman & Harrington 1997). These pathways are relatively unstable, leading to intermittent gas outflow. For pure bentonite, it has been observed that, under constant stress boundary conditions, the breakthrough of a gas into an initially water-saturated bentonite is only possible when the gas pressure exceeds the sum of the swelling pressure and water backpressure (Horseman & Harrington 1997, Gallé, 2000, Arnedo et al. 2008).
For pure bentonite, experiences highlighted that in most cases, there is no progressive desaturation of bentonite by increasing the gas pressure. Horseman et al. (1999) suggests that for highly compacted clay the air entry pressure necessary to overcome the tension of water in pores is so important that the theory of a visco-capillary flow with displacement of the water of the pores is not applicable. This can be explained by the extreme narrowness of inter-particle voids, but also by the extremely strong physicochemical interaction between molecules of water and layers of clay. This is why, according to Horseman, the bentonite will always fissure before the apparent air entry pressure is reached.

It is currently unclear what the dominating two-phase flow process in S/B mixtures really is and to what extent the mixing ratio and the dry density has an influence on the water and gas transport behaviour. If the dominating transport process is visco-capillary flow there should be no or only little stress or strain reaction due to gas transport (coupling through swelling) and changing the stress boundary conditions should have no influence on gas entry pressure nor on gas transport capacity.
2 Material preparation, experiment setups and results

2.1 Material characterization

2.1.1 Composition

The investigated material is a mixture of grey quartz sand and MX-80 Bentonite (Wayoming) in proportion 80/20 in dry mass.

The MX-80 is a sodium granular bentonite, named mixture ‘E’, which has been delivered at the EPFL in June 2009. The index properties of the MX-80 bentonite are shown in Tab. 2. The MX-80 bentonite is mainly composed of 84.9 ± 1.2 % montmorillonite, 4.8 ± 0.8 % muscovite, 3.7 ± 0.5 quartz, 5.2 ± 0.8 % feldspar and 1.3 ± 0.2 % calcite (Nagra, 2007).

MX-80 mixture ‘E’ bentonite shows exchangeable cations of 52.4 meq/100 g Na, 13.2 meq/100 g Mg and 1.4 meq/100 g K (Nagra, 2007).

Tab. 2: General properties of MX-80 bentonite (Müller-Vonmoos and Kahn, 1983; Lajudie et al., 1996; Seiphoori, in prep.).

<table>
<thead>
<tr>
<th>Bentonite</th>
<th>Specific gravity, $G_s$</th>
<th>Liquid limit, $w_L$ (%)</th>
<th>Plastic limit, $w_P$ (%)</th>
<th>Shrinkage limit, $w_s$ (%)</th>
<th>Specific surface area, SS (m²/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MX - 80</td>
<td>2.74-2.76</td>
<td>411 ± 10</td>
<td>47 - 70</td>
<td>14</td>
<td>562</td>
</tr>
</tbody>
</table>

The role of bentonite is to reduce the hydraulic conductivity of the mixture thanks to the swelling capacity of the smectite minerals.

The quartz sand, which has been used for all experiments, is a grey quartz sand from Carlo Bernasconi AG (carloag.ch). Its specific density is 2.65 g/cm³. The mineralogy of this quartz sand consists mainly of Silicon dioxide (97.40%), Alumina (1.35%), Potassium and Sodium oxides (0.8%), small amounts of Titanium dioxide, Periclase and Lime.

The presence of sand in the material reduces the shrinkage of the mixture when the water content decreases and thus reduces the risk of cracking. This risk is much more present in a pure bentonite (Graham, 2002), while at low bentonite contents, the sand particles are in direct contact with each other. Knowing the particle density of the two components, the particle density of the mixture was obtained using the following expression:

$$\rho_{s, mix} = \frac{100 \%_{sand}}{\rho_{s, sand} + \%_{bent}} = 2.67 \text{ g/cm}^3$$

2.1.2 Specimen preparation

The mixture used for the experiments was prepared following the outcomes of the Mock-up experiment (see Rueedi et al., 2010).

The sand and the granular bentonite, at the initial water content, were initially sieved at 0.5 mm, and then they were mixed manually. Once a good homogenization of mixture was obtained, the 11% of distilled water in weight was added. The obtained water content corresponds to the optimum water content determined by means of the modified Proctor test, carried out at
according to the Swiss Standard SN 670 330, under specific compaction energy of 3.4 J/cm$^3$ (Fig. 3). The wet mixture was carefully mixed again until reaching a good homogenization (Fig. 4). Finally the mixture was stored in hermetic containers for at least 3 days in order to obtain a homogenous water distribution over the whole mass of material.

Fig. 3: Modified Proctor test on S/B mixture in proportion 80/20 (Minon et al. 2010).

Fig. 4: S/B mixture prepared at $w = 11\%$.

Fig. 5: S/B mixture poured into the oedometric ring.
For the oedometric tests the compaction of the specimen was performed directly inside the oedometric ring (Fig. 5). Two filter papers were placed along the two bases. The specimens were prepared by static compaction. Two different presses were used for the compaction. The first one, from Wikeham Farrance Eng. LTD, was equipped with a load cell (maximum vertical force of 22 kN, accuracy of 0.1% of the full range) and the piston displacement was monitored by a LVDT (accuracy of 1 μm) (Fig. 6). The second press is a Walter & Bai type which is instrumented with a load cell (maximum vertical force of 100 kN, accuracy of 0.1 of the full range) and a LVDT (range of 20 mm and accuracy of 0.2%). The second press was used to compact specimens with dry density higher than 1.6 g/cm³. A constant compression rate of 0.5 mm/min was used for the preparation of all the specimens. For the preparation of the specimens tested in the triaxial cell, the same compaction procedure was applied with the help of a cylindrical mould.

Referring to Fig. 6, the vertical displacement of the piston (δ_{comp}) required to obtain the target dry density was determined as follows:

\[ \delta_{comp} = H_{1,IN} - H_{1,FIN} - \delta_{el} \]  

where \( H_{1,IN} \) is the initial surplus height of the piston, carefully measured with a calliper when the mixture is non-compacted yet, \( H_{1,FIN} \) is the final surplus height of the piston (final target height), and \( \delta_{el} \) is the elastic rebound that the material exhibits when the vertical stress is released.

\( H_{1,FIN} \) is obtained as:

\[ H_{1,FIN} = H_{1,0} + H_{sp} + t_{fp} \]  

where \( H_{1,0} \) is the surplus height of the piston in phase 1 when the cell is empty (Fig. 7), \( H_{sp} \) is the target specimen height, and \( t_{fp} \) is the thickness of the filter papers.

Knowing the high of the not-compacted specimen (\( H_{sp,i} = H_{1,IN} - H_{1,0} \)), the dry mass of the material (\( M_s \)) which is poured inside the cell and the initial dry density (\( \rho_{d,i} \)) of the specimen before compaction, the evolution of the specimen height (\( H_{sp} (\sigma_i) \)) and its dry density can be computed from the LVDT readings (\( \Delta H (\sigma_i) \)) as:
\[ H_{sp}(\sigma_v) = H_{sp,t} - \Delta H(\sigma_v) \]  \hspace{1cm} (3)

\[ \rho_d(\sigma_v) = \frac{M_s}{A_{sp} \cdot H_{sp}(\sigma_v)} \]  \hspace{1cm} (4)

The compaction is carried out until the target displacement of the piston \( \delta_{\text{comp}} \) is reached.

A compressive stress from 0.2 MPa to 20 MPa was applied to obtain specimen with a dry density that vary from 1.3 g/cm\(^3\) to 1.9 g/cm\(^3\).

Fig. 7: Specimen compaction phases.

Typical curves obtained during specimen compaction are showed in Fig. 8. In this Figure both the variation of the specimen height and the dry density with the applied vertical stress are presented. The curves are used to quantify the elastic rebound associated to the vertical stress release.

Fig. 8: Evolution of the specimen height (left) and the dry density (right) with the applied vertical stress during the specimen compaction.
2.2 Microstructural characterization

Due to the swelling capacity of the smectite minerals, the pore structure of the backfilling material changes as the degree of saturation changes. Wetting and drying processes affect remarkably the pore size distribution of the material; in particular the size of the macropores is reduced by the swollen bentonite whereas the microporosity remains essentially unaffected (Agus & Schanz, 2005).

The pore size distribution of the S/B mixture has been investigated using the mercury intrusion porosimetry (MIP) technique. Details on the device used to perform the experiments are given in the following section. Then the results of the tests are presented.

2.2.1 Mercury intrusion porosimetry (MIP)

In the MIP technique, a non-wetting fluid (mercury) is forced, by applying pressure, to penetrate into the specimen pores. During the test, the volume of the intruded mercury is measured as a function of the applied pressure. A cylindrical shape of the pores is assumed for the interpretation of the test results. With this assumption, the Washburn equation is used to relate the pore diameter ($D$) to the pressure at which the mercury intrudes the pore ($P$):

$$
D = \frac{4\sigma_{nw} \cdot \cos \theta_{nw}}{P}
$$

(5)

where $\sigma_{nw}$ and $\theta_{nw}$ are the surface tension and the contact angle of the non-wetting fluid, respectively. For the analysis of the test a value of $\sigma_{nw} = 0.480$ N/m was adopted. The contact angle was assumed equal to 140°.

The MIP tests on S/B mixture were performed using a Pascal 140/440 Porosimeter from Thermo Electron Corporation. In the first part of the test, the Pascal 140 unit (low pressure) was used. Once the specimen was inserted in the dilatometer the vacuum was applied for 15 minutes and then the intrusion of mercury was started at low pressure. By the first filling of the dilatometer, the apparatus provides information on the volume of the specimen and, consequently, on its bulk density. The mercury pressure was then slowly increased to reach a maximum pressure of 100 kPa, corresponding to a pore diameter of 151.3 µm. During this process the intruded volume was recorded by the unit. At the end of the measurement with the low-pressure unit, the dilatometer was transferred into the high-pressure unit (Pascal 440) where the pressure was increased up to 400 MPa, corresponding to a pore diameter of 3.8 nm. At the end of the pressure increase phase the apparatus computes the apparent volume of the grains. This value is in general higher than the real volume due to the fact that the total specimen porosity cannot be completely filled by the mercury due to the limitation of the MIP technique. After the maximum pressure was reached, the pressure was progressively reduced and the extruded mercury volume was registered. It is worth to note that MIP technique can be used to detect the macro and micro pores but it cannot be used to detect nanopores which are generally smaller than 3.8 nm.

2.2.2 MIP results

The first group of MIP tests were performed on S/B mixture specimens compacted at a dry density of 1.5 g/cm³ and 2.0 g/cm³ with 11% of water content (distilled water). Thus, the influence of the dry density on the pore size distribution was investigated. To study the effect of water saturation on the pore size distribution, an additional test was performed on a specimen prepared initially with a dry density of 1.5 g/cm³ and then saturated under constant volume conditions with distilled water.
The three tested specimens were dried by the freeze drying technique in order to minimize the changes in volume and pore size distribution during the extraction of water (Penumadu & Dean, 2000). The technique consists in placing the specimen in a vacuum chamber (at 0 mbar and at a temperature of -52°C) where it is dried by sublimation for 24 hours.

The results of the MIP tests are depicted in Fig. 9. All test results show a bi-modal pore size distribution. The criterion adopted to divide the macro by the micro porosity is the one suggested by Romero (1999), which fixes the limit between macro and micro porosity at 0.1 μm. The macroporosity region is characterized by modal values of the pore diameter equal to 51 and 72 μm for the dry densities of 2.0 g/cm³ and 1.5 g/cm³, respectively. The macropores are associated with the voids between the sand particles and at the contact zones between clay assemblages and sand particles. The microporosity region has a peak at the value of about 16 nm; it is associated to porosity within the clay assemblages and it remained unchanged for all the three specimens (Tab. 3). As it was expected the saturation process and the dry density change affect remarkably the pore structure of the material. Due to the increase in dry density the sands particles, as well as the bentonite pellets, become closer and consequently a reduction of the macroporosity was observed. The comparison between the MIP curves for the as compacted and the saturated specimen with dry density of 1.5 g/cm³ showed a reduction of the mode diameter from 72 μm to 4 μm. This reduction is attributed to the penetration of the water inside the interlayer clay space (nanoporosity) which is induced by osmotic process. In constant volume condition the increase in nanoporosity was compensated by a decrease of macroporosity.

Tab. 3: Characteristics of the tested specimens for the MIP.

<table>
<thead>
<tr>
<th>Specimen code</th>
<th>Dry density (g/cm³)</th>
<th>Specimen state</th>
<th>Mode of the pore diameters in the macropores region, d_M (μm)</th>
<th>Mode of the pore diameters in the micropores region, d_m (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S/B-1.5</td>
<td>1.5</td>
<td>As compacted</td>
<td>≈72</td>
<td>≈16</td>
</tr>
<tr>
<td>S/B-1.9</td>
<td>2.0</td>
<td>As compacted</td>
<td>≈51</td>
<td>≈16</td>
</tr>
<tr>
<td>S/B-1.5_SATd</td>
<td>1.5_SATd</td>
<td>Saturated with distilled water</td>
<td>≈4</td>
<td>≈16</td>
</tr>
</tbody>
</table>

Fig. 9: Pore size density function of three S/B specimens.
2.3 Experiments on swelling behaviour

2.3.1 Oedometric cells for swelling tests

The apparatus used for the swelling tests consists of a rigid stainless steel cylinder of 79 mm of internal diameter and 110 mm of external diameter. This rigid cell ensures the oedometric condition during the test. The base of the cell is connected to a flushing line, allowing the elimination of entrapped air bubbles. Inside the cell, 15 mm high specimens were placed between two filter papers and two coarse porous stones. The water in the apparatus was injected at 2 kPa of pressure using a pressure-volume (PV) controller connected to the base of the cell.

For the constrained swelling test the oedometric cell was positioned inside a rigid frame in order to maintain the constant volume condition during the test (Fig. 10). Three screws were used to compress the rubber O-ring placed between the oedometric ring and the bottom plate; hence, any leakage is prevented. A load cell (maximum vertical force of 50 kN), in contact with a stainless steel loading ram, installed at the top of the specimen was used to measure the swelling pressure developed during the test. A PVC lid on the top of the cell prevented the evaporation of the water from the upper base of the specimen. The unwanted change in volume of the specimen during the swelling was monitored by means of a LVDT; the maximum registered volumetric strain was less than 0.1%.

![Fig. 10: Scheme of the apparatus for constrained swelling tests.](image)

The set-up used for the free swelling tests is showed in Fig. 11. In this configuration, the upper part of cell was closed with a stainless steel lid. The specimen deformation was measured by a LVDT placed at the top of a movable rod in contact with the specimen. The upper base of the specimen was covered with a plastic foil to limit water evaporation.

For both, the constrained and the free swelling test, the transducers and the controllers were connected to a HBM data acquisition system, model QuantumX MX840A, allowing a continuous record of the measurements.
2.3.2 Resulting swelling capacity

The term swelling capacity includes both the swelling pressure and the swelling deformation. S/B mixture exhibits its swelling capacity due to the presence of high percentage of smectite minerals in the MX-80 bentonite (Section 0).

The swelling characteristics of bentonites and S/B mixtures, and the parameters that influence the maximum swelling pressure or swelling deformation of these materials, have been studied by several authors (Agus & Schanz 2005, Herbert et al. 2008, Studds et al. 1998, Komine & Ogata 1999, Komine & Ogata 2004, etc.). The following behaviours were highlighted with the mentioned studies:

- the initial dry density controls the magnitude of the swelling pressure and the swelling deformation of compacted bentonite and sand bentonites mixtures; in the case of S/B mixtures the swelling capacity of these materials is also controlled by the percentage of bentonite;
- by increasing the applied vertical pressure the swelling deformation decreases;

Depending on the salt concentration of the water used as saturating fluids a reduction or even a total loss of the swelling capacity of bentonites or S/B mixtures materials can be observed.

Under repository condition the S/B mixture could be saturated with three different types of water:

- formation water from the surrounding Opalinus Clay entering in the S/B mixture in the early pressurization phase;
- ditto, but flowing through the cementitious backfill before reaching the S/B seal;
- formation water from the above higher-permeability formation.
The montmorillonite minerals in contact with high salinity and cemented solutions undergo changes in morphology, crystallinity, particle surface, interlayer charge and chemistry of the octahedral layers (Herbert et al., 2004 and Herbert et al. 2008). Due to chemical alteration a certain percentage of the montmorillonite in the bentonite is transformed in kaolinite. These processes may lead to a significant or even total loss of the swelling capacity of the material (Herbert et al. 2004).

Available results on 80/20 S/B mixtures are limited. In this sense, an extensive laboratory programme was planned and carried out with the aim to evaluate the importance of parameters such as the initial dry density and the chemistry of the saturating fluid. Swelling pressure and swelling deformation tests were performed on specimens prepared at an initial dry density that was varied from 1.3 g/cm³ to 1.9 g/cm³. In these experiments two types of water, distilled and synthetic water were used as saturating fluid. The synthetic water was prepared in laboratory with the same chemical composition of the water expected in the underground repository (Traber, 2011). Its chemical composition is reported in Tab. 20. For this water, an osmotic suction of 0.9 MPa was measured with the WP4c.

Figs. 12 and 13 show the results of the swelling pressure tests performed on the S/B mixture (80/20) using distilled and synthetic water, respectively. The evolutions of the swelling pressure and the water volume entering into the specimen are depicted. The results show that the swelling pressure generated very fast in the early stage of the tests (up to 50 hours for distilled water and up to 25 hours for the synthetic one). After this stage the swelling pressure increasing rate reduced significantly. Comparison between the volumes of water that entered into the specimen at the different dry densities, Fig. 12-b, shows a significant decrease for the specimen with higher dry density. This reduction is attributed to the reduction of the permeability of the specimen caused by the reduction of the macropores as shown by the MIP tests (see Section 2.2.2) for the specimen with higher dry density. The results of the swelling tests on the specimens saturated with synthetic water are presented in Fig. 13. The swelling test on the specimen compacted at 1.3 g/cm³ of dry density was stopped after 17 hours due to the fact that no swelling pressure was generated during this test. Moreover, a remarkable water inflow equals to 2.98E-9 m³/s was measured, confirming the inhibition of the swelling capacity of the bentonite by the synthetic water. Similar to the distilled water case, the increase of swelling pressure and reduction of water inflow for the specimens with higher dry densities were observed.

