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## Estimation of water retention behaviour of MX-80 bentonite partially saturated with saline solution

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**Abstract.** The water retention behaviour of partially saturated MX-80 bentonite with pure water is relatively well investigated. However, in practical cases, the water contains a number of chemical compounds which affects the bentonite behaviour. In particular, bentonite is used in buffer applications of geological repositories for spent nuclear fuel, where the concentration of total dissolved solids in groundwater is expected to increase significantly with time. This paper investigates water retention behaviour of MX-80 type bentonite partially saturated with deionised water as well as 1M NaCl solution. In the experiments the specific volume of bentonite has been kept approximately constant.

### 1 Introduction

A wide range of bentonite and other swelling clay materials have been studied extensively, partially because of their potential for creating nearly impermeable self-healing barriers. Those barriers are useful for isolating toxic waste material, such as nuclear waste. Due to the very long service life of such barriers, the understanding of the barrier material must be very thorough. Likewise, the accuracy of the barrier material characterisation must be exceptional.

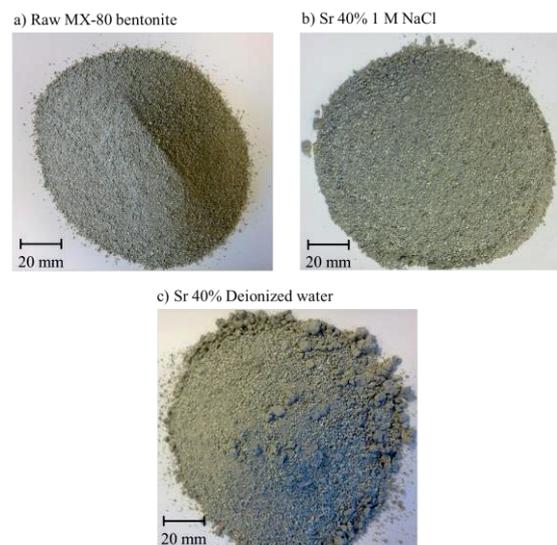
The present study describes the water retention behaviour of MX-80 bentonite partially saturated with: (1) deionised water and (2) 1 molar solution of NaCl. The paper adds to significant existing body of knowledge on MX-80 bentonite water retention behaviour e.g. [1-8].

Establishing the water retention curve is essential for determination of its behaviour upon wetting and drying. Water retention must be known not only for deionised water, but also for wide range of saline solutions. This study uses mainly filter paper technique to estimate the matric and total suctions of partially saturated bentonite. Filter paper data has been compared to the psychrometer measurements for selected bentonite samples.

### 2 Materials and Methods

#### 2.1 Material

MX-80 bentonite has been formed as a result of hydrothermal alteration of volcanic ash during the



**Figure 1.** MX-80 bentonite from Wyoming, USA: a) raw bentonite powder, b) hydrated with 1M NaCl solution (target saturation after compaction 40%), and c) hydrated with deionized water (target saturation after compaction 40%).

Cretaceous period. No characterisation of the material has been performed in this study. Based on [1], MX80 bentonite is composed mainly of montmorillonite (65–82%), quartz (4–12%), feldspars (5–8%), and smaller quantities of cristobalite, calcite and pyrite. The liquid limit and plastic limits of the MX-80 bentonite are 350%–570% and 70%, respectively. The density of the bentonite grains in the study was taken as 2.82 Mg/m<sup>3</sup> [1].

The MX-80 bentonite utilised in the study was supplied in a powder form with the original gravimetric

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water content being around 11-12%. Deionised water and 1 M NaCl water solution were used for hydrating the samples to the target saturation levels.

## 2.2 Methods

### 2.2.1 Sample Preparation

In order to prepare the cylindrical samples (50mm diameter, 20mm height), the oven dried (105 °C) MX-80 bentonite powder was mixed with water (pure or saline, as required). The amount of water has been chosen such that the desired target degree of saturations and specific volume should be reached after pressing the samples to the correct height.

