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Deep repository – engineered barrier system

Wetting and homogenization processes in backfill materials

Laboratory tests for evaluating modeling parameters

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Abstract

SKB in Sweden and Posiva in Finland are developing and implementing similar disposal concepts for the final disposal of spent nuclear fuel. A co-operation and joint development work between Posiva and SKB with the overall objective to develop backfill concepts and techniques for sealing and closure of the repository have been going on for several years.

The backfill materials investigated where: Asha 230B and Friedland for use as block materials and Cebogel QSE, MX-80, Minelco and Friedland for use as pellets material. The issues investigated were:

- 1. Homogenization processes of highly compacted backfill blocks and pellet filling during saturation. The influence of different materials and water salinity was studied.
- 2. Water uptake processes for different materials and different water types. Clay specimens were put in contact with water at one end and after a certain time the tests were terminated and the water content distribution determined.
- 3. The water retention curve was determined for the two block materials.

The results of these investigations will be used for modeling purposes either directly (retention curve) or indirectly by modeling of the tests.

Sammanfattning

SKB i Sverige och Posiva i Finland, utvecklar och planerar för liknande förvarskoncept för slutförvaret av utbränt kärnbränsle. Ett samarbete och gemensamt utvecklingsarbete mellan Posiva och SKB med målet att utveckla återfyllningskoncept och teknik för att täta och försluta ett förvar pågår sedan flera år.

De återfyllningsmaterial som har undersökts är: Asha 230B och Friedland som är tänkta att användas som blockmaterial samt Cebogel QSE, MX-80, Minelco och Friedland som är tänkta att användas som pellets material. Följande frågeställningar undersöktes:

- 1. <u>Homogeniseringen</u> av kompakterad återfyllningsblock och pelletsfyllning under bevattning. Inverkan av typ av material samt salthalten i vattnet undersöktes.
- 2. <u>Vattenupptagningen</u> av återfyllningsmaterial. Vid dessa försök fick provkroppar tillgång till vatten från en ända under en bestämd tid varefter fördelningen av vattenkvoten och densiteten bestämdes. Inverkan av typ av material samt salthalten i vattnet undersöktes.
- 3. Vattenhållningskurvan (retention curve) för två olika återfyllningsmaterial har bestämts.

Resultaten från dessa undersökningar kommer att användas för modelleringsändamål antingen direkt (vattenhållningskurvan) eller indirekt genom modellering av testerna.

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1 Introduction

The investigations described in this report belong to the third phase of the joint SKB-Posiva project "*Backfilling and Closure of the Deep Repository, BACLO*". The overall objective of the BACLO project is to develop a backfilling concept for the deep repository that can be configured to meet SKB's and Posiva's requirements in the chosen repository sites. The project was divided into four phases, of which two have already been finished. The second phase of the BACLO project consisted of laboratory tests and deepened analyses of the investigated backfill materials and methods and resulted in recommendation to focus on the development and testing of the block placement concept with three alternative backfill materials. The third phase investigations comprise laboratory and large-scale experiments aiming at testing the engineering feasibility of the concept. In addition, how site-specific constraints, backfilling method & materials affect the long-term functions of the barriers will be described and analysed in order to set design specifications for the backfill.

The work described in this report belongs to the group of laboratory investigations performed in third phase of the BACLO programme. The investigations performed in this subproject belong to Phase 3 of the BACLO project. The results from the investigations described in this report aim to evaluate additional modeling parameters regarding the modeling of the wetting and homogenization processes.

2 Test layouts and materials

2.1 General

Backfilling of deposition tunnels is planned to be done by emplacement of pre-compacted blocks of bentonite, see Figure 2-1. The blocks will be piled, filling up 60–80% of the tunnel volume (investigations regarding the proportion between pellets and blocks are ongoing in different projects but no decisions are made). The remaining space between blocks and rock is to be filled with bentonite pellets. During and after emplacement of blocks and pellets a number of scenarios can develop due to water inflow from the rock. These scenarios will affect the materials and perhaps also the functioning of the backfill material. A number of critical issues have earlier been identified and investigated. This report describes a number of additional issues. The outcome from the tests will be used in the modeling work.



Figure 2-1. Schematic drawing showing an example of the geometry of a deposition tunnel backfilled with blocks and pellets.

2.2 Test types

The investigations performed in this subproject belong to Phase 3 of the BACLO project. In Phase 2 a number of different test types were used aiming to study the behavior of the backfill materials during emplacement and during the early saturation. In Phase 3 it was decided to perform some complementary tests regarding erosion and piping in order to get more knowledge of the processes and also to do a number of other laboratory investigations. The modeling of the wetting and homogenization processes require however a number of complementary tests in order to evaluate additional modeling parameters. The following investigations have been done and are presented in this report:

- 1. Homogenization of backfill blocks and pellets. Tests have been performed in standard oedometers with a diameter of 50 mm and the height 40 mm. Compacted block was placed in the oedometer and a pellet filling of different thickness (4, 8 and 12 mm) was placed above. The samples had access to water only from the pellet side. The tests were performed with different clay materials, of both the blocks and the pellets, with different water salinity and of different test times. At termination the water content and density distribution were determined.
- 2. Water uptake. Tests samples were compacted in a cylinder with a diameter of about 100 mm and a height of 100 mm. The samples had access to water from one end during specified time periods. After this time period the distribution of water content and density of the samples were determined. The tests were made with two different materials and with two different salt contents in the water.
- **3. Water retention curve.** The retention curve has been determined for Asha 230B and for Friedland clay. The curve is an important material property for modeling of the behavior during saturation.

The tests will be described in chapter 3–5 in more detail.

2.3 Materials and water used in the tests

Different materials (both block materials and pellets types) have been used in the various tests undertaken. All materials are under consideration as future candidates for use as backfill:

- Friedland clay. Mixed layer clay from Germany with a smectite content of about 25–35%. /Karnland et al. 2006/
- Asha 230 B. Na-bentonite from India. A minor laboratory investigation have been done with this material /Johannesson et al. 2006/ and the liquid limit was here determined to w_L=472%. This together with the retention curve (see chapter 5) shows that the smectite content probably is rather high. Material from the same supplier and from the same deposit has been investigated in /Karnland et al. 2006/ showing a smectite content of about 80%. No data sheet or reference regarding the material used in the tests in this report is available.

