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Preliminary investigation on the water retention behaviour of cement bentonite mixtures

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Abstract. Cement bentonite mixtures are often used to build slurry walls for the containment of both aqueous and non aqueous pollutants, due to their quite low hydraulic conductivity and relatively high ductility and strength. Although their hydro-mechanical behaviour in saturated conditions has been studied in the past, a part of the slurry wall is expected to rest above the groundwater level. The hydraulic characterization in unsaturated conditions is then particularly relevant to evaluate the performance of the barrier, especially when it is aimed at containing non aqueous pollutant liquids which are lighter than water (LNAPL). These non wetting fluids rest above the water table and their penetration is possible just if the barrier is unsaturated. This paper presents some preliminary results of a laboratory characterization of the water retention behaviour of three different cement bentonite mixtures. The mixtures, prepared at cement – bentonite mass ratios ranging from 4:1 to 6:1, were immersed in water and cured for 28 days. Their water retention behaviour was then determined along drying and wetting paths through different techniques, namely axis translation, filter paper and vapour equilibrium. In the high suction range, the water content – suction relationship was found to be independent of cement -bentonite ratio. In the low suction range, the water content at a given suction was found to decrease for increasing cement bentonite ratios.

1 Introduction

Slurry trench cut-off walls are widely used for the containment of pollutants. Trenches of about 0.40 – 0.80 m thickness are dug around polluted areas, totally, or partially enclosing the polluted site. The construction process is generally performed in one single phase: a series of alternate panels are excavated, using the cement-bentonite slurry as a stabilizing fluid. The same material is then left in the trench to harden at the end of the excavation [1, 2, 3]. The hardening process conveys the material both relatively high strength and low hydraulic conductivity. According to most design codes, after one month of curing, laboratory samples of the hardened slurry should have a hydraulic conductivity K lower than 10^{-9} m/s and a uniaxial strength higher than 200 kPa [3].

The hydro-mechanical behaviour of cement-bentonite mixtures thus varies with time, achieving better hydro-mechanical properties, since hydraulic conductivity decreases and mechanical strength increases [3]. Their constituents are very sensitive to the chemical composition of the pore fluid, which affects the hydration reactions of the cement and the electrostatic forces acting between the bentonite particles. Chemical compatibility is thus very relevant for the long time performance of cut-off walls, as shown in [4, 5] among others.

Depending on the hydrogeological conditions, a part of the cut-off walls can extend above the water table. Notwithstanding that, very little attention has been paid to the behaviour in unsaturated conditions of cement-bentonite mixtures. Preliminary studies point out that laboratory samples cracked when left to dry at laboratory conditions. However, cracking was not found to be relevant for sample cyclically drained and wetted when immersed in a box and surrounded by sand [3].

The present study is related to a larger research project, developed in the frame of a collaboration with the Italian Ministry of Economic Development, aimed at evaluating the efficiency of evolved cut-off walls (equipped with dedicated devices, [6,7]) in the containment of contaminants that may either be miscible or immiscible in water. In particular, the percolation of non-aqueous liquid contaminants that are lighter than water (LNAPL) might be enhanced if the hardened mixture is unsaturated. Desaturation may be induced by water table flotation or, more likely, by low relative humidity environments, which can occur in arid climates or during summer in continental climates. This paper shows some experimental results about the water retention properties of different cement-bentonite mixtures after 28 days of curing.

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2 Materials and sample preparation

2.1 Materials

The cement used in this work is a commercial CEM I 32,5 N, Portland 325, cement. The bentonite is a commercial mixed sodium - calcium bentonite provided by Laviosa Chimica Mineraria (Livorno, Italy). According to preliminary characterization, the liquid limit of the bentonite is $w_L = 616 \%$ and the plastic limit is $w_P = 42 \%$. The specific gravity is $G_S = 2.95 \text{ Mg/m}^3$ and the hygroscopic water content of the bentonite powder at laboratory conditions is $w = 14 \%$.

