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# Filtres de céramique pour tensiomètres à haute capacité

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**RÉSUMÉ.** La succion de sols (pression capillaire de traction de l'eau interstitielle) est un paramètre crucial pour l'étude du comportement hydromécanique des sols partiellement saturés. La seule façon courante de mesurer directement la succion des sols est par des tensiomètres à haute capacité. Ces tensiomètres sont typiquement constitués par un filtre céramique avec une valeur d'entrée d'air très élevée, un réservoir d'eau et un transducteur de pression. La plage de mesure des tensiomètres est limitée par la valeur d'entrée d'air du filtre céramique qui, à son tour, est directement dépendante de la taille maximale des pores.

Dans le contexte de cette étude, la distribution de la taille des pores de deux céramiques, provenant respectivement d'un fournisseur américain, fournisseur du RU et d'un fournisseur allemand, a été analysée. La porosité a donc été étudiée par intrusion au mercure et adsorption d'azote selon les différentes plages des dimensions des pores. Des résultats préliminaires ont montré que les céramiques du fournisseur allemand et anglais ont une taille maximale des pores plus élevée que celle-là des céramiques du fournisseur américain. Par conséquent, les céramiques des fournisseurs allemand et anglais ont une valeur d'entrée d'air plus faible que la céramique du fournisseur américain. Cette différence est évidente lorsque les céramiques, employées pour la construction des tensiomètres à haute capacité avec dessins similaires, conduisent à différences considérables dans les mesures maximales de la tension interstitielle de l'eau dans les sols: si les céramiques allemandes permettent d'atteindre 700 kPa, les céramiques américaines peuvent mesurer jusqu'à 1500kPa.

**ABSTRACT.** Soil suction (that is tensile pore water pressure) is an important parameter for the study of the hydro-mechanical behaviour of unsaturated soils. At the present, soil suction can be directly measured by means of high capacity tensiometers. The typical design of a high capacity tensiometer comprises a high air entry value ceramic filter, a water reservoir and a pressure transducer. The measuring range of these sensors is believed to be directly related to the air entry value of the filter, which in turn increases with decreasing size of the largest pore inside the ceramic.

This work presents a study of the pore size distribution of three different ceramic filters manufactured by an American, English and German supplier. Mercury intrusion porosimetry (MIP) and nitrogen adsorption (NA) tests are performed to investigate the pore size distribution of these ceramics. Results from the MIP tests suggest that the largest pore sizes of the ceramics from the German and English suppliers are considerably larger than the largest pore size of the ceramic from the American supplier. Hence, the air entry values of the ceramics from the German and English suppliers should be lower than the air entry value of the ceramic from the American supplier. This is indeed confirmed by tests where suction was measured using tensiometers incorporating different ceramics but built according to the same design. These tests indicate that the maximum measured value of suction was about 700kPa for the tensiometer incorporating the German ceramic and increased to 1500kPa for the tensiometer incorporating the American ceramic.

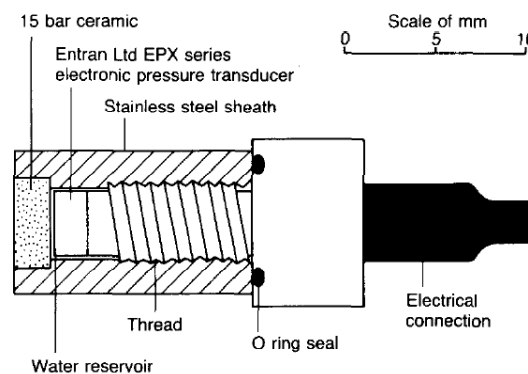
**MOTS-CLÉS :** tensiomètre ; succion de sols ; sols partiellement saturés.