In the homogenization tests pellets/granules of the following types have been used:

- Cebogel QSE. A commercial bentonite pellets with a montmorillonite content of about 80% (according to data sheet from the deliverer). The pellets are delivered by Cebo Holland BV. The material is used in ongoing field tests at Äspö. The pellets are extruded cylindrical rods with a diameter of 6.5 mm and a length of 5–20 mm.
- Minelco granules. Tests are also ongoing at Äspö using Minelco material. The grain size distribution of this material varies a lot (55% is > than 4 mm and 99% > 1 mm). The material origins from Milos and was delivered as a raw material that could be used for backfilling. No data regarding smectite content is available.

- Friedland granules. Friedland clay crushed to granules that are even-grained with a size of about 8 x 8 x 4 mm.
- MX-80. Specially made pellets consisting of MX-80 Wyoming bentonite with a smectite content of about 80% /Karnland et al. 2006/. The pellets are "pillow" shaped with the dimensions 18 x 18 x 8 mm. In addition there are a few percent fine materials present.

In order to use the different pellets/granules in the homogenization tests in rather small slot widths, the pellets were crushed somewhat.

The following water types were used in the tests:

- Water1: tap water.
- Water2: water with salinity of 1% (50/50 NaCl/CaCl₂).
- Water3: water with salinity of 3.5% (50/50 NaCl/CaCl₂).

The water type that most likely will leak into the repository during the installation phase is type 2, but due to possible "upconing effect" in Olkiluoto /Posiva 2006-05/ a number of tests were also made with a higher salinity of the water (3.5%) which also have been a design salinity used throughout all Baclo investigations. Some supplementary tests were made with tap water.

2.4 Uncertainties

All tests described in this report are performed in a geotechnical laboratory. Sensors used are carefully calibrated using controlled artifacts with traceability.

All determinations of mass are made by using a laboratory balance.

The number of tests performed is limited and it has not been possible to test the repeatability of the results within this project.

3 Homogenization of backfill blocks/pellets

3.1 General

The backfill concept includes compacted bentonite blocks that are piled on the tunnel floor. After piling there will be a remaining slot between the blocks and the rock wall, see Figure 2-1, which will be filled with bentonite pellets. During the saturation phase the blocks will swell and homogenization of the blocks and the pellets will start.

The test series were aimed to study the homogenization process and the influence of different materials and waters.

3.2 Test description

The tests were performed using oedometers of standard type, see Figure 3-1. Block specimens (d=50 mm and h=40 mm) were manufactured using either Friedland or Asha 230B. The specimens were compacted at the optimal water content /Johannesson et.al 2006/ (10–11% for Friedland and about 17% for Asha 230B) and with a compaction pressure of 25 MPa. The compacted specimen was placed in an oedometer and then a pellet filling of different thickness (4, 8 or 12 mm) was placed above. After preparation the sample got access to water from the pellets side i.e. the simulated rock wall. The axial force was continuously measured with a load cell.



Figure 3-1. Schematic view of the test equipment. An oedometer was used in the test series.

Test variables

- Two block types (Friedland and Asha 230 B).
- Four pellets/granules types (MX-80, Minelco, Cebogel and Friedland).
- Four pellets slot widths (4, 8 and 12 mm).
- Two water types (1% salt and 3.5% salt).

Laboratory determinations

After termination of the tests, the specimens were divided in slices with a thickness of 5 mm (2.5 mm in some positions). Density and water content of each slice was then determined.

Water content

Half part of each slice was placed in an aluminum baking tin and the bulk mass (m_b) of the specimen determined by use of a laboratory balance. The specimen was dried in an oven for 24 hours at a temperature of 105°C. The dry solid mass (m_s) of the specimen was then determined immediately after removal and the water mass (m_w) was calculated according to Equation 3-1:

$$m_w = m_b - m_s \tag{3-1}$$

The water content (w) of the specimen determined according to Equation 3-2:

$$w = \frac{m_w}{m_s} \tag{3-2}$$

Density

The other half of each slice was used for determining the bulk density. The specimen was weighed, first in air (m_b) and then submerged into paraffin oil (m_{bp}) . The volume of the specimen was then calculated

$$V = (m_b - m_{bp}) \times \rho_p \tag{3-3}$$

where ρ_p is the paraffin oil density. The bulk density of the specimen was calculated according to Equation 3-4:

$$\rho_b = \frac{m_b}{V} \tag{3-4}$$

Dry density and degree of saturation

After determining the water content and the bulk density of each specimen it was possible to calculate the dry density (ρ_d):

$$\rho_d = \frac{\rho_b}{1+w} \tag{3-5}$$

Since the density of the particles (ρ_s), the bulk density of the sample (ρ_b) and the density of water (ρ_w) are known the degree of saturation (S_r) can be calculated:

$$Sr = \frac{w \cdot \rho_b \cdot \rho_s}{\left[\rho_s \cdot \left[1 + w\right] - \rho_b\right]} \rho_w$$
(3-6)

Different ρ_s have been used for the different materials /Karnland et al. 2006/:

MX-80 2,780 kg/m³

_

Friedland	2,780 kg/m ³
Minelco	2,780 kg/m ³
Asha 230 B	2,900 kg/m ³

Test matrix

The complete test matrix for the homogenization tests is shown in Table 3-1. The performed tests are marked with shaded boxes.

Friedland blocks, height 40 mm						
Pellets material	Pellets material 1 % salt in water 3.5 % salt in water					
	Pellets height			Pellets height		
	4 m m	8 m m	12 m m	4 m m	8 m m	12 m m
MX-80	F101	F102	F103	F104	F105	F106
Minelco	F201	F202	F203	F204	F205	F206
Friedland	F301	F302	F303	F304	F305	F306
Cebogel	F401	F402	F403	F404	F405	F406

Table 3-1. Test matrix for the homogenization tests. The test numbers marked with shaded boxes are the ones performed.