2.2 Sample preparation

2.2.1 Preparation of the mixtures

Bentonite powder was firstly thoroughly mixed with tap water in a mixer, until a homogeneous plastic paste was obtained. The water to solid ratio imposed on mass basis was 18:1. The paste was covered with an impermeable sheet and left to rest for 24 hours to ensure full hydration of the bentonite and homogenization of the water content. A known mass of cement powder was added to the hydrated bentonite, and then the mixture mixed again. The following ratios between the cement and the bentonite dry masses were considered: 4:1, 5:1 and 6:1, the corresponding mixtures being named M1, M2 and M3. The main characteristics of the three mixtures are summarized in Table 1.

Table 1. Cement Bentonite mixtures tested in this study.

Mixture	M1	M2	M3
Water / Bentonite mass ratio (-)	18:1	18:1	18:1
Cement / Bentonite dry mass ratio (-)	4:1	5:1	6:1
Average water content after 28 days of curing, w_0 (%)	338	269	219
Specific weight, G_s (-)	2.68	2.70	2.72

2.2.2 Curing

The cement-bentonite mixtures were finally gently poured into plastic moulds placed at the bottom of large boxes. The moulds were designed and built in the geotechnical laboratory of the Politecnico di Torino, using a 3D printer [10]. Depending on type of test to be carried out, moulds of different size have been used, as shown in following section. After pouring, the boxes were filled with tap water, and the samples were left to cure under water for 28 days.

Figure 1 and Figure 2 show one mould used to prepare specimens for the suction controlled oedometer and the box containing the specimens during curing.



Fig. 1. Mould for suction controlled oedometer test [8].

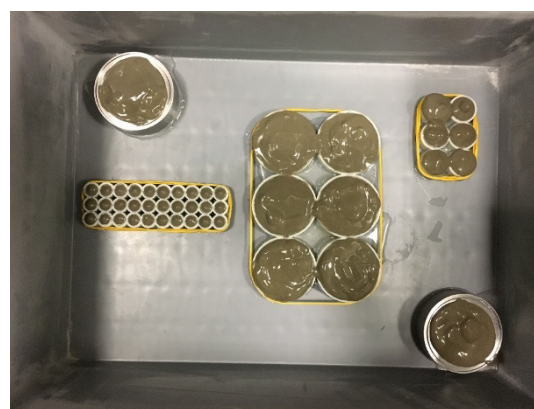


Fig. 2. Samples curing in a water bath [9].

3 Experimental techniques

Different techniques were used to determine the water retention behaviour of the cement-bentonite samples over a wide suction range.

3.1 Controlled suction techniques

3.1.1 Axis translation technique

The axis translation technique was used to impose matric suction s on M1 samples having initial size of 50 mm diameter and height of 20 mm. Specimens were placed in a suction controlled oedometer, which allows the independent control of total stress and of air and water pressures. A bottom porous stone with a nominal High Air Entry Value of 500 kPa was used to control water pressure within the sample. A total axial stress $\sigma = 370 \text{ kPa}$ was gradually imposed, together with an air pressure $u_a = 330 \text{ kPa}$ (corresponding to an axial net stress $\sigma_{net} = 40 \text{ kPa}$). The total stress and the air pressure were kept constant during the tests, while the water pressure was changed to impose the desired matric suction. Changes in the volume of water within the specimen were measured through a volume indicator connected to the water line. Vertical displacements of

the specimen were recorded by a LVDT placed at the top of the sample.

After maturation, specimens were placed in the oedometer for the determination of the main drying branch of the water retention curve. The main wetting branch was determined on samples that were preliminarily dried by exposing them to the laboratory environment for two weeks. The drying process caused a noticeable isotropic shrinkage of the specimen. The reduced diameter of the dried specimen made its use in the determination of the wetting branch problematic, because the oedometer ring (internal diameter $D = 50$ mm) is rigid and cannot follow radial shrinkage of the mixture. From the mechanical point of view, a specimen with a diameter smaller than D subjected to a vertical loading would fail in uniaxial conditions (null radial stress) rather than be compressed in oedometer conditions (null radial strain). Therefore, the side of the dried specimen was first coated with a layer of silicon grease and then placed inside the oedometer ring. The annular gap between the coated specimen and the steel ring was filled by pouring an epoxy resin. The sample and the fluid resin were left resting overnight to allow for hardening of the epoxy. The sample was then placed within the suction controlled cell for testing.

