

The Use of Hydrophobic and Hydrophilic Tensiometers in the Measurement of Water and NAPL Suctions and Determination of SLRC

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Abstract. This technical note presents some preliminary results obtained with the use of hydrophobic and hydrophilic tensiometers to measure liquid pressures in three-phase systems (air-NAPL-water). The porous tips of the hydrophobic tensiometers underwent a surface treatment known as silanization. The silanized tensiometers demonstrated little influence of the interstitial water in the measured values of NAPL suction, the contrary occurring in the case of the standard ones. Due to water preferential wettability, the water tensiometers with standard porous stone tips presented adequate hydrophilic behavior, measuring water suction satisfactorily. These tensiometers were also used to determine the soil-liquid retention curves (SLRC) of an eolian sand by vaporization. Water and diesel were used in the performed tests. The performance of the vaporization technique was satisfactory, reducing the time required for test accomplishment and presenting repeatable results. In the case of diesel, due its low vapor pressure and the selective nature of its vaporization process, the use of this technique was shown to be limited. Sample heating was used to accelerate the vaporization process.

Keywords: vaporization technique, soil-liquid retention curves, NAPL, multiphase flow.

1. Introduction

Multiphase flow normally involves several processes that occur simultaneously in the soil during the transport of a non aqueous phase liquid (NAPL) from the soil surface to the water table. NAPLs are divided into two groups: Those denser than water (DNAPL) and those lighter than water (LNAPL). During LNAPL migration when the water table is reached, there is an accumulation and posterior spreading of the liquid in the contact between the two phases. Contrarily, DNAPL moves continuously down (below the water table) until an impervious layer is found. The main phenomena occurring during NAPL transport through the subsoil are: free phase flow (NAPL flow as a separate liquid phase), NAPL free phase adsorption/desorption by the soil solid particles, dissolution and transport of the NAPL soluble part (which in its turn involves adsorption, advection and diffusion) and volatilization and transport of the NAPL vapor involving the same phenomena cited above.

One of the most important aspects of NAPL transport is the NAPL free phase flow from soil surface to water table across the vadose zone or the unsaturated layer of soil. This layer acts naturally protecting the water table from NAPL contamination and the adoption of remediation techniques before NAPL reaches the saturated zone always leads to less expensive remediation procedures.

On the other hand, unfortunately, the simultaneous transport of three or more liquid phases in unsaturated soils is a complex matter. Liquid distribution inside soil pores will be a function of their wettability order which in turn can vary with the amount of organic matter in the soil as it is a function of the ranking of the superficial tension values of the liquids involved in the process. Water usually presents the highest values of superficial tension when in contact with most mineral surfaces whereas NAPL presents higher values of superficial tension when in contact with organic surfaces. In the case of unsaturated soils with a negligible organic content, wettability order is water-NAPL-air (more to less wettable). In the case of soils presenting high amounts of organic matter the wettability order changes and we have NAPL-water-air. As the water has the highest wettability in most cases involving multiphase flow, it tends to occupy the smaller pores of the soil. NAPL tends to occupy the remaining spaces and the interstitial air fills only the larger voids in the soil. As a consequence, the concept of suction must be extended in comparison to traditional unsaturated soil mechanics. At least two values of suction can be adopted: one calculated as the difference between the pressures of NAPL and water and the other considering the pressures of air and NAPL.

As regards the suction of water and NAPL due to capillarity effects, not only is the water superficial tension important but also the NAPL superficial tension and the

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Submitted on September 29, 2010; Final Acceptance on February 14, 2011; Discussion open until April 30, 2012.

tension in the interface NAPL-Water. Besides this, NAPL normally presents a low dielectric constant (ϵ_r) or low polarity compared to water ($\epsilon_{rw} = 80$ and $\epsilon_{mapl} \sim 2$). In this case, the thickness of the NAPL double layer is smaller due to a less pronounced electrical attraction between the solid particle surface in the NAPL molecules. Therefore it can be said that the adsorption phenomena will be much more pronounced in the case of water than NAPL.

