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# Modeling of saturation and swelling effects in clays under different saline conditions

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## Abstract

For the safety assessment of underground repositories predictive models are needed which can describe the process of infiltration and swelling pressure in compacted bentonites used as buffer and sealing materials. The infiltration can be well described for laboratory scale experiments by the Second Fickian Law. However, there is no proof yet, that application of this law is permissible when its useage is extended to the large scale of a repository with much more complex boundary conditions. Different models exist for the prediction of the swelling pressure of bentonites in contact with water and saline solutions. All of them are applicable only to systems with homoionic bentonite in contact with pure water or NaCl solution. None of these models is applicable to mixed salt solutions at ionic strengths as they are encountered naturally in repositories in salt formations. A pragmatic GRS approach for the solving of this problem is described in this paper. A three years GRS research programme on this subject has revealed that the main factors influencing the swelling pressure of the bentonite-brine system are the brine composition, the density of the compacted bentonite and the hydrostatic pressure. The boundary conditions which have to be observed are discussed and the principle of the new approach is described.

## Introduction

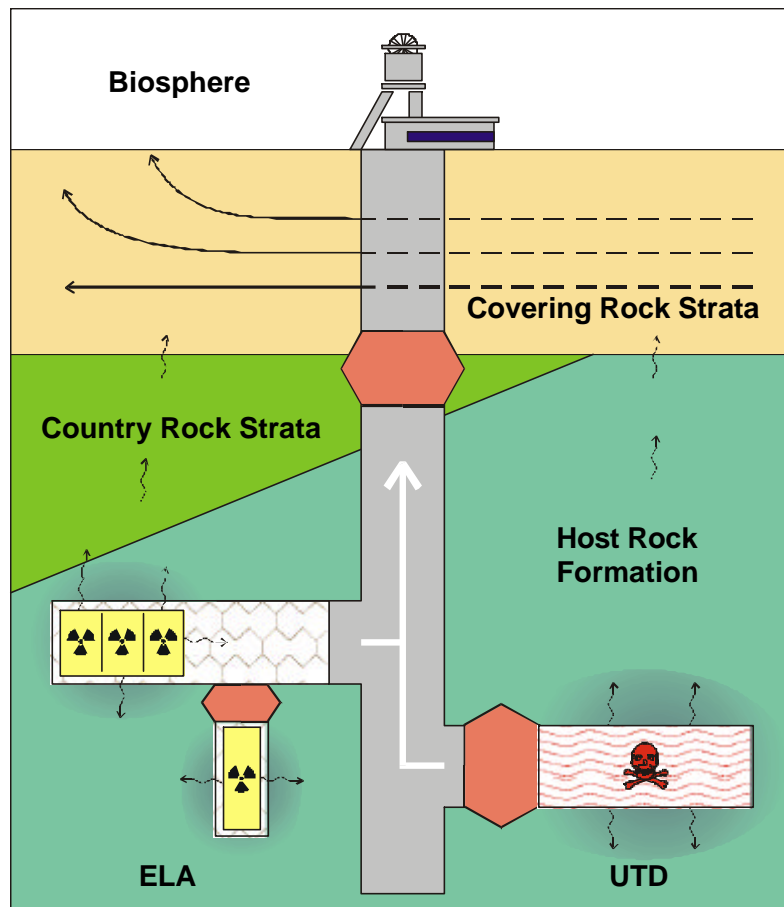
In the multibarrier system of repositories for radioactive and chemical wastes (figure 1) compacted bentonites may be used as construction materials for the technical barriers. Bentonites are considered to be ideal sealing and backfilling materials because of their high swelling capacity. For the safety assessment of a repository the prediction of the long term behaviour of compacted clays is required. Extensive experimental data are the basis for existing predictive models which are used for the description of infiltration and swelling pressure in compacted bentonites. These models are able to reproduce the experimental data fairly well as long as the solutions which are causing the swelling have low salinities.

The host rocks of German repositories for radioactive and hazardous wastes, however, are salt formations where very high salinities with complex composition of the brines must be considered. This fact poses certain problems concerning the use of bentonites. Salt solutions tend to reduce the swelling capacity, and no swelling pressure will develop if the capacity is reduced to an extent where the actual volume is not filled by the maximum swollen clay. For very high salt contents the existing models are not able to predict or reproduce the experimental results. In order to either extend existing models or to create a new model the governing parameters for these processes must be identified.