Asha blocks, height 40 mm							
Pellets material	1 % salt in	water		3.5 % salt in	water		
	Pellets he	ight	t Pellets height				
	4 m m	8 m m	12 m m	4 m m	8 m m	12 mm	
MX-80	A101	A102	A103	A104	A105	A106	
Minelco	A201	A202	A203	A204	A205	A206	
Friedland	A301	A302	A303	A304	A305	A306	
Cebogel	A401	A402	A403	A404	A405	A406	

In addition to the tests in Table 3-1 a number of extra tests were performed:

- The standard test duration was two months. In order to study the influence of time on the homogenization process two additional tests were performed with a test length of 2 weeks and 4 months respectively i.e. one shorter time than the standard and one longer. Both these tests were performed with the same test layout as F305 i.e. Friedland block with Friedland granules in an 8 mm thick slot.
- In order to study the influence of friction between the bentonite and the steel walls on the final water content and density distribution an extra test with lubricated walls was performed. This test was also performed with the same layout as test F305 i.e. Friedland block with Friedland granules in an 8 mm thick slot. The lubrication was made with "Molycote" (MoS2).
- In order to study the influence of the water inlet position an extra test where the sample had access to water from both bottom and top was performed This test was also performed with the same layout as F305 i.e. Friedland block with Friedland granules in an 8 mm thick slot.

3.3 Results

3.3.1 General

The results from the tests are presented in the following sub chapters in order to show the influence of the varied parameters as clearly as possible:

- 1. Influence of wall friction, water access direction and test duration.
- 2. Influence of different pellet material.
- 3. Influence of slot width.
- 4. Influence of salt content in the water.
- 5. Tests with different block material.

In each sub chapter, diagrams that show the distribution of water content, dry density and the degree of saturation are presented.

For each test also diagrams showing the swelling pressure development during test time are provided.

A table is provided in Appendix G showing data from specimen preparation and also measurements done after finishing the tests. An attempt to measure the width of the former pellets filled slot was done on each sample after finishing. The accuracy of these measurements is rather rough depending on the difficulties to determine the interface between pellets and block. When the block and the pellets were of different material it was possible to see an interface but if the block and pellets were of the same material it was almost impossible to see the interface. The results from these measurements are also provided in the table in Appendix G. In addition pictures of each specimen after finishing the tests are shown in Appendix C–F.

In the following a brief analysis of the results will be done. However, in order to fully evaluate and accomplish the results from the tests they need to be modeled.

3.3.2 Influence of test duration, wall friction and water access on the homogenization process

Five tests with the same test layout (Friedland block with an 8 mm slot filled with Friedland granules) were performed. Water with a salinity of 3.5% was used in all tests.

The diagrams in Figure 3-2 show the results from the tests. The black straight line shows the initial conditions at test start.



Figure 3-2. Water content (upper), dry density (middle) and degree of saturation (lower) plotted as function of the distance from water inlet for the five samples made of Friedland blocks performed in order to investigate the influence of wall friction, water inlets and test duration.

Influence of test duration

The green dots show the results from the standard test which was running for two months. The red and purple dots show the results from the tests with the same layout but terminated after two weeks and 4 months respectively.

The diagrams show that there is a logical influence of the time regarding the homogenization of the specimens. The water content in the outermost pellets slot was about 5% higher for the two weeks test than for the four month test. The same tendency can be seen in the diagram showing the dry density distribution. In the same positions, the outermost pellets slot, the dry density was somewhat higher for the four month tests (1.57 g/cm³) compared to the two week test (1.48 g/cm³). It seems however that the main part of the homogenization takes place during the first two weeks but reaching a completely homogenization will probably take very long time. The time dependence can also be seen when studying the measurements of the swelling pressure, see Figure 3-3.

An observation is that the degree of saturation is very high for all samples along their whole length; although the two week test has somewhat lower values. The homogenization after water saturation is driven by the differences in swelling pressure that will give a pore pressure gradient. The processes will need modeling.

Influence of wall friction

The influence of wall friction was investigated by lubricating the walls in the test cell with "Molycote" MoS2 (black dots). When comparing the results from this test with the standard test (green dots) almost no difference in water content or density could be determined.

Influence of access to water

One test was performed with access to water from both ends i.e. both from the pellets side and the block side (blue dots). The only difference in results between this test and the standard test (green dots) was that there was somewhat higher water content at the filter side of the block. The influence of when water is available seems to be very small which means that there is no early compaction of the pellets by swelling of that part of the block.

Swelling pressure

The swelling pressure measurements for the five tests are shown in Figure 3-3. The results were all gathered around 500 to 700 kPa except for the four month tests which had a swelling pressure of about 1,050 kPa. The difference is caused by the fact that this specimen had a slightly higher average density which also could be seen in Figure 3-2.

Earlier determinations of the swelling pressure of Friedland clay /Johannesson 2008/ showed that with a dry density of 1.8 g/cm3 (average density for the specimens including both block and pellets) the swelling pressure will be about 1,500 kPa. Since the swelling pressure only is measured on the "pellets side", see Figure 3-1, where the density is between 1.4–1.6 g/cm₃, the figures for the measured pressures are considered very reasonable.

The diagram in Figure 3-3 shows that after about two weeks test duration, the swelling pressure has reached its maximum and after that there are only small changes.



Figure 3-3. Swelling pressure as function of time for the five samples made of Friedland blocks performed in order to investigate the influence of wall friction, water inlets and test duration.

3.3.3 Influence of different pellet/granules in the slot

Different materials

The diagrams in Figure 3-4 show a comparison between the four tests performed with Friedland blocks and with an 8 mm thick slot filled with four different pellet materials. Water with a salinity of 3.5% was used in all of the tests. All four tests were running for two months.