In order to model water unsaturated flow the hydraulic conductivity and soil-water retention curves of the soil must be known. The model proposed by Van Genuchten (1980) is most commonly used model to represent the soil-water retention curve. Despite the fact that this model does not address the possibility of completely dry soil, as in the model of Fredlund & Xing (1994), it is simple and can be used for the values of suction normally found in the field. In the case of problems involving shallow depths in dry and hot climates and/or very low water content values, however, the use of the Fredlund & Xing (1994) equation becomes more attractive.

$$\theta = \theta_r + \frac{\theta_s - \theta_r}{[1 + (\alpha\psi)^n]^m} \quad (1)$$

where θ is the soil volumetric water content; θ_r (-) is the soil residual volumetric water content; θ_s (-) is the soil saturated volumetric water content, ψ is the water suction (kPa) and n (-), m (-), and α (kPa⁻¹) are soil parameters.

From a historical perspective, the physics of three phase flow on porous media has appeared in the petroleum reservoir literature. These models are used to model oil and natural gas recovery from petroleum reservoirs (Collins, 1961; Corey, 1986, Aziz & Settari, 1979 and Marle, 1981). Due to the arduous experimental procedures to determine truly multiphase soil-liquid retention curves (SLRC), several attempts have been made to transform one curve obtained for a pair of liquids (mostly air-water) to a multiphase curve or to one of the possible two phase systems (air-NAPL or NAPL-water). In many cases in such situations the Van Genuchten (1980) equation is employed.

Capillary scaling is the most common idea used to transform one soil-liquid retention curves into another one. Lenhard & Parker (1988) and USEPA (1997) present some scaling rules to transform soil-liquid curves from one pair of fluids to multiphase curves. In the case of USEPA (1997), the main assumptions are listed below. Equations (2) and (3) define the suctions involving the immiscible phases of the system

$$\psi_{nw} = \psi_{nw}(\theta_w) = u_n - u_w \quad (2)$$

$$\psi_{an} = \psi_{an}(\theta_{nw}) = u_a - u_n \quad (3)$$

In these equations, the subscripts *a*, *w* and *n* stand for the phases air, water and NAPL, respectively. θ_w (-) means the total volumetric content of liquids (water and NAPL) in

the system. u_a , u_w and u_n (kPa) are the interstitial pressures of air, water and NAPL, respectively. It can be noted from Eq. (3) that the suction between the phases air and NAPL is a function of θ_w . This equation brings implicitly the idea of wettability of the phases. As NAPL has intermediate wettability, in soil pores with water and NAPL, NAPL will be in contact with the interstitial air. Water will preferentially occupy the inner part of the pore and/or the the smaller pores of the soil. In this case, the radius of the air-NAPL meniscus will be a function of the total amount of liquids, which is represented by θ_w .

The suction air-water is calculated using Eq. (4). Besides this, the values of suction between the immiscible phases are scaled using the values of interfacial tensions along the interfaces between the air and water phases (σ_{aw}), the NAPL and water phases (σ_{nw}) and the air and NAPL phases (σ_{an}), according to Eq. (5) (Leverett, 1941).

$$\Psi_{aw} = \psi_{an} + \psi_{nw} \quad (4)$$

$$\frac{\Psi_{aw}}{\sigma_{aw}} = \frac{\psi_{an}}{\sigma_{an}} + \frac{\psi_{nw}}{\sigma_{nw}} \quad (5)$$

In order for Eqs. (4) and (5) to be compatible, another assumption must be made which is represented by Eq. (6). As can be seen, the interfacial tension air-water (or the water superficial tension) is assumed to be equal to the sum of the interfacial tensions NAPL-water and air-NAPL.

$$\sigma_{aw} = \sigma_{an} + \sigma_{nw} \quad (6)$$

The wettability order used in this paper is controversial among some authors such as Bradford & Leij (1995 and 1996), mainly due to the fact that NAPL is a mixture of many different chemicals and/or when the soil is mineralogically heterogeneous and contains a significant amount of organic matter. In this case of the experiments performed by the authors a 5% solution of octadecyltrichlorosilane, OTS, in ethanol was added to a sand (25% fine, 50% medium and 25% coarse) to form a totally hydrophobic medium, which was mixed in different proportions with pure sand in order to form a medium with fractional wettability. The constraints of the Eqs. (4) to (7) are also questioned by authors such as Wilson *et al.* (1990) and McBride *et al.* (1992).