## Boundary conditions in a repository in salt formations

It is not necessary to predict a very large variety of chemical and physical conditions in the clay-salt solution system. For practical purposes it is enough if the infiltration and the resulting swelling

pressures can be predicted for the relevant range of expected repository conditions. These conditions are:



**Figure 1: Multibarriersystem in underground repositories for radioactive wastes (ELA) and hazardous wastes (UTD); compacted clays may be used as are part of the technical barriers i.e. sealing, backfilling and buffer materials**

- a dry density of the compacted bentonite seal element of  $1.6 \text{ g/cm}^3$ ,
- brine compositions varying in molities between 0 – 6,1 (Na), 0 – 1,6 (K), 0 – 4,8 (Mg), 0 – 0,006 (Ca), 6,1 – 9,2 (Cl), and 0 – 0,9 (SO<sub>4</sub>)
- hydrostatic pressure of 100 bars. This pressure corresponds to a brine-filled repository in 800 m depth.

## Modeling

The results of a literature review and a short description of the models in the light of the actual problem is given below.

### Infiltration

#### *Basic concepts*

The term "infiltration" stems from soil science and denotes the migration of water in soil following the gravitational field. Infiltration in soil may be described in space and time following mathematical expressions for advection and dispersion. One key parameter for such systems is the pore size distribution as a measure of how conductive is the matrix for fluid transfer. Usually such a spot of soil

may be envisaged as system of partially interconnected pores, woven into a structure of solid particles of various sizes.

Depending on the mean pore size, advection eventually becomes unimportant, leaving diffusion as the dominant process for the migration of water or solutes. At low particle sizes, clay minerals become enriched. Due to the lamellar constitution of aluminosilicates, an understanding of the migration of water in clay matrices can not be won by simply extending concepts of flow modeling through more coarse structures. This is because the high specific surface of clays in conjunction with their hydrophilicity and the hydration of adsorbed cations add to the process of diffusion. Unlike to structures which are (ideally) built up by sand grains and whose pore space is not altered by the process of infiltration, clayey soils are changed by the contact with solution.

With compacted bentonites we end up in a situation where the structure of pore space (and hence the conductivity for water) is not constant in time and may change in response to the composition of solution. Further, by the swelling of clay particles upon the hydration of their surfaces, hitherto open pathways for solution close.

For the development of a model for infiltration the following steps may be of help. Imagine dry, compacted bentonite enclosed in fixed walls permeable only for water. Within the bentonite we have a total pore space of  $V_t = V_p$ . The first increment of water will strive for the stablest bound possible, which will lead it inevitably into the hydration sphere of interlayer cations. This is to say that the first water entering the clay barrier becomes immobile as far as we restrict mobility to advection. The process of hydration will go along as long as there are unhydrated cations left (or, in terms of thermodynamics, until the chemical potential of interlayer water equals that of remaining pore water). Simultaneously, the pore space accessible for liquid water reduces in response to the swelling of the clay. It is only after the immobile fraction of total pore volume is filled that additional water may protrude further into the clay barrier. The pore space left for advection is the total pore space minus the "immobile volume",  $V_p = V_t - V_{im}$ .

Depending on the initial dry density of the compacted bentonite the remaining volume for "mobile" water may be diminished to a measure that virtually no advection takes place anymore and any further distribution of water into the barrier is exclusively due to diffusion and capillary forces. Also water transport in the vapor phase will be diffusive.

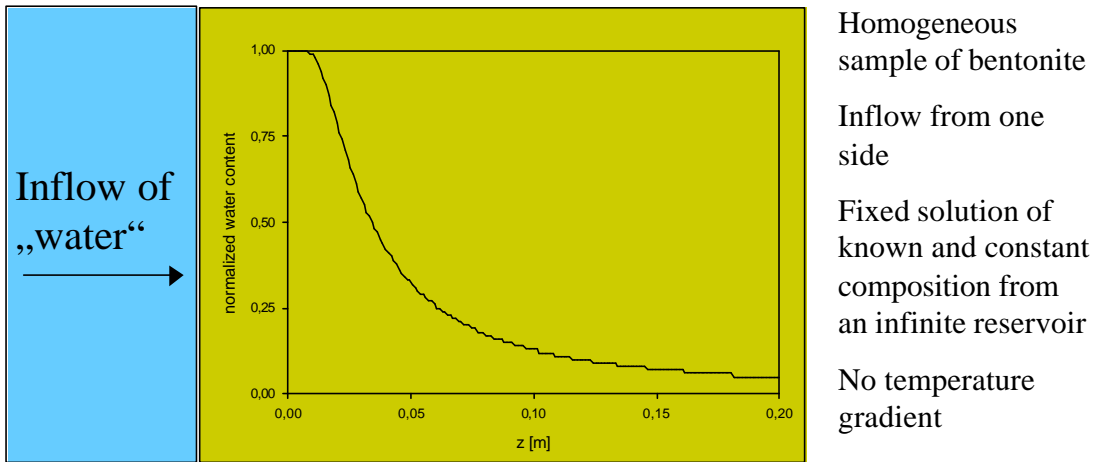
### *Modeling*

Considering the processes in the preceding section it is not surprising, that a description of infiltration into cylindrically shaped samples of compacted bentonites with the Second Fickian Law was successful in many cases (e. g. [i], [ii], [iii]). It may also be understood that the Second Fickian Law fails to describe experiments with samples with a content of clay as low as 10% [iv]. Now, as the clay content in engineered barrier systems are clearly higher, we could be inclined to lean back and say: "The problem is solved." Is it really ?