Upon mixing, the saline water yielded a fairly uniform mixture of MX-80 with small lumps, while the deionized water produced larger lumps (Fig. 1). The mixture was then placed in an air-tight plastic bag and sealed. The lumps were crushed manually inside the sealed plastic bag to ensure a uniform distribution of moisture within the mixture. Once the mix was uniform, it was left in the sealed bag for two weeks to equilibrate at room temperature.

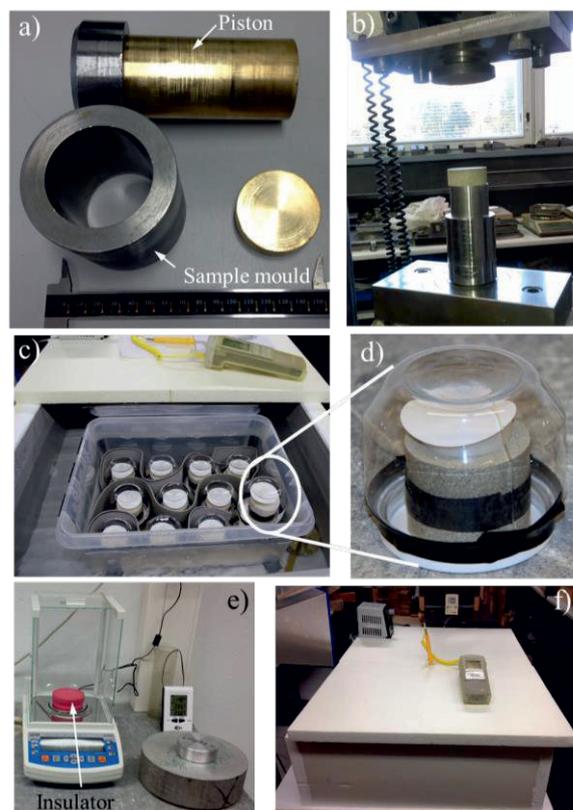
The pressing procedure has been the same for all the samples. Following the equalisation period of at least two weeks, carefully weighted amount of bentonite powder at water content correct for the targeted degree of saturation has been put into the steel mould. The mould was coated with a very thin layer of PTFE lubricant. The top and the bottom of the sample were isolated from the piston and background by thin plastic sheets (so almost no moisture could escape the bentonite during pressing).

Then the samples have been uniaxially pressed at a loading rate of around 1mm /min till reaching the desired height of 20 mm (Fig. 2 a,b). Due to noticeable relaxation of the samples after the initial pressing (around 0.6 mm), they were pressed multiple-times. The target dry density of all the samples was 1.6 g/cm<sup>3</sup>. See Figure 3 for typical stress-displacement curves.

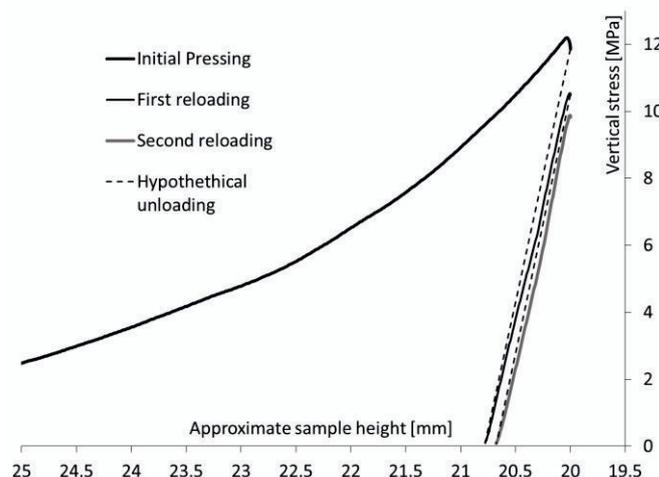
Samples have been measured multiple times in order to estimate their volume. Knowing the sample volume, water content and wet weight, dry densities were computed (Fig. 4). However, despite using high accuracy press and significant care, some variation of dry density (as indicated in Fig. 4) was unfortunately impossible to avoid. However, the achieved uniformity of dry density is similar to that of previous studies [1]. The dry-density variation seems to be correlated with the sample degree of saturation, as well as with the salinity of pore fluid. To achieve more uniform dry density distribution of samples, instead of following the same preparation procedure for all the samples, the procedure perhaps should be modified for drier and wetter samples. The drier samples should be pressed to lower height and the wetter samples to higher height.