Using the aforementioned assumptions the equations below summarize some of the possibilities of the use of the capillary scaling to enable Eq. (1) to be used in the case of multiphase flow.

$$\theta_w = f(\psi_{nw}) = \theta_{wr} + \frac{\theta_s - \theta_{wr}}{[1 + (\alpha\psi_{nw})^n]^m} \quad (7)$$

$$\theta_{nw} = f(\psi_{an}) = \theta_{nwr} + \frac{\theta_s - \theta_{nwr}}{[1 + (\alpha\psi_{an})^n]^m} \quad (8)$$

where θ_w (-) is the volumetric content of water (the same of θ in Eq. (1)); θ_{nw} (-) is the volumetric content of NAPL; θ_{nr} (-) is the volumetric content of liquids (water + NAPL); θ_{wr} (-) is the residual volumetric content of water; and θ_{nr} (-) is the residual volumetric content of liquids (water + NAPL).

The value of the volumetric water content of air, θ_a (-) is obtained using the equation below:

$$\theta_a = n - \theta_{nr} \quad (9)$$

where n (-) is the soil porosity.

Table 1 presents the values of interfacial tension of some liquids. As can be noted the interfacial tension air-liquid (also called superficial tension) of the NAPLS are about one third of the value obtained for water. On the other hand, comparing the values of interfacial tensions air-liquid and NAPL-water the assumption embodied in Eq. (6) is only fairly fulfilled in the case of benzene and toluene. In the case of diesel which is a mixture of different chemicals Eq. (6) can not be applied.

Several experimental methods have been proposed and reported to determine the soil-water retention curves in bi-phase systems (Machado & Dourado, 2001, Lenhard & Parker, 1988; Mahler & Oliveira, 1998; Feuerharmel *et al.* 2004, Oliveira & Marinho, 2008, Fredlund & Rahardjo, 1993, etc.). Axis translation and its variations is still the most widely spread technique used to determine the soil-water retention curve, however, some other techniques such as osmotic devices, air relative humidity and temperature control, paper filter and vaporization techniques have gained attention in recent years. Vaporization (or evaporation) are techniques where soil is left to evaporate its liquid content in a controlled way. The attention of many researchers has focused on the case of the determination of SLRC using materials with a high vapor pressure. According to Oliveira (1995), in sandy soils, the combination of the vaporization technique and the use of contact tensiometers for soil suction measurements resulted in similar results to those obtained using *tempe cells*. In this case, only bi-phase systems (air-NAPL and air-water) were used. The interstitial liquids were water, ethylene glycol, 4-clorotoluene and n-hexanol. With the use of the vaporization technique the time required to finish the tests was reduced substantially.

In the case of multiphase systems, however, using the axis translation technique or otherwise, there is the need for an additional medium to separate the interstitial pressures of water and NAPL. This is normally achieved transform-

Table 1 - Values of interfacial tension of some liquids.

Liquid	Air-liquid interfacial tension (dynes/cm)	NAPL-water interfacial tension (dynes/cm)
Benzene	28.9 (20 °C)	35.0(25 °C)
Toluene	29.0 (20 °C)	36.1(25 °C)
Diesel	26.9 (25 °C)	22.3 (25 °C)
Water	71.97 (25 °C)	-

ing a standard (hydrophilic) porous medium, such as a high entry value ceramic disk, into a hydrophobic one. The silanization technique is one of the alternatives to perform this conversion, as described by Lenhard & Parker (1988).

This paper presents the performance of hydrophilic and hydrophobic contact tensiometers of small dimensions, developed by the authors, used to measure interstitial pressures of water and NAPL (Diesel) in a multiphase flow system. Contact tensiometers, saturated with oil and water, were also used in the vaporization technique in order to obtain SLRC in an eolian sand. The obtained results are compared with the results provided by Eqs. (4) to (8).