### *The real case*

Comparison of figure 2 and 3 reveals that with respect to performance assessment a lot more aspects need to be taken into account.

## Infiltration ... in column-experiments

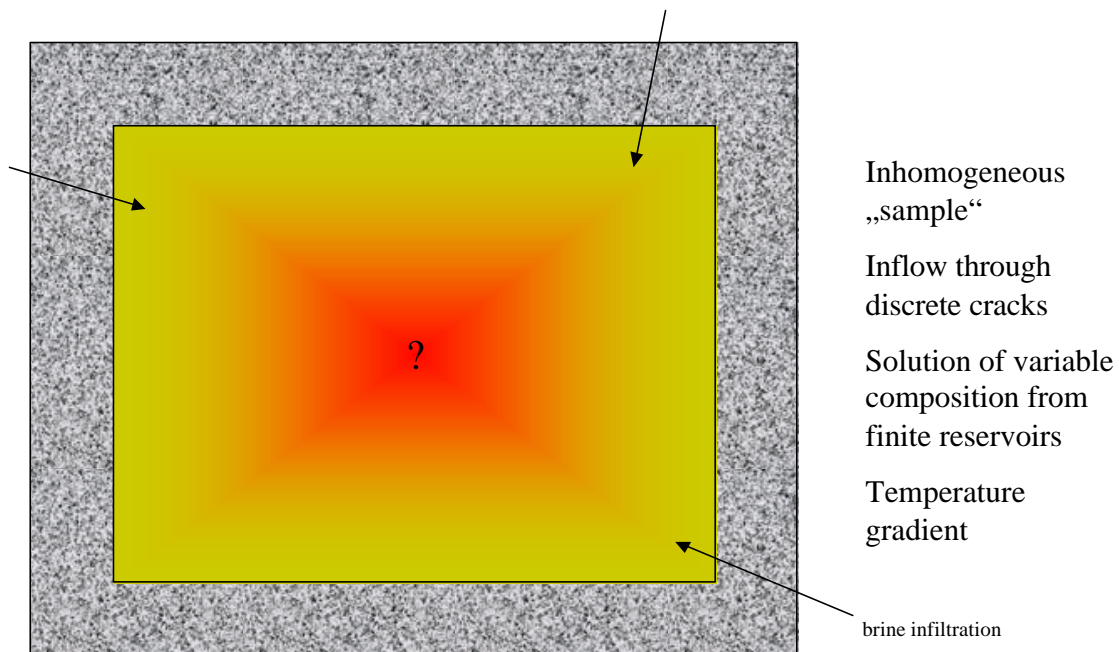


—————> Description by the 2. Fickian Law

**Figure 2: Schematic view of a column experiment for the infiltration of water into compacted bentonite (laboratory conditions)**

The Second Fickian Law is a partial differential equation whose solution is subject to strict boundary conditions. In the case shown we have an infinite reservoir of solution, constant in composition. The process is 1-dimensional and the investigated sample is homogeneous with respect to dry density and composition. No preferential pathways are open and temperature gradients are absent.

## Infiltration ... in reality



—————> THM-Modeling

**Figure 3: Schematic view on in-situ conditions influencing the infiltration of water into compacted bentonite**

In the real case (figure 3) we note that solution may not enter from a single point (or plane) but through discrete cracks in the surrounding host rock. If the connection between these cracks and other solution filled reservoirs in the host rock offers less conductivity than the compacted bentonite, then the volume of solution standing in line for infiltration can be said to be limited, which puts the first mentioned boundary condition into question (which may be changed, of course, giving rise to other solutions for the Second Fickian Law).

Secondly, the shear size of a 1:1 "model" of a real repository makes it probable, that the condition of homogeneity of the buffer is difficult to fulfill. Other "real case" factors may be changing solution composition and temperature gradients within the backfill. This may have a tremendous impact on the solution composition which eventually will reach the high-level waste canister: Villar et al. found that with decreasing distance to heaters built into compacted bentonite, the salinity of pore solutions increased [v].