**KEY WORDS:** tensiometer ; soil suction ; unsaturated soils.

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## 1. Introduction

The direct measurement of matric suction, or tensile (capillary) pore water pressure, is important for the characterisation of the hydro-mechanical behaviour of unsaturated soils and is normally performed by means of high capacity tensiometers (HCTs). The development of high capacity tensiometers is mainly restricted to university research but the prototypes fabricated so far have measured relative high values of soil suction, from 100kPa up to 2000kPa. The first HCT was developed at Imperial College London by Ridley and Burland [RID 1993]. The design of the HCT developed at Imperial College is shown in figure 1 and comprises : a ceramic filter with an air entry value of 1500kPa ; a small water reservoir of about 3mm<sup>3</sup>; and a pressure transducer with a working range of 3500kPa. Since then, similar designs have been published in the literature ([RID 1995]; [GUA 1997]; [MEI 2002]; [TAR 2002]; [TAK 2003]; [LOU 2008]; [CUI 2008]; [MEN 2013]; among others).



**Figure 1.** High capacity tensiometer developed by Ridley and Burland at Imperial College [RID1993].

To measure soil suction, the HCT needs to be first fully saturated by water, which is normally achieved by pre-pressurising the sensor, i.e. by pushing water at relatively high pressure through the ceramic filter to saturate both the filter and the reservoir. If the filter is then placed in contact with an unsaturated soil sample, the suction in the soil will draw water out of the ceramic and reservoir, thus deflecting the sensing membrane of the transducer until equilibrium is attained between the water pressure inside the HCT and the soil. The reading by the transducer at equilibrium will provide the required measurement of soil suction. However, if suction exceeds the measurement range of the HCT, cavitation occurs and air bubbles will emerge from the ceramic filter into the water reservoir as shown by Mendes and Buzzi [MEN 2013]. Cavitation of HCTs is easily detected as it produces a noticeable and immediate reduction of the measured suction down to values between 80 and 100kPa. Fast evaporation tests, where the HCT is exposed to atmosphere until it cavitates, are used to assess the maximum measurement range of these sensors. In evaporation tests, the reading immediately before cavitation is taken as the maximum value of suction that can be measured by the HCT.

The different components of the HCT (pressure transducer, water reservoir and ceramic filter) have a direct impact on its performance. Ideally, the pressure transducer should be symmetrical with respect to the sensing membrane. This is because the calibration of the HCT is usually performed in the positive (compressive) pressure range and the symmetry of the sensor is required in order to extrapolate this calibration to the negative (tensile) pressure range [TAR 2002]. The working pressure of the pressure transducer should be, at least, in the same range as the air entry value of the ceramic filter. The material of the outer sheath and the size of the water reservoir have little influence on the suction measurement range ([MEN 2013] and [MEN 2014]). However, the size of the water reservoir does have an influence on the time required to attain equilibrium during measurements and on the time necessary to saturate the HCT during pre-pressurisation. Among all components, the ceramic filter, or more precisely the air entry value (AEV) of the ceramic filter, is probably the factor that has the largest influence on the performance of the HCT. The AEV is the maximum value of suction, i.e. the maximum differential pressure between the air outside the filter and the water inside the filter, which can be sustained by the ceramic before air breaks inside the pores, hence causing cavitation of the HCT. The AEV of the ceramic is related to the size of the largest pore whereby a smaller size of the largest pore corresponds to a higher value of the AEV, which in turn means a higher value of the maximum suction measured by the HCT.

This manuscript presents a study about the pore size distributions of three ceramic filters with the objective of identifying the most suitable filter to be used in a commercial HCT which is being developed within a collaborative industry-academia project funded by the European Commission (project MAGIC - Monitoring systems to Assess Geotechnical Infrastructure subjected to Climatic hazards). Mercury intrusion porosimetry (MIP) and nitrogen adsorption (NA) tests were performed to determine the pore size distribution of three ceramic filters provided by an American, English and German supplier, respectively, named herein ceramic A, ceramic E and ceramic G. The full pore size distribution of the three ceramics was measured over a large pores range from 7nm to 0.5mm (MIP tests) as well as smaller pores range from 2nm to 80nm (NA tests). Ceramic A and ceramic G were also used in the construction of two HCTs with similar designs. The maximum values of suction measured by these two HCTs during fast evaporation tests were 800kPa, for the HCT built with ceramic G, and 1500kPa, for the HCT built with ceramic A, which corroborated the results obtained from the MIP and NA analyses.

## 2. Materials and methods

Before proceeding with the construction of the two HCTs, the internal pore structure of the three ceramics was studied by mercury intrusion porosimetry (MIP) and nitrogen adsorption (NA). These tests allowed the determination of the largest pore size of each ceramic, which is directly related to the maximum value of suction that can be measured by the HCT.