The pellets materials used in the tests have different smectite contents which cause different properties and behavior during the tests. Some comments to the tests:

- The degree of saturation is very high along all specimens.
- For the specimens with different materials in the slot (MX-80, Cebogel and Minelco) compared to the compacted material, there will be remaining gradients in water content and density depending on the different properties of the materials. The main reason for the differences in dry density in the slot is that MX-80, Minelco and Cebogel have higher smectite content than Friedland clay and will be in equilibrium with the highly compacted Friedland block regarding suction and swelling pressure at higher water contents (lower dry densities), see also chapter 5.

Swelling pressure

The swelling pressure measurements for the tests are shown in Figure 3-5. The specimen with Friedland granules in the slot has the lowest pressure (550 kPa) while the two specimens with MX-80 and Cebogel have a swelling pressure of just below 700 kPa. The specimen with Minelco granules in the slot has the highest swelling pressure, about 1,150 kPa. This depends probably on that the average density for this specimen is somewhat higher, see Figure 3-4.



Figure 3-4. Water content (upper), dry density (middle) and degree of saturation (lower) plotted as function of the distance from water inlet for four specimens made of Friedland blocks. The slot width was the same for all tests (8 mm) but different pellet material was used.



Figure 3-5. Swelling pressure as function of time for four specimens made of Friedland blocks. The slot width was the same for all tests (8 mm) but different pellet material was used.

3.3.4 Influence of different slot width

Three tests with Friedland blocks and with different slot widths (4, 8 and 12 mm) were performed. For all three tests the slot was filled with Friedland granules.

The results from the tests are shown in Figure 3-6. The homogenization rate is strongly influenced by the slot width. With a small slot width, 4 mm, the homogenization rate seems to be considerable faster since the required swelling of the block is small. The dry density in the outermost parts of the former slot was determined to 1.67 g/cm^3 for the specimen with 4 mm slot but for the two specimens with 8 and 12 mm slot width the density was determined to $1.48-1.49 \text{ g/cm}^3$.

Swelling pressure

The swelling pressure measurements for the three tests are shown in Figure 3-7. The two specimens with a slot width of 8 and 12 mm had a swelling pressure of about 550 kPa and the specimen with a slot width of 4 mm have a swelling pressure of about 1,300 kPa. The difference depends on the fact that the specimen with the higher swelling pressure also has a higher average density, see Figure 3-6.



Figure 3-6. Water content (upper), dry density (middle) and degree of saturation (lower) plotted as function of the distance from water inlet for three specimens made of Friedland blocks with same pellet material (Friedland granules) but different slot width.



Figure 3-7. Swelling pressure as function of time for three specimens made of Friedland blocks with same pellet material but different slot width.

3.3.5 Influence of salt content in the water

The main part of the tests was performed using water with a salinity of 3.5%. In order to study the influence of the water salinity on the homogenization process, two tests were made using water with a salinity of 1%. The tests were performed with Friedland blocks and with a slot width of 8 mm. The slot was filled with either Friedland granules or MX-80 pellets.

The results from the two tests performed with 1% salt in the water are provided in Figure 3-8 together with the results from the corresponding tests performed with 3.5% salt. The diagrams indicate that the influence of the water salinity regarding the homogenization process is very small, at least for the used salinities. The influence of the different pellets material in the slot was much stronger.

Swelling pressure

The results from the swelling pressure measurements are shown in Figure 3-9. The two specimens that have had access to water with 1% salt content have a higher swelling pressure than the specimens saturated with 3.5% salt in the water. Because of the rather low density of the specimens on the pellets side the sensitivity for salt in the water is rather strong.



Figure 3-8. Water content (upper), dry density (middle) and degree of saturation (lower) plotted as function of the distance from water inlet for four specimens made of Friedland blocks with two different pellet materials and saturated with two different salt solutions.



Figure 3-9. Swelling pressure as function of time for four specimens made of Friedland blocks with two different pellet materials and saturated with two different salt solutions.

3.3.6 Influence of another block material

Five tests have been performed where the blocks were made of Asha 230B and with a pellet filled slot with a width of 8 mm. Four different pellets material were tested. Two tests were performed with MX-80 pellets and different water salinity (1% and 3.5%).

The variation in water content and density after two months testing was very small for all samples except for the one with Friedland granules. This is logical since the Friedland material also has the lowest smectite content i.e. the lowest swelling pressure.

Swelling pressure

The results from the swelling pressure measurements are shown in Figure 3-11. The swelling pressure is for all samples between 2,000–3,100 kPa. The results are very logical and correspond very well to earlier measurements made by /Johannesson et.al 2008/.



Figure 3-10. Water content (upper), dry density (middle) and degree of saturation (lower) plotted as function of the distance from water inlet for five specimens made of Asha 230 blocks and different pellet material and saturated with two different salt solutions.



Figure 3-11. Swelling pressure as function of time for five specimens made of Asha 230 blocks and different pellet material.

4 Water uptake tests

4.1 General

In order to simulate and calculate the water uptake and the homogenization of a backfill consisting of pre-compacted blocks it is of great importance to study how and at which rate the backfill material can take up water. In the laboratory it is possible to vary parameters which affect the water uptake and homogenization processes, such as material type, initial density and water content and the chemistry of the water. This type of tests can also be used as "benchmark tests" where different models can be tested for simulating the water uptake and homogenization.

Test description

The tests were performed as follows:

- Material was compacted into a cylinder (ø = 101 mm and h = 100 mm) in five layers (see Figure 4-1)
- The specimens had access to water from the bottom during a specified time. The water was not pressurized (less than 10 kPa pressure). During the water uptake the sample was prevented to expand.
- After the water uptake phase the specimen was removed from the cylinder and the density and water content of the material were determined. The determinations were made on every 10 mm of the specimen. The results from the measurements were plotted as function of the distance from the water inlet.

Two different materials were used; Friedland and Asha 230 B. The materials were compacted to two different densities, one representing the assumed density of the blocks and one representing the average density of the filling after saturation. Furthermore the tests were made with two different water types (1% salt and 3.5% salt).



Figure 4-1. The equipment used for the tests.