**DISTILLED WATER**

![Distilled Water Results](image)

**Fig. 12:** Results of the swelling pressure test using distilled water in terms of swelling pressure and water volume exchange for the tested specimens. In the graph the average swelling pressure measured for each specimen is indicated.
The dry density, at which the specimen is compacted, has a strong impact on the developed swelling pressure; Fig. 14 shows the swelling pressure as a function of the specimen dry density for both distilled and synthetic water; in the figure the values of the swelling pressure reported correspond to the weighted average of the swelling pressures measured (after 50 hours for distilled water and after 25 hours for the synthetic one). An exponential trend for the swelling pressure with the dry density was taken as best fit for both water types.

The use of synthetic water reduced remarkably the swelling pressure. The rate of reduction of the swelling pressure ($R_{P_{sw}}$) with the dry density was calculated as proposed by Komine et al. (1999):

$$R_{P_{sw}} = \frac{P_{SW,\text{DW}} - P_{SW,\text{SW}}}{P_{SW,\text{DW}}}$$

where $P_{SW,\text{DW}}$ is the swelling pressure in distilled water and $P_{SW,\text{SW}}$ is the maximum swelling pressure in synthetic water. The reduction is in the range of 67% - 45% for the dry density in the range of 1.3 - 1.9 g/cm$^3$ when the mixture was in contact with synthetic water. The reduction factor trend shows that the loss in swelling capacity due to the saturation with synthetic water, in the range of dry densities under consideration, can be partially compensated by the increase in dry density.
Fig. 14: Comparison between the swelling pressures developed with distilled water and in situ pore water.

Fig. 15 shows the results of the free swelling tests carried out on the S/B mixture at different dry densities and using both distilled and synthetic water. The evolutions of the swelling strain and the water volume entering into the specimen are depicted. Fig. 15-a shows an increase of the swelling strain by increasing the dry density. This increasing trend was also observed for the free swelling tests with synthetic water; however the maximum swelling strain is much lower for these tests with respect to the tests with distilled water.

The swelling strain in these experiments was defined as:

$$\varepsilon_{sw} = \frac{H(t) - H_0}{H_0}$$  \hspace{1cm} (7)

where $H(t)$ is the specimen height measured in time with the differential transducer, and $H_0$ is the initial height of the specimen.

The evolution of the swelling strain with time ($\varepsilon_{sw}(t)$) can be derived from the experimental results by fitting the data with a hyperbolic function (Komine & Ogata, 1999):

$$\varepsilon_{sw}(t) = \frac{t}{a + bt}$$  \hspace{1cm} (8)

where $a$ and $b$ are curve fitting parameters.

The asymptote of the hyperbole represents the maximum swelling strain of the material reached at the end of the saturation process and it is calculated as:

$$\varepsilon_{sw, max} = \lim_{t \to \infty} \frac{t}{a + bt} = \frac{1}{b}$$  \hspace{1cm} (9)
The parameter a and b, and the maximum swelling strain, determined at different dry densities, are summarized in Tab. 4 and 5 for distilled and synthetic water, respectively.

### DISTILLED WATER

<table>
<thead>
<tr>
<th>$\rho_d$ (g/cm³)</th>
<th>a (-)</th>
<th>b (-)</th>
<th>$\varepsilon_{sw, max}$ (-)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.5</td>
<td>141.61</td>
<td>2.20</td>
<td>0.45</td>
</tr>
<tr>
<td>1.6</td>
<td>93.82</td>
<td>1.83</td>
<td>0.55</td>
</tr>
<tr>
<td>1.8</td>
<td>121.02</td>
<td>0.94</td>
<td>1.06</td>
</tr>
</tbody>
</table>

### IN SITU WATER

<table>
<thead>
<tr>
<th>$\rho_d$ (g/cm³)</th>
<th>a (-)</th>
<th>b (-)</th>
<th>$\varepsilon_{sw, max}$ (-)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.45</td>
<td>1.67</td>
<td>8.89</td>
<td>0.11</td>
</tr>
<tr>
<td>1.62</td>
<td>8.93</td>
<td>4.59</td>
<td>0.22</td>
</tr>
<tr>
<td>1.75</td>
<td>55.04</td>
<td>1.94</td>
<td>0.52</td>
</tr>
</tbody>
</table>

Fig. 15: Results of the free swelling test using distilled water (plot a and b) and in situ pore water (plot c).
In these experiments the rate of reduction of the free swelling capacity with the dry density was calculated as (Komine et al., 1999):

$$R_{e_{sw}} = \frac{\varepsilon_{SW,DW} - \varepsilon_{SW,SW}}{\varepsilon_{SW,DW}}$$

(10)

where $\varepsilon_{SW,DW}$ is the maximum free swelling in distilled water and $\varepsilon_{SW,SW}$ is the maximum free swelling pressure in synthetic water. The rate of reduction of the free swelling, in the considered range of dry density was calculated from the value of the free swelling taken from curve fittings of the experimental data for both distilled and synthetic water.

Fig. 16 depicts the results of the free swelling tests carried out using both distilled and synthetic water. The values of swelling strain reported in the figure are the maximum swelling strain expected at the end of the swelling process ($\varepsilon_{SW,max}$); these values were indicated in Tabs. 4 and 5. The experimental data has been fitted using a linear function as suggested by Komine (1999).

Due to the reduction of the swelling capacity of the mixture in contact with synthetic water the time required for the swelling test was strongly reduced, from about 200 hours for distilled water to less than 50 hours for the synthetic one. Supposing a linear correlation between swelling strain and dry density the minimum dry density required to have an observable swelling deformation of the mixture is about 1.3 g/cm$^3$ when distilled water is used and about 1.4 g/cm$^3$ for synthetic water. This latter observation is in agreement with the results of the constrained swelling tests in which, for the test carried out with synthetic water, no remarkable swelling pressure was observed for a dry density of 1.3 g/cm$^3$.

**Fig. 16:** maximum swelling strain as function of the initial dry density for S/B mixture 80/20.
The rate of reduction of swelling strain decreased very fast for a dry density that varies from 1.4 to 1.6 g/cm³. For dry densities higher that 1.6 g/cm³ the curve tends to an asymptotic value of 0.4.

2.3 Permeability tests

2.3.1 Permeameter cell

The permeameter cell was used to determine the water permeability, the gas permeability and the breakthrough pressure of fully saturated specimens.

The permeameter consists of a stainless steel ring of 51 mm of internal diameter and 20 mm high situated between two plates that are fixed together with four screws (Fig. 17). The screws allow the compression of two O-rings which ensure the tightness of the cell. Each plate contains a central hole which is closed with a valve and connected to PV controller. Three more holes are located around the central hole to allow the preliminary flushing operation of the lines. Inside the cell a specimen is placed between two coarse porous disks and two filter papers which enable a good distribution of the water along the specimen surface.

Fig. 17: Photographs of permeameter cell.

2.3.2 Triaxial cell

The used triaxial cell is a Bishop-Wesley system manufactured by GDS Instruments. The bottom cap is equipped with two injection holes which allow performing the flushing prior to water or gas injection. The top cap is equipped only with one injection hole. The triaxial cell was used to perform water permeability tests and gas injection tests at different confining pressures in order to verify the influence of the stress state on the permeability and the breakthrough pressure of the S/B mixture.

The specimen inside the cell is 50 mm in diameter and 15 mm high. The short height of the specimen allowed speeding up the saturation process. The specimen is wrapped in two membranes in order to prevent any leakage of either water or gas due to the possible damage of one of the membranes during the test. In triaxial conditions the presence of radial confinement ensures the good contact between the specimen and the membranes and prevents the formation of preferential flow paths along the specimen lateral surface.
2.3.3 Modular column cells

The modular column cell (MC) was used to perform permeability tests and breakthrough tests on fully saturated specimens (Fig. 18). The MC consists of two plates (bottom and top) and several modules that can be assembled together allowing the selection of the total height. The plates contain two injection and extraction lines each, in order to allow the flushing operations. The modules are cylindrical shaped elements of 60 mm high, 101 mm inner diameter and 136 mm outer diameter (Fig. 19-a). The different modules are assembled together by means of 6 screws. The sealing between each module is ensured by rubber O-rings. The MCs are made of stainless steel and they can sustain pressures up to 20 bars. The mixture can be directly poured and compacted in the columns. During the preliminary phase of the FORGE project the MC has been modified by adding an inlet in which a pressure sensor is inserted. The inlet for the sensor consists of a steel cylinder covered at its end with a ceramic filter cap (pore size equal to 1 μm) and connected to a manometer and to a pressure sensor (Fig. 19-b). The ceramic filter cap, which allows maintaining the circuit saturated as well as stopping small particles entering the pressure sensor line, is placed at the centre of the module. The pressure sensor line is first composed by a manometer (pressure range between 0 to 25 bars) followed by two way valves and ended by a pressure transducer (pressure range 0 to 10 bars) which gives a higher precision for the pressure measurement. A dummy ensures the water tightness between the inside and the outside of the module. In the setup for the MC cell, thin layer of resin was add on the walls of the cell in order to prevent the formation of preferential flow path.

Fig. 18: Scheme of a column with two modules and two sensor ports with installed manometers.

Fig. 19: Module assembled with a sensor line, b) Sensor line with the filter cup, the dummy, the manometer and the two way valve.
2.3.4 Experiment setup for water permeability testing

The water permeability tests were carried out in steady state conditions by applying pressure gradients between two specimen ends. The inlet and outlet water fluxes were monitored by two PV controllers (Fig. 20). In the first phase of the test the specimen was saturated maintaining a low pressure difference (e.g. 10 kPa). The specimen was considered fully saturated when the measured inflow and outflow matched (Fig. 21).

Fig. 20: Example of the experimental setup for water permeability test with the permeameter cell.

![Diagram](image)

Fig. 21: Example of the saturation phase of the specimen compacted at $\rho_d = 1.45 \text{ g/cm}^3$. The height of the specimen is 15 mm.
In the case of the test performed in the triaxial cell, the confining pressure was increased while ensuring drained conditions to the specimen. Then vacuum was applied at the top and the bottom of the specimen to remove the air from the specimen pores; during this phase -100 kPa of vacuum was reached. This pressure was lower than the maximum pressure experienced by the specimen during the compaction phase. Thus the modification of pore structure by this pressure was considered as negligible. The full saturation of the mixture was ensured when the equality in inflow and outflow was reached.

In both type of tests, the hydraulic conductivity \((K\) in m/s) was computed using Darcy’s law for incompressible fluids and neglecting the difference in elevation:

\[
K = \frac{Q \cdot \gamma_w \cdot L}{A \cdot (P_{up} - P_{dw})}
\]  

(11)

where \(Q\) is the volumetric flux, \(A\) is the specimen surface, \(P_{up}\) and \(P_{dw}\) are the upstream and the downstream pressure, \(\gamma_w\) is the specific weigh, and \(L\) is the drainage length. Knowing the hydraulic conductivity \(K\), the intrinsic permeability of the medium \((k)\) is calculated as:

\[
k = \frac{K \cdot \mu}{\gamma_w}
\]  

(12)

where \(\mu\) is the dynamic viscosity of the water.

The permeability tests were performed under isochoric condition, using the permeameter cell and the MC cell, on specimen compacted at dry density of 1.5 g/cm\(^3\). Further tests were performed in triaxial conditions by applying a confining pressure of 100 kPa on a specimen compacted at a dry density of \(\approx\)1.46 g/cm\(^3\) and a confining pressure of 300 kPa on a specimen compacted at a dry density of \(\approx\)1.47 g/cm\(^3\). In Tab. 6 the code and the characteristic of the tested specimens, the test condition, and the obtained experimental results are reported.

Tab. 6: Results of the permeability tests carried out at different boundary conditions.

<table>
<thead>
<tr>
<th>Specimen code</th>
<th>Test condition</th>
<th>Dry density (g/cm(^3))</th>
<th>Hydraulic conductivity (m/s)</th>
<th>Intrinsic permeability (m(^2))</th>
</tr>
</thead>
<tbody>
<tr>
<td>TX-C100</td>
<td>(P_c = 100) kPa</td>
<td>1.46±0.02</td>
<td>(6.04\times10^{-11})</td>
<td>(5.47\times10^{-18})</td>
</tr>
<tr>
<td>TX-C300</td>
<td>(P_c = 300) kPa</td>
<td>1.47±0.02</td>
<td>(2.86\times10^{-11})</td>
<td>(2.59\times10^{-18})</td>
</tr>
<tr>
<td>MC-dd1.5</td>
<td>Constant volume</td>
<td>1.51</td>
<td>(2.78\times10^{-11})</td>
<td>(2.52\times10^{-18})</td>
</tr>
<tr>
<td>PERM-dd1.5</td>
<td>Constant volume</td>
<td>1.50</td>
<td>(5.35\times10^{-11})</td>
<td>(3.88\times10^{-18})</td>
</tr>
</tbody>
</table>

The two values of hydraulic conductivity measured under different confining pressure are very close to each other. At this level of stress the reduction in the hydraulic conductivity due to the increase of confining pressure can be considered as negligible and this slight change is attributed to the accuracy in the determination of the dry density obtained during specimen preparation.

The comparison with the results from permeability tests carried out on the same type of mixture in a previous study is reported in Fig. 22.
2.3.5 Validity of Darcy’s law

In order to verify the validity of the Darcy’s law for the S/B mixture the water permeability tests were conducted on the specimen no. PERM-dd1.5 by applied different pressure gradients while measuring the corresponding flows. The permeameter cell was selected for this test. These results were also compared with the ones obtained for the specimens TX-C100, TX-C300 and MC-dd1.5.

The results of the tests are presented in Fig. 23. An exponential trend for the specific discharge with the hydraulic gradient was taken as best fit for the experimental data.
2.4 Determination of retention properties

2.4.1 Chilled mirror dewpoint psychrometer (WP4c)

The WP4c is a dewpoint psychrometer manufactured by Decagon Devices (US) which enables to measure the total suction of a specimen in the range of 3 - 300 MPa, in a relative short time (about 1 hour for each measurement) (Leong et al. 2003; Cardoso et al. 2007).

The device consists of a sealed chamber where the following components are located (Fig. 24):

- a mirror, whose temperature is precisely controlled by a thermoelectric (Peltier) cooler;
- a photoelectric cell, which detects the exact point at which the condensation first appears in the mirror;
- an infrared thermometer, which detects the specimen temperature;
- a fan, whose purpose is to reach equilibrium more quickly and to control the boundary layer conductance of the dew point sensor.

In the WP4c the soil specimen, in equilibrium with the surrounding air, is placed in the sealed chamber. Then humidity (mole fraction of vapour water) inside the chamber is defined by the specimen. The temperature of the mirror is reduced by means of a thermoelectric cooler. When the temperature in the mirror reaches the dew point, condensation occurs (which is observed by the photoelectric cell), at the same time, a thermocouple attached to the mirror records the dew-point temperature.

During the process, the air around the mirror is saturated gradually; while the mole fraction and the partial pressure of vapour water remain constant. Therefore, the saturated pressure of water vapour at the dew point temperature indicates the absolute humidity of the air.

The relative humidity, which is the ratio between the saturated vapour pressure of water at the dew point and the saturated vapour pressure of water at air temperature, is obtained from the difference between the dew point temperature and the temperature of the soil specimen. It is assumed that the two temperatures are measured simultaneously.
2.4.2 Microcage

The microcage (Fig. 25) is an innovative cell recently designed and manufactured at the Laboratory of Soil Mechanics of the EPFL. It has a cylindrical shape and it is made of brass. The cell is composed of two lids that can be tightened together. It is 50 mm in diameter and 11 mm high. The specimen inside the cell has a diameter of 40 mm and a height of 7.5 mm. The top and the bottom of the cell are perforated in order to allow the water/vapour circulation. The sealing of the cage is provided by an O-ring placed between the two lids. The cell was manufactured with the aim of analysing the retention properties of the S/B mixture at high suction values. To this purpose, the cell dimensions were fixed in order to allow the cell to be positioned directly inside the WP4c chamber. In this way suction measurements can be carry out without opening the cell, maintaining the constant volume condition, and preserving the swelling pressure inside the cell. Moreover, the cell can be used in the freeze-drying device; in this sense, the freeze drying can be performed without opening the cell with the aim to minimize specimen disturbances associated to the opening of the cell and the consequent swelling pressure release.

![Photograph of the microcage.](image)

2.4.3 Water retention curve using the WP4c

The WP4c and the microcage were used to obtain the retention behaviour of the S/B mixture at high suction values. The use of the microcage allows the determination of the retention curve on a single specimen with the possibility to better highlight hysteretical features of the retention behaviour.

**Specimen preparation and procedure**

In order to follow a main wetting path, the mixture was prepared following the procedure reported in section 0 with an initial water content of 4 %. The specimen was then compacted inside the microcage. The microcage was closed and sealed in a hermetic container for 1 day to allow moisture redistribution inside the specimen. After that the initial suction was measured with the WP4c.

For the wetting path the material was saturated in steps, by putting the microcage in an ambient with 100 % of relative humidity (zero total suction). Inside the saturating chamber the material
tends to equilibrate with the imposed suction and so to adsorb water from the humid ambient. After a certain time that varies from 12 to 24 hours for each step, the saturation process was stopped and the microcage was extracted from the chamber. Then, it was sealed with paraffin tape for 1 day in order to let the adsorbed moisture to distribute inside the specimen. After the equalization, the suction was measured with the WP4c (Fig. 26). The weight of the cell was continuously monitored in order to compute the evolution of the water content.

1) 2) 3) 4)

Fig. 26: Phases of the wetting path: 1) Saturation; 2) Sealing; 3) weight measurement; 4) Suction measurement.