3.1.1 Vapour Equilibrium Technique

The Vapour Equilibrium Technique was adopted to impose total suction ψ . Specimens having initial diameter of 20 mm and height of 10 mm were placed in in hermetically closed jars, suspended over saturated salt solutions. The list of the salt solutions and corresponding total suction adopted is as follows: KNO_3 ($\psi = 9.59$ MPa), $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ ($\psi = 13.69$ MPa), KCl ($\psi = 22.76$ MPa), NH_4Cl ($\psi = 33.99$ MPa), NaCl ($\psi = 37.06$ MPa), NaNO_3 ($\psi = 38.13$ MPa), NaNO_2 ($\psi = 54.96$ MPa). The actual suction values of the aforementioned solutions were double checked by means of a WP4 psychrometer. The mass of the samples, measured with a high precision (accuracy 10^{-3} g) balance, equilibrated after 2-3 weeks. Volume changes were determined by measuring the current diameter and height with a calliper.

3.2 Controlled water content techniques

3.2.1 WP4 chilled mirror psychrometer

A chilled mirror psychrometer WP4 by Decagon was used to measure the total suction along the main drying and main wetting branches of specimens with an initial diameter of 20 mm and height of 10 mm. Immediately after curing, saturated samples were routinely exposed for a couple of hours to the laboratory atmosphere (relative humidity $\text{RH} = 38.5\%$, temperature $T = 21^\circ\text{C} \pm 0.5^\circ\text{C}$, corresponding to $\psi = 129$ MPa according to the psychrometric law). Specimens were then closed within an impermeable foil and left to homogenize in a closed jar -while suspended above distilled water- for about 48 hours. Their total suction was finally measured with

WP4, together with the gravimetric water content. Their current volume was determined through measurements with a calliper.

3.2.2 Filter paper technique

The filter paper technique was adopted to measure matric suction s along both drying and wetting. Sheets of Scheicher & Schuell no. 589 WH filter paper were used in the experiments. Two soil samples having diameter $D = 50$ mm and height $H = 10$ mm were used for each mixture and branch (drying and wetting). Along drying, the specimens were first exposed to the laboratory atmosphere for a couple of hours to allow water mass loss due to evaporation. They were then placed in an airtight container that was sealed with a plastic electrical tape, and left in the laboratory at controlled temperature for 2 weeks for equilibration of the water potentials between the sample, the air within the container and the filter papers. Three dry sheets of filter paper were sandwiched between the two samples for matric suction determinations. The weight of the central filter paper was then measured to determine their respective water contents. The whole procedure was repeated after exposing the sample again to the laboratory air, for determinations at lower water contents. New dry sheets of filter paper were used for each determination.

Measurement along wetting were made in a similar manner. In this case, the cement bentonite samples were exposed to dry laboratory air for two weeks after curing. Then, a known amount of water was added to the sample at each wetting step, followed by water content equilibration in the sealed container for two weeks. Both the weight of the filter papers and the one of the samples were measured for the determination of the main wetting branch.

Besides relying on the literature relationships quoted in [8], a specific calibration was carried out to determine the relationship between the gravimetric water content of the filter paper w_f and suction. To this extent, sheets of filter paper were first dried in the oven and then placed within a hermetically sealed container, where they were suspended over the same solutions described in Section 3.1.1. The experimental data can be fitted with the relationship:

$$\psi (\text{kPa}) = 145150 \cdot e^{(-0.185w_f)} \quad (1)$$

According to [11], it is assumed that relationship (1) holds for both total and matric suction determinations. Figure 3 provides the comparison between the data of this calibration with the literature relationships in [11]. The experimental data agree quite well with the expression of *Greacen et al. (1987)* suggested in [12].

Note that the calibration was done only for relatively high suctions and low water contents w_f . However, *Greacen et al. (1987)* and [12] report a bilinear logarithm water retention for the filter paper. At high water contents ($w_f \geq 54\%$), this is given by:

$$\log \psi = 1.882 - 0.0102 \cdot w_f \quad (2)$$

In absence of a proper calibration, eq. (2) was then used whenever $w_f \geq 54\%$, while eq. (1) was used in the remaining cases.