4.2 Test matrix

The complete test matrixes for the water uptake tests are shown in Table 4-1 and Table 4-2. The preformed tests are shaded in the tables. Altogether 10 tests were performed on each material. The average dry densities of the specimens together with target densities (Density1 and Density 2) are also shown. The Friedland specimens were fairly well compacted to the decided densities (i.e. 1,708 kg/m³ and 2,000 kg/m³) while the densities of the Asha specimens were lower compared to the target densities. The variation between the specimens was also larger for the Asha samples.

Table 4-1. Full test matrix for the water uptake tests on Friedland clay. The test numbers in
shaded boxes were performed. The target densities were: Density 1 = 1,780 kg/m ³ and Density
2 = 2,000 kg/m³ (dry densities).

		Duration		
	1 week	2 weeks	4 weeks	8 weeks
Density 1, 1% salt	Friedland 11:	Friedland 12: ρ _d = 1,751 kg/m³	Friedland 13:	Friedland 14: $\rho_d = 1,745 \text{ kg/m}^3$
Density 2, 1% salt	Friedland 21:	Friedland 22: ρ _d = 2,003 kg/m³	Friedland 23:	Friedland 24: ρ _d = 2,011 kg/m³
Density 1, 3.5% salt	Friedland 31:	Friedland 32: ρ _d = 1,758 kg/m³	Friedland 33:	Friedland 34: ρ _d = 1,748 kg/m³
Density 2, 3.5% salt	Friedland 41: $\rho_d = 2,014 \text{ kg/m}^3$	Friedland 42: $\rho_d = 2,013 \text{ kg/m}^3$	Friedland 43: $\rho_d = 2,011 \text{ kg/m}^3$	Friedland 44: $\rho_d = 2,003 \text{ kg/m}^3$

Table 4-2. Full test matrix for the water uptake tests on Asha clay. The test numbers in shaded boxes were performed. The target densities were Density $1 = 1,540 \text{ kg/m}^3$ and Density $2 = 1,700 \text{ kg/m}^3$ (dry densities).

Duration					
	1 week	2 weeks	4 weeks	8 weeks	
Density 1, 1% salt	Asha 11:	Asha 12: ρ _d = 1,461 kg/m³	Asha 13: ρ _d = 1,488 kg/m³	Asha 14:	
Density 2, 1% salt	Asha 21:	Asha 22: ρ _d = 1,602 kg/m³	Asha 23: ρ _d = 1,659 kg/m³	Asha 24:	
Density 1, 3.5% salt	Asha 31:	Asha 32: ρ _d = 1,461 kg/m³	Asha 33: ρ _d = 1,490 kg/m³	Asha 34:	
Density 2, 3.5% salt	Asha 41: ρ _d = 1,617 kg/m³	Asha 42: ρ _d = 1,607 kg/m³	Asha 43: ρ _d = 1,602 kg/m³	Asha 44: ρ _d = 1,591 kg/m³	

4.3 Test results

The measured water content for the Friedland specimens no. 41, 42,43 and 44 with the average dry density of about 2,000 kg/m³ and saturated with water of 3.5% salinity, are plotted in Figure 4-2a as function of the distance from the water inlet i.e. from the bottom of the samples. The water content is increasing with the time. After 4 weeks the water content had increased in all parts of the specimens (also close to the top of the sample although less than 1%). In Appendix A the measured degree of saturation and dry density are plotted. The degree of saturation was determined by using the density of the solid particles 2,780 kg/m³ /Karnland et al. 2006/. The degree of saturation for all of the samples was almost 1.00 close to the water inlet. The dry density was rather even over the samples, with the exception close to the water inlet where a decrease in density could be observed.

In Figure 4-2b and Figure 4-2c the influence of the initial density and water salinity regarding the saturation of the specimens can be analyzed. The diagrams show that the influence of the water salinity on the water uptake is very small but the influence of the initial dry density is large. Corresponding plots for the degree of saturation and dry density are shown in Appendix A. The tests made with the higher density had initially a high degree of saturation (~80%) and was after 2 weeks higher than 90% up to a distance of 35 mm from the water inlet. Corresponding distance for the specimens which had had access to water during 8 weeks was about 70 mm. For the specimens the degree of saturation was higher than 80% up to a distance of 30 mm from the water inlet. Corresponding distance for the specimens the degree of saturation was higher than 80% up to a distance of 30 mm from the water inlet. The water inlet water inlet water inlet. The water inlet water inlet water inlet. The water inlet water inlet water inlet water inlet. The water inlet water inlet water inlet water inlet water inlet. The water inlet water inlet water inlet water inlet water inlet. The water inlet water inlet water inlet water inlet water inlet. The water inlet water inlet water inlet water inlet water inlet. The water inlet water inlet water inlet water inlet water inlet. The water inlet water inlet water inlet water inlet water inlet. The water inlet water inlet water inlet water inlet water inlet. The water inlet water inlet water inlet water inlet water inlet. The water inlet water inlet water inlet water inlet water inlet. Water inlet water inlet water inlet water inlet water inlet. Water inlet water inlet

The evaluated water content for the Asha 230B specimens no. 41, 42,43 and 44 with the average dry density of about 1,600 kg/m³ and saturated with water of 3.5% salinity, are plotted in Figure 4-3a as function of the distance from the water inlet. The water content increases with time. After 4 weeks the water content had increased in all parts of the specimens. The specimen which had access to water for 8 weeks, increased its water content close to the top of the sample from 17% at the start to about 23%. In Appendix B the measured degree of saturation and dry density are plotted. The degree of saturation was determined by using the density of the solid particles 2,900 kg/m³ /Karnland et al. 2006/. The degree of saturation for all specimens was 1.00 close to the water inlet. The 8 weeks sample increased its degree of saturation from the initial value of about 60% to 82% at the top. The plot of the dry density of the specimens shows that the part of the specimens closest to the water inlet had expanded i.e. a decrease in dry density causing an increase of the density for the rest of the samples. This was valid also for the sample which had been saturated for only one week. The decrease in dry density can be observed to a distance of about 50 mm from the water inlet.