In a conventional filter paper test, a single soil specimen is cut into two halves and the ‘contact filter paper’ for matric suction measurements is placed in between. However, since the bentonite samples were almost impossible to cut into two pieces, as an alternative, both

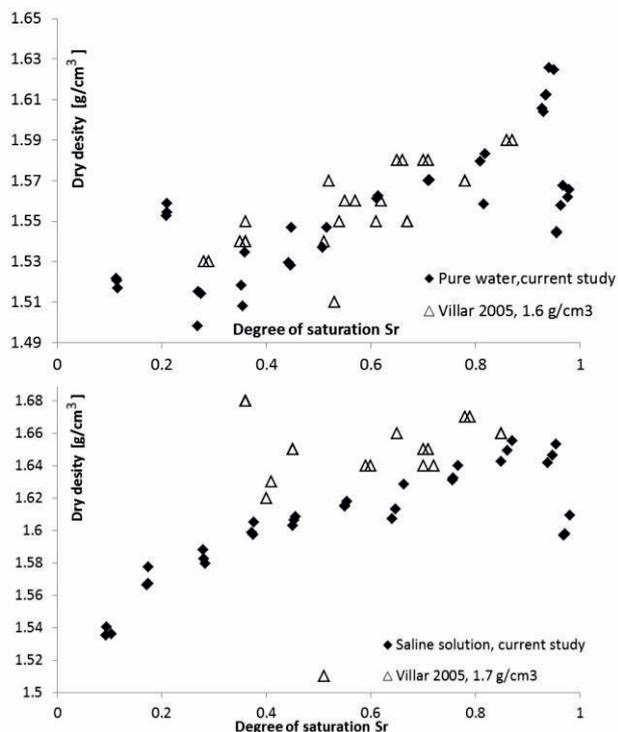
halves were pressed separately (the target height of each was 20 mm) to produce quite identical samples. Pressed samples were then sealed in air-tight plastic bags and left for moisture equalisation for at least one night before commencing the filter paper test.



**Figure 2.** a) tools used for sample pressing, b) compressed specimen being extracted from the sample mould, c) samples kept in the constant-temperature water bath, d) filter paper test assembly, e) insulation placed between the balance and moisture tin, and f) well insulated water bath with temperature control.



**Figure 3.** Vertical stress and displacement during sample pressings. No unloading data is available – the unloading paths are hypothetical. The vertical stress value is based on the press force readout. The target sample height was 20 mm. Pressing resulted in sample of Sr=0.59 and dry density 1.56 g/cm<sup>3</sup>



**Figure 4.** Variation of dry density in the current study and in [1]. For comparison, data for samples with target dry density of 1.6 and 1.7 g/cm<sup>3</sup> are shown [1].

### 2.2.2 Filter Paper Technique

Soil suction measurement with filter paper technique is well established method of estimation of suction. The major advantage of this technique is the ability to measure both total and matric suction quite reliably without expensive equipment.

The filter paper technique measures suction indirectly by relating the water absorbed by the filter paper with suction by means of a calibration curve for the particular filter paper used. The total suction is measured with a filter paper not being in contact with the sample; hence the paper is wetted via the water vapour. The contact filter paper measures matric suction.

The Whatman<sup>®</sup> Grade 42 type filter paper was used in the present study. The filter paper was cut to fit the diameter of the bentonite samples. The experimental procedure was very similar to these in [9,10].

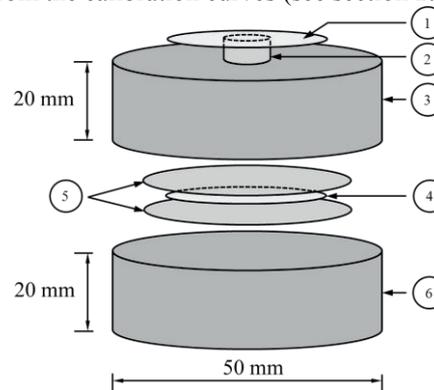
In the filter paper test, a contact filter paper, sandwiched between the upper and lower halves of the soil sample, measures the matric suction. The contact filter paper is enclosed by two high-permeable protective filter papers to avoid any contamination. The non-contact filter paper, placed on top of the soil specimen measures the total suction (Fig. 5).