For the drying path a similar procedure as for the wetting path was adopted. In this case the cage was dried at the laboratory ambient, in which the temperature is kept at 22°C (±1 °C) and the relative humidity is 41 % (corresponding total suction of 120 MPa). During the drying process the weight of the cell was monitored in order to compute the evolution of the water content. The process was stopped after certain time that varies from 1 to 24 hours, depending on the reached water content, by sealing the microcage with the paraffin tape. As the water evaporation occurred from the holes of the microcage, so it started from the external surface of the specimen, the cage was kept sealed in paraffin for at least one day in order to let the total suction to homogenise inside the specimen. The drying process was stopped once the suction reached the initial suction value obtained after compaction. The microcage was opened at the end of test and the contact of the specimen with the cell was assessed.

Fig. 27 presents the water retention curves of the mixture for two specimens compacted at 1.53 g/cm³ and 1.8 g/cm³, respectively. The results show that for a given degree of saturation, the measured total suction was higher for the specimen compacted at 1.8 g/cm³. This result is attributed to the reduction of the macroporosity due to the increase in the dry density and in the consequent increment of the capillary component of the total suction.

The experimental data were fitted with the equation proposed by van Genuchten (1980):

\[
S_r = \left( \frac{1}{1 + \left( \frac{\psi}{\alpha} \right)^n} \right)^m
\]

(13)

where \( S_r \) is the degree of saturation, \( \alpha \), \( n \) and \( m \) are material parameters. The parameters obtained by fitting the experimental data are presented in Tab. 7.

The lower plots in Fig. 27 show the data obtained by measuring the total suction with the WP4c together with the data obtained by imposing a matric suction with the axis translation technique.
(for dry densities of about 1.5 g/cm³). The van Genuchten parameters for that wetting branch are then recalculated for the entire range of suction (form 0.001 to 20 MPa).

Fig. 27: Top plots: Water retention curves of the S/B mixtures compacted at 1.53 cm³ and 1.80 g/cm³ for saturation (wetting) and desaturation (drying). Suction versus degree of saturation (left) and suction versus water content (right). Lower plots: total suction data (with the WP4c) and data obtained by applying the axis translation technique for a similar dry density of around 1.5 g/cm³.

Tab. 7: Van Genuchten parameters calculated for specimen compacted at 1.53 and 1.8 g/cm³ of dry density. Fitting parameters for total suction measurements only (ψ > 3 MPa); Fitting parameter for the entire range of suction measurements (0.003 < s < 0.5 MPa; 3 < ψ < 20 MPa).

<table>
<thead>
<tr>
<th>Van Genuchten parameters</th>
<th>Dry density² = 1.53 g/cm³</th>
<th>Dry density² = 1.8 g/cm³</th>
<th>Dry density² = 1.5 g/cm³</th>
</tr>
</thead>
<tbody>
<tr>
<td>α (MPa)</td>
<td>0.13</td>
<td>0.19</td>
<td>0.36</td>
</tr>
<tr>
<td>m</td>
<td>17.01</td>
<td>16.38</td>
<td>16.97</td>
</tr>
<tr>
<td>n</td>
<td>0.02</td>
<td>0.02</td>
<td>0.02</td>
</tr>
</tbody>
</table>
2.5 Gas permeability experiments

The gas permeability tests were conducted on partially saturated specimens using the permeameter cell. The specimens were prepared following the procedure described in section 0. At the 11% of water content the initial degree of saturation of as compacted specimens varies from 28% to 59%, for a dry density in the range of 1.3 g/cm³ - 1.8 g/cm³.

The gas permeability test was measured using the transient method which consists in monitoring the gas pressure decay of a pressurized gas reservoir, connected to one end of the specimen while the gas is flowing through the specimen. The other end of the specimen was maintained at atmospheric pressure. Before starting the gas test, the degree of saturation of the specimen was controlled by injecting water in the cell with a PV controller.

2.5.1 Methodology

The method used to measure the gas permeability was the transient method. For this method the inlet of the permeameter was connected to an airtight gas reservoir of known volume while the outlet was maintained at atmospheric pressure (the valve at the outlet remained open). The gas reservoir was initially filled with gas at a known pressure. The upstream line was instrumented with a pressure probe which recorded the pressure decay in the reservoir in time (Fig. 28). The initial degree of saturation of the as compacted specimen was increased in controlled way by injecting a known quantity of water into the specimen by means of a PV controller. This preliminary phase was necessary due to the high gas permeability of the as compacted specimens. In this condition the pressure of upstream gas reservoir decayed too fast and it could not be recorded with the data acquisition system (DAS). After the water injection saturation, the permeameter was closed for four days in order to reach a homogeneous distribution of the water over the whole specimen volume. The weight of the permeameter was measured before and after the gas permeability test in order to assess the possible change in the water content due to water displacement by gas injection. As the degree of saturation after water injection was not known a priori, at the end of the gas permeability test the water content of the specimen was measured by drying it in an oven at 105 °C for 24 h, in agreement with the SN 670 3406.

![Experimental setup for gas permeability test.](image-url)
Considering the isochoric condition and neglecting the compressibility of the grains the governing equations of gas transport mechanism for a rigid porous medium were considered.

Referring to Fig. 29 the excess of inflow over outflow during a short time interval, \(dt\), through the surface of the control volume, that is perpendicular to the \(x\) direction is expressed as:

\[
\dot{I}_x dx
dy
dz
dt - \left(\dot{I}_x dx
dy
dz
dt + \frac{\partial \dot{I}_x}{\partial x} dx
dy
dz
dt\right)
\] (14)

where \(\dot{I}_x\) is the mass flux of the fluid.

For the principle of mass conservation, the excess of flow is balanced by the variation of mass with time within the control volume:

\[
n_g \frac{\partial \rho_g}{\partial t} dx
dy
dz
dt = - \frac{\partial \dot{I}_x}{\partial x} dx
dy
dz
dt
\] (15)

where \(n_g = n(1-S_r)\) is the porosity occupied by the gas phase, and \(\rho_g\) is the gas density.

The mass flux is expressed by:

\[
\dot{I}_x = \rho_g \bar{q}_g
\] (16)

where \(\bar{q}_g\) is the Darcy’s velocity, which is equal to:

\[
\bar{q}_g = - \frac{\rho_g \cdot k_{g,x} \cdot \ddot{g}}{\mu_g} \frac{1}{\rho_g} \frac{\partial P}{\partial x}
\] (17)
Combining equations 15, 16 and 17 and considering an homogeneous and isotropic porous medium \((k = \text{const})\), we obtain:

\[
n_g \frac{\partial \rho_g}{\partial t} = \frac{k_{g,e}}{\mu_g} \frac{\partial}{\partial x} \left( \rho_g \frac{\partial P}{\partial x} \right) \tag{18}
\]

The density of ideal gas \((\rho_g)\) is a function of its pressure \((P)\) and its temperature \((T)\). This relationship is expressed by the gas state equation:

\[
\rho_g = \frac{m_g P}{RT} \tag{19}
\]

where \(R\) is the universal gas constant and \(m_g\) is the molar mass of the gas.

Introducing equation 19 into equation 18, the governing equation of gas transport in a rigid porous medium is obtained (Bear, 1972):

\[
n(1 - S_g) \frac{\partial P}{\partial t} = \frac{k_{g,\text{eff}}(S_g)}{2\mu_g} \nu^2 p^2 \tag{20}
\]

Where \(k_{g,\text{eff}}\) is the effective permeability with respect to the gas phase. The effective permeability is a function of the space available in the pores for one fluid to flow and it depends on the degree of saturation of the considered fluid and the porosity of the medium. It is expressed as:

\[
k_{g,\text{eff}} = k_i \cdot k_{rel} \tag{21}
\]

where \(k_i\) is the intrinsic permeability of the medium at 100 % of degree of saturation of the considered phase and \(k_{rel}\) is a number that depends on the degree of saturation of the considered phase. For a fully dry medium the relative gas permeability is equal to 1 while the relative water permeability is equal to zero.

The boundary conditions for the permeability were written considering that the pressure was equal to the atmospheric pressure at the downstream, while the upstream was connected to the airtight reservoir of know volume, that was initially pressurized at a known pressure. At the upper boundary the outflowing mass from the gas reservoir \((\frac{\partial M_{g,up}}{\partial t})\) is equal to the mass flux through the specimen \((\frac{\partial M_g}{\partial t})\).

The two mass fluxes can be expressed as:

\[
\frac{\partial M_g}{\partial t} = \frac{V_{up} \frac{\partial P_{up}}{\partial t}}{RT} \tag{22}
\]

\[
\frac{\partial M_{g,up}}{\partial t} = \rho_g \cdot \frac{\partial P_{up}}{\partial t} \tag{23}
\]

where \(V_{up}\) and \(P_{up}\) are the volume and the pressure of the airtight reservoir, respectively, \(M_{g,up}\) is the mass of the gas in the reservoir and \(A\) is the specimen surface.

Combining equations 22 and 23 the upper boundary condition is written as:

\[
\frac{\partial P_{up}}{\partial t} = \frac{k_{eff} A}{\mu_g V_{up} P_{up}} \frac{\partial P_{up}}{\partial x} \tag{24}
\]
Finally, the following system must be solved in order to simulate the gas injection process:

\[
\begin{align*}
\frac{n(1 - S_r)}{\partial t} & = k_{g,\text{eff}}(S_r) \frac{\partial^2 p}{\partial x^2} \\
\frac{\partial P_{\text{up}}}{\partial t} & = \frac{k_{\text{eff}} A}{\mu_g} \frac{P_{\text{up}}}{\partial x} & \text{for } & \forall \ t; x = 0 \\
P_{\text{dw}} & = 0 & \text{for } & \forall \ t, x = L \\
P & = P_{\text{in}} = P_{\text{dw}} & \text{for } & t = 0; \forall \ x
\end{align*}
\]

This system was solved numerically using a finite difference scheme. The numerical solution was then fitted with the experimental data in order to find the value of the effective permeability (see an example in Fig. 30).

![Fig. 30: Results of the gas permeability test (\(\rho_d = 1.54; S_r = 0.69\)).](image)

The gas permeability of the S/B mixture was determined for a dry density of about 1.5 g/cm³ at two different degrees of saturation. The results of the test are reported in Tab. 8.

Tab. 8: Results of the gas permeability test.

<table>
<thead>
<tr>
<th>Specimen n°</th>
<th>Dry density, (\rho_d) (g/cm³)</th>
<th>Water content, (w) (-)</th>
<th>Degree of saturation, (S_r) (-)</th>
<th>Effective permeability, (k_{g,\text{eff}}) (-)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>(\sim 1.54)</td>
<td>0.20</td>
<td>0.69</td>
<td>(1.23 \cdot 10^{12})</td>
</tr>
<tr>
<td>2</td>
<td>(\sim 1.50)</td>
<td>0.25</td>
<td>0.85</td>
<td>(8.43 \cdot 10^{15})</td>
</tr>
</tbody>
</table>
2.5.2 Gas penetration tests

In the present experimental program, gas penetration tests were carried out applying different boundary conditions and using different saturating fluids in order to evaluate the influence of the boundary condition and pore water chemistry on the gas transport capacity of the fully saturated S/B mixture.

In total, five gas penetration tests were performed on specimens compacted at a target dry density of 1.5 g/cm$^3$. The first preliminary test was done using the MC cell which allows monitoring the pressure at the middle height of the column by means of a pressure transducer. Then the test was conducted in triaxial condition, at two different confining pressures, in order to verify the influence this latter parameter on the threshold pressure. Several cycles of gas injection were applied in triaxial conditions to verify the repeatability of the breakthrough pressure determination. The last test was done on a specimen saturated in constant volume condition with synthetic water using the permeameter cell.

2.5.3 Test procedure

In all the performed tests, after saturation, the pore water back pressure was set equal to a constant value at both ends of the specimen. Once the water exchange between the specimen and PV controller became negligible, the water pressure was considered as homogenous inside the specimen. After the pressure equalisation phase, one side of the cell was connected to the gas injection line while the other side remained connected to the PV controller. Before starting the gas injection, the inlet duct was flushed with gas at a pressure lower than 10 kPa in order to evacuate the water from the porous stones. The upstream gas pressure, initially set equal to the water pressure, was increased step by step while at downstream the water pressure was maintained constant. At each step of the test the gas pressure was controlled by means of a pressure regulator and the mixed outflow was monitored by means of the PV controller. The test was considered as finished once a steep increase of the outflow was observed; in this case, an increase of pressure in the PV connected to the outlet of the devices was systematically observed. This increase is due to the inability of the PV to compensate the sudden increase in the outflow.

2.5.4 Results

For the sake of clarity, all the results of the gas penetration tests are presented starting from the moment in which the gas injection started and they are divided in phases. Each phase correspond to an increment of the upstream gas pressure.

MC cell:

The results of the gas injection test in the MC cell are shown in Fig. 31. In this test, after the cell saturation, the water pressure was initially set to 50 kPa at the bottom plate and it was maintained constant by means of a PV controller. The gas (dry air) was injected from the top plate of the column starting from 40 kPa; then the gas pressure was increased from 40 to 50 kPa and then from 50 to 100 kPa. Different phases of this gas injection test are summarized in Tab. 9. In this table it also reported the normalized flux ($q_n$), defined as volumetric flux divided by the specimen area, measured for each gas injection phase.

During phase 1 a constant normalized flux of 8.32E-10 m/s, from the PV controller toward the specimen, was observed. The pressure registered by the pressure transducer at the middle height of the specimen decreased until a constant value of 40.7 kPa. The constant exiting water flux from the controller indicates that a steady state condition was reached while the specimen was fully saturated. The water pressure difference of 9.3 kPa (= 50 – 40.7 kPa) between the bottom
and the centre of the specimen allowed to calculate a water permeability of 2.66E-12 m/s. This value is in good agreement with all previous data on the S/B mixture with an initial dry density of 1.5 g/cm$^3$ (see above). Considering a linear distribution of the water pressure along the specimen height, the water pressure at the top of the specimen was estimated at 31.4 kPa. Consequently, knowing the value of the gas and water pressure at the top boundary a matric suction (Pa-Pw) of 8.6 kPa was calculated. This matric suction was lower than the air entry value of the mixture (about 9 kPa) that can be estimated from the MIP results.

During phase 2 (Pa = 50 kPa), the water continued to flow toward the specimen and the measured pressure by the transducer (at the middle height) increased up to 45.9 kPa. By applying the same considerations as for phase 1, a permeability of 2.89E-11 m/s was obtained. The matric suction at the top of the specimen was 8.2 kPa, so lower than the estimated air entry pressure.

For the third phase, a gas pressure of 100 kPa was applied. In contrary to the two previous phases, a flow toward the water PV controller was observed. This flow could be explained considering that the applied suction on the top of the specimen was higher than the mixture air entry suction and consequently the specimen underwent to a drying process (the air started to invade the specimen pores). The pressure monitored by the pressure transducer increased until reaching a constant value of $\approx$73 kPa. The pressure stabilization occurred after about 80 h of gas injection at 100 kPa which indicates that, at this time, the desaturation front reached the middle height of the column. A sudden increase of the normalized outflow, from 1.91E-9 m/s for phase 3.a to 4.30E-7 m/s for phase 3.b, occurred at t = 620h, this, after about 160 h of gas injection at 100 kPa (phase 3.b). This delayed response indicates that the desaturation front reached the bottom of the specimen after about 600 hours and a continuous gas pathway was created inside the column.

Fig. 31: Gas injection test in the MC cell.
Tab. 9: Data from gas penetration test in Modular cell.

<table>
<thead>
<tr>
<th>Phase no</th>
<th>$P_{g, top}$ (kPa)</th>
<th>$P_{w, bot}$ (kPa)</th>
<th>$P_{probe}$ (kPa)</th>
<th>$q_m$ (m/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>40</td>
<td>50</td>
<td>50 to 40.7</td>
<td>$8.32 \cdot 10^{-10}$</td>
</tr>
<tr>
<td>2</td>
<td>50</td>
<td>50</td>
<td>40.7 to 45.7</td>
<td>$4.16 \cdot 10^{-10}$</td>
</tr>
<tr>
<td>3.a</td>
<td>100</td>
<td>50</td>
<td>45.7 to 73.3</td>
<td>$1.91 \cdot 10^{-9}$</td>
</tr>
<tr>
<td>3.b</td>
<td>100</td>
<td>50 to 54</td>
<td>73.3</td>
<td>$4.30 \cdot 10^{-7}$</td>
</tr>
</tbody>
</table>

Triaxial cell:

For the gas injection tests in triaxial condition, the gas was injected from the bottom of the specimen while the water pressure at the top was maintained constant with a PV controller. As it was mentioned in section 2.3.2, once the specimen was placed in the triaxial cell the confining pressure was increased up to the target value. Then vacuum was applied at the top and the bottom of the specimen to evacuate the air present in the specimen porosity and in the drainage system. Finally, the specimen was saturated by injecting water from top and bottom. After the specimen was saturated the gas injection was started.

Three tests were performed. In the first test a confining pressure of 100 kPa was applied and the gas was injected starting from a pressure of 20 kPa. The second test was carried out on a specimen subjected to 300 kPa of confining pressure and the gas pressure was initially set equal to 34 kPa. The last test consisted of a second cycle of gas injection carried out on the same specimen used for the second test after re-saturation; in order verify the reproducibility of the breakthrough. The results of these tests are shown in Figs. 35 to 37 and listed in Tabs. 10 to 12.

Comparing phase 2 of the test at 100 kPa of confinement (test TRX1) and phase 1 of the test carried out at 300 kPa of confinement (test TRX2), where similar pressure difference were applied (10 kPa and 14 kPa, respectively), a small reduction of the mixed normalized outflow is observed (from $7.36 \cdot 10^{-9}$ to $3.25 \cdot 10^{-9}$ m/s) due to the increase of the confining pressure.

In the case of the first gas test in triaxial condition ($P_{\text{conf}} = 100$ kPa, TRX1) a steep increase in the outflow was observed when the gas pressure reached the value of 40 kPa, at a pressure difference of 20 kPa between water and gas. After the increase in the outflow was registered, the pressure in the PV started to increase. In the case of the second gas test in triaxial condition ($P_{\text{conf}} = 300$ kPa, TRX2) a higher gas pressure (around 55 kPa) was needed to get the sudden increase of the outflow. This result highlights the effect of confining pressure on gas breakthrough pressure. The third test (TRX3) showed essentially the same behaviour registered for the test TRX2, highlighting a sort of reversible behaviour of the breakthrough process after resaturation.
Fig. 32: Test TRX1: gas injection test in the triaxial cell at 100 kPa of confining pressure.