Small samples were cut from the ceramic filters from each supplier in order to fit inside the testing tubes of the NA and MIP devices. Before the tests, samples were purified under vacuum (<0.013kPa of absolute pressure) at a temperature of 105°C for 24 hours using a Micrometrics VacPrep 061.

After the MIP tests, the samples were safely disposed due to the health risks posed by the presence of mercury inside the pores. Because of this, each sample was first tested by NA and subsequently by MIP. After the NA test, the ceramic pores are only filled by nitrogen, which can be easily removed by re-purifying the sample under vacuum and temperature as previously explained. The same sample can then be used for the MIP test.

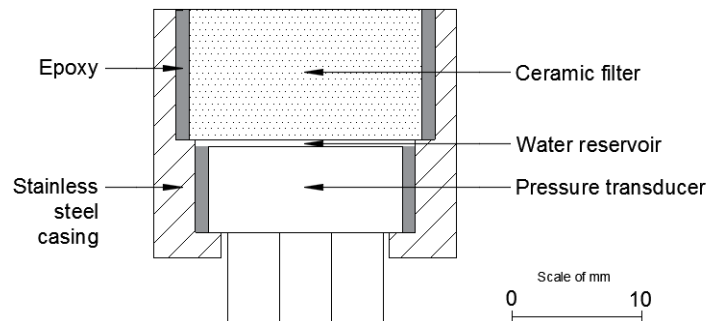
The NA tests were performed by using a Micrometrics TriStar II device. The nitrogen adsorption-desorption measurements were interpreted according to the method proposed by Barrett, Joyner and Halenda, usually referred to as the BJH method [BAR 1951], to determine the pore size distribution over the range from 2nm to 50nm. The BJH method estimates the pore size distribution by measuring both adsorption and desorption of nitrogen on the pore surface at different values of relative vapour pressure and at a constant temperature of 77°K. Distinct isothermal adsorption and desorption curves are obtained because of the hysteresis induced by the capillary condensation and evaporation of nitrogen inside the pores. The pore volume is estimated from the desorption curve at each value of relative pressure. Further information on the BJH method for the determination of pore size distribution by using NA can be found in Barrett et al. [BAR 1951].

The MIP tests were performed by using a Micrometrics AutoPore IV device. The mercury intrusion-extrusion measurements were interpreted according to the Young-Laplace theory (Eq. 1), assuming a cylindrical pore geometry, to determine the pore size distribution over the range from 7nm up to 0.5mm. In Eq. 1, the pore size (measured by the pore radius  $r_{\text{pore}}$  or pore diameter  $d_{\text{pore}}$ ) is determined from the mercury surface tension ( $\gamma$ ), the contact angle ( $\theta$ ) between the mercury and the pore surface and the pressure difference ( $\Delta P$ ) across the curved mercury-air interface, whose shape is defined by the principal radii of curvature  $r_1$  and  $r_2$  [GIE 2006]. In this work, we consider a spherical shape for the mercury-air interface, so that the two principal radii of curvature are identical ( $r_1 = r_2 = r_{\text{pore}}$ ), the surface tension of the mercury-air interface is assumed to be 485mN/m and the contact angle for the mercury is taken to be 130°. The pore volume is then determined from the intruded mercury volume at each value of mercury pressure. Other significant data that can be obtained from this test are the porosity, the specific surface and the apparent skeletal and bulk densities of the ceramics [GIE 2006].

$$\Delta P = \gamma \left( \frac{1}{r_1} + \frac{1}{r_2} \right) = \frac{2\gamma \cos \theta}{r_{\text{pore}}} \Leftrightarrow d_{\text{pore}} = \frac{4\gamma \cos \theta}{\Delta P} \quad [1]$$

The two ceramics with better characteristics in terms of pore size distribution and toughness were those from the American and German suppliers (see next section). These two ceramics were then used for the construction of

two HCTs with similar design as schematically shown in figure 2. Fast evaporation tests were performed as to determine the maximum suction measured by each HCT, which helped to validate the results obtained from the previous analysis of the pore structure. Before each evaporation test, the HCTs were saturated according to the pre-pressurisation procedure of Mendes and Buzzi [MEN 2014].