Once the contact-filter paper was in position, an electrical tape was used to tape both halves of the samples together to enforce firm contact between the filter papers and the samples. The filter paper assembly was then immediately enclosed in an air-tight transparent plastic jar and the lid was taped with the electrical tape to prevent any possible exchange of humidity with the

environment. The jars in the present study were oriented upside-down (Fig. 4d) (lid on the floor). This orientation facilitates the emplacement and removal of the bentonite sample and the non-contact filter paper without much disturbance in shorter period of time compared to the orientation proposed in [9].

The jar was then stored inside an air-tight plastic container (Fig. 2c). The plastic container was subsequently placed in a constant temperature water-bath (+25°C ±0.2°C) for equalisation over a period of two weeks (Fig. 2f). The equilibration period was longer than often recommended 1 week (e.g. [9]) to make sure the equilibrium is fully reached.

Once the equalisation time is over, the assembly was removed from the water bath and the gravimetric water content of the filter papers were determined with a high precision scale of 0.0001g accuracy (Fig. 2e). Insertion of a piece of thermal insulation material between the metal plate of the balance and the aluminium tin containing the wet filter paper helped to minimise the waiting time for a stable reading. Corresponding suction values were obtained from the calibration curves (see section 2.2.3).



- |    |   |
|----|---|
| 1. | Non-contact filter paper for total suction measurement        |
| 2. | Ring support for non-contact sample filter paper              |
| 3. | Upper-half of the bentonite sample                            |
| 4. | Contact filter paper for matric suction measurement           |
| 5. | Two protective filter papers sandwiching contact filter paper |
| 6. | Lower-half of the bentonite sample                            |

**Figure 5.** Schematic of the filter paper total and matric suction measurement techniques.

### 2.2.3 Calibration curves

To determine the matric suction of the MX-80 specimens, calibration curves for Whatman 42 filter paper by Habmlin [11] (eq. 1), Leong et al. [12] (eqs. 2 & 3) alongside the standard ASTM [10] (eqs 4 & 5) as well as using calibration using van Genuchten [13] curve (eq. 6, [14]) were tested in the present study:

$$\log \Psi = 8.022 - 3.683 \log w_f \quad (1)$$

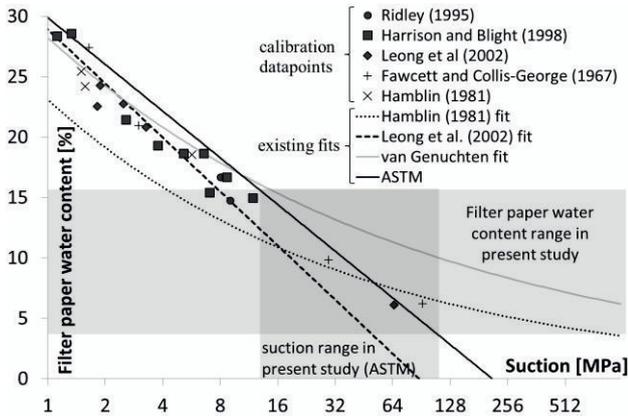
$$\log \Psi = 2.909 - 0.0229 w_f, w_f \geq 47 \quad (2)$$

$$\log \Psi = 4.945 - 0.0673 w_f, w_f < 47 \quad (3)$$

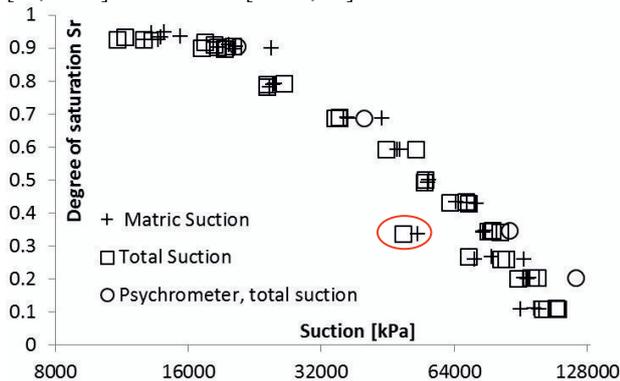
$$\log \Psi = 5.327 - 0.0779 w_f, w_f < 45.3 \quad (4)$$

