Shot-clay MX-80 bentonite: An assessment of the hydro-mechanical behaviour

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Abstract

This study presents the results of an experimental study conducted to characterise the hydro-mechanical behaviour of shot-clay MX-80 bentonite. In the shot-clay process, granular bentonite was mixed continuously with water and shot on the walls of a tunnel section at the Grimsel Underground Research Laboratory (URL), Switzerland. The shot-clay was placed to create a layer of bentonite in direct contact with the host rock to avoid preferential water and/or gas flow along the tunnel wall. Samples for an experimental programme were collected during the shooting. The index properties, microstructural features, swelling potential and water retention properties of the shot-clay were analysed. An experiment was then conducted under controlled total suction to analyse the hydro-mechanical behaviour of the material along a predefined stress path involving suction and confining stress variations. Based on the results of this test, the expected behaviour of the shot-clay bentonite when subjected to the environmental conditions in the repository was determined. The test results were compared with data on the observed behaviour of compacted MX-80 granular bentonite to assess the effects of the shot-clay emplacement technique on the behaviour of the MX-80 bentonite. The results highlight the role of the emplacement dry density on the behaviour of bentonite.

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1. Introduction

Engineered gas transport systems (EGTS) are used to limit gas overpressures in the backfilled underground structures of nuclear waste repositories to acceptable levels without compromising the radionuclide retention capacity of the engineered barrier system (EBS) (Nagra, 2008). The main design elements of an EGTS are (i) specially designed backfill materials for the emplacement caverns, characterised by high porosity and high compressive strength, and (ii) gas-permeable tunnel seals consisting of sand/bentonite (S/B) mixtures with bentonite contents of 20% to 30%.

The Gas-Permeable Seal Test (GAST) was initiated in 2010 and conducted at the Grimsel Test Site (GTS, Switzerland) in 2011 and 2012. An S/B seal with a length of 8 m and a diameter of 3 m was emplaced to demonstrate the effective functioning of gas-permeable seals at realistic scales and under realistic hydraulic boundary conditions ('proof of concept').

Technological gaps may generate preferential flow paths within the swollen S/B mixture (Wang et al., 2013). To avoid preferential water and/or gas flow along the tunnel wall, a bentonite ring with a target hydraulic conductivity of at least 1E–12 m/s, corresponding to a dry density of the bentonite of approximately 1.4 Mg/m³, had to be emplaced around the S/B seal. The first emplacement option assessed was "shot-claying", which involved mixing MX-80 granular bentonite with water (at a volumetric water-to-bentonite ratio of 3 to 11.7), feeding the mixture into a hydraulic pump and shooting the mixture onto the tunnel wall (Fig. 1). A shot-clay test was performed at the Grimsel URL as part of the pre-testing for the final experimental setup (Teodori et al., 2012).

Dixon et al. (2011) presented a comprehensive summary of granular and bentonite pellet installations to fill technological gaps in high-level nuclear waste repositories. This summary highlighted the difficulties related to the low angle of repose of dry pellets when poured and pellet damage that occurs when the material is blown in dry conditions. The summary by Dixon et al. (2011) also reported on wet-blowing installations, highlighting the ability of wet-blown bentonite to be placed on near-vertical surfaces and the lack of information on the hydro-mechanical behaviour of the emplaced material.

In the experiment discussed in the present study, after the layer of emplacing the bentonite on a tunnel wall, the layer was subjected to several hydro-mechanical processes, some of which resulted from the experimental emplacement and others of which were tested in the lab to assess their potential to optimise the final dry density of the
bentonite ring after full saturation. The laboratory tests primarily assessed the impact of a ventilation phase immediately following shot-claying and the compaction due to the emplacement of the S/B seal inside the bentonite ring. The sampling procedure at GTS is described in the next section. The material was characterised in terms of its index properties, microstructural features, swelling potential and water retention capacity. A comprehensive stress path was designed, and a controlled-suction test was carried out to assess the probable hydro-mechanical response of the material during the various expected phases of environmental conditions in the repository. The results of the experiments were compared with other data on the behaviour of compacted MX-80 granular bentonite to assess the effects of the shot-clay emplacement technique on the behaviour of MX-80 bentonite.