2. Materials and Methods

The soil used was a uniform eolian sand, typical of the dunes found around Salvador, Bahia (coast material, quaternary age). The physical properties of the dune were obtained using the following Brazilian Standards: NBR 6457/1986, NBR 6502/1995, NBR 6508/1984, NBR 13292/1995 and NBR 7181/1984. Samples presented average values of porosity $n = 0.37$, 100% of sand fraction (mainly fine to medium sand) and unit weight of solid particles $\rho_s = 2.68 \text{ g/cm}^3$. Soil samples were classified as SP (poor graded in the USCS). Liquids used in the performed tests were water and diesel. Table 2 summarizes the main properties of interest of the liquids used. Interfacial tensions are given in Table 1.

Average values of fluid conductivity (k) were $k = 1.2 \times 10^{-2} \text{ cm/s}$ (water) and $k = 4.34 \times 10^{-3} \text{ cm/s}$ (Diesel). These values are compatible with the concept of intrinsic permeability of Nutting (1930).

$$k = \frac{K \cdot \rho \cdot g}{\mu} \quad (10)$$

where K is the soil intrinsic permeability [L^2], ρ is the fluid density [ML^{-3}], μ is the fluid dynamic viscosity [$ML^{-1}T^{-1}$] and g is the gravity acceleration [LT^{-2}].

Table 2 - Main liquid properties.

Liquid	ρ (g/cm^3)	μ (cp)	Vapor pressure, 20 °C (mmHg)	ε (-)	Water solubility (mL/L)
Water	1.00	1.00	17.535	80.0	∞
Diesel	0.83	3.75	< 5	2,31	298

Vaporization tests used two different types of vaporization cells. In the first set of tests a stainless vaporization cell was used similar to that used by Oliveira (1995), with nominal internal dimensions of 50 x 20 mm. Standard tensiometers, saturated with oil or water, were employed. The pressure transducers used had a pressure range of 0-100 kPa (absolute pressure) and the porous stone tips of the tensiometers had an air entry value of 100 kPa (Model 0604D04-B01M1, Soilmoisture Equipment Corp.). Tensiometers had an internal chamber of about 200 mm³. Due to their relatively large inner chamber they are able to sustain suctions only up to 70 kPa. Figure 1 presents the cell and tensiometers used in the first set of tests. The maximum time interval for suction measurement stabilization was about 10 min (suctions of 8 and 16 kPa were applied to the water reservoir containing the tensiometer porous tip). The obtained time-response curves were considered satisfactory taking into account the vaporization rate of the experiments. All the tensiometers were saturated (with water or Diesel) under vacuum before tests in a vacuum chamber designed specially for this purpose. Figure 2 presents details of the developed tensiometers. The picture below shows the coupling of the pressure transducer to the tensiometer tip and its cross section, the ceramic tips used and the coupling of the tensiometers to the flow channel (not used in this technical note).

Soil samples were statically compacted in thin layers of 2 mm in the vaporization chamber until desired dry density was reached ($\rho_d = 1.69 \text{ g/cm}^3$). Sample saturation was carried out by the use of vacuum pressure and a drip of the desired liquid. This procedure was adopted to fully saturate



Figure 1 - Vaporization chamber used in the first set of tests, tensiometer and porous tip.

the samples before the tests. During the performed tests samples were exposed to the atmosphere in a chapel with exhaustion, allowed to evaporate its liquid content for some period, weighed and then closed using the cell cover. The tensiometer was then placed in contact with the sample until the suction measurement stabilized. This process was repeated until the evaporation rate became negligible. As the evaporation rate decreased during the test, the periodicity of the readings at the beginning of the tests was higher. Similar procedures were used for both water and diesel.