$$\log \Psi = 2.412 - 0.0135 w_f, w_f > 45.3 \quad (5)$$

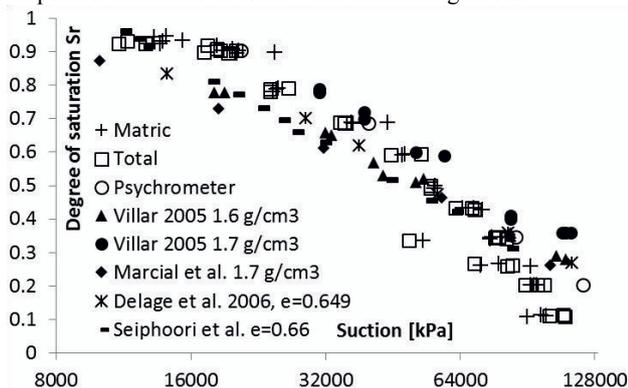
$$\Psi = 0.051 [(248/w_f)^{9.615} - 1]^{0.473} \quad (6)$$



**Figure 6.** Calibration curves for filter paper and available calibration points matched with approximate range of suctions and filter paper water content in current study. Data courtesy Ms Acikel, after [15-16]. Calibration data points taken from [12,17-19] and fits from [10-12, 14].



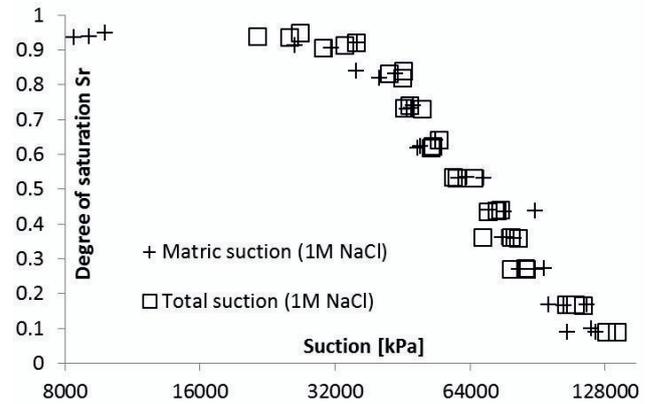
**Figure 7.** Obtained total and matric suctions for MX-80 bentonite partially saturated with deionised water. Points in the ellipse not taken into account when calculating errors.



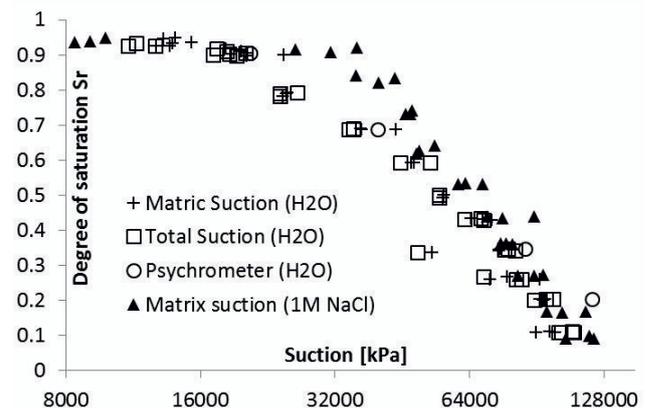
**Figure 8.** Obtained water retention curve vs literature data on the same material [1, 3, 5-6].

where  $\Psi$  is soil suction (kPa) and  $w_f$  is filter paper water content (%). Unfortunately, there is relative lack of calibration data in high suction ranges typical for bentonite (corresponding to suctions above 10 MPa), as shown in Figure 6. In the calculations, ultimately the ASTM calibration has been used.