2. Tested material

2.1. Sampling procedure

Laboratory tests were carried out on material samples collected on site during the emplacement of the shot-clay. After the emplacement of the first bentonite layer, the shooting was suspended to allow the installation of several cylindrical steel tubes (with diameters of 9.5 cm and heights of 12 cm), that were pushed approximately 1 cm into the bentonite layer (Fig. 2.a). The mixture of bentonite and water was then shot directly into the sampling tubes (Fig. 2.b), and eventually, the cylinders were extracted, trimmed (Fig. 2.c), sealed and transported to the laboratory. Visual inspection of the shot-clay front on the tunnel surface suggested that good homogenisation of water and bentonite was obtained during the shooting. Examination of the trimmed core (Fig. 2.c) suggested that the bentonite grains were dry in their interiors and that water was absorbed by the finer particles and on the surfaces of the grains only.

2.2. Index properties

MX-80 bentonite was used for the shot-clay experiment. It contains 85% Na-smectite clay, which confers the well-known high swelling capacity, and has a specific area of 523 m²/g (Plötze and Weber, 2007). The index properties of the shot-clay MX-80 bentonite were determined in the laboratory. A specific gravity \( G_s = 2.74 \) was measured with a pycnometer, using Kerdane rather than water because its non-polarity prevents the development of diffuse double layers. A liquid limit \( w_l = 4.30 \) was measured using the fall cone penetration technique (Wood, 1985; BSI, 1990). A plastic limit \( w_p = 0.65 \) was determined in accordance with ASTM D4318. The average water content was 0.39 for the samples that were extracted immediately from the shot layer and sealed and 0.32 for the samples that were exposed to the site conditions for approximately 30 min before the tubes were sealed. Determination of the grain size distribution of the granular bentonite (apparent grain size distribution) was performed by sieving the bentonite grains at their hygroscopic water content; the analysis revealed a sand-sized fraction of 60% and a gravel-sized fraction of 30%.
The bulk density and the dry density were in the ranges of 1.58–1.63 Mg/m³ and 1.14–1.17 Mg/m³, respectively. The average void ratio was 1.4. Complementary density measurements were performed on samples cored out from the bentonite layer after the shot-clay was completed. The density values of these samples were in the same range of those obtained for the samples collected in the tubes installed before the shooting.

The initial total suction was measured using a chilled-mirror dew-point psychrometer (WP4C, Decagon Device Inc.) (Leong et al., 2003; Cardoso et al., 2007). The device measures the total suction using the psychrometric law, which relates the water potential of the material to the relative humidity established in the measuring chamber in which the specimen is placed. Initial total suction values of 4 MPa and 6 MPa were measured for the samples at water contents of 0.38 and 0.32, respectively.

2.3. Microstructural features

Mercury intrusion porosimetry (MIP) was used to determine the pore size density (PSD) function of the shot-clay. A cubic specimen approximately 5 mm in size was trimmed from one of the collected samples and freeze-dried to remove the pore water. The porosimetry test was carried out using a Thermo Electron Corporation porosimeter. The maximum applied intrusion pressure was 400 MPa (corresponding to an entrance pore size diameter of approximately 4 nm). The pressure was changed incrementally, and the intruded volume was measured continuously. The compressibility of the various components of the equipment was considered in the analysis.

Fig. 3 shows the cumulative intrusion void ratio (eHG) and the pore size density function (PSD = −ΔeHG / Δ(log d)) versus the entrance pore diameter (d). The difference between the cumulative void ratio and the void ratio measured on the samples (0.45) is associated with the inter-lamellar bentonite porosity that cannot be accessed by the mercury and with the largest pores on the sample surface being filled by the mercury before the intrusion process starts. A multimodal pore size density function was obtained and is shown in Fig. 3.b. A peak with a modal value of approximately 20 nm is observed. This peak is commonly observed in compacted granular bentonites and is associated with the intra-aggregate porosity, i.e. the pores between the bentonite particles within the aggregates (e.g., Romero et al., 2011). Pore sizes in the range of 4 × 10⁻² to 10 μm correspond to the swollen intra-aggregate pores in assemblages that were hydrated during the shot-clay process. Pore sizes larger than 10 μm correspond to inter-bentonite-assemblage pores and represent the macro-pores in the shot-clay bentonite.