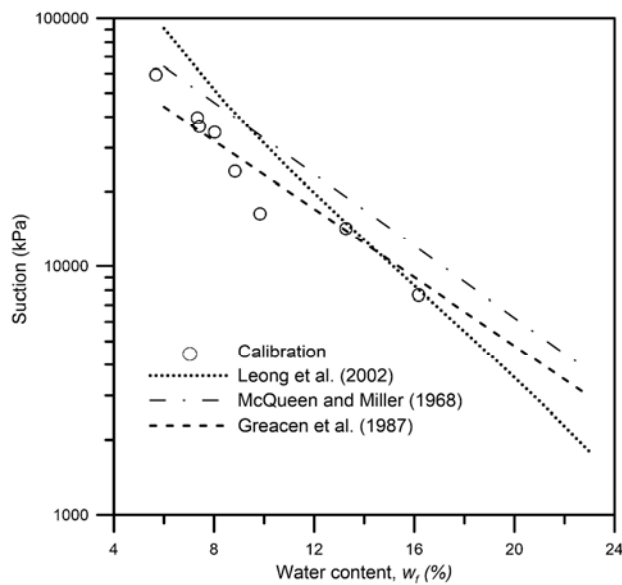


Fig. 3. Calibration of the filter paper S&S 589

3.2 Evaluation of osmotic suction

The chemical composition of the pore water of cement bentonite mixtures depends on the water used in the preparatory stage, on the cation exchange capacity, on the type of cations of the bentonite powder and on the chemical reactions triggered by cement hydration. These factors generally suggest that the osmotic suction π might be not negligible. The osmotic suction of the water in the baths after 28 days of curing was determined with WP4 measurements and it was found to be $\pi_0 \cong 285$ kPa.

It was then assumed that during drying, only H_2O molecules evaporate. The osmotic suction at the current water content, required to determine matric suction in tests where total suction was either imposed or measured, was then assumed to increase linearly with the decrease in water content, according with the expression:

$$\pi = w/w_0 \cdot \pi_0 \quad (3)$$

where w_0 is the average gravimetric water content of the sample of the different mixtures after 28 days of curing and before testing, as reported in Table 1. The matric suction of the WP4 and VET measurements was then evaluated as $s = \psi - \pi$.

4 Results

4.1 Volume change along drying and wetting

All the mixtures experienced significant volume changes during drying. The current volume of each specimen was measured at each stage by means of a calliper (or LVDT in case of the axis translation tests), with the exception of M1 samples when exposed to the high suctions with the VET. These samples were very fragile and it was

impossible to detect their volume change because they easily broke when handled. Such a problem did not occur with M2 and M3 samples in the same conditions. Figure 4 presents the experimental relationship between void ratio and matric suction for M1 and M3 samples (the behaviour of M2 was intermediate and is not reported because of space limitations).

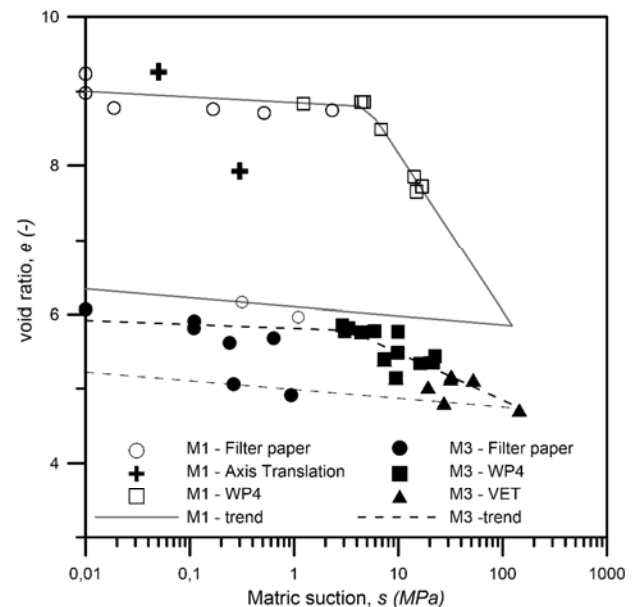


Fig. 4. Evolution of void ratio along drying-wetting cycles.

A phenomenological interpretation of the experimental results is put forward observing that the changes in the void ratio are linearly related to the logarithm of matric suction. A threshold matric suction s_t , of about $4 \div 5$ MPa for all the mixtures, separates a lower suction range $s < s_t$ from a higher suction range $s > s_t$. The logarithmic compliance (*i.e.* the slope of the void ratio – log of suction curve) of samples of different mixtures is about the same in the low suction range, while in the high suction range it decreases for increasing cement-bentonite ratios.