Filter paper, when initially wetted by a saline solution and subsequently dried, will change its mass due to the salt residual from absorbed water. To take that effect into account, we note that in each dm<sup>3</sup> of 1M saline solution there is 58.44g of NaCl and that the density of such



**Figure 9.** Obtained water retention curve for MX-80 bentonite partially saturated with 1 molar solution of NaCl.



**Figure 10.** Differences between suctions for samples partially saturated with deionised (H<sub>2</sub>O) and salty water (NaCl)

solution at 25 °C is approximately 1042.54 g/dm<sup>3</sup>. Hence, for 1M solution of NaCl, the salt mass remaining in the filter paper after evaporation of water is equal to the evaporated water mass multiplied by  $[58.44 / (1042.54 - 58.44)]$ . The filter paper water content was then calculated as the quotient of the evaporated water mass and the clean dry filter paper mass, which is, in turn, the difference of the weighted dry filter paper mass and the mass of salt remaining on the filter paper:

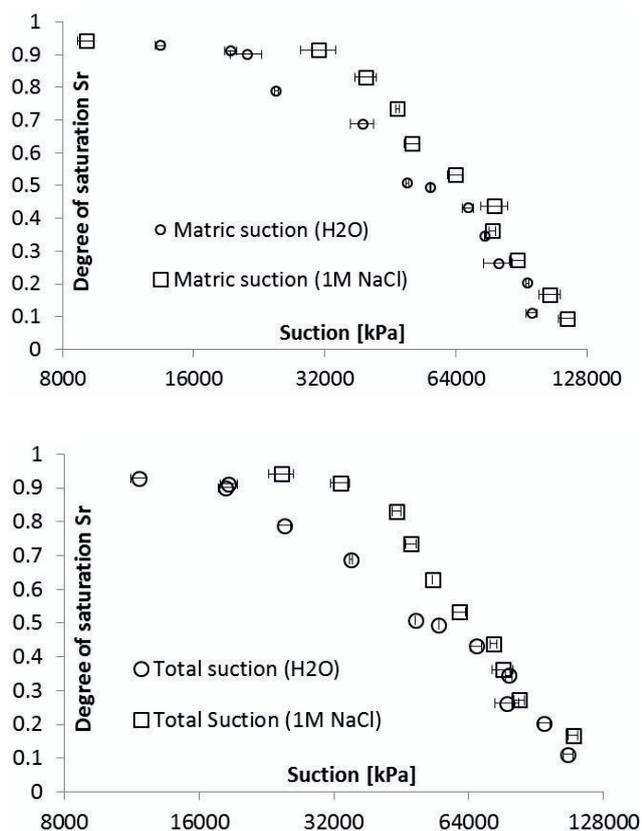
$$w_f = \text{water mass} / (\text{dry filter paper mass} - \text{salt mass}) \quad (7)$$

### 3 Results and discussions

In the experimental program 36 samples partially saturated with pure water and 33 samples partially saturated with 1 molar solution of NaCl were tested. The degree of saturation of samples varied from approximately 10% to above 95%.

#### 3.1. Measured suction

The results for bentonite saturated with pure water are shown in Figure 7. Figure 8 shows that those suctions are within the range of the typical published values, e.g. [1, 3, 5-6] and are further confirmed by psychrometer readouts (Table 1). The water retention curve for bentonite partially saturated with 1 molar NaCl (Fig. 9) is



**Figure 11.** Standard errors (computed based on 3 samples for each average value) for matric and total suctions.

**Table 1.** Comparison of filter paper and psychrometer results.

Sample	Sr [%]	Suction [MPa]		Difference [%]
		Filter Paper	Psychrometer	
51_w_20_3	20.3	98.7	120.3	18
11_w_40_5	34.8	78.0	85.1	8.4
35_w_80_5	68.8	35.2	39.9	11.9
45_w_100_3	90.5	20.2	20.7	2.3

somewhat different to that of deionised water. Matric suctions for pure and salty water are shown in Figure 10. It is quite clear that most significant difference in the water retention behaviour for pure and salty water is at higher saturations. The likely reason is that saturating the bentonite with the salty solution leads to creation of different microstructure and pore sizes in the material. That, in turn, leads to quite different water retention characteristic. However, some part of the differences may be attributed to the variation in dry densities of the samples saturated with pure water and salty solution.