3. Experimental programme

The behaviour of the shot-clay MX-80 bentonite was assessed through an intense laboratory campaign. The aim of the tests was to analyse the response of the material to the environmental conditions expected in the repository from emplacement through full saturation. The material’s swelling potential, water retention behaviour and volumetric response under hydro-mechanical loading were analysed. All of the tests were performed on specimens cored out from the tube samples collected on site. The initial characteristics of the tested specimens in the various experiments are listed in Table 1. Distilled water was used in all of the experiments. All of the tests were run in a temperature-controlled laboratory (22 ± 0.5 °C). Details of the testing procedures are given in the following sections.

3.1. Swelling behaviour

To evaluate the swelling potential of the shot-clay MX-80 bentonite, constrained and free swelling tests were performed on specimens 1.88 cm in height and 7.53 cm in diameter. Each specimen was placed in a rigid-walled steel cell, resting on a porous disc connected to a water inlet. The upper part of the specimen was in contact with a coarse porous stone, and a flushing line was connected to the upper part of the cell. The swelling pressure was measured by inserting the cell containing the specimen into a rigid frame to ensure constant-volume conditions. To capture any swelling associated with the homogenisation of the

<table>
<thead>
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<th>Test</th>
<th>Water content</th>
<th>Void ratio</th>
<th>Degree of saturation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Constrained swelling</td>
<td>0.380</td>
<td>1.38</td>
<td>0.75</td>
</tr>
<tr>
<td>Free swelling</td>
<td>0.319</td>
<td>1.38</td>
<td>0.63</td>
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<td>Water retention</td>
<td>0.390</td>
<td>1.43</td>
<td>0.75</td>
</tr>
<tr>
<td>Controlled-suction hydro-mechanical test</td>
<td>0.320</td>
<td>1.38</td>
<td>0.63</td>
</tr>
</tbody>
</table>

Table 1: Initial characteristics of the tested shot-clay specimens in the experimental activities.

Fig. 4: Schematic layout of the experimental setup for the controlled hydro-mechanical test.
water content in the material after the shooting, the specimen was taken to the laboratory and placed in the test apparatus as soon as possible (approximately 2.5 h after the shooting time). In this phase, no access to water was provided. A load cell (with an accuracy of 10 kPa with respect to the area of the specimen) was placed between the top cap and the frame to measure the swelling pressure. An initial total vertical stress of 80 kPa was applied to ensure good contact of the various parts of the apparatus. The specimen was then inundated with water, and the swelling pressure was continuously monitored until it stabilised.

A free swelling test was carried out by saturating the specimen and measuring the vertical heave with an LVDT system. A constant pore swelling pressure was continuously monitored until it stabilised. A stress of 5 kPa was applied to ensure the evacuation of air from the top of the cell during the rising of the wetting front.

3.2. Water retention behaviour

The water retention curve for the main drying path was analysed using a controlled air drying process in which the total suction was measured with a chilled-mirror dew-point psychrometer. Several specimens were cored from one sample using a special holder that could be positioned directly in the device for the suction measurement. After the initial water content and total suction of the sample were determined, a drying path was initiated in each specimen by controlled air drying at the ambient laboratory conditions (T = 22 °C, RH = 40%). The evolution of the water content was monitored for each specimen by continuously weighing the sample holder. The air-drying process was stopped once each specimen reached its target water content value. The sample holder was then sealed, and the sample was cured for 2 days to ensure good distribution of the water within the specimen. The total suction of the specimen was then measured using the WP4C device. After the suction was measured, the volume of the specimen was measured, and the water content was determined by oven-drying.

3.3. Controlled-suction hydro-mechanical test

A controlled-suction hydro-mechanical test was carried out to measure the volume change and water content variation in the material over the range of environmental conditions expected in the repository. A schematic diagram of the experimental arrangement is shown in Fig. 4. The cell holds a sample with a diameter of 7.53 cm and a height of 1.88 cm. The vertical load is controlled by a level arm system. The applied vertical stress is assumed to be representative of the radial stress of the shot-clay layer in the tunnel. The vertical displacement is measured by an LVDT installed on the top of the cell. The computed vertical strains in the cell are equivalent to the radial strains of the shot-clay in the tunnel. The total suction is controlled using the vapour equilibrium technique. The pore water potential of the material is represented by the migration of water molecules through the vapour phase, relative to a reference system of a known water potential, until equilibrium is achieved (Romero, 2001). At the end of the equalisation phase, the pore water and the vapour phase have the same potential. Through this equality and by assuming that the water vapour obeys the ideal gas law, it is possible to express the total suction of the material as a function of the relative humidity of the closed system in which the specimen is placed, according to the psychrometric law. The relative humidity (RH) and the absolute temperature (T) are related to the total suction (ψ) through the following expression (Fredlund and Rahardjo, 1993):