Clearly, as the observed system become more complex, it is necessary to consider a multitude of coupled processes. Considerable efforts were spent to accomplish the task of "thermo-hydraulic mechanical modeling". On the experimental part it is to be mentioned that a large scale in-situ experiment which can be used for the checking of the applicability of the model is running since several months in the salt mine Sondershausen in Thuringia in Eastern Germany. In a drift of the abandoned potash mine a dam was constructed with a sealing element of compacted bentonite. Another example which covers both experimental and modeling aspects is the DECOVALEX II project. The project aimed to conduct a 1:1-scale experiment with electrically-driven heaters placed in boreholes and surrounded by compacted bentonite. For more detailed information we refer to the Technical Report [vi]. Four models were applied to the calculation of the temperature evolution in buffer and host rock, water content and total pressure in the buffer, pore pressure in the rock and vertical displacement of the containment. Up to 29 parameters were needed to do the various calculations.

#### *Open questions*

Of course there were many questions left at the end of the report, and there is no use to repeat them all at this place. We would like to direct attention to the following aspects:

Even for small lab-experiments covering the effect of temperature – and these experiments do not pose the fundamental problem of irreproducibility like in-situ experiments – it is not possible to state

- how much water enters the bentonite in time,
- which solution arrives at the canister, leading to corrosion, and
- which swelling pressure develops.

All given aspects in mind it must remain questionable whether full water saturation of the entire clay barrier is attained at all. But even then, canister material will still be subject to enhanced corrosion due to higher salinities of adjacent solution.

#### **Swelling pressure**

One key parameter is the swelling pressure which develops when compacted bentonite is reacted with aqueous solution. This is, because it has impact on the time necessary for any solution to reach the canister, and the migration of radionuclides released from therein. While the processes responsible for the evolution of swelling pressure are well known in principle, it has not been accomplished yet to create a mathematical link between solution composition and swelling pressure.

Striving for a means to calculate swelling pressure diverse approaches were applied which may be subdivided into three groups:

- empirical models,
- double layer models, and
- thermodynamic models

#### *Empirical models*

Philip Low proposed an empirical model of the form

$$\ln(P_s + 1) = \alpha \frac{m_c}{m_w} + \ln B \quad [1]$$

where  $P_s$  = swelling pressure,  $m_c$  and  $m_w$  are masses of clay and water, respectively, and  $\alpha$  and  $B$  are adjustable parameters [vii]. Direct measurement of the swelling pressure on 35 different montmorillonites showed a relationship between  $P_s$  and the mass ratio of montmorillonite to water  $m_w/m_w$ .  $\alpha$  and  $B$  are constants that are characteristic of the montmorillonite and probably of the solution applied. That is to say, the actual impact of solution composition on the swelling pressure is "hidden" in the empirical parameters. Further, this model does not take into consideration the degree of compaction of the montmorillonite. This equation somehow resembles an expression used by engineers:

$$\log P_s = -\frac{A_0}{r_{red}} + B_0 \quad [2]$$

with  $r_{red}$  being the reduced dry density and  $A_0$ ,  $B_0$  as adjustable parameters. The latter must be determined for each solution, and as yet no clear relation has been established to solution composition.

### *Double Layer Models*

There are several similar theories concerning the distribution of ions between charged surfaces which may generally be identified as double-layer models. The diffusive double layer model was independently developed by Guy and Chapman 1919. The purpose of the model, with respect to clay swelling, is to determine the mid point electrical potential between two parallel montmorillonite clay flakes. If the potential is properly determined for a certain physico-chemical condition it should in principle be possible to calculate the repulsive forces arising between the flakes.

Komine and Ogata (1996) calculated the swelling pressure on the basis of the DDL-Theory and compared the results with laboratory data for different ion concentrations [viii]. This Model is not applicable either to high salinities. It doesn't work for other ions in solution than Na. Saturated NaCl solution (6.1 M) gives total loss of swelling pressure for all clay densities, which is in clear contradiction to experimental data obtained during the last three years in different research institutes in Germany. It should further be mentioned that a total of 11 parameters were necessary for the model to work, not included "trivial" constants like Avogadro's number and the like. The concentration of ions in pore water is one of the not trivial values needed.

Double layer models not only suffer from the presence of other ions than sodium and chloride. Even more intricate problems are associated with the simple fact that in compacted bentonite we face a whole distribution of particle separation distances, which under the circumstances given may be well below 10 Å. This is to say that the double layer for which the theory is designed has no chance to extend undisturbedly into pore space. The very small dimensions therein pose yet another problem: what is the concentration of any ion in the pore space? Depending on the size of the pores it clearly will not be the same as in a hypothetical bulk solution in equilibrium with the same cations attached to the clay surface. The theory of electrical double layers gives us the notion of the sorbed cations arranged within a diffuse swarm in the vicinity of the clay surface. When the mean diameter of the pores in the compacted bentonite become smaller than twice the value of the thickness of the double layer the question arises whether we have any pore water at all - or adsorbed water. Within the layer of adsorbed water the content with regard to cations is different compared to that in the bulk solution. For further theoretical treatment it becomes necessary to define precisely which part of the cations belong to the clay surface and are therefore considered as being adsorbed, and which part belongs to the bulk solution. For example, if we state that the volume made up by the total external surface of the bentonite and the mean thickness of the electrical double layer represents the "surface volume" of the system, only that fraction of total pore volume which exceeds the surface volume may be addressed as "bulk phase" or "pore solution" with some justification. The dependency of expelled "pore water" composition from the degree of compaction has been shown by Muurinen and Lehikoinen [ix].