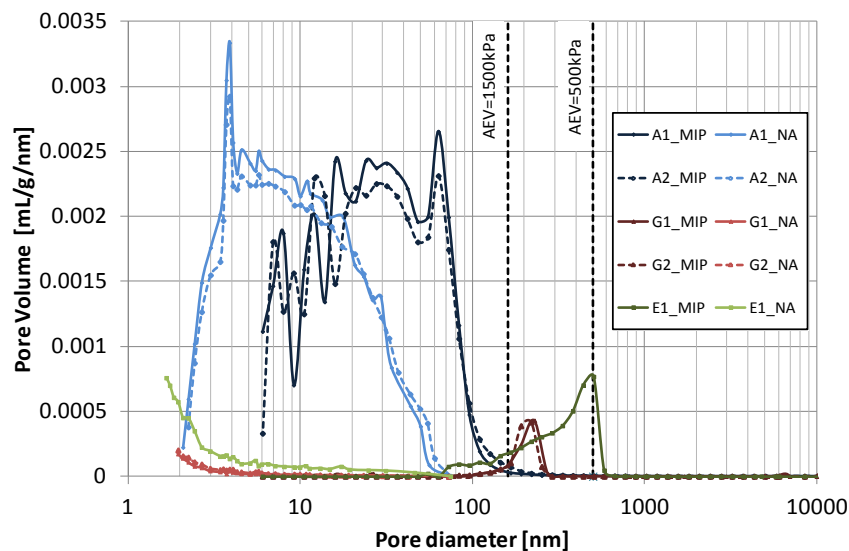


**Figure 2.** Scheme of the high capacity tensiometer developed at UPPA.

### 3. Results

MIP and NA tests were performed on two ceramics from the American supplier, two ceramics from the German supplier and one ceramic from the English supplier. The intention was to perform at least two tests for each type of ceramic in order to verify the repeatability of the measurements. However, this was not possible for the ceramic from the English supplier (ceramic E) due to the unavailability of a second ceramic filter. Results from the MIP and NA tests are presented in figure 3 and in table 1.

In figure 3, each test is labelled according to the format  $xy_z$ , where  $x$  refers to the ceramic supplier (A, G and E for the American, German and English suppliers respectively);  $y$  refers to the ceramic number; and,  $z$  refers to the different type of test (MIP for mercury intrusion porosimetry and NA for nitrogen adsorption). In the following, we use the term “mesopores” for the class of pores whose sizes are comprised between 2 and 50nm while we use the term “macropores” for the class of pores whose sizes are above 50nm.



**Figure 3.** Pore size distributions of the different ceramics.

Figure 3 and table 1 show that, for all ceramics, the sizes of the largest pores fall always inside the macropore range (>50nm in diameter), even though the pore size distributions of the three ceramics are rather different. Ceramic E was found to have the biggest size of the largest pore (580-670nm in diameter), the highest porosity (about 50%) and an intermediate value of bulk density ( $1.9\text{g/cm}^3$ ). Moreover, the pore size distribution of this

ceramic covers most of the macropore range (60-670nm) but does not extend into the smaller mesopore range. Ceramic G showed an intermediate size of the largest pore equal to 250-290nm, the lowest porosity (around 13%) and the highest bulk density (around  $2.6\text{g/cm}^3$ ) of all ceramics, with a pore size distribution that falls again in the macropore range (95-290nm). Finally, ceramic A had the smallest size of the largest pore (165-190nm) of all ceramics, with an intermediate value of average porosity equal to about 33% and the lowest value of bulk density equal to  $1.4\text{g/cm}^3$  on average. Unlike the former two ceramics, the pore size distribution of ceramic A covered both the macropore and mesopore ranges (2-190nm).

Inspection of figure 3 indicates a good repeatability of both the MIP and NA tests for the A and G ceramics. This also suggests a good quality control of the manufacturing process for these two types of ceramic.