$$\psi = -\frac{\rho_w \cdot R \cdot T}{M_w} \cdot \ln(\text{RH})$$

where R is the universal gas constant and $\rho_w$, and $M_w$ are the density and the molecular mass of water, respectively.

Variably saturated NaCl solutions and a saturated Mg(NO₃) solution were used to control the relative humidity and hence the total suction of the reference system. Romero (2001) and Witteveen et al. (2013) provided the relationship between the solution concentration and the applied suction. A pump was used to force a humid air flow through the pores of the specimen to reduce the time required to reach equilibrium (Pintado et al., 2009).

Fig. 5. Stress path implemented for the investigation of the hydro-mechanical behaviour of the shot-clay MX-80 bentonite.

Fig. 6. Results of the swelling tests: swelling pressure evolution vs. time under constant volume conditions (a); swelling heave and water content evolution under free swelling conditions (b).
The applied stress path was based on the range of environmental conditions that the shot-clay material is expected to experience in the evolution of the repository. The following steps were applied and are depicted in Fig. 5:

1) In the initial state (point A), the specimen was placed in the cell and equalised to a total suction of 6 MPa using a 1.3 M NaCl solution. This suction was the value measured in the sample immediately following the shooting (Section 2.2). A vertical stress of 8 kPa was applied to ensure good contact between the specimen and the loading system.

2) The vertical load was increased to 67 kPa to simulate an initial compaction of the material (path A-B) to reproduce the emplacement of the buffer material (such as bentonite blocks) in the repository.

3) A drying path (B-C) was applied to reflect the ventilation phase of the repository, during which the material will be exposed to the relative humidity of the tunnel. A suction of 87 MPa was applied using a saturated Mg(NO₃) solution, corresponding to a relative humidity of 50% at the laboratory temperature, which was the average relative humidity at the Grimsel URL.

4) A loading path (C-D) was then applied increasing the vertical stress to 249 kPa at a constant total suction of 87 MPa. This phase is intended to reproduce the development of the swelling pressure in the buffer material when it is wetted through the vapour phase. This adsorption is justified when considering that the buffer material is usually emplaced at its hygroscopic water content (e.g. 5% for the granular MX-80 bentonite at RH = 34%, corresponding to a total suction of 148 MPa) and exposed to the relative humidity in the tunnel.

5) The path D-E reflected the wetting of the material upon water vapour uptake from the surrounding host rock. The wetting was performed using a saturated NaCl solution, resulting in a total suction of 37 MPa (corresponding to a relative humidity of 75%). During this phase, the volume was kept constant to reflect the presence of the buffer material. The axial strain was limited to the range of ±0.25% by increasing the vertical stress in small steps, to prevent swelling of the material. This process was repeated until the specimen did not exhibit any further tendency to swell.

6) Lastly, the specimen was flooded with distilled water (path E-F) to measure the maximum swelling potential associated with the full saturation of the material. To accomplish this operation, the cell was removed from the loading system and installed in a highly rigid frame equipped with a load cell. An initial vertical stress equal to the swelling pressure obtained at the end of path D-E was applied.

To better observe the material behaviour during the application of the stress path described above, the cell was opened for visual observation at the ends of steps B-C, D-E and E-F. This operation was performed quickly to minimise disturbance of the test. After each inspection, the conditions of suction and vertical stress at the end of the imposed step were re-stabilised before the following step was applied. No vertical strain or water content change was observed during this equalisation phase, confirming that the opening of the cell did not cause disturbance of the test procedure.

4. Test results

4.1. Swelling potential of the as-shot material

The results of the constrained swelling test are shown in Fig. 6.a, in which the measured swelling pressure is plotted with respect to time. The initial homogenisation of the water content after the shooting did not produce any appreciable swelling pressure. A maximum swelling pressure of 990 kPa was observed 10 days after the cell was flooded under constant-volume conditions.