### *Thermodynamic Models*

The approach of equilibrium thermodynamics is in a sense opposed to the previous one as it doesn't require any internal parameter of the clay-water system to be known, but entirely depends on those variables of state which are truly in equilibrium. Thermodynamics with regard to swelling pressure takes refuge to macroscopic quantities, directly accessible to laboratory measurements and control [x]. This is

paid for in that the thermodynamic approach gives no answer as to the internal mechanism of swelling. Further, any calculations following the thermodynamic approach are only valid to the extent that non-equilibrium factors are not likely to be important in the respective system. Recalling own experiments with compacted clays and considering a certain degree of irreversibility attached to some processes with clay we regard this condition as questionable.

Spósito proposed for the swelling pressure

$$P_s = -\frac{\Delta m_w}{\bar{v}_w} \quad [3]$$

where  $\Delta m_w$  is the change in water chemical potential when a unit mass of water is transferred from pure liquid water into the clay paste, and  $\bar{v}_w$  is the partial molar volume of water at constant T, P and mass of clay. Low and Anderson suggested an analogy between swelling pressure and the osmotic pressure of the solution in equilibrium with the clay [xi]. Further development of equation [3] leads to the relation [xii]:

$$P_s = -\frac{RT}{M_w \bar{v}_w} \ln \frac{p}{p_0} \quad [4]$$

where  $M_w$  is the molar weight of water and  $p, p_0$  are the vapor pressure of adsorbed and pure water, respectively. In practice, water adsorption and desorption isotherms need to be recorded to gain the vapor pressure of adsorbed water for any water content of the clay. Reversely, knowing the water content, the resulting swelling pressure can be calculated with equation [4].

In principle, equation [4] may also be applied to clays equilibrated with a pure binary electrolyte solution. Then  $p_0$  in equation [4] is replaced by the vapor pressure of the electrolyte solution, which is smaller than that of pure water. In accordance with experimental findings this leads to a reduction of swelling pressure. For saturated NaCl-solution, however, a swelling pressure of zero is predicted, which is in contradiction to reality.

Another, more principal problem arises from the fact that it is impossible to separate an equilibrated clay from its high-saline hexary salt solution without changing the composition of adsorbed cations. The thermodynamic (and in fact, also the double layer models) worked with systems where homoionic bentonite was reacted with pure water or binary electrolyte solution (NaCl). Now, if the ionic strength of pure NaCl-solution in contact with Na-covered clay is reduced by washing and centrifuging steps, the composition of the adsorbed phase is still bound to consist entirely of sodium cations. The dried clay will therefore exhibit the same (or say: comparable) affinity to water like in suspension. A clay, however, which is at a given temperature in cation exchange equilibrium with a hexary solution will exhibit precisely that adsorbed composition on its surface which matches the equilibrium under the conditions of solution composition and ionic strength. Any washing step, necessary to separate the clay from suspension to eventually gain a dried sample for recording a water adsorption isotherm, will disturb cation exchange equilibrium giving rise to a change in the composition of adsorbed cations.

Predictions are improved for NaCl if equation [4] is supplemented with a term which accounts for an osmotic pressure component. It demands absolute values for positive charges ( $\text{Na}^+$ ) present in the clay prior to contact with solution and in the solution.

For the reasons given above this approach will also fail as soon as hexary electrolyte solutions are regarded.

## Results of GRS experimental programme

The underlying idea of this study may be outlined as follows: Intracrystalline swelling is considered the principle process from which the macroscopically detectable swelling pressure in highly salinar solutions arises. Interlayer water is located primarily within the hydration sphere of cations sorbed therein. The number of water molecules within the hydration sphere is different, depending on the charge density of the cation involved. Hence, the composition of interlayer cations influences the interlayer water content.