Tab. 10: Data from gas penetration test in triaxial condition at 100 kPa of confinement

<table>
<thead>
<tr>
<th>Phase n°</th>
<th>$P_{g,bot}$ (kPa)</th>
<th>$P_{w,top}$ (kPa)</th>
<th>$q_w$ (m/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>$\approx 20$</td>
<td>$\approx 20$</td>
<td>$1.84 \times 10^{-9}$</td>
</tr>
<tr>
<td>2</td>
<td>30</td>
<td>20</td>
<td>$7.36 \times 10^{-9}$</td>
</tr>
<tr>
<td>3</td>
<td>40</td>
<td>20 to 30</td>
<td>$5.24 \times 10^{-6}$</td>
</tr>
</tbody>
</table>
Fig. 33: Test TRX2: gas injection test in the triaxial cell at 300 kPa of confining pressure – Cycle 1.

Tab. 11: Data from gas penetration test in triaxial cell at 300 kPa of confinement – cycle 1.

<table>
<thead>
<tr>
<th>Phase n°</th>
<th>( P_{g,bot} ) (kPa)</th>
<th>( P_{w,top} ) (kPa)</th>
<th>( q_n ) (m/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>34</td>
<td>20</td>
<td>( 3.25 \times 10^{-9} )</td>
</tr>
<tr>
<td>2</td>
<td>45</td>
<td>20</td>
<td>-</td>
</tr>
<tr>
<td>3</td>
<td>55</td>
<td>20 to 40</td>
<td>( \approx 4.99 \times 10^{-6} )</td>
</tr>
</tbody>
</table>
Fig. 34: Test TRX3: gas injection test in the triaxial cell at 300 kPa of confining pressure – Cycle 2.

Tab. 12: Data from gas penetration test in triaxial cell at 300 kPa of confinement – Cycle 2.

<table>
<thead>
<tr>
<th>Phase n°</th>
<th>$P_{g,bot}$ (kPa)</th>
<th>$P_{w,top}$ (kPa)</th>
<th>$q_n$ (m/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>20</td>
<td>20</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>32</td>
<td>20</td>
<td>$7.07 \times 10^9$</td>
</tr>
<tr>
<td>3</td>
<td>40</td>
<td>20</td>
<td>$3.54 \times 10^{-6}$</td>
</tr>
<tr>
<td>4</td>
<td>51</td>
<td>20 to 40</td>
<td>$5.41 \times 10^{-6}$</td>
</tr>
</tbody>
</table>

Permeameter:

The gas injection test in the permeameter was performed on a specimen saturated with synthetic water (Tab. 13). To saturate the specimen, the water was injected at a pressure of 6 kPa from both sides of the permeameter. The drainages were periodically opened in order to remove the air that was present in the specimen porosity which accumulated continuously in the porous disks and injection ducts. After flushing the inlet of the permeameter with gas (dry air) injected at low pressure, the gas pressure was set at 6 kPa. As shown in Fig. 35, the gas injection consists in three phases. In the first phase, gas and water pressure are the same, a mixed normalized out flux of $1.14 \times 10^{-8}$ m/s was measured. This flux is attributed to the diffusion process through the fully saturated specimen. In the second phase the gas pressure was slightly increased up to 7.7 kPa. At the beginning of this phase a higher flux was observed at the outlet. This flux became stationary at the mid-time of the phase, suggesting that the specimen reached a stationary degree of saturation. In phase 3, the gas pressure was increased up to 10.2 kPa and the breakthrough
was reached immediately. At this point a normalized flux equal to 1.04E-5 m/s was measured. This flux is one order of magnitude higher than the flux observed for the tests in triaxial conditions and two order of magnitude higher than the flux measured after breakthrough in the MC cell (in isochoric condition), even if the applied gradient is much lower. The gas breakthrough pressure was also strongly reduced for the specimen saturated with synthetic water. This difference could be attributed to the use of distilled water to saturate the specimens in triaxial and MC cells. As shown above, the use of synthetic water inhibits the swelling capacity of bentonite and affects the S/B mixture porosity.

![Figure 35: Gas injection test in the permeameter after saturation with synthetic water.](image)

**Fig. 35:** Gas injection test in the permeameter after saturation with synthetic water.

**Tab. 13:** Data from gas penetration test in the permeameter saturated with synthetic water.

<table>
<thead>
<tr>
<th>Phase n°</th>
<th>$P_{\text{g,bot}}$ (kPa)</th>
<th>$P_{\text{w,top}}$ (kPa)</th>
<th>$q_{\text{in}}$ (m/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>6</td>
<td>5-6</td>
<td>$1.14 \times 10^{-8}$</td>
</tr>
<tr>
<td>2</td>
<td>7.7</td>
<td>5-6</td>
<td>-</td>
</tr>
<tr>
<td>3</td>
<td>10.2</td>
<td>6 to 10</td>
<td>$1.04 \times 10^{-5}$</td>
</tr>
</tbody>
</table>

### 2.6 Positron Emission Tomography (PET) experiment

The main aim of the PET experiment performed at HZDR (Leipzig, Germany) was to obtain 4D (3D space and time) images of water saturation and gas invasion into an 80/20 S/B sample.
2.6.1 Experiment Setup

The experiment column for the PET experiment was specifically constructed to: a) withstand 20 bar hydraulic pressure; and b) provide as little as possible shielding for CT (Computer tomography) and PET measurements.

The central part (sample area) of the S/B-Column consists of a special Epoxy-Resin with mechanical characteristics comparable to Plexiglas. The two end plates are made of Plexiglas and are belted together with screws made of carbon fibre armed synthetics (CFK) (Fig. 36).

To prevent material transport out of the sample chamber at each end of the columns, filter plates are installed. The pore size of the glass-ceramic filters is 1 µm. The plates are equipped with O-rings to seal the connection of the filter plates to the central part of the S/B-Column. Additionally, the correct torque must be applied to the CFK-screws to ensure the proper connection. The geometrical dimensions of the column are given in Tab. 14.

Tab. 14: Dimensions of PET column

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Outer length</td>
<td>160mm</td>
</tr>
<tr>
<td>Outer diameter</td>
<td>115mm</td>
</tr>
<tr>
<td>Thickness of the side walls</td>
<td>30mm</td>
</tr>
<tr>
<td>Length of the sample space</td>
<td>110mm</td>
</tr>
<tr>
<td>Diameter of the inner sample space</td>
<td>55mm</td>
</tr>
</tbody>
</table>

Fig. 36: Technical drawing of PET column.

The column was tested with water doped with helium at a pressure of about 22 bar. During the test, the column pressure and the room temperature were correlating. The tightness test confirms
that the system is tight up to a water pressure of 20 bar. The sniffle probe with a helium detector gave no indication for a leakage.

In a second step, the system was pressurized with helium to nearly 6 bar and the tightness was checked with a helium detector. No helium leakage was recognizable during the test. Furthermore, the displacement of the whole system was measured during the pressurization of the system. No remarkable material expansion was recorded (Fig. 37).

The columns was filled at EPFL with 80/20 S/B mixture applying the same materials and the same emplacement procedure are explained in Chapter xx. Due to fact that the column was constructed for a maximal pressure of 20 bars the uniaxial compaction pressure was 20 bars leading to an emplacement dry density of about 1.45 g/cm³.

![PET column after emplacement of S/B.](image)

The column was mounted onto a flow board to perform controlled water and gas injection and monitoring (Fig. 38). For column saturation a water flow of 0.002 ml/min was applied for 20 d. Then a conservative PET tracer solution with $[^{58}\text{Co}](\text{Co(CN)}_6)^3-$ (70.86 days half-life) was injected with 0.001 ml/min for 42 days.
An artificial water solution with a composition as shown in Tab. 15 was used for injection to simulate similar conditions as for Grimsel Test Site.

Tab. 15: Chemical composition of artificial injection water.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ionic strength [mmol/L]</td>
<td>0.63</td>
</tr>
<tr>
<td>NaF [mmol/L]</td>
<td>0.30</td>
</tr>
<tr>
<td>NaCl [mmol/L]</td>
<td>0.15</td>
</tr>
<tr>
<td>NaSO₄ [mmol]</td>
<td>0.06</td>
</tr>
<tr>
<td>El. Cond. [μS/cm]</td>
<td>197</td>
</tr>
<tr>
<td>pH</td>
<td>9.60</td>
</tr>
<tr>
<td>Particle concentration [10⁹ pt/L]</td>
<td>6 - 40</td>
</tr>
<tr>
<td>Particle diameter [nm]</td>
<td>40 - 1000</td>
</tr>
</tbody>
</table>

**PET-CT measurements**

CT analysis was applied to assess the homogeneity of the S/B material after emplacement and after saturation. A PET-CT “Biograph 16” was used for the measurements with 412 x 512 pixel axial resolution leading to a resolution of about 0.9766 x 0.9766 mm. Measurements were
performed after compaction and 10 days after starting saturation. Fig. 42 shows a raw picture along the column after these 10 days.

PET measurements were performed regularly after injection of the PET tracer.

PET tracer

As mentioned above the cobalt complex \([^{58}\text{Co}](\text{Co(CN)}_{6})_{3}^{-}\) (70.86 days half-life) was used as tracer to this experiment. The injection water was doped with 50 MBq in 100ml of injection water. Note that the complex is negatively charged. It is, therefore, expected that the tracer will not enter the clay interlayers but only move within the pore space accessible to advective water flow.

2.6.2 Results

CT measurements

The S/B column was measured by computer tomography (CT) before and after saturation. The initially emplaced density was about 1.64 g/cm\(^3\) (dry density 1.45 g/cm\(^3\)). Fig. 39 shows the estimated distributions of gas and water in the column after 10 days of water injection.

Water and gas saturations were obtained by displaying the data in Hounsfield scale (CT number) (H=-1000 for air and H=0 for water). Based on this value the porosity \(\Phi\) can be calculated according to

\[
\Phi = 1 - \frac{\mu_{\text{por}} \cdot H}{1000 \cdot (\mu_{\text{max}} - \mu_{\text{por}})} = 1 - h_{\text{v,por}} \cdot H
\]  

(26)
Where, for 50 keV, the coefficient $h_c$ is equal to 0.000246 for air and 0.000367 for water and $\mu$ is the mass reduction coefficient for matrix (mtx) and voids (por), respectively. The water and gas saturation is calculated according to

$$S_w = \frac{V_w}{V_{tot}} = \frac{1}{\Phi} \frac{V_{w}}{V_{tot}} = \frac{\Phi_w}{\Phi}$$

(27)

Bluish areas in Fig. 40 show highly saturated parts of the S/B column, whereas reddish areas indicate high amounts of air remaining. The blue areas at the outer interface appear to be highly saturated but this may be an artefact from image interpretation.

![Image: Interpretation of water and gas saturations in S/B column after 10 days of water injection. Water inflow is from the right side.](image)

Fig. 40: Interpretation of water and gas saturations in S/B column after 10 days of water injection. Water inflow is from the right side.

The average densities along the S/B column obtained by averaging density values at each cross section is shown in Fig. 41. The figure shows that density variations are of the order of 10 % on the observed scale of about 10 cm. Furthermore, a clear increase in density is visible from about 1.65 to about 1.75 g/cm$^3$. This fits well with the about 29 ml of water injected over the 10 days of water injection. Additionally, there seems to be a consistent density increase between about 40 to 110 mm along the column.

First water outflow was observed after 15 days (or 22 ml). Together with the initial water volume in column of about 50 ml only about half of the pore space of about 125 ml was saturated at that time.
Fig. 41: Axial density profile along the S/B column after compaction (black dots) and after 10 days of saturation (red dots). Saturation was performed from the right side. The cross sections with Pb markers attached can be seen as low density points (due to shielding).

PET measurements

Column saturation

Water volumes and concentrations were observed throughout the experiment. Fig. 42 shows the cumulative volumes of injected water during the experiment phases and the observed water activities at the outflow. Note that the activity of the injection water is 0.5 MBq/ml. First arrival of the tracer was after injecting 27 ml of tracers (or 24 days injection) and a peak was observed after 60 ml of traced water injected (or 51 days injection). Adding the 30 ml of clean water previously injected into the column the total water injected was 90 ml until peak arrival – and including the initial water content of column it leads to about 140 ml of water, compared to an approximate pore volume of 125 ml.

The estimated hydraulic permeability after saturation was 0.4 μDarcy or 4E-19 m². The peak activity of about 0.11 MBq/ml indicates that tracer may be slightly sorbing on the clay minerals of the S/B column. This would as well explain the higher activities seen towards the injection side of the column (see Fig. 46)
Fig. 42: Cumulated water injection volumes and activities (decay corrected) of outflowing water during time.
Fig. 43: PET images at different times after starting tracer injection.
Gas test with helium

After saturation was completed He gas was injected at 20 bar pressure. Fig. 44 shows that gas breakthrough occurred after ca 10 hours, where a first bubble appeared already after about 7 hours. The total gas volume injected was 500 ml at 20 bars (or 10 nL). Note that only small amounts of water outflow (≈ 2 ml) were observed due to gas injection.

Fig. 44: A) Gas inflow and B) pressures at both sides of the columns. C) outflow and D) pressures during first gas injection phase and breakthrough. He flow in cm³/min is 1.45 times the rate given in the upper figure.
Gas permeability tests were performed several times after gas breakthrough by applying varying gas flow rates (Fig. 45). The tests show consistently increasing gas permeabilities from initially about 5 μDarcy to about 30 μDarcy (or 5E-18 to 3E-17 m²).

The column was investigated by PET again after the gas tests have been completed. The activity differences before and after gas testing are displayed in Fig. 46, where bluish colours indicate reductions and reddish colours indicate increase of activity. It appears that activities tend to decrease towards the gas inlet, whereas in most of the column differences are balanced. Looking at the radially averaged image shown in Fig. 47 the discrepancy seems much less evident but there seems to be a shift of concentrations towards the outflow side. However, it needs to be kept in mind that some variations are due to repositioning the sample from one measurement session to the next.

Fig. 45: pressure evolution at the gas inflow (lowest plot), applied gas flow rates (middle plot) and calculated gas permeabilities for multiple gas tests using He gas.
Fig. 46: Difference of activities before and after gas injection. Bluish colours indicate reduction, red colours indicate increases.

Fig. 47: Radial average activities in S/B column displayed against distance (in mm) from gas outlet.
2.6.3 Conclusions

A new column for water and gas testing at 20 bar pressure under PET-CT scanner was successfully developed, constructed and emplaced. The main challenge of producing a column without steel parts in the main observation section while maintaining constant volume under pressure loading could be reached and numerous tests were run to assess the capability of CT and PET measurements.

The CT measurements have revealed valuable insights into the spatial distribution of water during saturation. However, the limited CT resolution of about 1mm seems not sufficient to distinguish between soil, water and gas phases on the pore scale, with the consequence that densities measured always show an average of the three media in place.

Water outflow was observed already after 15 days (or 22 ml water injected), when saturation was only about 50% (compared to 40% after compaction). This indicates that the main water flow occurs either through a very limited part of the porosity and/or that preferential water flow occurs along the column wall. The latter mechanism was tried to be hindered by covering the column wall with resin before S/B emplacement but, as shown in Fig. 48, there may be some preferential flow paths along or near the column interface. However, the Figure shows as well that there are many localised high concentration areas near the column outflow.

PET tracer measurements during the water injection phase have successfully been applied to provide a 4D picture of water movement inside the S/B column. The accumulation of activity toward the water inlet indicates some degree of sorption, making a direct interpretation of the results rather difficult.

Injection of helium at 20 bar into the column saturated with radioactive tracer has shown that breakthrough appears after about 7 hours and that only a very small amounts of radioactive water (about 2 ml) is expelled due to gas invasion and breakthrough.

![PET image with axial and radial cross sections of activity measurements (after 8 days of tracer injection).](image-url)
2.7 TDR column experiment

2.7.1 Background
The TDR column was originally set up to calibrate the TDR probes emplaced inside the GMT experiment.

The design, construction and initial saturation of the TDR test are described in GMT/M 02-06 (AN/02-193). Updates on the TDR saturation are included in the GMT annual reports from FY2002 to FY2006. An updated summary of results was amalgamated in 2007 together with some modelling results obtained during the GMT experiment (Lanyon, 2007).

2.7.2 Column setup
The column test layout was designed to:

- Obtain data to calibrate the TDR signals and control the test.
- Inject water into the bottom of the cylinder in as a homogeneous manner as possible.
- Use densities and initial water contents of the bentonite/sand mixture similar to those in the GMT EBS.
- The cylinder should have a water tightness of at least 20 bar (maximum design pressure).
- Emplacement of the sensors in the bentonite/sand similar to that of the EBS instrumentation (good contact between the sensors and the bentonite/sand).
- Avoid preferential flow paths along the boundaries as much as possible.

The experimental set-up (Fig. 49) consists of a steel column with a length of about 0.90 m and an inner diameter of 0.20 m. The column is instrumented with 3 tensiometers (T1 - T3) and three piezometers (PE1 - PE3), which are located 0.15, 0.45 and 0.75 m above the bottom of the column. A single rod TDR was placed along the central axis of the column. Finally, a total pressure cell (vibrating wire type) was fixed at the inner side of the top flange. Water injection is conducted through a thin gravel layer at the bottom of the column. The top outlet of the column is at atmospheric pressure. The column was designed for injection pressures of up to 2 MPa.

The cylinder was filled upside-down to ensure a good contact between the total pressure cell and the bentonite/sand. The pre-mixed bentonite/sand was hand-compacted with a variety of different diameter plastic rods and the emplaced density calculated from the volume of the column sections and the mass of bentonite/sand emplaced. The calculated bulk density was approximately 2.1 g/cm$^3$ at a gravimetric water content of 0.11. Great care was taken in the compaction around the instruments (especially the TDR and ceramic tensiometer caps).