The result of the free swelling test, in terms of the axial strain and water content evolution over time, is shown in Fig. 6.b. A final swelling strain of 324% and a final water content of 280% (corresponding to a calculated degree of saturation of 99%) were reached after approximately 64 days.

4.2. Water retention behaviour

The water retention behaviour of shot-clay MX-80 bentonite along a drying path is depicted in Fig. 7 in terms of the void ratio, water content

![Void ratio vs. suction for drying path in shot-clay MX-80 bentonite material (a); water retention behaviour of shot-clay MX-80 bentonite in terms of water content (b), and degree of saturation (c).](image-url)
and degree of saturation versus the total suction. The initial conditions measured on the whole sample from which the specimens were extracted are also reported. The measured void ratio of the specimens was initially higher than the sample void ratio. This difference is attributable to the rebound of the material during the specimen extraction. This observation seems to be confirmed by the change in slope in Fig. 7.a once the void ratio of the sample was reached during the drying process. A residual degree of saturation of approximately 40% was obtained.

4.3. Controlled suction hydro-mechanical behaviour

The initial characteristics of the tested specimen are reported in Table 1. The evolution of the water content and axial strain over time

![Fig. 8. Variation of the water content and axial strain of the specimen during the different steps of the applied stress path shown in Fig. 5.](image)

![Fig. 9. Results of the controlled-suction hydro-mechanical test.](image)
for the different steps of the imposed stress path are shown in Fig. 8. Fig. 9 depicts the equilibrium states in the planes of total suction vs. vertical stress, axial strain vs. vertical stress, total suction vs. water content and axial strain vs. water content. During the initial equalisation phase (A), no significant deformation of the specimen was recorded, and only limited wetting was observed. This wetting is attributed to initial drying of the specimen during the cell installation. The water content reached (34%) is consistent with the water retention curve shown in Fig. 7.b. The loading path (A–B) at constant total suction caused an axial strain of approximately 1% without any expulsion of water. The following drying step (B–C) took 33 days to reach equilibrium and resulted in significant shrinkage of the material (9%) and a reduction of the water content to 14.2%, consistent with the water retention curve shown in Fig. 7.b. At this stage, the cell was opened to assess the loss of lateral contact with the oedometric ring and the formation of drying cracks. Lateral shrinkage of approximately 4% in radial strain was calculated by analysing the collected images (Fig. 10.a). Interestingly, the creation of drying cracks was also observed on site, three to four days after the shooting. A loading path was applied by increasing the vertical stress to 249 kPa (C–D) while maintaining constant total suction. As a result, the axial strain increased up to 11% while the water content increased slightly. The wetting path D–E, which was applied using an unsaturated NaCl solution in the vapour phase, resulted in an increase of the water content to approximately 0.20, while the variation in the axial strain was maintained in the range of ±0.25%. A swelling pressure of 495 kPa was measured during that intermediate saturation step (Fig. 11.a). The cell was also opened for visual observation after this path. As Fig. 10.b shows, the specimen swelled due to water vapour uptake, reversing the lateral shrinkage around the perimeter that occurred during the drying procedure along path B–C. The cracks observed earlier in the centre of the sample were only partially healed and were still clearly observed.

The flooding of the specimen (path E–F) at constant volume led to the development of a final swelling pressure of 1250 kPa after approximately 6 days (Fig. 11.b). The cell was finally dismantled and the full homogenisation of the specimen was observed due to saturation (Fig. 10.c). A final water content of 0.41 (corresponding to a computed final degree of saturation of 99%) was measured.

5. Discussion of test results

Some conclusions concerning the effects of the emplacement technique on the material behaviour can be drawn from the experimental results obtained. The experimental results were compared with the results of complementary tests performed on compacted samples of granular MX-80 bentonite with a similar apparent grain size distribution, prepared at various dry densities (Seiphoori, 2014).