In the case of the diesel however, the evaporation rate became negligible even with the sample clearly presenting high values of saturation. A process of selective evapora-

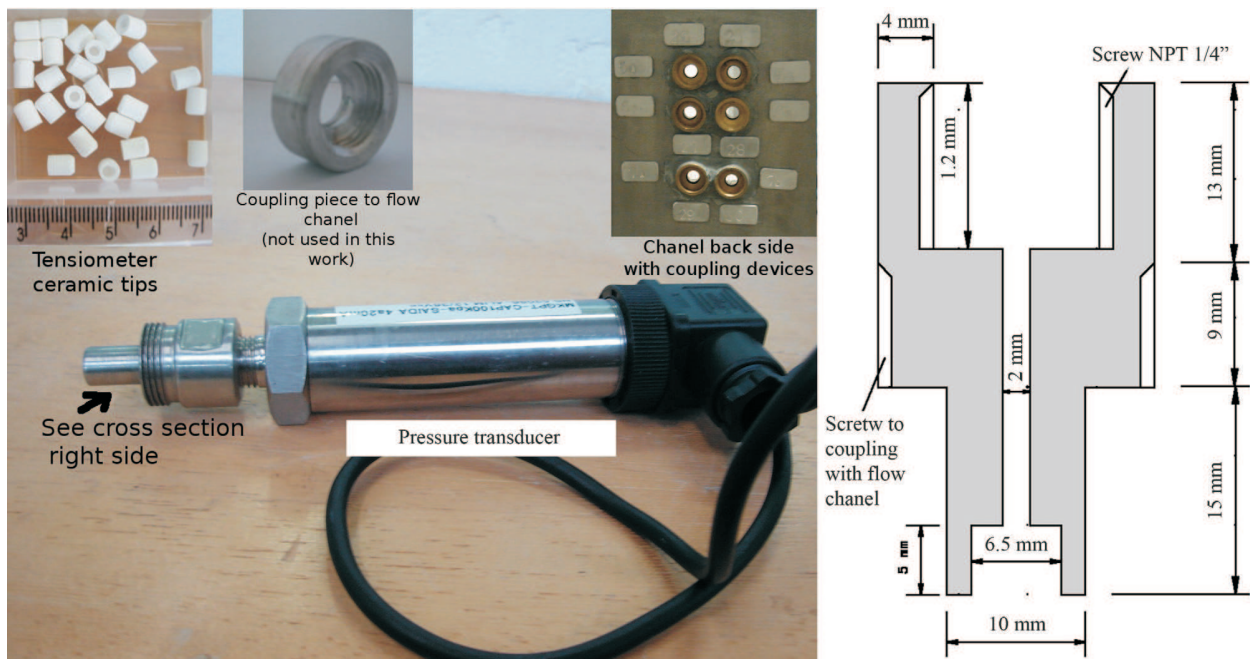


Figure 2 - Details of the tensiometer tip and couplings with pressure transducer and flow channel.

tion of the diesel components was observed in such a way that this product changed its characteristics during the evaporation process, decreasing its vapor pressure drastically and almost ceasing the evaporation process. In order to increase the vapor pressure of the remaining diesel components, soil samples were submitted to progressive heating up to 220 °C. During this process, for each heating step, samples were cooled and weighed and the suction measured as described above.

A second set of tests used the apparatus illustrated in Fig. 3. In this case both standard and silanized tensiometers were used. The silanization process modifies the surface of silica and metal oxides with the introduction of a layer chemically bonded to the substrate. There are many silanization techniques, however, the organic silanization in anhydrous conditions is the most commonly used for silica modification. Although there is the possibility that some parts of the porous stone surface are not accessible to the silanization agent, this process is considered irreversible and the hydrophobic nature of the transformed porous stone is considered permanent (Lenhard & Parker, 1988 and Kecharvazi *et al.* 2005)

In this paper, the silanization process was similar to that proposed by Lenhard & Parker (1988). After heating the porous stones for water removal (100 °C, 24 h), they were cooled to environment temperature in a dissector and then immersed in chloretrimetilsilane (PA; 98%), in an hermetic chamber for two hours. After this samples were washed with toluene (PA; 99.5%) and methanol (PA; 99,9%).

The apparatus used to perform the multiphase tests is composed of a Nylon cell with internal nominal dimensions



Figure 3 - Multiphase system suction measurement.

of 50 mm x 6 mm, a balance and a pair of tensiometers, one saturated with water and the other with oil, which was installed to measure the interstitial pressures. Two types of tensiometers saturated with oil were used, with silanized and standard tips. The idea was to observe the benefits of the silanization technique in the tensiometers performance. The compaction process was similar to that used in the first set of tests, however, in the case of the second set of tests the evaporation process was carried out continuously as well as the suction and mass measurements.