In Figure 4-3b and Figure 4-3c the influence of the initial density and water salinity regarding the saturation of the specimens can be analyzed. The figures show that the influence of the water salinity on the water uptake is very small but the influence of the initial dry density is large. This is obvious for the tests with duration of 2 weeks. For test no. 23 with the water salinity of 1% (4 weeks test, see Figure 3) the water content was lower compared to test no. 43 (3.5% salt). The difference in water content can be explained by the relatively high dry density for test No 23 ($\rho_d = 1,660 \text{ kg/m}^3$) compared to test No 43 ($\rho_d = 1,602 \text{ kg/m}^3$). Corresponding plots for the degree of saturation and dry density are shown in Appendix B.



Figure 4-2. Water content as function of the distance from water inlet for the tests made with Friedland material: a) Upper diagram shows the influence of time, b) Middle diagram showing the influence of salt content and c) Lower diagram showing the influence of the specimen density.



Figure 4-3. Water content as function of the distance from water inlet for the tests made with Asha 230B material. a) Upper diagram shows the influence of time, b) Middle diagram showing the influence of salt content and c) Lower diagram showing the influence of the specimen density.

5 Determination of retention curves

5.1 General

The water retention curve is a relation between the water content and the energy state or potential of the soil water expressed as relative humidity or suction. The retention curve is here presented as water content vs. relative humidity. The so called specific retention curve is determined under free swelling condition and starting with an initial water content deviating from 0%.

The method used is an experimentally simple method described below; see also /Wadsö et al. 2004/.

In addition to the retention curves the relative humidity was determined above the saline water (1% and 3.5%) referred to in sections 3 and 4.

5.2 Terminology

The water content, w, used is defined according to section 3.2. The relative humidity is the ratio between the partial vapour pressure p and the vapour pressure at saturation p_s

in %, $RH = 100 \cdot \frac{p}{p_s}$.

5.3 Experimental set up

5.3.1 General

The specific retention curve was determined for Asha and Friedland materials. The initial water contents used are shown in Table 5-1. The specimens were placed in jars with tight lids at different controlled *RH* for about 3 months or until a steady state equilibrium was reached. The jars were placed in an oven with a constant temperature of 25°C. During the course of the experiment, the specimens were weighed at regular intervals and the development of increase in mass was monitored.

Table 5-1.	Initial	water	content.
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Material	Initial water content (%)
Asha 230B	1.5
Friedland	7.8

The specimens were determined to have reached an equilibrium state when two specified conditions were fulfilled:

- the parameter Ω (Equation 5-1) was less than 5.10⁻⁹ s⁻¹.
- the rate of change of water content per 100 h was less than 0.02%.

$$\Omega = \frac{dm}{dt} \times \frac{(RH_f - RH_i)/100}{m(t) - m_i}$$
(5-1)

Here, *m* is the mass (g) and *t* is the time (s). The parameter Ω and the limit value of this parameter were suggested by /Wadsö et al. 2004/. The subscripts *f* and *i* refer to the initial and final states, respectively.

At the end of the experiment, the final water content was measured on each of the specimens.

5.3.2 Test equipment

About 8.5 g of the Friedland material and 6.5 g of the Asha material were used in the experiment. Results from previous experiments (Sandén et al. 2008) show that the Asha material is more prone to swelling when exposed to water than the Friedland material and therefore a smaller amount of material was used to ensure that there would be no loss of material during the experiment.

The specimens were placed in metal cages and hung inside glass jars with a tight lid. A rod passing through the lid made it possible to weigh the specimens below the balance without having to remove them from the jars. A plastic sealing washer was placed around the rod such that the generated *RH* was maintained during the course of the experiment. The design is presented in Figure 5-1.



Figure 5-1. Schematic view showing the test equipment.

Each jar contained a salt solution, which generated a constant relative humidity. The measurements were carried out at eight different *RH* values (0%, 11%, 33%, 58%, 76%, 84%, 93%, 97%). These *RH* values were achieved using various saturated salt solutions (LiCl, MgCl₂, NaBr, NaCl, KCl, K₂SO₄) except in the cases of *RH* = 0% and 93% were molecular sieve and an unsaturated NaCl solution, respectively, were used. The *RH* values for the saturated salt solutions were taken from /Greenspan 1977/ and the vapour pressure above the 2 molal NaCl solution was taken from / Clarke and Glew 1985/. The jars were placed in an oven at a constant temperature of 25°C. The unsaturated solution was replaced with a new mixture on a weekly basis such that a constant humidity would be maintained in the jar.

5.3.3 Test matrix

The test matrix is presented in Table 5-2. One specimen was made for each material and *RH*, with the exception of the jar with saturated NaCl solution, where an extra specimen was added for comparison.

Chemical	RH at 25°C (%)	Asha	Friedland
Molecular sieve	0	A	1
LiCI (saturated)	11.3	В	2
MgCl ₂ (saturated)	32.8	С	3
NaBr (saturated)	57.6	D	4
NaCl (saturated)	75.5	E and Ell	5 and 5II
KCI (saturated)	84.3	F	6
NaCl (2 molal)	93.1	G	7
K ₂ SO ₄ (saturated)	97.3	Н	8

Table 5-2. Test matrix and labels.

Table 5-3. Retention curves for the Asha and Friedland materialsobtained at 25°C.

RH at 25°C (%)	Water content – Asha (%)	Water content – Friedland (%)
0	0.1	0.2
11.3	2.6	1.6
32.8	6.2	3.6
57.6	12.6	6.1
75.5	20.8*	8.0*
75.5	20.6*	8.1*
84.3	24.8	9.7
93.1	32.5	13.4
97.3	45.8	21.4

*Double samples

5.4 Results

The retention curves for the Asha and Friedland materials obtained at 25°C are presented in tabular form in Table 5-3. The results are also shown in Figure 5-2. In this figure results for MX-80 (Dueck and Nilsson 2008) are also shown, for comparison reason. The initial water content of the MX-80 material was 9.8%, the determination was made at 20°C and the results were obtained with the same methodology as was used for the materials Asha and Friedland.