The dry density achieved by the shot-claying procedure (approx. 1.15 Mg/m³) was similar to the density that would be obtained if granular bentonite were simply poured on the ground (approximately 1.2 Mg/m³) but lower than the one that would be obtained using a granular material optimised to a Fuller distribution (1.4–1.5 Mg/m³), as is foreseen for the use of granular bentonite as a buffer material, in accordance with the Swiss concept of high-level nuclear waste disposal (Plötze and Weber, 2007). The lower dry density achieved by the shot-claying procedure is attributed to the fast swelling of the bentonite grains during mixing with water in the pump system, and it is strongly dependent on the initial properties of the material and the amount of water added during shot-claying.

![Fig. 11. Swelling pressure development during the wetting phase: step D–E (a) and step E–F (b) in Fig. 5.](image-url)
To compare the micro-structural features associated with the different emplacement techniques, the results of an MIP test (in terms of pore size distribution) on a poured granular bentonite sample (with a dry density of 1.5 Mg/m$^3$) was compared to the PSD of the as-shot bentonite (Fig. 12). The comparison confirmed that the bentonite grains swell during mixing with water. This swelling resulted in the creation of larger void spaces particularly in the pore size range attributed to inter-bentonite-grain pores. This structure also resulted in a lower dry density for the shot-clay. Evolution with time of the microstructural features of the shot-clay bentonite may be expected as a result of ageing effects (Delage et al., 2006).

The initial low dry density and high water content after shooting were also responsible for the reduced swelling pressure of the bentonite of approximately 1 MPa. In Fig. 13, the swelling pressure measured in this study is compared with the results of swelling pressure tests carried out on granular bentonite compacted at various dry densities (Seiphoori, 2014). Interestingly, the swelling pressure for the shot-clay bentonite fits the general exponential trend for the compacted material well. The results of this comparison suggest that the result of the emplacement technique on the behaviour of the material could be assessed directly by considering the dry density achieved in the shooting.

This tendency also seems to be confirmed by the results of the analysis of the water retention properties of the materials. The water retention behaviour of the shot-clay and the compacted MX-80 bentonite at different initial dry densities along drying paths are compared in Fig. 14. The water retention behaviour observed in the controlled-suction oedometric test (point C) is also represented in this plot. The water retention properties of the samples prepared by different emplacement techniques follow a unique trend for a wide range of total suction values, despite their different initial dry densities. This unique trend between the total suction and the water content is valid for the domain governed by the adsorptive storage mechanism, according to which water is retained in the intra-aggregate pores (Salager et al., 2013). The effect of the emplacement technique can be observed in the reduced air entry value for the shot-clay bentonite. This is a direct consequence of the lower initial dry density.

The controlled-suction test provided information on the hydro-mechanical behaviour of the material under various loading conditions. The most severe changes in strain and water content were associated with drying during the ventilation phase. The test also highlighted the tendency of the material to crack during the drying step at a relative humidity of 50%, which is typical for GTS in the winter. The shot-clay bentonite showed, however, good self-sealing properties once full saturation was attained. The formation of drying cracks at the imposed relative humidity could be a consequence of the low emplacement dry density (Kleppe and Olson, 1985; Péron et al., 2009).

6. Conclusions

Based on the above discussion, it can be concluded that the shot-clay emplacement technique does not change the overall behaviour of the granular MX-80 bentonite, except that the low initial dry density leads to reduced swelling capacity and a reduced air entry value and an increasing tendency to form drying cracks, compared to the compacted MX-80 bentonite. The dry density achieved with the shot-clay technique was lower than the target value. In order to reach a higher dry density, the method could be improved by optimising the original granular bentonite material and reducing the amount of water added during shot-claying (Koch, 2002; Teodori et al., 2012). The shot-clay emplacement technique was capable of providing a homogeneous layer of bentonite along the entire section of the tunnel; such layer would not be realizable by simply pouring the granular bentonite. The laboratory test results show that the final density (after full saturation under constrained conditions) can be improved considerably by ventilating the tunnel at 50% RH, but the results also show that post-compaction of the material has little impact on the final density.
Overall, the shot-clay technique has high potential to achieve the final dry density of 1.4 Mg/m³ in MX-80 bentonite, which was the target density in this experiment. However, the shot-clay application of the bentonite on the tunnel surface, which permits free swelling, may be more limited than the application into a closed volume, as performed by Dixon et al. (2011), because in that case, the early swelling of the material is limited by the confined volume of the backfilled void space.

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References