**Table 1.** Porosimetry data of the different ceramics.

| Supplier | Ceramic | MIP                 |                                 |  |                                  | NA            |  |
|----------|---------|---------------------|---------------------------------|--|----------------------------------|---------------|--|
|          |         | Largest pore*<br>nm | Pore volume distribution*<br>nm | Cumulative pore volume<br>$\text{cm}^3/\text{g}$ | Bulk density#<br>$\text{g/cm}^3$ | Porosity<br>% | Cumulative pore volume<br>$\text{cm}^3/\text{g}$ |
| American | A1      | 165-190             | 6-190                           | 0.229  | 1.31                             | 30.1          | 0.0740   |
|          | A2      | 190-220             | 6-220                           | 0.235  | 1.53                             | 36.2          | 0.0750   |
| German   | G1      | 250-290             | 95-290                          | 0.051  | 2.68                             | 13.7          | 0.0007   |
|          | G2      | 250-290             | 95-290                          | 0.035  | 2.57                             | 12.1          | 0.0008   |
| English  | E1      | 580-670             | 60-670                          | 0.262  | 1.90                             | 49.8          | 0.0042   |

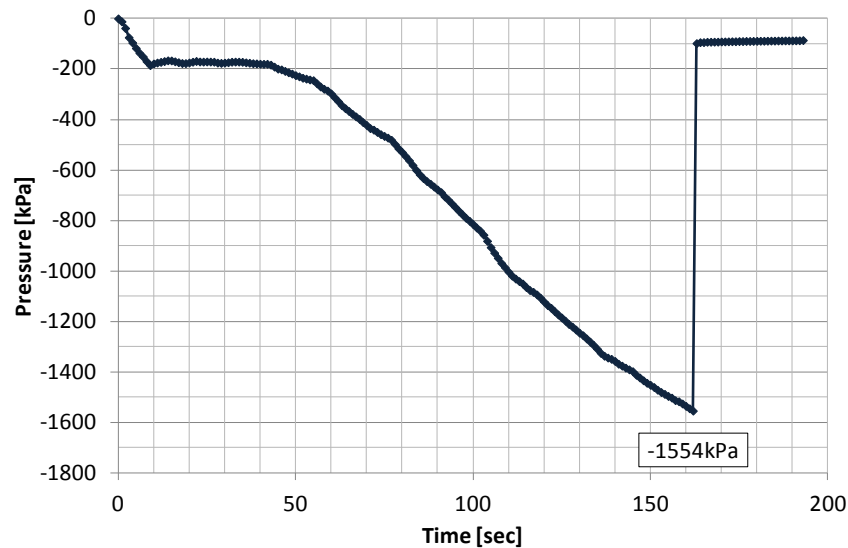
\* - pore diameter. # - at 1.45kPa of absolute pressure.

As mentioned before, the size of the largest pore inside the ceramic filter is related to the AEV of the filter and, hence, to the maximum value of suction that can be measured by the HCT. For comparison purposes of the AEV of the ceramic filter and pore size, AEVs of 500 and 1500kPa were determined by using the Young-Laplace equation (Eq.1), which are also included in figure 3 as vertical dashed lines. Therefore, from the MIP and NA results, it can be concluded that the ceramic from the American supplier (ceramic A) should have the highest AEV because it has the smallest size of the largest pore.

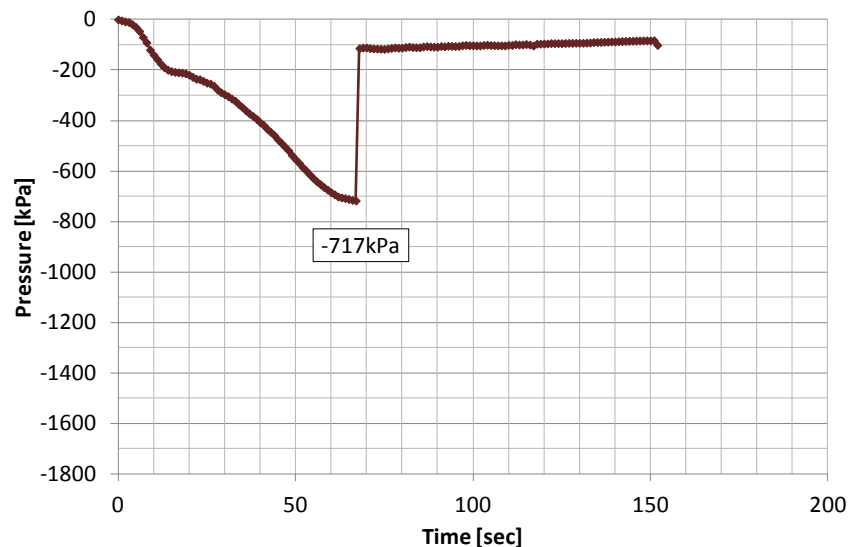
Ceramic A was also used to build a HCT according to the design shown in figure 2. After saturation, the HCT was subjected to a fast evaporation test and the results from this test are shown in figure 4. The negative (tensile) water pressure measured by the HCT immediately before cavitation was -1554kPa, which indicates that the measurement range of suction is about 1500kPa for this HCT. This value is similar to the AEV advertised by the ceramic supplier and is also consistent with the pore size distribution shown in figure 3.