### 3.2. Accuracy of the results

As a first check of the filter paper technique accuracy, the obtained suctions have been compared against values from psychrometer tests. Suctions obtained with the filter paper technique seem to match the psychrometer results reasonably well (see Table 1), with the biggest difference

at very high suction where filter paper measurements are known to be less reliable.

Furthermore, it is expected that, on average, the difference between total and matric suction in case of samples saturated with deionised water should be low (as the osmotic suction component should be zero assuming no salt is present in the natural bentonite; very small amounts of salt may be present though [20]). That is indeed confirmed – the average difference between the matric and total suction measurements (for all the samples partially saturated with water) is just 0.083 MPa. However, the standard error of that mean value is significant and equal to 1 MPa. In case the error in for each value is computed in % of the total suction, the standard error of the mean is approximately 2%. Similarly, for salty solution the average difference between matric and total suction is 3.95 MPa. That value matches the estimated value of osmotic component of suction (4.6 MPa [14]) somewhat poorer than in the case of deionised water. The standard error of the difference between total and matric suction is also higher (approximately 2.05 MPa). Hence the obtained mean value of 3.95 MPa is within reasonable statistical error. Finally, omitting the correction for salt residue in the dry filter paper decreases the average value of the osmotic suction to 3.67 MPa (standard error remains the same).

All the measurements were made for three samples with the same target degree of saturation. Those samples have some unavoidable variation in dry density (see Fig. 4). That variation, unfortunately, affect the suction values in the samples. Nonetheless, having sets of three samples for each degree of saturation, it is possible to compute the average value and standard deviation of each set (Fig. 11), even though such error estimates based only on three data-points should be treated with caution.

Averaging the average standard error for each three samples set gives approximately 1.5 MPa (matric suction, water), 1.4 MPa (total suction, water), or approximately 3% and 2.7% of the suction value, respectively. Those values for matric (2.9 MPa, 4.6%) and total suction (2.7 MPa and 3.7%) in samples partially saturated with 1M NaCl solution are again higher. It is unclear why, despite following exactly same procedure for samples saturated with pure water and salty solution, the inaccuracies are larger for salty solution.

The standard error of the degree of saturation value is very small for most of the samples. The averaged values for all the samples are 0.0023 (0.47%) and 0.0039 (0.73%) for samples partially saturated with pure water and saline solution, respectively. The errors are so small that they could not be shown in Figure 11.

### 3.3. Discussion of the results

In general, the obtained water retention characteristic of bentonite fit reasonably well into the known body of data. However, there are some interesting features that attract attention.

In particular, there is somewhat significant variance between the matric and total suction values close to saturation seen in samples partially saturated with each

liquid (in case of salty solution, beyond the expected value of osmotic suction) which are difficult to explain (Figs 7 & 9).

Furthermore, there are clear differences in matric suctions in the region of high saturations between samples saturated with pure and saline liquid (Fig. 10). Those differences may be due to differences in microstructure caused by saline solution, as well as some variation of dry density. Should they be confirmed in further studies, they may be one reason of the altered bentonite behaviour when saturated with salty solutions.

## 4 Conclusions & future work

The paper provides further data on water retention behaviour of MX-80 bentonite. The aim of the study was to obtain high quality consistent set of water retention data for bentonite. Here only the results from the first part of the testing program is shown – the investigation of the effects of various saline solutions will be continued in summer 2016. Presented data will also be further confirmed by psychrometer measurements of total suction. Subsequent microstructural investigations are also planned.

The planned testing program will offer further insights on how salinity affect the water retention behaviour of bentonite, as well as perhaps provide some further link between changes in microstructure, salinity of wetting liquid and water retention behaviour. These results, in turn, will allow for more accurate prediction of long term behaviour of bentonite barriers. Providing such predictions is the final goal of this research program.

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