The bentonite/sand was emplaced with an initial gravimetric water content of 11% and bulk density of 2.1 g/cm$^3$ equivalent to a dry density of 1.87 g/cm$^3$, porosity of 0.30 and saturation of about 0.7. In all, some 26.76 liters of the mixture was emplaced. The volume of water required to fully saturate the bentonite/sand is expected to be between 2.4 and 2.5 liters.
Fig. 49: Sketch of TDR column setup and b) photo during construction of the TDR column showing the filling and instrumentation of the mid cylinder section. In photo b) the following instrumentation can be seen: On the left side the ceramic cap of the TE/Lab/2 and on the right side the stainless steel filter of PE/Lab/2. In the middle part of the picture the lower parts of the TDR probe.

The design and manufacturing of the TDR cylinder was completed during the first half of February 2002. On February 19, 2002 the cylinder was filled with the bentonite/sand mixture and the sensors were installed. On February 28, 2002 the cylinder was transported from the Solexperts laboratories to the GMT site at the Grimsel Test Site (GTS) and the sensors were connected to the GMT Data Acquisition System.

2.7.3 Results

Saturation

Initial saturation was controlled by low flow constant-rate injections for the first few days of the test. After the initial saturation period, saturation has been controlled by regulating the injection pressure at the base of the column. The pressure has been raised in a series of steps from 600 kPa (11/03/02) to the current pressure of 1850 kPa (the column was pressure tested to 2 MPa during construction).

The upper outlet port has been open until 2 February 2009 (Tab. 16). Water flow from the top outlet was first detected in March 2006. From 16 June 2006 outflow has been measured (with some interruptions) using a water column flow meter.
Tab. 16: Injection steps and other major events during saturation

<table>
<thead>
<tr>
<th>Date</th>
<th>Column boundary condition</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>01/03/2002</td>
<td>Sensor installation</td>
<td></td>
</tr>
<tr>
<td>06/03/2002</td>
<td>0.18 ml/min fixed rate</td>
<td></td>
</tr>
<tr>
<td>07/03/2002</td>
<td>0.15 ml/min fixed rate</td>
<td></td>
</tr>
<tr>
<td>11/03/2002</td>
<td>Injection pressure set to 600 kPa</td>
<td></td>
</tr>
<tr>
<td>03/09/2002</td>
<td>Injection pressure set to 1200 kPa</td>
<td></td>
</tr>
<tr>
<td>18/06/2003</td>
<td>Injection pressure set to 1800 kPa</td>
<td></td>
</tr>
<tr>
<td>16/09/2003</td>
<td>Injection shut-in</td>
<td>WCFM used in GMT field test</td>
</tr>
<tr>
<td>07/10/2003</td>
<td>Injection pressure set to 1800 kPa</td>
<td></td>
</tr>
<tr>
<td>13/05/2004</td>
<td>Injection pressure set to 1850 kPa</td>
<td></td>
</tr>
<tr>
<td>15/03/2006</td>
<td>Water outflow first observed</td>
<td></td>
</tr>
<tr>
<td>16/06/2006</td>
<td>Start of measurement of outflow</td>
<td></td>
</tr>
<tr>
<td>27/11/2007</td>
<td>Start tracer injection (uranine + rhodamine)</td>
<td>Tracer with 0.010 g/l Uranine and 0.029 g/l Rhodamine</td>
</tr>
<tr>
<td>29/02/2008</td>
<td>Start injection with He back pressure</td>
<td>He back pressure measured with pressure transducer since 10/06/08</td>
</tr>
<tr>
<td>11/07/2008</td>
<td>He back pressure set to about 2000 kPa</td>
<td>Maximum He back pressure between 1900 and 2100 kPa</td>
</tr>
<tr>
<td>02/02/2009</td>
<td>Move the column to the VE cavern</td>
<td>Stop out flow measurements</td>
</tr>
<tr>
<td>04/02/2009</td>
<td>Restart injection at 20 bar</td>
<td>Continuous decrease of He back pressure. Leakage due to loss of quality of the injection system used already since 2002</td>
</tr>
<tr>
<td>03/11/2009</td>
<td>Replace the old water column flow meter (WCFM) with a new one</td>
<td>Improvement of back pressure stabilisation but still slight decrease</td>
</tr>
<tr>
<td>02/09/2010</td>
<td>Improve tightness of the column after observation of leak at TDR sensor cable</td>
<td>Additional tightness installed at TDR cable</td>
</tr>
<tr>
<td>06/05/2011</td>
<td>Installation extraction side setup</td>
<td></td>
</tr>
<tr>
<td>22/06/2011</td>
<td>Set extraction pressure to 15 bar. Injection pressure at 20 bar</td>
<td>Continuous decrease of extraction pressure. Several tightness tentative</td>
</tr>
<tr>
<td>27/01/2012</td>
<td>Modification of the extraction setup by removing the hydraulic hose. Calibration of the extraction setup volume</td>
<td>Stabilisation of the extraction pressure</td>
</tr>
<tr>
<td>07/03/2012</td>
<td>Start gas injection</td>
<td>Injection pressure 20 bar with N₂ instead of He. Extraction pressure 15 bar kept stable using a control switch valve</td>
</tr>
</tbody>
</table>
**Pressure evolution**

Fig. 50 shows the pressure (as measured by the three piezometers) and the total pressure (at top of column) from the start of saturation. The response to the different pressure steps can be clearly seen with PE/Lab/1 responding most quickly. Very sharp responses are seen to the 1800 kPa pressure step in both PE/Lab/1 and PE/Lab/2.

Once pressure at PE/Lab/3 stabilises during 2006 and 2007, the pressure drop between PE/Lab/1 and PE/Lab/2 is significantly larger (~900 kPa) than that between PE/Lab/2 and PE/Lab/3 (~500 kPa) as shown in Fig. 50. This may indicate either some effect of partial saturation in the column or heterogeneity (e.g. channelled interfacial flow in the lower column).

The total stress (as measured by the total pressure cell at the top of the column) reacts almost instantaneously to changes in injection pressure. After this instantaneous reaction the measured stress drops slowly before returning to a slow increase. The measured stress in July 2007 was a little over 400 kPa which is considerably less than the injection pressure at the base of the column. The response of the TP cell is currently not fully understood but a few points shall be discussed in more detail.

A more detailed discussion of pressure response can be found in Lanyon (2007).

[Fig. 50: Overview of pressure and total pressure during saturation.]

The pressure evolution since starting gas injection on 7 March 2012 is displayed in Fig. 51. The test will be performed by injecting gas at 20 bar pressure and keep the extraction side at a constant pressure of 15 bar, thus applying a delta pressure of 5 bars. It can be seen that the pressure in the injection tank continuously decreases and the estimated flow is equal to the former water injection flow. This is because the water in the injection chamber has to be
replaced first before a continuous gas phase is present below the S/B seal. The zigzag shaped pressure evolution at the outflow side shows that the water/gas separation system worked quite well after some difficulties at the beginning of gas injection test. The upper figure shows the water inflow and outflow from the column.

Evolution of water saturation and suction

The volumetric water content as calculated from the revised linear relationship with travel time is shown in Fig. 52. The general form is a roughly linear increase until mid 2004 (after the increase in injection pressure to 1850 kPa) followed by a stabilisation at a water content of about 0.245. Variations around this value may relate to the environmental conditions at the site (e.g. humidity within TDR cabinet) rather than the conditions within the column. Overall, the nearly 2 years of constant water content indicate that the TDR measurements are very stable over a long period.

Inspection of the TDR reflectograms suggested a uniform increase in saturation along the probe as opposed to a sharp saturation front previously assumed.
The fact that the maximum volumetric water content reached during the experiment is not 0.3 (initial total porosity) could be because:

- not only the shift but as well the slope of the calibration is wrong (see above)
- the initial porosity decreased due to increasing pressure from atmosphere to 1.8 MPa

Verification of the actual water content in the column near the TDR probe and thus adaptation of the calibration line cannot be done without dismantling the column, though sampling for porosity analysis will be difficult due to pressure release.

Fig. 53 shows the data from the piezometer and tensiometer pairs. The instrument pairs are located at the same height but on opposite sides of the column at 0.145, 0.465 and 0.765 m above the base. Tensiometer 2 required regular removal of air and refilling as part of the maintenance (it is not clear why this tensiometer dried out more quickly than the others).

PE/Lab/1 shows an early pressure rise (~30 kPa) (see Fig 50) prior to opening of PE/Lab/1 to check saturation. At this point the tensiometer on the opposite side of the column had reached atmospheric pressure. The higher pressure in PE/Lab/1 may indicate some minor heterogeneity (injection pressure was 600 kPa at this time).

The data from level 2 shows a roughly consistent picture (given the problems with the tensiometer) with saturation being reached at the end of 2002.
At level 3 the tensiometer pressure approaches atmospheric pressure in mid 2003 after refilling and removal of air in July and September 2002, but the piezometer pressure does not start to increase until mid 2004 indicating a slow gradual saturation.

Fig. 53: Piezometer and tensiometer data comparison for left) PE&TE/Lab/1, right) PE&TE/Lab/2. Note that time scale differs for each plot.

**Water inflow/outflow**

Fig. 54 shows the inflow and outflow flowmeters over the year 2007. It can be seen that the inflow is roughly 0.7 ml/day while the outflow has increased from 0.45 ml/day to about 0.5 ml/day during the same period. Unfortunately the inflow meter is rather noisy and shows some unexpected variations. The outflow meter typically shows a more regular response (the flowmeter was removed for a short while during May/June in 2007). The best estimate is therefore that approximately 70 % of the inflow is being produced.

![Inflow and outflow water column flowmeter data.](image_url)
Fig. 55: Inflow data deduced from the water column flow meter.
Fig. 56: Outflow data deduced from the water column flow meter.

**Mass balance**

Two "back of the envelope" calculations are given here to provide simple ways of estimating the saturation and permeability of the bentonite/sand in the TDR column.
Saturation

The average state of saturation of the column can be estimated from the pore volume, initial water content and total injected water (Tab. 17).

Tab. 17: Calculation of water required for saturation.

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
<th>Comment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Porosity of bentonite/sand</td>
<td>0.296</td>
<td>Compacted bulk density (stated as 2.1 g/cm³) and the gravimetric water content of the emplaced material specified as 0.11. Estimated initial dry density is 1.87 g/cm³. Grain density 2.655 g/cm³</td>
</tr>
<tr>
<td>Initial saturation</td>
<td>0.69</td>
<td>Based on gravimetric water content and grain density</td>
</tr>
<tr>
<td>Column internal volume</td>
<td>27.42 liter</td>
<td>Total volume 27.65 less approximately 0.23 liters taken up by the central TDR (52 cm long and 2.35 cm diameter) neglecting volume associated with the piezometers and tensiometers</td>
</tr>
<tr>
<td>Column pore volume</td>
<td>8.12 liter</td>
<td>Assuming gravel layer had a porosity similar to the bentonite/sand. Assumes all tubing saturated or of negligible volume.</td>
</tr>
<tr>
<td>Water required to saturate</td>
<td>2.48 liter</td>
<td>From saturation and pore volume</td>
</tr>
</tbody>
</table>

The calculation of the total injected water is more difficult due to the very wide range of flow rates used. Initial flooding of the pore volume was at about 0.2 ml/min equivalent to 288 ml/day (see Fig. 54) for a short period while subsequently flow rates dropped to the current value of 0.7 ml/day. In addition data was lost due to a problem with the DAS PC during the early high flow rate period from 08/03/02 to 11/03/02. After this period flow rates were below the resolution of the flow controller and from 20/03/02 a measurement scale was used to estimate the flow. From 21/05/03 a water column flow meter was used.

An approximate sum of the total flow is given in Tab. 18. Periods of poor or missing data have been assumed to relate to zero flow conditions in the calculations. It is difficult to gauge the accuracy of the different flow measurement methods or to set an absolute minimum for the injection flow; however, the data indicate that the column was highly saturated when flow was first detected from the top of the column in March 2006.

Tab. 18: Estimated flow rates and cumulative injection.

<table>
<thead>
<tr>
<th>Period</th>
<th>Volume injected (liters)</th>
<th>Cumulative Volume (liters)</th>
<th>Saturation at end</th>
<th>Mean flow rate (ml/day)</th>
<th>Comment</th>
</tr>
</thead>
<tbody>
<tr>
<td>06/03/02-20/03/02</td>
<td>0.34</td>
<td>0.34</td>
<td>0.73</td>
<td>24.28</td>
<td>Assumes no inflow during data loss period</td>
</tr>
<tr>
<td>21/03/02-15/05/03</td>
<td>1.19</td>
<td>1.53</td>
<td>0.88</td>
<td>2.83</td>
<td>Some periods of negative flow indicated by weighing scale (scale</td>
</tr>
<tr>
<td>Period</td>
<td>Volume injected (liters)</td>
<td>Cumulative Volume (liters)</td>
<td>Saturation at end</td>
<td>Mean flow rate (ml/day)</td>
<td>Comment</td>
</tr>
<tr>
<td>----------------</td>
<td>--------------------------</td>
<td>----------------------------</td>
<td>-------------------</td>
<td>-------------------------</td>
<td>-------------------------------------------------------------------------</td>
</tr>
<tr>
<td>15/05/03-17/06/03</td>
<td>0.02</td>
<td>1.55</td>
<td>0.88</td>
<td>0.59</td>
<td>carrying pressure vessel gained weight)</td>
</tr>
<tr>
<td>25/06/03-31/12/03</td>
<td>0.246</td>
<td>1.79</td>
<td>0.91</td>
<td>1.30</td>
<td>WCFM installed</td>
</tr>
<tr>
<td>01/01/04-31/12/04</td>
<td>0.144</td>
<td>1.94</td>
<td>0.93</td>
<td>0.39</td>
<td>Flow controller defective due to lightning over voltage 12/05/04-17/12/04</td>
</tr>
<tr>
<td>01/01/05-31/12/05</td>
<td>0.308</td>
<td>2.24</td>
<td>0.97</td>
<td>0.84</td>
<td>Some high readings caused by bubbles in flow lines Early January</td>
</tr>
<tr>
<td>01/01/06-31/12/06</td>
<td>0.252</td>
<td>2.50</td>
<td>0.998</td>
<td>0.69</td>
<td>Flow from outlet March 16</td>
</tr>
<tr>
<td>01/01/07-28/05/07</td>
<td>0.129</td>
<td>2.63</td>
<td>1.01</td>
<td>0.87</td>
<td>Some poor high flow data excluded (see Fig.)</td>
</tr>
<tr>
<td>28/05/07-27/11/07</td>
<td>0.121</td>
<td>2.75</td>
<td></td>
<td>0.66</td>
<td>27/11/07 injection with uranine and rhodamine</td>
</tr>
<tr>
<td>28/11/07-29/02/08</td>
<td>0.056</td>
<td>2.81</td>
<td></td>
<td>0.64</td>
<td>26/02/08 move the column to create space 29/02/08 Sta injection with He</td>
</tr>
<tr>
<td>01/03/08-11/07/08</td>
<td>0.073</td>
<td>2.9</td>
<td></td>
<td>0.55</td>
<td>Instable flowrate due to instable He backpressure probably due to slight leaking of He 11/07/08 injection 20 bar</td>
</tr>
<tr>
<td>12/09/08-03/12/08</td>
<td>0.044</td>
<td>2.94</td>
<td></td>
<td>0.53</td>
<td>11/11/08 Stop outflow</td>
</tr>
<tr>
<td>04/12/08-15/12/08</td>
<td>0</td>
<td>2.94</td>
<td></td>
<td>0</td>
<td>No flow. Injection stopped due to leak. 16/12/08 repair leak</td>
</tr>
<tr>
<td>16/12/08-02/11/09</td>
<td>0.112</td>
<td>3.11</td>
<td></td>
<td>0.35</td>
<td>02/02/09 move the tank to VE cavern</td>
</tr>
<tr>
<td>03/11/09-08/09/10</td>
<td>0.170</td>
<td>3.26</td>
<td></td>
<td>0.55</td>
<td>New wcfm 03/11/09 02/09/10 new fittings</td>
</tr>
<tr>
<td>09/09/10-11/10/10</td>
<td>0.007</td>
<td>3.28</td>
<td></td>
<td>0.22</td>
<td>Repair TDR cable fitting change of slop</td>
</tr>
<tr>
<td>12/11/10-15/02/11</td>
<td>0.039</td>
<td>3.33</td>
<td></td>
<td>0.41</td>
<td></td>
</tr>
</tbody>
</table>
Effective permeability – hydraulic conductivity

A simple estimate of the effective saturated permeability of the bentonite/sand can be made by assuming homogeneous linear flow along the column between the piezometers. The calculation can be performed for the different piezometer pairs (PE/Lab/1-PE/Lab/2 and PE/Lab/2-PE/Lab/3. The permeability \( k \) (m\(^2\)) can be estimated from the average flow rate using:

\[
Q = \frac{k \cdot A \cdot \Delta P}{\mu \cdot \Delta l} \quad \text{[m}^3\text{/s]} \tag{28}
\]

where \( A \) is the cross-sectional area of the column (0.0314 m\(^2\)), \( \Delta P \) the pressure difference (Pa) between the sensors, \( \Delta l \) the distance between the sensors (0.3 m) and \( \mu \) is the viscosity (10\(^{-3}\) Pa·s).

Estimated permeability and calculated average flow rates for different time periods (between inflow WCF refills) are given in Tab. 19. The permeability has been calculated using the inflow rate. Outflow rate was measured from June 2006 onwards but column refill times do not always match those of the inflow meter. The permeability for both intervals is about 10\(^{-19}\) m\(^2\) (hydraulic conductivity of approximately 10\(^{-12}\) m/s). The permeability in the lower section (typical value 8·10\(^{-20}\) m\(^2\)) is about 20 % lower than that in the upper section (typical value 1.1·10\(^{-19}\) m\(^2\)).
Tab. 19: Average flowrates and estimated permeability for selected periods from January 2006.