For the Friedland material the retention curve was also determined by a sorption balance. With this method both absorption from the initial water content of 0% and desorption from higher water content are determined. The method is further described by e.g. /Wadsö et al. 2004/. From RH = 0% a stepwise change of 9.5% up to RH = 95% and back to RH = 0% was performed during a total time of 100h. The dry weight of the specimen used was 22 mg and the measurement was performed at 21°C. The test on Friedland material was performed by L. Wadsö at Building Materials at Lund University. In Figure 5-3 the result is shown with result from Figure 5-2 where SB in the label indicates results from the sorption balance.



Figure 5-2. Retention curves for the Asha and Friedland materials obtained at 25°C shown with previous results for MX-80 at 20°C. The labels show material and initial water content (%). The arrows show the moisture paths.



Figure 5-3. Specific retention curves for the Friedland material obtained with the jars and with a sorption balance (label SB). The arrows show the moisture paths.

For the modelling of the test results from sections 3 and 4 the suction of the water used was needed. The relative humidity was measured above the solutions 1% salt and 3.5% salt (cf. section 2.3). The measurements were made by capacitive sensors and psychrometers according to Table 5-4.

(1)

Suction was calculated from the Kelvin equation according to Equation 1

$$\psi = -\frac{R \cdot T}{V_{w\theta} \cdot \omega_v} \ln(\frac{p}{p_s})$$

where

 ψ = suction (kPa)

T = absolute temperature (K)

R = universal gas constant (8.31432 J/(mol K))

 v_{w0} = specific volume of water (1/ ρ_w m³/kg)

 ρ_w = density of water (kg/m³)

 ω_v = molecular mass of water vapour (18 kg/kmol)

p = partial pressure of pore-water vapour (kPa)

 p_s = saturation pressure of water vapour over a flat surface of pure water of the same temperature (kPa)

After the measurements shown in Table 5-4 measurements were made above salt solutions with tabulated *RH* values (75% and 98% for the capacitive sensors and 98% and 99% for the psychrometers). The deviations between the measured values and the tabulated values were less than $\Delta RH < 0.1\%$ for the capacitive sensors and less than $\Delta RH < 0.2\%$ for the psychometers.

Table 5-4. Measured relative humidity above salt solutions.Average values of measurements by different sensors.

Salt solution	RH %	T °C	Suction kPa	Determined by
1% salt	99.3	19.0	930	psychrometers
3.5% salt	98.1	20.2	2,650	psychrometers and capacitive sensors

5.5 Time criteria

The two samples with the highest *RH* value in the jar (Asha jar H and Friedland jar 8) changed substantially in weight after about 2,500 h, having previously been close to an equilibrium state, cf. Figure 5-4. The Asha sample returned to its previous equilibrium mass after a few weeks, whereas the Friedland sample continuously decreased in weight – seemingly towards a different equilibrium state. After dismantling the samples, it was found that there were white crystals in the Friedland clay and holes had developed in the cage. There were no such findings in the Asha material and equipment.

The parameters, which were used to determine whether the specimens were in an equilibrium state, are presented in Table 5-5. At least one of the conditions was fulfilled in all samples. For the evaluation of Ω , the approximate initial relative humidity 8% and 70% were used for the Asha and Friedland materials, respectively.



Figure 5-4. Changes in mass with time for the two samples with the highest RH – Asha jar H and Friedland jar 8.

Table 5-5.	Equilibrium	-determinina	parameters	for the A	sha and	Friedland	materials
	-94	aotoning	paramotoro	101 110 / 1	ona ana		matorialo

Asha			Friedland						
Specimen	Ω (s ⁻¹)	100·(dw/dt) (%/100 h)	Specimen	Ω (s ⁻¹)	100·(dw/dt) (%/100 h)				
A	5.1E–10	2.3E-03	1	1.0E-09	3.8E-03				
В	-4.0E-10	-5.4E-03	2	-4.0E-09	-1.5E-02				
С	-3.8E-09	-2.8E-02	3	-2.3E-09	-8.9E-03				
D	1.4E–21	1.2E–14	4	-1.6E-09	-6.6E-03				
E	-5.5E-10	-5.6E-03	5	4.3E-09	1.3E-02				
EII	8.1E–10	8.2E-03	511	1.7E-09	7.3E-03				
F	-1.2E-09	-1.3E-02	6	7.6E-10	3.9E-03				
G	-5.3E-10	-6.9E-03	7	-2.9E-10	-2.6E-03				
Н	-2.6E-10	-4.6E-03	8	6.8E-10	1.2E-02				

5.6 Discussion

The results show that at absorption the three materials Asha, Friedland and MX-80 will reach different water contents at equilibrium with a specific relative humidity.

At each series made with the method with jars one specimen was made as a doublet specimen and the deviation was less than 0.2%, cf. Table 5-3. Uncertainties of the method with jars were commented on by /Wadsö et al. 2004/.The results from Figure 5-5 show that the dominating part of the retention curve determined with jars agree with the desorption part of the curve determined with the sorption balance. This corresponds with the fact that the initial water content of the material used in the jars was 7.8% and that less water content was reached after desorption.

6 Summary and conclusions

6.1 General

The tests described in this report are intended to investigate several important properties of different backfill candidate materials, considered for use in backfilling of tunnels in the KBS-3V concept. The investigations will be used to evaluate modeling parameters for some backfilling candidate materials regarding wetting and homogenization processes.

This chapter summarizes the main conclusions from these tests.

6.2 Homogenization tests

The materials used in this investigation have different properties regarding the behavior during water uptake and saturation reflected by the different appearances of the retention curves and mainly caused by differences in smectite content. When different materials are used together e.g. for backfilling of a deposition tunnel (backfill blocks made of one material and another material used for the pellets in the remaining slots) the different retention and mechanical properties imply that at otherwise similar conditions regarding load and suction different water contents and densities will be reached at equilibrium. During the saturation the low density part will be compressed by the swelling pressure from the high density part. All measurements of the swelling pressure have shown stabilization after about 2 weeks testing.