Cation exchange equilibria, interlayer spacing, and water content were determined for the Na-bentonite MX-80 equilibrated with a set of eleven 5,9 N model solutions. The model solutions were constant in K- and Ca-content and varied with respect to Na- und Mg-content. The obtained cation exchange isotherm is shown in figure 4. The regression was calculated utilizing an operative selectivity coefficient, which is based exclusively on data directly measured:

$$K \equiv \frac{m_{\text{Na}} \bar{m}_{\text{A}}}{m_{\text{A}} \bar{m}_{\text{Na}}} \equiv R \frac{\bar{m}_{\text{A}}}{\bar{m}_{\text{Na}}} \quad [5]$$

For further treatment it is assumed that CEC is constant, and that sorbed Mg and Ca have a constant effective charge  $z_{\text{eff}}$ .

$$\bar{m}_{\text{A}} = (CEC - \bar{m}_{\text{K}} - \bar{m}_{\text{Na}}) z_{\text{eff}}^{-1} \quad [6]$$

$z_{\text{eff}}^{-1} = 1$  indicates an effective valence of sorbed Mg and Ca of 1, while  $z_{\text{eff}}^{-1} = 0,5$  correspondingly gives an effective valence of sorbed Mg and Ca of 2. It follows:

$$\bar{m}_{\text{Na}} = \frac{R z_{\text{eff}}^{-1} (CEC - \bar{m}_{\text{K}})}{K + R z_{\text{eff}}^{-1}} \quad [7]$$

Analysis of cation exchange equilibria indeed indicate the sorption of univalent chloro complexes of the type  $\text{MCl}^+$  ( $\text{M} = \text{Mg}, \text{Ca}$ ), the proportion in relation to sorbed  $\text{Mg}^{2+}$  or  $\text{Ca}^{2+}$  decreasing with increasing solution content of Mg in relation to Na. Interlayer spacing (Figure 5) and total water content of compacted bentonite samples increased with increasing Mg-content [xiii].

Further experiments were carried out with a second set of solutions and four different clay types, two bentonites (MX-80 and Calcigel) and two nonswelling clays (Roter Salzton and Grauer Salzton) from salt formations. The composition of the solutions was that of naturally occurring brines in potash salt formations. The contents of Na, K, Ca, Mg, Cl and  $\text{SO}_4$  of the brines were variable. Figure 6 shows the relationship between the interlayer spacing and K content in solution for the bentonites and the nonswelling clays. Figure 7 shows the relationship between water content and K content in solution for the different clay types. The conclusion drawn from this second set of experiments was that the governing factors of the water uptake, the interlayer spacing and consequently the swelling pressure of compacted bentonites are the K and Mg contents of the NaCl-saturated natural brines. Potassium is the dominating parameter. So far on the basis of the data it is not possible to distinguish reliably quantitatively between interlayer water and pore water. Nevertheless it is concluded that bentonites maintain enough of their swelling capacity in contact with brines in order to be promising candidates for sealing materials in repositories in salt formations.