<table>
<thead>
<tr>
<th>Start</th>
<th>End</th>
<th>Inflow rate (ml/day)</th>
<th>Outflow rate (ml/day)</th>
<th>$K_{P1-P2}$ ($m^2$)</th>
<th>$K_{P2-P3}$ ($m^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>26/01/2006</td>
<td>13/03/2006</td>
<td>0.71</td>
<td>Not measured</td>
<td>8.6.E-20</td>
<td>1.1.E-19</td>
</tr>
<tr>
<td>17/03/2006</td>
<td>23/04/2006</td>
<td>0.72</td>
<td></td>
<td>8.6.E-20</td>
<td>1.1.E-19</td>
</tr>
<tr>
<td>27/04/2006</td>
<td>15/06/2006</td>
<td>0.70</td>
<td></td>
<td>8.4.E-20</td>
<td>1.1.E-19</td>
</tr>
<tr>
<td>18/06/2006</td>
<td>09/07/2006</td>
<td>0.67</td>
<td>0.41</td>
<td>8.3.E-20</td>
<td>1.0.E-19</td>
</tr>
<tr>
<td>12/07/2006</td>
<td>13/08/2006</td>
<td>0.67</td>
<td></td>
<td>8.3.E-20</td>
<td>1.1.E-19</td>
</tr>
<tr>
<td>15/08/2006</td>
<td>28/09/2006</td>
<td>0.65</td>
<td></td>
<td>8.1.E-20</td>
<td>1.0.E-19</td>
</tr>
<tr>
<td>30/09/2006</td>
<td>26/10/2006</td>
<td>0.94</td>
<td></td>
<td>1.2.E-19</td>
<td>1.6.E-19</td>
</tr>
<tr>
<td>28/10/2006</td>
<td>18/12/2006</td>
<td>0.63</td>
<td></td>
<td>8.3.E-20</td>
<td>1.1.E-19</td>
</tr>
<tr>
<td>20/12/2006</td>
<td>16/01/2007</td>
<td>0.61</td>
<td></td>
<td>8.2.E-20</td>
<td>1.1.E-19</td>
</tr>
<tr>
<td>10/02/2007</td>
<td>09/03/2007</td>
<td>1.10</td>
<td></td>
<td>1.5.E-19</td>
<td>2.0.E-19</td>
</tr>
<tr>
<td>14/03/2007</td>
<td>28/03/2007</td>
<td>1.32</td>
<td>0.44</td>
<td>1.9.E-19</td>
<td>2.4.E-19</td>
</tr>
<tr>
<td>22/04/2007</td>
<td>08/05/2007</td>
<td>0.76</td>
<td>0.45</td>
<td>1.0.E-19</td>
<td>1.2.E-19</td>
</tr>
<tr>
<td>12/05/2007</td>
<td>24/05/2007</td>
<td>0.67</td>
<td>0.46</td>
<td>8.6.E-20</td>
<td>1.1.E-19</td>
</tr>
</tbody>
</table>

Gas injection test

The long saturation phase of this experiment offered the unique opportunity to test gas flow behaviour of a highly compacted S/B mixture at an intermediate scale. The N$_2$ gas injection was started on 07.03.2012 to assess both gas flow during the early phase, when gas starts entering the column, and gas flow rates after breakthrough. A differential pressure of 5 bars was applied, while constant pressure at the outflow of 15 bars was maintained (i.e. injection pressure of 20 bars).

2.8 FORGE Mock-up experiment

2.8.1 Objectives

The mock-up experiment was set up to demonstrate the impact of chemical interaction between cement and bentonite on water and gas transport properties on a more realistic spatial scale and with more realistic boundary conditions (i.e. cement as source term, saturated and unsaturated conditions). The main objectives are:

- Provide experimental data on the water saturation process and gas invasion on an intermediate scale
- Obtain samples from the mortar-bentonite interface for post mortem analysis
- Test QA procedures for later full-scale experiment(s)
- Provide improved model parameters for full-scale modelling
2.8.2 Experimental setup and procedure

The influence of the expected clogging layer on the water and gas transport properties across the material interfaces will be looked at in detail through dedicated hydraulic and gas migration experiments. Final dismantling of the column will provide the necessary information to connect changes observed in material properties with observations from the column experiment.

The column to be used for this experiment is shown in Fig. 57. It consists of a steel cylinder with numerous inlets for measuring pressures/suctions, etc.

The experiment aims at simulating the interface between porous mortar backfill and the S/B plug/seal (Fig. 57). Plugs and seals will be constructed with abutments on both sides to prevent horizontal displacement and subsequent deterioration of the water sealing properties.

The experiment was installed in a 920 mm long, 620 mm OD/600 mm ID, stainless steel (AISI-316L) cylinder equipped with flange sealings at both ends (see Fig. 1) and a large number of ducts. The maximum working pressure of the cylinder is 20 bar and the calculated empty cylinder weight with flanges is about 400 kg. The cylinder is placed in a horizontal position such that the flanges are vertical.

Details about material properties, instrument types and parallel laboratory experiments can be found in Nagra (2010).

Fig. 57: Schematic display of mock-up experiment showing the two mortar discs at both ends (grey), the central S/B body (brown) and the granular bentonite (beige); and the following instruments: combined tensiometer / pressure sensors (with white tips), total pressure cells at both sides, 3-finger TDRs and ring TDRs (black rings).
**Water and gas injection systems**

The schematic drawing of the gas and water injection system is given in Fig. 58. The water injection flowboard consists of the following units:

- The N$_2$ bottle with the pressure reduction valve
- The piston pressure vessel PV1 with separated gas/water phases
- The flowcontroller WFC to control the injection
- The water inlet into the system.

The gas/water extraction flow is measured with:

- Gas-water separator PV2 with the differential pressure transducer DPT1.

The gas injection flow board consists of same units as the water injection flowboard with the following configuration:

- The N$_2$ bottle with the pressure reduction valve
- The piston pressure vessel PV1 filled with gas phase
- The gas inlet into the system
- The differential pressure transducer DPT2 to measure the amount of injected gas.
- The gas/water extraction flow is measured with the pressure vessel for gas and water outflow measurements PV2 with the differential pressure transducer DPT1.
Fig. 58: Schematic drawing of experimental setup for water and gas injection.

### Water volume in PV1:

<table>
<thead>
<tr>
<th>Piston length (mm)</th>
<th>Water volume (l)</th>
</tr>
</thead>
<tbody>
<tr>
<td>345</td>
<td>12.547</td>
</tr>
<tr>
<td>330</td>
<td>12</td>
</tr>
<tr>
<td>375</td>
<td>10</td>
</tr>
<tr>
<td>138</td>
<td>5</td>
</tr>
<tr>
<td>28</td>
<td>1</td>
</tr>
</tbody>
</table>
Initial material properties

First, sand and bentonite were mixed with atmospheric water content during 5 minutes. Then the necessary water to reach 11% of water content was added in three steps. For water homogenisation, 5 minutes of mixing were let between each step. Finally, the mixture was let in the mixer switched on for 15 minutes. After the mixing, the S/B mixture was stored in air tightness boxes at least for 2.5 hours.

Tab. 20 summarises some characteristic measured for each layer of S/B. Fig. 59 shows the evolution of the dry unit weight and the apparent unit weight all along the sample. One can note a good homogeneity of the sample in terms of density and the targeted value of 1.8 g/cm$^3$ in terms of dry unit weight have been reached with an absolute accuracy of 0.1 g/cm$^3$.

Tab. 20: Experimental results obtained with the permeability tests.

<table>
<thead>
<tr>
<th>Layers</th>
<th>Weight S+B (kg)</th>
<th>Weight dry sand (kg)</th>
<th>Weight dry bentonite (kg)</th>
<th>Height from top (cm)</th>
<th>Height (mm)</th>
<th>Apparent unit weight</th>
<th>Dry unit weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>10.91</td>
<td>8.73</td>
<td>2.18</td>
<td>74.47</td>
<td>26.2</td>
<td>2.24</td>
<td>2.02</td>
</tr>
<tr>
<td>2</td>
<td>8.26</td>
<td>6.61</td>
<td>1.65</td>
<td>71.85</td>
<td>22.2</td>
<td>2.00</td>
<td>1.80</td>
</tr>
<tr>
<td>3</td>
<td>12.87</td>
<td>10.29</td>
<td>2.57</td>
<td>68.23</td>
<td>36.2</td>
<td>1.91</td>
<td>1.72</td>
</tr>
<tr>
<td>4</td>
<td>6.69</td>
<td>5.35</td>
<td>1.34</td>
<td>66.39</td>
<td>18.4</td>
<td>1.95</td>
<td>1.76</td>
</tr>
<tr>
<td>5</td>
<td>11.53</td>
<td>9.22</td>
<td>2.31</td>
<td>63.32</td>
<td>30.7</td>
<td>2.02</td>
<td>1.82</td>
</tr>
<tr>
<td>6</td>
<td>11.53</td>
<td>9.22</td>
<td>2.31</td>
<td>60.22</td>
<td>31</td>
<td>2.00</td>
<td>1.80</td>
</tr>
<tr>
<td>7</td>
<td>11.53</td>
<td>9.22</td>
<td>2.31</td>
<td>57</td>
<td>32.2</td>
<td>1.92</td>
<td>1.73</td>
</tr>
<tr>
<td>8</td>
<td>11.53</td>
<td>9.22</td>
<td>2.31</td>
<td>53.9</td>
<td>31</td>
<td>2.00</td>
<td>1.80</td>
</tr>
<tr>
<td>9</td>
<td>10.78</td>
<td>8.63</td>
<td>2.16</td>
<td>51.09</td>
<td>28.1</td>
<td>2.06</td>
<td>1.86</td>
</tr>
<tr>
<td>10</td>
<td>10.78</td>
<td>8.63</td>
<td>2.16</td>
<td>48.21</td>
<td>28.8</td>
<td>2.01</td>
<td>1.81</td>
</tr>
<tr>
<td>11</td>
<td>10.41</td>
<td>8.33</td>
<td>2.08</td>
<td>45.34</td>
<td>28.7</td>
<td>1.95</td>
<td>1.76</td>
</tr>
<tr>
<td>12</td>
<td>13.39</td>
<td>10.71</td>
<td>2.68</td>
<td>41.57</td>
<td>37.7</td>
<td>1.91</td>
<td>1.72</td>
</tr>
<tr>
<td>13</td>
<td>13.39</td>
<td>10.71</td>
<td>2.68</td>
<td>37.88</td>
<td>36.9</td>
<td>1.95</td>
<td>1.76</td>
</tr>
<tr>
<td>14</td>
<td>10.78</td>
<td>8.63</td>
<td>2.16</td>
<td>35.02</td>
<td>28.6</td>
<td>2.03</td>
<td>1.83</td>
</tr>
<tr>
<td>15</td>
<td>10.97</td>
<td>8.78</td>
<td>2.19</td>
<td>32.02</td>
<td>30</td>
<td>1.96</td>
<td>1.77</td>
</tr>
<tr>
<td>16</td>
<td>10.97</td>
<td>8.78</td>
<td>2.19</td>
<td>29.22</td>
<td>28</td>
<td>2.11</td>
<td>1.90</td>
</tr>
<tr>
<td>17</td>
<td>10.97</td>
<td>8.78</td>
<td>2.19</td>
<td>26.3</td>
<td>29.2</td>
<td>2.02</td>
<td>1.82</td>
</tr>
<tr>
<td>18</td>
<td>10.97</td>
<td>8.78</td>
<td>2.19</td>
<td>23.52</td>
<td>27.8</td>
<td>2.12</td>
<td>1.91</td>
</tr>
<tr>
<td>19</td>
<td>11.19</td>
<td>8.95</td>
<td>2.24</td>
<td>20.48</td>
<td>30.4</td>
<td>1.98</td>
<td>1.78</td>
</tr>
<tr>
<td>20</td>
<td>11.19</td>
<td>8.95</td>
<td>2.24</td>
<td>17.44</td>
<td>30.4</td>
<td>1.98</td>
<td>1.78</td>
</tr>
</tbody>
</table>
Fig. 59: Evolution of the apparent unit weight a) and the dry unit weight b) all along the sample.

Injection water

The scenario to be demonstrated with the mock-up experiment is the saturation of a repository seal by water from the overlying formation(s). It is therefore expected that the inflowing water will have a similar composition as the one in the Opalinus Clay and the above "Brown Dogger". Tab. 21 below shows the artificial pore water (APW) for Opalinus Clay - "Brown Dogger", which was chosen for experiments by ETHZ. The same recipe was applied for the injection water of the FORGE mock-up experiment.

Tab. 21: Modelled composition and other geochemical parameters of the APW for ETHZ and the reference pore water for Opalinus Clay.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>OPA reference (Maeder, 2009)</th>
<th>APW (ETHZ / Valther)</th>
<th>Comment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature</td>
<td>25</td>
<td>25</td>
<td></td>
</tr>
<tr>
<td>pH</td>
<td>7.203</td>
<td>7.861</td>
<td></td>
</tr>
<tr>
<td>pCe</td>
<td>-2.781</td>
<td>--</td>
<td>Siderite-pyrite equilibrium</td>
</tr>
<tr>
<td>Concentration units</td>
<td>mmol/kg</td>
<td>mmol/kg</td>
<td></td>
</tr>
<tr>
<td>Na (total)</td>
<td>164.4</td>
<td>163.8</td>
<td></td>
</tr>
<tr>
<td>K (total)</td>
<td>2.604</td>
<td>2.551</td>
<td></td>
</tr>
<tr>
<td>Ca (total)</td>
<td>12.51</td>
<td>11.91</td>
<td></td>
</tr>
<tr>
<td>Mg (total)</td>
<td>9.625</td>
<td>9.166</td>
<td></td>
</tr>
<tr>
<td>Sr (total)</td>
<td>0.2106</td>
<td>--</td>
<td></td>
</tr>
<tr>
<td>Fe^{II} (total)</td>
<td>0.0524</td>
<td>--</td>
<td></td>
</tr>
</tbody>
</table>
Parameter | OPA reference (Maeder, 2009) | APW (ETHZ / Valther) | Comment
---|---|---|---
Fe$^{III}$ (total) | 3.31·10^{-9} | -- |
Si (total) | 0.1779 | -- |
Cl (total) | 160.0 | 160.0 |
SO$^{IV}$ (total) | 24.72 | 24.00 |
Si$^{III}$ (total) | 12.4·10^{-9} | -- |
Cl$^{IV}$ (total) | 2.506 | 0.5431 |
F | -- | -- |
Alkalinity | 2.308 | 0.5494 |
P(CO$^{2}$) | $10^{-2.20}$ | $10^{-3.5}$ [bar] |
Ionic strength | 0.2299 | 0.2264 [m] |
SI (calcite) | 0.00 | 0.00 |
SI (dolomite, ord.) | 0.00 | 0.00 |
SI (celestite) | 0.00 | -- |
SI (gypsum) | -0.28 | -0.31 |
SI (strontianite) | -0.98 | -- |
SI (quartz) | 0.00 | -- |
SI (fluorite) | -- | -- |
SI (siderite) | 0.00 | -- |
SI (pyrite) | 0.00 | -- |

Calculations performed with Phreeqc (version 2.15.07) and Nagra-PSI data base Version 01/01.

2.8.3 Results and discussion

Column saturation

The saturation was started on July 13, 2010. The main activities and events are listed in Tab. 22 below.

Tab. 22: Main activities and events since start of saturation.

<table>
<thead>
<tr>
<th>DATE</th>
<th>TIME</th>
<th>ACTIVITY</th>
</tr>
</thead>
<tbody>
<tr>
<td>25.06.10</td>
<td></td>
<td>Start saturation of TE sensors</td>
</tr>
<tr>
<td>06.07.10</td>
<td></td>
<td>Saturate TE sensors</td>
</tr>
<tr>
<td>13.07.10</td>
<td>08:00</td>
<td>Saturation of TE sensors: TE 700_E and TE 385_N OK, 465_NW and 465_SE</td>
</tr>
<tr>
<td>DATE</td>
<td>TIME</td>
<td>ACTIVITY</td>
</tr>
<tr>
<td>---------</td>
<td>-------</td>
<td>--------------------------------------------------------------------------</td>
</tr>
<tr>
<td>16.07.10</td>
<td>07:30</td>
<td>Repair leakage</td>
</tr>
<tr>
<td>10:50</td>
<td></td>
<td>Saturation with max. 1.5 bar backpressure and with WFC</td>
</tr>
<tr>
<td>22.07.10</td>
<td>12:30</td>
<td>Pressurize pressure vessel</td>
</tr>
<tr>
<td>11.08.10</td>
<td></td>
<td>Mortar saturation, drilling of the filter at injection port (the filter was clogged)</td>
</tr>
<tr>
<td>03.09.10</td>
<td></td>
<td>Saturation tensiometer TE sensors and measure tension pressure</td>
</tr>
<tr>
<td>23.09.10</td>
<td></td>
<td>Saturation TE sensors and measure tension pressure</td>
</tr>
<tr>
<td>13.10.10</td>
<td></td>
<td>Start injection for the saturation phase with 5 bar backpressure and 10 ml/h flow rate</td>
</tr>
<tr>
<td>18.10.10</td>
<td>12:56</td>
<td>Increase injection pressure to 20 bar</td>
</tr>
<tr>
<td>22.10.10</td>
<td></td>
<td>Tensiometer TEs measure above atmosphere, saturation reached</td>
</tr>
<tr>
<td>23.11.10</td>
<td></td>
<td>Replace the TE sensors with 20 bar PE sensors</td>
</tr>
<tr>
<td>03.12.10</td>
<td>10:30</td>
<td>Shut in injection due pressure decrease and flow decrease to 0</td>
</tr>
<tr>
<td>06.12.10</td>
<td></td>
<td>Remove the injection tank. The piston of the tank is leaking. Tank transported to Solexerts for repair. Injection stopped</td>
</tr>
<tr>
<td>08.12.10</td>
<td></td>
<td>Installation of the tank after repair and restart injection</td>
</tr>
<tr>
<td>04.02.11</td>
<td>12:26</td>
<td>TDR multiplexer check</td>
</tr>
<tr>
<td>04.04.11</td>
<td></td>
<td>Sampling from PE 775-C. Installation double filter before the flowmeter</td>
</tr>
<tr>
<td>19.04.11</td>
<td></td>
<td>Circulation for degassing. 6 L circulation volume using the tank</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Take sample from the tank and from PO-920 N (extraction port during degassing)</td>
</tr>
<tr>
<td>04.05.11</td>
<td></td>
<td>Circulation for degassing within the mortar using a pump</td>
</tr>
<tr>
<td>11.05.11</td>
<td></td>
<td>Stop circulation within the mortar and resume injection</td>
</tr>
<tr>
<td>23.05.11</td>
<td>15:28</td>
<td>Injection flow meter clogged completely, flowmeter bypassed</td>
</tr>
<tr>
<td>30.06.11</td>
<td></td>
<td>Installation new DAS for TDR measurement</td>
</tr>
<tr>
<td>28.07.11</td>
<td>16:40</td>
<td>Reduce injection pressure (TP 175 N over 30 bar)</td>
</tr>
<tr>
<td>15.09.11</td>
<td></td>
<td>Set injection pressure to 16 bar</td>
</tr>
<tr>
<td>20.09.11</td>
<td></td>
<td>Refill the injection tank</td>
</tr>
<tr>
<td>02.11.11</td>
<td>11:40</td>
<td>Installation of flowmeter after repair and continue injection through the flowmeter</td>
</tr>
<tr>
<td>07.03.12</td>
<td>17:12-17:45</td>
<td>Start saturation at the gas side port PE 175 C. Stop the injection due to high pressure over 33 bar at</td>
</tr>
<tr>
<td>20.03.12-</td>
<td></td>
<td>Saturation of the mortar by injecting from the gas side using a gear</td>
</tr>
<tr>
<td>DATE</td>
<td>TIME</td>
<td>ACTIVITY</td>
</tr>
<tr>
<td>----------</td>
<td>------------</td>
<td>------------------------------------------------------------------------------------------------------------------------------------------</td>
</tr>
<tr>
<td>02.04.12</td>
<td></td>
<td>pump</td>
</tr>
<tr>
<td>02.04.12</td>
<td></td>
<td>Start injection from both injection point and PE 175 C which is at the gas side. Port PO-N 95 is open</td>
</tr>
<tr>
<td>10.04.12</td>
<td></td>
<td>Close PO-N 95 and open PE-385 N</td>
</tr>
<tr>
<td>01.05.12-03.05.12</td>
<td></td>
<td>Injection only from gas side, water injection port closed</td>
</tr>
<tr>
<td>03.05.12</td>
<td>13:06</td>
<td>Injection from both sides: water side and gas side PE-175 C</td>
</tr>
<tr>
<td>15.05.12</td>
<td></td>
<td>Fill the injection tank with Pearson water</td>
</tr>
<tr>
<td>05.07.12</td>
<td></td>
<td>Replace TDR Muxes and function check. TDR cable leakage at Muxes plug, cut the cable before the Mux at TDR-700</td>
</tr>
<tr>
<td>13.09.12</td>
<td>12:00</td>
<td>Bypass injection flowmeter due to clogging</td>
</tr>
<tr>
<td>26.10.12</td>
<td></td>
<td>Tightness of TDR cable after leakage observed at TDR 465 and TDR 245</td>
</tr>
<tr>
<td>31.10.12</td>
<td></td>
<td>Installation new flowmeter 15 g/min instead of the clogged flowmeter 10 g/h. Water leak from the cable cut</td>
</tr>
</tbody>
</table>