4.2 Changes in the degree of saturation along main drying and wetting

The water retention behaviour was interpreted in terms of the evolution of the effective degree of saturation with matric suction. At each suction value, the degree of saturation was determined on the basis of the measured water content and void ratio. For the VET data of the M1 mixture, where volume changes were not measured, the phenomenological trend shown in Figure 4 was used to estimate void ratio changes at high suctions. The experimental data were fitted with the expression of Gallipoli *et al.* [13]

$$S_r^e = \left[\frac{1}{1 + (\alpha e^\beta \cdot s)^n} \right]^m \quad (4)$$

where S_r^e is the effective degree of saturation and α , β , n and m are model parameters. S_r^e is defined as:

$$S_r^e = \frac{S_r - S_r^{irr}}{1 - S_r^{irr}} \quad (5)$$

where S_r^{irr} is the irreducible degree of saturation, that is assumed to be the one determined at the highest suction imposed with the VET ($s \cong 140$ MPa for all mixtures). Equation (4) was chosen among other water retention models because it explicitly takes into account the void ratio changes occurring along the drying path, whose relevance has been described in the previous section. The experimental data, together with the fit provided by equation (4) for M1 samples are presented in Fig. 5: the values of the hydraulic parameters are provided in the following table:

Table 1. Retention parameters for M1 sample

M1	α (MPa ⁻¹)	β (-)	n (-)	m (-)
drying	0.12	0.50	2	0.80
wetting	5	0.50	1	0.65

4.3 Changes in gravimetric water content

The phenomenological expressions relating suction to the void ratio and suction to the degree of saturation were combined determining the relationships between suction and gravimetric water content for the three mixtures. The experimental data and their fit through Equation (4) for the M1 specimens are presented in Figure 6. Comparison between Figure 5 and Figure 6 shows that a limited, although appreciable, reduction of water content occurs when suction increases while the sample remains saturated (approximately $0 \leq s \leq 100$ kPa). During this stage, the water content reduced from $w = 220\%$ to $w = 210\%$ for M3 samples and from $w = 338\%$ to $w = 316\%$ for M1 samples. For all cement:bentonite ratios, most water is lost in the suction range $1 \text{ MPa} < s < 10 \text{ MPa}$.

4.4 Influence of cement – bentonite ratio

A comparison between the behaviour of the three mixtures is here resumed. To allow clarity, it limits to the fits obtained with eq. (4), while the experimental data are not reported. When the interpretation is done in terms of degree of saturation (Figure 7), the cement to bentonite ratio has little impact on the behaviour along main drying. However, it impacts on the main wetting curve, and increase in cement content leads to a higher retention capacity.

The interpretation is somehow reverted in terms of relationship between water content and suction (Figure 8). Because of the differences in the initial void ratio and in the volume strains caused by suction changes, the drying curves of the three mixtures are different, although when $s > 2$ MPa the behaviour of mixtures M2 and M3 is very similar, and a single curve is obtained for all the mixtures when $s > 20$ MPa. On the other hand, for suction $s > 100$ kPa, the $w - s$ relationships of the three mixtures appears to be quite comparable. However, at smaller suctions, differences remain, since the capacity

to accommodate water within the porous space increases as the cement – bentonite ratio decreases.

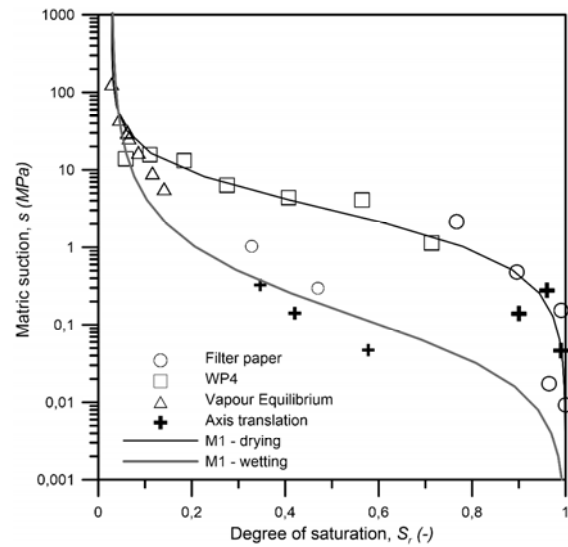


Fig. 5. Water retention of M1 samples: $S_r - s$.