A compilation of the test results is provided below:

- *Influence of time.* The influence of time was tested by performing three identical tests with the same test layout and with three different test durations (2 weeks, 2 months and 4 months). The tests were performed with blocks made of Friedland clay and with a slot filled with Friedland granules (slot width = 8 mm). The tests showed that the main part of the homogenization seemed to take part during the first two weeks which was supported by the swelling pressure measurements. The differences between the results from these three tests were very small regarding water content and density distribution.
- *Influence of wall friction*. The influence of wall friction regarding the homogenization was tested by performing one test where the inner wall of the test cell was lubricated with "Molycote" (MOS2). It was not possible to detect any differences between this test and a similar test performed without any lubrication.
- *Influence of access to water*. In one test the specimen had access to water from both the pellets side and the block side. The only difference between this test and the standard type was that the water content was somewhat higher at the filter side of the block.
- *Influence of different slot widths.* The influence of the slot width regarding the homogenization rate was as expected rather large. In the performed test series, three tests with different slot widths (4, 8 and 12 mm width) were performed. The results showed that in the test with the 4 mm slot the time to completed homogenization had been considerable shorter.
- *Influence of salt content in the water.* Tests were performed using water with two salinities, 1% and 3.5% and with either MX-80 pellets or Friedland granules in the slot. The results showed that the influence of salt regarding the homogenization was very small.
- *Influence of material in the slot.* The difference in water content and density distribution after finishing the tests varied strongly depending on which material that was used in the slot. However there will always be remaining differences depending on the different material properties.

• *Influence of block material.* The two tested block materials, Friedland and Asha 230B, are very different regarding the smectite content. This results in that there is a major difference in the initial dry density of the tested block materials (Friedland=2,000 kg/m3 and Asha=1,620 kg/m3). This difference in initial density is one reason for the fact that after termination there was still large difference in dry density between the pellets filled slot and the block when using Friedland clay but when using blocks made of Asha 230B this difference was considerably lower.

6.3 Water uptake tests

- The water uptake tests show that the saturation rate of the backfill is much depending on the type of material.
- The water uptake rate of a material is much depending on the initial dry density.
- The water uptake rate is very little depending on the salinity of the water used for saturating the specimen.
- The water uptake tests will be used as "bench mark" tests for evaluating different models used for simulating the water uptake.

6.4 Retention curve

- Retention curves for the two backfill candidate materials Asha 230B and Friedland were determined.
- Relative humidity was measured above 1% and 3.5% salt solutions used for the saturation in sections 3 and 4.

6.5 Further work and recommendations

The process of homogenization and water uptake can be well described by the test types used in this investigation. However, in order to fully evaluate the results the tests need to be modelled. This report includes the behaviour of two backfill materials. If additional material will be of interest similar tests should be repeated on the new material. It could also be useful to do some parallel tests in order to study the repeatability of this kind of tests.

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Appendix A







Appendix B







Appendix C



Appendix D



Appendix E



Appendix F



Appendix G

	Specimen preparation								After finishing the tests					
	Block Pellets Block						Block+Pellets	Specimen (block and pellets)						
Specimen	Material	w initial	m	h	d	Material	w initial	m	h	Dry density*	m	h	d	Pellets slot**
		%	g	mm	mm		%	g	mm	g/cm ³	g	mm	mm	mm
F102	Friedland	10.19	167.01	39.92	49.38	MX-80	12.75	16.40	8	1.766	202.88	49.30	50.14	6.4
F105	Friedland	10.19	166.57	39.30	49.40	MX-80	12.75	16.40	8	1.785	201.95	48.75	50.10	7
F205	Friedland	10.19	168.09	40.10	49.40	Minelco	18.70	18.70	8	1.782	203.73	48.61	50.10	7.4
F302	Friedland	10.19	166.76	40.30	49.42	Friedland	6.95	17.60	8	1.770	203.10	48.94	50.15	7.5
F304	Friedland	10.19	166.83	39.74	49.42	Friedland	6.95	8.80	4	1.859	189.88	45.13	50.12	-
F305	Friedland	10.19	167.24	40.15	49.40	Friedland	6.95	17.60	8	1.780	205.36	49.15	50.12	6.1
F305 Mb	Friedland	10.19	167.42	40.05	49.40	Friedland	6.95	17.60	8	1.785	205.95	49.28	50.11	6
F305 2xwater	Friedland	10.19	166.57	40.00	49.40	Friedland	6.95	17.60	8	1.779	204.19	48.69	50.13	6
F305 4months	Friedland	10.19	166.71	40.11	49.40	Friedland	6.95	17.60	8	1.776	200.88	48.13	50.11	5
F305 2weeks	Friedland	10.19	166.97	39.88	49.40	Friedland	6.95	17.60	8	1.787	203.12	48.33	50.12	6
F306	Friedland	10.19	166.70	40.08	49.40	Friedland	6.95	26.40	12	1.721	214.39	52.25	50.12	8.2
F405	Friedland	10.19	166.97	39.90	49.40	Cebogel	19.16	17.60	8	1.769	203.19	48.93	50.10	6.5
A102	Asha	17.43	145.20	40.53	49.60	MX-80	12.75	16.40	8	1.451	***	***	***	7
A105	Asha	17.43	144.87	40.50	49.59	MX-80	12.75	16.40	8	1.449	***	***	***	7.4
A205	Asha	17.43	144.97	40.50	49.59	Minelco	18.70	17.60	8	1.452	***	***	***	7.5
A305	Asha	17.43	145.10	40.80	49.60	Friedland	6.95	17.60	8	1.462	191.15	49.51	50.10	6.7
A405	Asha	17.43	144.75	40.54	49.60	Cebogel	19.16	17.60	8	1.449	188.72	48.62	50.18	7.5

* Calculated average dry density
 ** Estimated width of the former slot. The accuaracy of this measurement is low.
 *** No measurements available