Figs. 60 to 62 show the evolution of pressures (water and total) and flows in the Mock-up experiment. Water injection was performed as a constant flow injection at initially 5 bar back pressure and constant flow of 10 ml/h. As the pressure on the injection side approached the back pressure the back pressure was increased to the targeted 20 bars. It can be seen that the flow decreases when the back pressure was reached and stabilised at about 1 ml/h, thus at a lower rate than expected.

A surprising observation was made at one of the total pressure cells (TP-133-N) which was rising above 30 bars, while the other sensor located at the same side of the S/B showed only about 3 to 4 bar pressure (Fig. 61). Although the latter one was more likely the actual swelling pressure in the system, the observed 30 bar pressure would be considerably above the design pressure of the stelle vessel. As consequence the injection pressure was decreased to 16 bar. After starting water injection from the far end of the column both total pressure cells showed the same value although TP-133-N still shows the highest total pressure.

The observed evolution of water pressures provided a nice insight into the importance of local heterogeneities and how they affect the water saturation process. Fig. 62 shows that the first set of pressure sensors (showed in blue) were saturated almost at the same time in December 2010. Looking at the sensors in the centre of the column, shown in green, the saturation occurred over a period of about 4 months at the same axial position. This observation corresponds quite well with the observed water saturation in the area, as measure by the 3-finger TDR TDR-465-SW-C. Note that the TDR measurements average over a considerably larger volume (about 100 cm$^3$) than the point pressure measurements.

It yet not entirely clear how the data from the ring TDRs can be used to assess water saturation (Fig. 64). Their main function will be to detect preferential gas flow along the column wall during gas injection.
Fig. 60: Measurements in the injection system. Top: pressures at the water (blue and pink) and gas (green and yellow) injection side. Bottom: inflows at the water injection side.
Fig. 61: Top: Injection pressures at water (blue) and gas (green) injection side. Bottom: Total pressures at water (blue and pink) and gas (red and green) injection side.
Fig. 62: Pressure readings at the monitoring points inside the S/B seal.

Fig. 63: TDR travel time plots of the 3-finger TDR located in the centre of the S/B seal.
As the saturation time was considerably longer than expected the pressure data were assessed in more detail. The main question was why the permeability of the S/B was considerably lower than expected based on the pre-tests in the laboratory. This question has direct implications for the construction of a repository plug because the hydraulic permeability of the S/B is a design parameter which cannot be determined in the field.

The analysis was done on two lines of evidence, namely an analysis of the gradients observed in the mock-up at different times, and the measurement of S/B permeabilities in the lab using the exact same materials as used for the mock-up experiment.

The pressure analysis was performed at four different times during the saturation phase, where the dates chosen had to be during times with as little disturbance as possible (Fig. 65). Based on these data the hydraulic permeability of each measurement point was assessed using Darcy's law.
\[ Q = \frac{-kA}{\mu} \left( p_s - p_i \right) \Delta x \]  

(29)

Where \( Q \) is the injection rate, \( k \) is the hydraulic permeability, \( \mu \) is the viscosity (1.216 mPa at 13 °C), \( A \) is the cross section of the S/B body, \( p \) is the pressure at injection and observation point and \( x \) is the distance from injection.

The resulting permeabilities are between 2 and 2.8E-19 m\(^2\), with an average over all sensors of 2.5E-19 m\(^2\), thus about 5 times lower than designed.

Fig. 65: Hydraulic pressures measured at all pressure sensors at four dates during column saturation (left) and evolution of permeabilities over time.

The laboratory analyses of the hydraulic permeability at different dry densities of the identical material are shown in Fig. 66. The figure shows clearly that the use of rounded sand (FORGE sand) as compared of the initially tested edged Leman sand has led to a considerable decrease of permeabilities. In fact the results coincide quite well with the observations of hydraulic gradients in the mock-up.

Additionally, it was tested what the impact is on this crucial relation when changing the clay material. Fig. 66 shows that there are only minor differences, which may as well represent some analytical uncertainties.
Chemical composition of injection water

Water samples were taken from the water injection side at different times since water injection was started. It can be seen that electric conductivities measured in the mortar water are considerably higher than those measured in the injection water (artificial pore water). Furthermore, the mortar seems to leach out considerable amounts of organic components, most likely related to the grinding aids added to the cement material. Tab. 23 shows that considerable amounts of Sr and Si are dissolved from the mortar, the later one mainly during the very early stage quickly reducing to more or less constant amounts.

Tab. 23: chemical composition of injection water. Ion concentrations are given in mg/L.

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Location</td>
<td></td>
<td>Injection tank</td>
<td>Injection (mortar) filter</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Field pH</td>
<td></td>
<td>12.79</td>
<td>12.61</td>
<td>12.63</td>
<td></td>
<td></td>
</tr>
<tr>
<td>EC(μS/cm)</td>
<td></td>
<td>26100</td>
<td>23700</td>
<td></td>
<td>8400</td>
<td></td>
</tr>
<tr>
<td>Eh</td>
<td></td>
<td>-139</td>
<td>-137</td>
<td>-135</td>
<td></td>
<td></td>
</tr>
<tr>
<td>T</td>
<td></td>
<td>15.0</td>
<td>15</td>
<td></td>
<td>14.1</td>
<td></td>
</tr>
<tr>
<td>Lab EC (μS/cm)</td>
<td></td>
<td>19240</td>
<td>28000</td>
<td>24200</td>
<td>24000</td>
<td>22900</td>
</tr>
<tr>
<td>Lab pH</td>
<td></td>
<td>7.86</td>
<td>9.24</td>
<td>12.59</td>
<td>12.33</td>
<td>12.43</td>
</tr>
<tr>
<td>AC (4,3)</td>
<td></td>
<td>0.54</td>
<td>0.4</td>
<td>78.75</td>
<td>67.6</td>
<td>66.8</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>------</td>
<td>-----</td>
<td>-----</td>
<td>-----</td>
<td>-----</td>
<td>-----</td>
<td></td>
</tr>
<tr>
<td>Na</td>
<td>3765.8</td>
<td>3860</td>
<td>3570</td>
<td>3130</td>
<td>3220</td>
<td>2910</td>
</tr>
<tr>
<td>K</td>
<td>99.7</td>
<td>96.2</td>
<td>975</td>
<td>415</td>
<td>369</td>
<td>206</td>
</tr>
<tr>
<td>Ca</td>
<td>477.3</td>
<td>470</td>
<td>389</td>
<td>770</td>
<td>719</td>
<td>1130</td>
</tr>
<tr>
<td>Mg</td>
<td>222.8</td>
<td>216</td>
<td>0.08</td>
<td>&lt; 0,1</td>
<td>&lt; 1</td>
<td>&lt; 0,2</td>
</tr>
<tr>
<td>Sr</td>
<td>0</td>
<td>&lt; 1</td>
<td>21.4</td>
<td>25.4</td>
<td>25.5</td>
<td>22.5</td>
</tr>
<tr>
<td>Cl</td>
<td>5672</td>
<td>5720</td>
<td>3900</td>
<td>4120</td>
<td>4220</td>
<td>4960</td>
</tr>
<tr>
<td>SO4</td>
<td>2304</td>
<td>2310</td>
<td>243</td>
<td>36.6</td>
<td>41</td>
<td>52.2</td>
</tr>
<tr>
<td>Al</td>
<td>0</td>
<td>0.06</td>
<td>0.035</td>
<td>1.45</td>
<td>0.055</td>
<td>0.045</td>
</tr>
<tr>
<td>Si</td>
<td>0</td>
<td>0.11</td>
<td>24</td>
<td>0.6</td>
<td>1.25</td>
<td>0.85</td>
</tr>
<tr>
<td>DOC</td>
<td>0</td>
<td>3.9</td>
<td>43.9</td>
<td>39.5</td>
<td>44.4</td>
<td>38.2</td>
</tr>
<tr>
<td>Acetate</td>
<td>0</td>
<td>&lt; 1</td>
<td>30.4</td>
<td>20.2</td>
<td>27.6</td>
<td></td>
</tr>
<tr>
<td>Formiate</td>
<td>0</td>
<td>1.5</td>
<td>7.2</td>
<td>5.2</td>
<td>&lt; 5</td>
<td></td>
</tr>
</tbody>
</table>

Fig. 67 shows the major ion concentrations in time. The dashed lines added indicate the concentrations of the APW injected into the mortar disc. It can be seen that Cl, SO₄ and Na are consumed within the mortar, while Ca, K, Sr and inorganic carbon are continuously dissolved from the OPC in the mortar disk.

As shown in Fig. 68 Na and K should be leached from the mortar in the early phase when portlandite is consumed and the pH decreases from about 13 to about 11. However, due to the high salinity of the injection water Na is dissolved rather than leached out. The decreasing concentrations of K and Na, however, can be nicely observed in the mortar water, although the pH observed in the mortar was never higher 12.8. As expected Ca concentrations are increasing during this early evolution stage.

SO₄ concentrations are likely reduced due to gypsum dissolution inside the mortar disk. This process should be confirmed when modelling the hydrochemical evolution of the experiment and/or when removing the material and analysing the different materials for their mineral compositions.

The leaching of Cl from the injection water is somewhat surprising because it was thought to be a conservative water component. An explanation could be that the high Cl concentration in the injection water leads to observable Cl diffusion into the mortar (gravel and lime), and thus to a loss of Cl in the pore solution analysed. With an approximate pore volume inside the mortar disc of 4200 ml and an inflow of 10 initially to 4 ml/h at a later stage leads to a residence time inside the disc of 17 to 44 days respectively.
Fig. 67: Major ion concentrations over time. Na and Cl concentrations relate to the left y-axis and K, Ca, SO$_4$ relate to the right y-axis.

Fig. 68: Schematic display of expected evolution of major high-pH plume components (top plot) and main phases/processes in the evolved concrete.

Fig. 69 shows the evolution of calculated water permeabilities over time. The results provide further evidence that the observed lower hydraulic permeabilities, as stated above, are not a consequence of the high-pH water injected into the S/B but merely a consequence of the S/B properties as compacted. The figure indicates that there is no observable decrease of hydraulic permeabilities over the time scale of the experiment (i.e. about 2 years). Post mortem analyses of the cement-S/B interface will provide direct evidence on the impact of high-pH water on the porosity distribution in the adjacent S/B seal.
Fig. 69: Evolution of calculated hydraulic permeability inside the S/B seal.
3 Phenomenological evidence & conceptual understanding

3.1 Fundamentals and basic understanding

As proposed in Nagra (2008) gas transport through low-permeability rock formations is
controlled not only by the hydraulic and mechanical properties of the rock mass (intrinsic
permeability, porosity, rock strength), but also by the gas pressure at the place of gas entry
and the hydromechanical state of the rock (i.e. water saturation, porewater pressure, stress state).
Phenomenological considerations suggest the following subdivision of the basic transport
mechanisms (Fig. 70):

- advective-diffusive transport of gas dissolved in the porewater
- visco-capillary two-phase flow
- dilatancy-controlled gas flow
- gas transport along macroscopic tensile fractures (hydro- and gas-fracturing)

It is worth mentioning that different terminologies for the description of gas transport processes
are found in the geoscientific literature. The term "capillary failure" is often used in oil & gas
industry to describe the two-phase flow regime in cap rocks (e.g. Clayton & Hay 1994). "Membrane
seal failure" describes gas transport through the pre-existing pore system of the
caprock (i.e. advection / diffusion of dissolved gas and two-phase flow), whereas "hydraulic
seal failure" is used to describe gas leakage due to hydromechanical processes in the seal
(microfracturing, reopening of existing faults, hydro/gas fracturing). A comprehensive literature
study on gas storage and gas transport phenomena is given in Evans (2008).

Fig. 70: Classification and analysis of gas transport processes in clay materials (from
Nagra, 2008).
3.2 Phenomenological evidence

3.2.1 Basic classification of material from index tests

The investigated material is a mixture of grey quartz sand and MX-80 Bentonite (Wayoming) in proportion 80/20 in dry mass.

The MX-80 is a sodium granular bentonite, named mixture ‘E’, which has been delivered at the EPFL in June 2009. The index properties of the MX-80 bentonite are showed in Tab. 24. The MX-80 bentonite is mainly composed of 84.9 ± 1.2 % montmorillonite, 4.8 ± 0.8 % muscovite, 3.7 ± 0.5 quartz, 5.2 ± 0.8 % feldspar and 1.3 ± 0.2 % calcite (Nagra, 2007).

MX-80 mixture ‘E’ bentonite shows exchangeable cations of 52.4 meq/100 g Na, 13.2 meq/100 g Mg and 1.4 meq/100 g K (Nagra, 2007).

Tab. 24: General properties of MX-80 bentonite (Müller-Vonmoos and Kahn, 1983; Lajudie et al., 1996, Seiphoori, in prep.).

<table>
<thead>
<tr>
<th>Bentonite</th>
<th>Specific gravity, ( G_s )</th>
<th>Liquid limit, ( w_L (%) )</th>
<th>Plastic limit, ( w_p (%) )</th>
<th>Shrinkage limit, ( w_s (%) )</th>
<th>Specific surface area, ( SS (m^2/g) )</th>
</tr>
</thead>
<tbody>
<tr>
<td>MX - 80</td>
<td>2.74-2.76</td>
<td>411 ± 10</td>
<td>47 - 70</td>
<td>14</td>
<td>562</td>
</tr>
</tbody>
</table>

The role of bentonite is to reduce the hydraulic conductivity of the mixture thanks to the swelling capacity of the smectite minerals.

The quartz sand, which has been used for all experiments, is a grey quartz sand from Carlo Bernasconi AG (carloag.ch). Its specific density is 2.65 g/cm\(^3\). The mineralogy of this quartz sand consists mainly of Silicon dioxide (97.40%), Alumina (1.35%), Potassium and Sodium oxides (0.8%), small amounts of Titanium dioxide, Periclase and Lime.

The presence of sand in the material reduces the shrinkage of the mixture when the water content decreases and thus reduces the risk of cracking. This risk is much more present in a pure bentonite (Graham, 2002), while at low bentonite contents, the sand particles are in direct contact with each other. Knowing the particle density of the two components, the particle density of the mixture was obtained using the following expression:

\[
\rho_{s,\text{mix}} = \frac{100}{\frac{\% \text{sand}}{\rho_{s,\text{sand}}} + \frac{\% \text{bent}}{\rho_{s,\text{bent}}}} = 2.67 \text{ g/cm}^3
\]

The mixture used for the experiments was prepared following the outcomes of the Mock-up experiment (Nagra, 2010).

The sand and the granular bentonite, at the initial water content, were initially sieved at 0.5 mm, and then they were mixed manually. Once a good homogenization of mixture was obtained, the 11% of distilled water in weight was added. The obtained water content corresponds to the optimum water content determined by means of the modified Proctor test, carried out at according to the Swiss Standard SN 670 330, under specific compaction energy of 3.4 J/cm\(^3\) (Fig. 71). The wet mixture was carefully mixed again until reaching a good homogenization (Fig. 72). Finally the mixture was stored in hermetic containers for at least 3 days in order to obtain a homogenous water distribution over the whole mass of material.
Fig. 71: Modified Proctor test on S/B mixture in proportion 80/20 (Minon et al. 2010).

Fig. 72: a) S/B mixture prepared at w = 11%. b) S/B mixture poured into the oedometric ring.