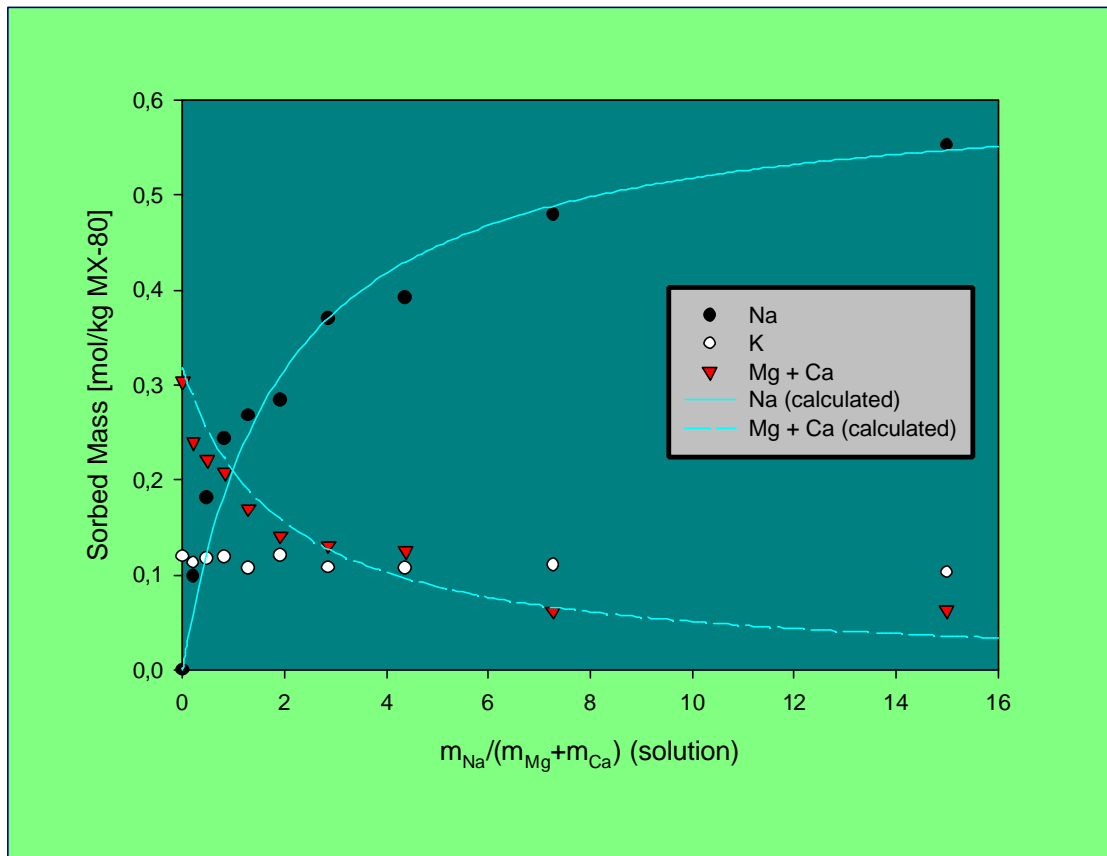


Figure 4: Cation exchange isotherms on MX-80 in highly saline solutions; m denotes mol/kg

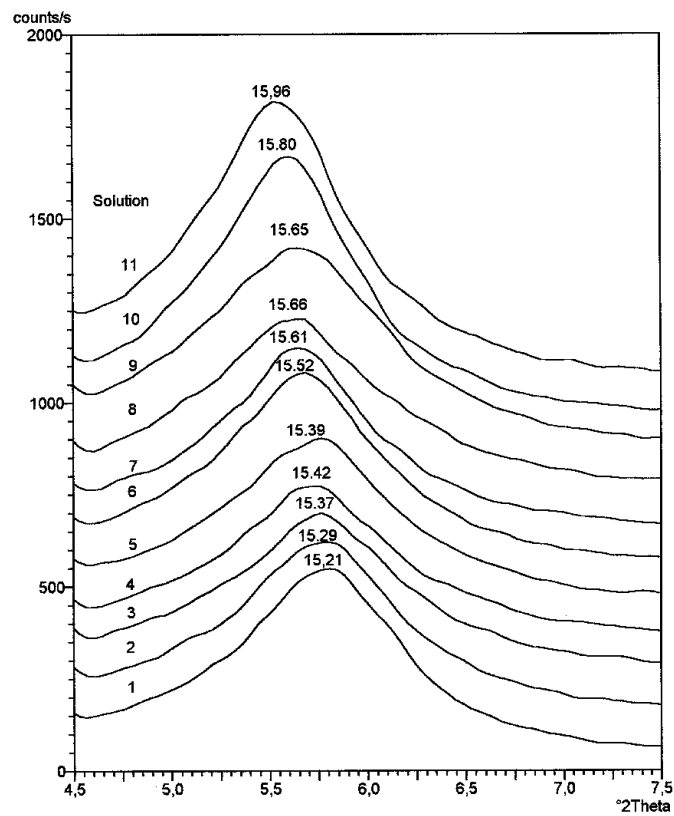
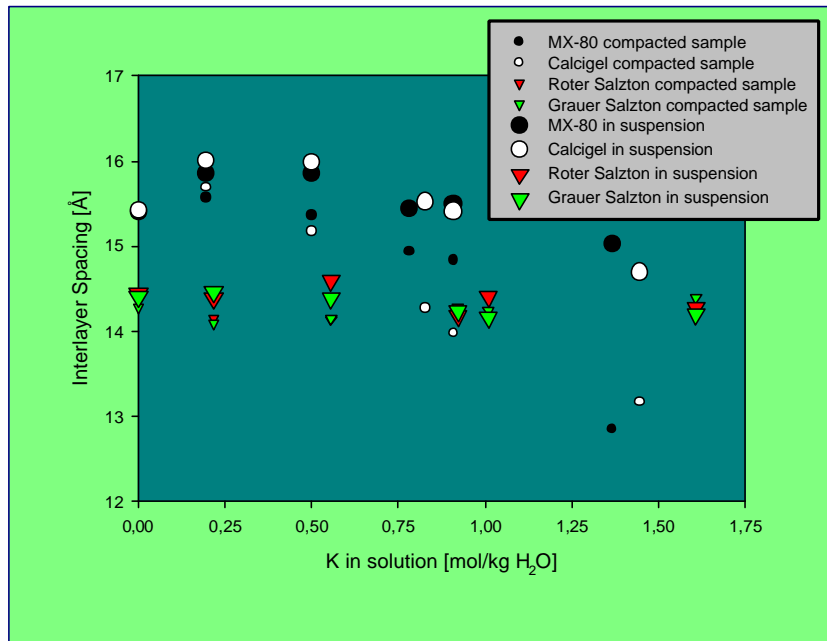
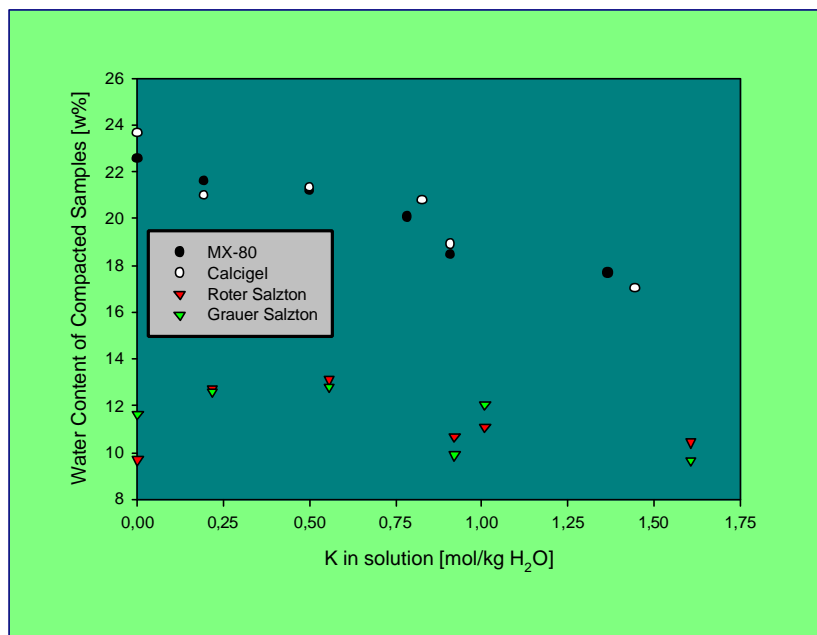