During preparation of the samples for the MIP and NA tests, it was noticed that ceramic G was considerably tougher than the other two ceramics. While it was relatively easy to cut ceramics A and E by simply using a pair of pliers, it was instead necessary to use a hammer and chisel to break ceramic G. The greater toughness of ceramic G makes this type of ceramic better suited for HCTs to be used in the field. A second HCT was built using this tougher ceramic according to the design shown in figure 2. As before, the HCT was first saturated and then subjected to a fast evaporation test whose results are shown in figure 5. Inspection of figure 5 indicates that, immediately before cavitation, the HCT recorded a value of tensile pore water pressure equal to -717kPa which suggests a smaller measuring range for this HCT compared with the previous one. This was expected because, as shown in figure 3 and table 1, the ceramic filter from the German supplier (ceramic G) has a bigger size of the largest pore compared to the ceramic filter from the American supplier (ceramic A).

No HCT was built with a ceramic filter from the English supplier (ceramic E) because of the unavailability of an extra ceramic filter from this supplier. Nevertheless, the MIP and NA tests performed on ceramic E suggest that a HCT built with such type of ceramic would have a very limited measuring range, even lower than the measuring range of the HCT built with the ceramic filter from the German supplier. However, because only one MIP and NA test was performed on ceramic E, there are still doubts about the validity of these results and it would therefore be advisable to perform a repeat test on another sample.



**Figure 4** - Evaporation test on a HCT incorporating a ceramic filter from the American supplier.



**Figure 5** - Evaporation test on HCT incorporating a ceramic filter from the German supplier.

#### 4. Conclusions

The pore size distributions of three different ceramics filters from three different suppliers (American, English and German) were analysed by mercury intrusion porosimetry and nitrogen adsorption with the aim of finding the most suitable filter to be used in the construction of high capacity tensiometers for the measurement of soil suction.

The comparison of the three pore size distributions indicates that the ceramic filter from the American supplier is the most suitable one for the construction of HCTs because this filter has the smallest size of the largest pore (165-220nm in diameter) and therefore the highest value of AEV (about 1500kPa), which corresponds to the largest suction measurement range. In comparison, the ceramic from the German supplier has a bigger size of the largest pore (250-290nm in diameter) and, therefore, a lower value of the AEV and a smaller suction measurement range. However, the ceramic from the German supplier is much harder and tougher than the other two ceramics, which makes it particularly suited for the construction of HCTs to be used in the field. The ceramic from the English supplier shows the biggest size of the largest pore (580-670nm) of all ceramics and is therefore considered to be the least suited for the construction of HCTs because of its low AEV and limited

measurement range. However, given that only one test has been performed on this type of ceramic, it is not possible at this stage to reach a full conclusion about the performance of this ceramic filter.

The two ceramic filters from the American and German suppliers were also used for the construction of two HCTs according to the same design. Results from fast evaporation tests indicate that the HCT built with the ceramic from the American supplier measured a maximum value of suction of about 1500kPa, which is greater than the maximum suction of about 700kPa measured by the HCT incorporating the ceramic filter from the German supplier. This is an expected result which is consistent with the differences in pore sizes between these two types of ceramic.

## 5. Acknowledgements

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