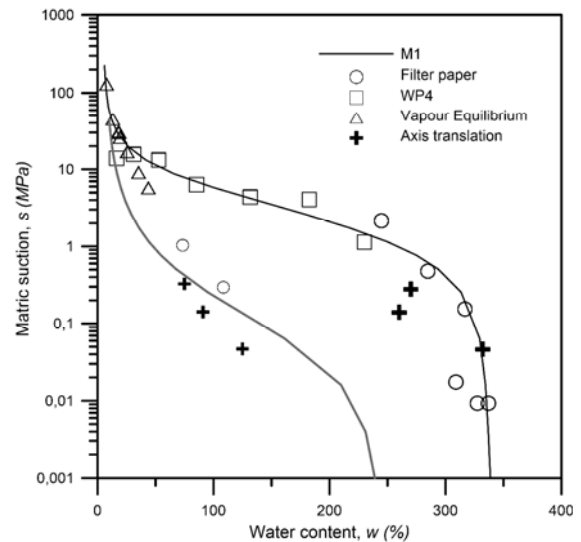


Fig. 6. Water retention of M1 samples: $w - s$.

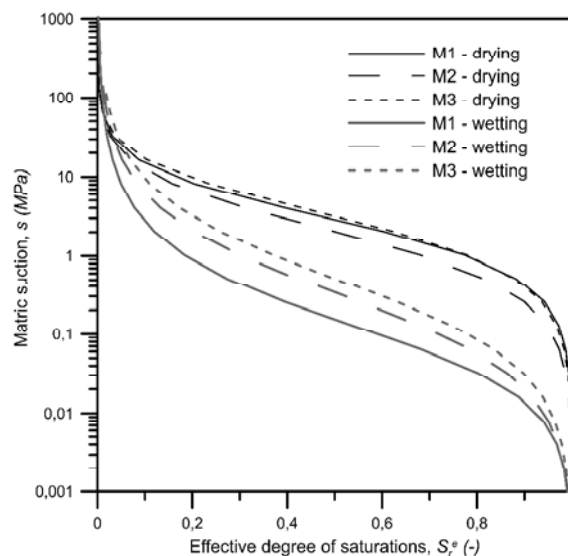


Fig. 7. $S_r^e - s$ fits for the three mixtures.

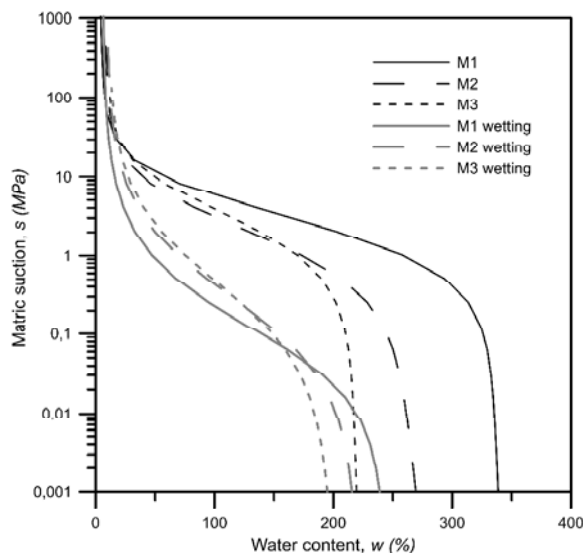


Fig. 8. Water content – matric suction fits for main drying and wetting branches of the three mixtures.

5 Concluding remarks

The preliminary results obtained in the current investigation show that cement bentonite mixtures have a remarkable tendency to shrink when dried. The volume – suction relationship can be reproduced quite adequately by a phenomenological semi-logarithmic relationship. The volume changes experienced along drying are not fully recovered upon wetting. The relevance of such a plastic behaviour increases as the cement – bentonite ratio decreases, also impacting on the water content retained under a given suction, which is higher the lower the cement – bentonite ratio along main drying, while it has a little dependency on the mixture type along main wetting.

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