Figure 5: Interlayer spacing of MX-80 in 11 model solution with varying Na and Mg contents but constant K content



**Figure 6:** Relationship between interlayer spacing and K content in solution for bentonites (MX-80 and Calcigel) and nonswelling clays (Roter Salzton, Grauer Salzton) from salt formations



**Figure 7** Relationship between water content and K content in solution for compacted bentonites (MX-80 and Calcigel) and nonswelling clays (Roter Salzton, Grauer Salzton) from salt formations

Swelling pressures were measured only for the compacted bentonite MX-80 with the two sets of salt solutions. These experiments were very time consuming and showed a rather poor reproducibility. Nevertheless the following general conclusions could be drawn:

The swelling pressure is largely dependant on the degree of compaction and the applied fluid pressure. Therefore these experiments were conducted using raw densities around 1,6 g/cm<sup>3</sup> for the bentonite samples and 100 bars for the fluid pressure

All swelling pressures with brines were much lower than those obtained with pure water, but higher as with saturated NaCl-solution.

If the potassium content is kept constant in the NaCl-saturated brines, the water uptake, the interlayer spacing and the swelling pressure increases with increasing Mg content.

In brines with varying K and Mg contents K plays the dominant role [xiv].

## GRS's empirical approach

Neither one of the different predictive models for the swelling pressure of bentonites in contact with saline solutions discussed above can describe the complex processes involved in the actual problem. Nevertheless it seems obvious, that only an extended thermodynamic model will ever be able to perform this task. Such a model should include partial Gibbs Free Energies of the water and all the ions in the pore space as well as in the interlayer space. These quantities of the pore solutions and the interlayer solutions are expected to be very different. Whereas it is possible to accurately calculate them for brines in general, so far no experimental procedures are in sight for their direct determination in a system of brine with compacted bentonite.

Therefore GRS suggests a pragmatic approach to the problem. The following model equation was set up:

$$\ln P_s = - \frac{A(m_{Na}, m_K, m_{Mg+Ca})}{r_{red}} + B(m_{Na}, m_K, m_{Mg+Ca}) \quad [8]$$

where  $m_i$  denotes molality of species  $i$  in solution, and A,B are adjustable parameters. With this equation we adopt the regression formula used by engineers (equation 2). Both adjustable parameters A, B are expected to be functions of solution composition. The task is to build up a matrix of experiments with differing values for  $m_{Na}$ ,  $m_K$ ,  $m_{Mg+Ca}$ , and  $r_{red}$ . In the experiments a state of equilibrium must be established. Once the experimental data are available and reliable i.e. reproducible, the expressions for A and B must be found, which match best the observed correlation between swelling pressure and variables. For this approach a large number of swelling pressure experiments is needed. However, with the new and much faster technique developed recently in the GRS we are optimistic. The developed model will be related to realistic conditions in the near field of repositories on entirely empirical grounds. For the same reason extreme caution must be applied that the bentonite investigated exhibits the same microstructure as is to be expected under in-situ conditions.

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