For the oedometric tests the compaction of the specimen was performed directly inside the oedometric ring (Fig. 5). Two filter papers were placed along the two bases. The specimens were prepared by static compaction. Two different presses were used for the compaction. The first one, from Wikeham Farrance Eng. LTD, was equipped with a load cell (maximum vertical force of 22 kN, accuracy of 0.1% of the full range) and the piston displacement was monitored by a LVDT (accuracy of 1 μm) (Fig. 6). The second press is a Walter & Bai type which is instrumented with a load cell (maximum vertical force of 100 kN, accuracy of 0.1 of the full range) and a LVDT (range of 20 mm and accuracy of 0.2%). The second press was used to compact specimens with dry density higher than 1.6 g/cm³. A constant compression rate of 0.5 mm/min was used for the preparation of all the specimens. For the preparation of the specimens tested in the triaxial cell, the same compaction procedure was applied with the help of a cylindrical mould.

3.2.2 Phenomena related to full saturation

Darcy’s law proposes a linear relationship between the specific discharge and the hydraulic gradient. This relationship is valid only when laminar flow occurs so the fluid movements are dominated by viscous forces.
According to Bear (1972) the upper limit of the linearity of the Darcy’s law, for a porous media, is at Reynolds number from 1 to 10. In this range the velocity is low and the viscous forces are predominant. For higher Reynolds number the flow becomes turbulent. Bear (1972) also mentioned the existence of a lower limit of the Darcy’s law. At a minimum gradient $i_0$, called threshold gradient, the flow in fine grain size soil is very little. This phenomenon is attributed to counter currents along the pore walls in a direction opposite of that of the main flow.

In order to verify the validity of the Darcy’s law for the S/B mixture the water permeability tests were conducted on the specimen n° PERM-dd1.5 by applied different pressure gradients while measuring the corresponding flows. The permeameter cell was selected for this test. These results were also compared with the ones obtained for the specimens TX-C100, TX-C300 and MC-dd1.5.

The results of the tests are presented in Fig. 73. An exponential trend for the specific discharge with the hydraulic gradient was taken as best fit for the experimental data.

![Fig. 73: Verification of Darcy's law for the S/B mixture.](image)

### 3.2.3 Phenomena related to solute transport

**PET experiment**

A plexiglass column was designed to perform PET (positron emission tomography) measurements during water saturation and later gas invasion. The column was mounted onto a flow board to perform controlled water and gas injection and monitoring. For column saturation a water flow of 0.002 ml/min was applied for 20 d. Then a conservative PET tracer solution with $[^{58}\text{Co}](\text{Co(CN)}_6)^{3-}$ (70.86 days half-life) was injected with 0.001 ml/min for 42 days.

Water flows/volumes and concentrations were observed throughout the experiment. First arrival of the tracer was after injecting 27 ml of tracers (or 24 days injection) and a peak was observed after 60 ml of traced water injected (or 51 days injection). Adding the 30 ml of clean water
previously injected into the column the total water injected was 90 ml until peak arrival – and including the initial water content of column it leads to about 140 ml of water, compared to an approximate pore volume of 125 ml.

The estimated hydraulic permeability after saturation was 0.4 \( \mu \)Darcy or 4E-19 m\(^2\). The peak activity at the outflow of about 0.11 MBq/ml indicates that tracer may be slightly sorbing on the clay minerals of the S/B column. This would as well explain the higher activities seen towards the injection side of the column (see Fig. 77).

Overall the test shows that water flow is quite homogeneous in the S/B body.
Swelling tests under changing hydrochemical conditions

Constrained and free swelling tests were carried out in order to determine the relationship between swelling capacity and the initial dry density of this material under different chemical boundary conditions. The results showed that the amount of swelling depends on the initial dry density and is strongly affected by the change in water salinity.

Figs. 75 and 76 show the results of the swelling pressure tests performed on the S/B mixture (80/20) using distilled and synthetic water, respectively. The evolutions of the swelling pressure and the water volume entering into the specimen are depicted. The results show that the
swelling pressure generated very fast in the early stage of the tests (up to 50 hours for distilled water and up to 25 hours for the synthetic one). After this stage the swelling pressure increasing rate reduced significantly. Comparison between the volumes of water that entered into the specimen at the different dry densities, Fig. 75-b shows a significant decrease for the specimen with higher dry density. This reduction is attributed to the reduction of the permeability of the specimen caused by the reduction of the macropores as shown by the MIP tests (see Section 2.2.2) for the specimen with higher dry density. The results of the swelling tests on the specimens saturated with synthetic water are presented in Fig. 13. The swelling test on the specimen compacted at 1.3 g/cm³ of dry density was stopped after 17 hours due to the fact that no swelling pressure was generated during this test. Moreover, a remarkable water inflow equals to 2.98E-9 m³/s was measured, confirming the inhibition of the swelling capacity of the bentonite by the synthetic water. Similar to the distilled water case, the increase of swelling pressure and reduction of water inflow for the specimens with higher dry densities were observed.

**DISTILLED WATER**

![Fig. 75: Results of the swelling pressure test using distilled water in terms of swelling pressure and water volume exchange for the tested specimens. In the graph the average swelling pressure measured for each specimen is indicated.](image)

**SYNTHETIC WATER**

![Fig. 76: Results of the swelling pressure test using synthetic water. In the graph the average swelling pressure measured for each specimen is indicated.](image)
The dry density, at which the specimen is compacted, has a strong impact on the developed swelling pressure; Fig. 77 shows the swelling pressure as a function of the specimen dry density for both distilled and synthetic water; in the figure the values of the swelling pressure reported correspond to the weighted average of the swelling pressures measured (after 50 hours for distilled water and after 25 hours for the synthetic one). An exponential trend for the swelling pressure with the dry density was taken as best fit for both water types.

The use of synthetic water reduced remarkably the swelling pressure. The rate of reduction of the swelling pressure ($RP_{sw}$) with the dry density was calculated as proposed by Komine et al. (1999):

$$RP_{sw} = \frac{P_{SW,DW} - P_{SW,SW}}{P_{SW,DW}}$$

where $P_{SW,DW}$ is the swelling pressure in distilled water and $P_{SW,SW}$ is the maximum swelling pressure in synthetic water. The reduction is in the range of 67% - 45% for the dry density in the range of 1.3 - 1.9 g/cm³ when the mixture was in contact with synthetic water. The reduction factor trend shows that the loss in swelling capacity due to the saturation with synthetic water, in the range of dry densities under consideration, can be partially compensated by the increase in dry density.

Fig. 77: Comparison between the swelling pressures developed with distilled water and in situ pore water.

Fig. 78 shows the results of the free swelling tests carried out on the S/B mixture at different dry densities and using both distilled and synthetic water. The evolutions of the swelling strain and the water volume entering into the specimen are depicted. The figure shows an increase of the swelling strain by increasing the dry density. This increasing trend was also observed for the
free swelling tests with synthetic water; however the maximum swelling strain is much lower for these tests with respect to the tests with distilled water.

Due to the reduction of the swelling capacity of the mixture in contact with synthetic water the time required for the swelling test was strongly reduced, from about 200 hours for distilled water to less than 50 hours for the synthetic one. Supposing a linear correlation between swelling strain and dry density the minimum dry density required to have an observable swelling deformation of the mixture is about 1.3 g/cm³ when distilled water is used and about 1.4 g/cm³ for synthetic water. This latter observation is in agreement with the results of the constrained swelling tests in which, for the test carried out with synthetic water, no remarkable swelling pressure was observed for a dry density of 1.3 g/cm³.

![Graph showing maximum swelling strain as function of the initial dry density for S/B mixture 80/20.](image)

The rate of reduction of swelling strain decreased very fast for a dry density that varies from 1.4 to 1.6 g/cm³. For dry densities higher that 1.6 g/cm³ the curve tends to an asymptotic value of 0.4.

### 3.2.3 Phenomena related gas invasion/drainage

#### Water retention curves

The water retention curve of the mixture was determined for two different dry densities (1.53 and 1.8 g/cm³) in both drying and wetting, in constant volume conditions, and the van Genuchten parameters were determined.

For the wetting path the material was saturated in steps, by putting the microcage in an ambient with 100% of relative humidity (zero total suction). Inside the saturating chamber the material tends to equilibrate with the imposed suction and so to adsorb water from the humid ambient.
After a certain time that varies from 12 to 24 hours for each step, the saturation process was stopped and the microcage was extracted from the chamber. Then, it was sealed with paraffin tape for 1 day in order to let the adsorbed moisture to distribute inside the specimen. After the equalization, the suction was measured with the WP4c (Fig. 79). The weight of the cell was continuously monitored in order to compute the evolution of the water content.

For the drying path a similar procedure as for the wetting path was adopted. In this case the cage was dried at the laboratory ambient, in which the temperature is kept at 22°C (±1°C) and the relative humidity is 41 % (corresponding total suction of 120 MPa). During the drying process the weight of the cell was monitored in order to compute the evolution of the water content. The process was stopped after certain time that varies from 1 to 24 hours, depending on the reached water content, by sealing the microcage with the paraffin tape. As the water evaporation occurred from the holes of the microcage, so it started from the external surface of the specimen, the cage was keep sealed in paraffin for at least one day in order to let the total suction to homogenise inside the specimen. The drying process was stopped once the suction reached the initial suction value obtained after compaction. The microcage was opened at the end of test and the contact of the specimen with the cell was assessed.

Fig. 79 (upper plots) presents the water retention curves of the mixture for two specimens compacted at 1.53 g/cm³ and 1.8 g/cm³, respectively. The results show that for a given degree of saturation, the measured total suction was higher for the specimen compacted at 1.8 g/cm³. This result is attributed to the reduction of the macroporosity due to the increase in the dry density and in the consequent increment of the capillary component of the total suction.

The experimental data were fitted with the equation proposed by van Genuchten (1980):

\[ S_r = \left( \frac{1}{1 + \left( \frac{\Psi}{\alpha} \right)^n} \right)^m \]  

where \( S_r \) is the degree of saturation, \( \alpha, n \) and \( m \) are material parameters. The parameters obtained by fitting the experimental data are presented in Tab. 25.

Fig. 79 (lower plots) shows the data obtained by measuring the total suction with the WP4c are showed together data obtained by imposing the matric suction with the axis translation technique for a similar dry density (about 1.50 g/cm³). The van Genuchten parameters are then recalculated for the entire range of suction (form 0.001 to 20 MPa).
Fig. 79: Top plots: Water retention curves of the S/B mixtures compacted at 1.53 cm³ and 1.80 g/cm³ for saturation (wetting) and desaturation (drying). Suction versus degree of saturation (left) and suction versus water content (right). Lower plots: total suction data (with the WP4c) and data obtained by applying the axis translation technique for a similar dry density of around 1.5 g/cm³.

Tab. 25: Van Genuchten parameters calculated for specimen compacted at 1.53 and 1.8 g/cm³ of dry density. †Fitting parameters for total suction measurement (0.003 < \( s < 0.5 \) MPa; 3 < \( \psi < 20 \) MPa).

<table>
<thead>
<tr>
<th>Van Genuchten parameters</th>
<th>Dry density = 1.53 g/cm³</th>
<th>Dry density = 1.8 g/cm³</th>
<th>†Dry density = 1.5 g/cm³</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Wetting</td>
<td>Drying</td>
<td>Wetting</td>
</tr>
<tr>
<td>( \alpha ) (MPa)</td>
<td>0.13</td>
<td>0.19</td>
<td>0.36</td>
</tr>
<tr>
<td>m</td>
<td>17.01</td>
<td>16.38</td>
<td>16.97</td>
</tr>
<tr>
<td>n</td>
<td>0.02</td>
<td>0.02</td>
<td>0.02</td>
</tr>
</tbody>
</table>

PET experiment

After injection of 140 ml of water into the available pore volume of about 120 ml, He gas was injected into the column with a pressure of 20bar. Gas breakthrough has occurred already after 7
hours and the PET column was investigated again after the gas tests have been completed. The activity differences before and after gas testing is displayed in Fig. 80, where bluish colours indicate reductions and reddish colours indicate increase of activity. It appears that activities tend to decrease towards the gas inlet, whereas in most of the column differences are balanced. Looking at the radially averaged image shown in Fig. 80 the discrepancy seems much less evident but there seems to be a shift of concentrations towards the outflow side. However, it needs to be kept in mind that some variations are due to repositioning the sample from one measurement session to the next.

Fig. 80: Difference of activities before and after gas injection. Bluish colours indicate reduction, red colours indicate increases.
Gas permeability tests

Gas permeability tests were performed to determine the effective gas permeability of the mixture at different degrees of saturation; nevertheless additional measurements are necessary to determine the relationship between the effective gas permeability and the degree of saturation.

For all the performed gas penetration tests, the gas breakthrough, corresponding to a steep increase of the outflow, occurred at a gas pressure of a few tens of kPa (Tab. 26), thus much lower than the sum of the swelling pressure and the pore water pressure (or the confining pressure, in case of triaxial condition). As shown in Fig. 82 the observed breakthrough pressures, corresponding with a (connected) pore radius in the range of 20 to 100 microns fits quite well with the observed pore size distributions for the same density of 1.5 g/cm³. All together this is a good indication that the main flow process in S/B material is visco-capillary flow. This result shows a clear difference to experiments performed with pure bentonite where the breakthrough pressure is equal or higher than the sum of swelling and water pressure (e.g. Horseman & Harrington 1997, Gallé, 2000).

It is yet unclear why the gas flow for the test in the modular column with 6 cm length showed about 10 times lower flow than those performed in the triaxial cell with 1-1.5 cm length. Future experiments will show if this observation is systematic or if it is only an experimental artefact from the different setups.
Tab. 26: Summary of gas tests performed on 80/20 sand bentonite.

<table>
<thead>
<tr>
<th>Density</th>
<th>Test setup</th>
<th>Thickness</th>
<th>Diameter</th>
<th>Water</th>
<th>$\Delta p$</th>
<th>Conf. Pressure</th>
<th>Norm. flow</th>
<th>$p_s$</th>
</tr>
</thead>
<tbody>
<tr>
<td>g/cm$^3$</td>
<td>cm</td>
<td>cm</td>
<td>kPa</td>
<td>kPa</td>
<td>m/s</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.5</td>
<td>V0</td>
<td>6</td>
<td>10.1</td>
<td>dest.</td>
<td>&lt;46 to 50</td>
<td>-</td>
<td>4.30E-07</td>
<td>75 to 85</td>
</tr>
<tr>
<td>1.5</td>
<td>TRX</td>
<td>1.5</td>
<td>5</td>
<td>dest.</td>
<td>10 to 20</td>
<td>100</td>
<td>5.24E-06</td>
<td>75 to 85</td>
</tr>
<tr>
<td>1.5</td>
<td>TRX</td>
<td>1</td>
<td>5</td>
<td>dest.</td>
<td>15 to 35</td>
<td>300</td>
<td>4.99E-06</td>
<td>75 to 85</td>
</tr>
<tr>
<td>1.5</td>
<td>TRX</td>
<td>1</td>
<td>5</td>
<td>dest.</td>
<td>11 to 31</td>
<td>300</td>
<td>4.99E-06</td>
<td>75 to 85</td>
</tr>
<tr>
<td>1.5</td>
<td>TRX</td>
<td>1</td>
<td>5</td>
<td>dest.</td>
<td>20 to 40</td>
<td>300</td>
<td>5.41E-06</td>
<td>75 to 85</td>
</tr>
<tr>
<td>1.5</td>
<td>V0</td>
<td>2</td>
<td>5.1</td>
<td>synth.</td>
<td>about 4</td>
<td>-</td>
<td>1.04E-05</td>
<td>25</td>
</tr>
</tbody>
</table>

Fig. 82: Pore size distributions for two densities (1.5 and 2 g/cm$^3$) and for initial (as compacted) and saturated state. Range of 20 to 100 microns is indicated in green.

Additional measurement of the specimen deformation once reaching the breakthrough pressure is needed to study the nature of the created gas pathway.

3.2.4 Coupled processes

The coupling of water saturation and swelling are already explained in Chapter 2.3. The tests showed that maximum swelling pressure is reached quicker for the saline water compared to
distilled water, although as mentioned above, the equilibrium reached under saline conditions is lower.

As shown in Tab. 25 increasing the confining pressure had little impact on gas breakthrough pressure and there is no significant influence on the gas flow after breakthrough. This may be explainable by the stiff grid provided by the sand matrix resisting the external stress, while the gas flow is dominated by the bentonite material in the constant macro pore space.

### 3.2.5 Scale effects

Preliminary interpretation of the pressure evolution in the mock-up experiment show that it is possible to design and emplace a large scale experiment with a defined target water permeability and that small scale experiments are sufficient to provide the necessary relation between density and hydraulic permeability. However, it needs to be noted that these tests must be done with the same material as the one finally applied. As shown in Fig. 83 varying the sand type from Leman to FORGE sand led to 10 times lower hydraulic permeability of the S/B seal. As a consequence the FORGE mock-up experiment was saturating much slower than designed initially based on the Leman sand.

Fig. 83: Relation of S/B (80/20) dry densities and resulting hydraulic permeabilities for two different sands and two different MX80 clay types.
4 Conclusions

The experimental work performed in the framework of the Forge project has successfully developed reproducible protocols for water and gas transport experiments on S/B mixtures. A particular challenge was the control/sealing of the interfacial flow along the test equipment, and the low suction ranges of at most a few MPa at initial saturation.

The swelling and water permeability tests performed for different dry densities have confirmed the exponential relation between dry density and swelling respectively hydraulic permeability. Varying both chemical and mechanical/stress boundary conditions aimed at investigation the impact of realistic (repository like) boundary conditions on swelling and permeability. The results show that increasing water salinity has a considerable impact on both swelling pressure and water permeability and that there was little impact from increasing external stress.

The gas tests performed consistently indicate that visco-capillary flow is the dominating transport process for water and gas. Additionally, the tests show that increasing the external stress conditions has little impact on entry pressure and gas transport capacity. However, a considerable decrease of swelling pressure was observed when saturating the sample with higher salinity synthetic pore water.

The experiment programme will be continued after FORGE to produce a broader database for water and gas transport in S/B mixtures and providing deeper insights into the processes underlying two-phase flow in these materials.
5 References