Samples were first saturated by water and then submitted to vaporization. One experiment of the second set of tests was performed keeping the room closed while the other was performed with all the windows of the room open (better ventilation conditions).

After the desired water suction was reached, samples were saturated with oil and the changes in the interstitial pressures were recorded. The water suction measurements made during the water vaporization stage were also used to obtain the soil-water retention curve of the samples.

3. Results and Discussion

Figure 4 presents the soil-water retention curves obtained using the two vaporization cells used in the tests. As can be observed, all the samples presented similar results, showing the equivalence of the techniques used. Figure 4 also shows the fitting of Eq. (1) to experimental results. Minimum square method was used in the fitting process. The obtained soil parameters were $\theta_s = 0.37$, $\theta = 0.25 \text{ kPa}^{-1}$, $\theta_r = 0.0004$, $m = 0.77$ and $n = 4.38$ ($R^2 = 0,99$). Figure 5 presents soil-liquid retention curves obtained for water and diesel.

As can be observed, diesel experimental points are located to the left of the average water curve. This had been expected since according to Eq. (5) and data presented in Table 1, due to the smaller air-diesel superficial tension compared to water, smaller suction values are expected for the same liquid content. Due to the nature of the soil used

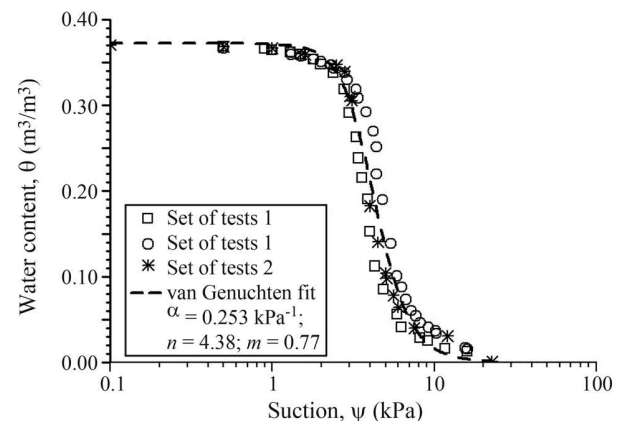


Figure 4 - Soil water retention curves using the two vaporization cells.

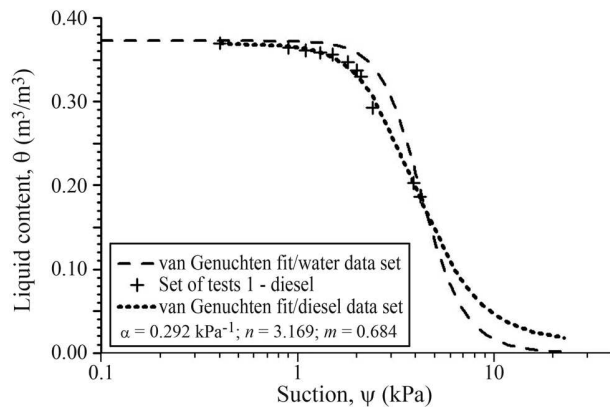


Figure 5 - Soil-liquid retention curves for water and Diesel.

(an eolian medium to fine sand) the influence of the adsorption phenomena in the soil suction values is considered negligible. Figure 5 also shows the fitting of Eq. (1) to experimental results. The obtained soil parameters were $\theta_s = 0.37$, $\theta = 0.29 \text{ kPa}^{-1}$, $\theta_r = 0.012$, $m = 0.684$ and $n = 3.17$ ($R^2 = 0.99$). Figure 6 presents the fitted SLRC (diesel and water) and the soil-diesel curve estimated using Eq. (5) (capillary scaling).

As can be observed, the use of the capillary scaling concept did not expected reasonable results. The observed reduction in the interstitial suction was much less than previewed by the use of Eq. (5). It must be noted that the capillarity scaling technique assumes suction as proportional to fluid superficial tension (air-liquid interfacial tension values shown in Table 1).

As the tested samples presented similar physical indexes and the soil structure is not a matter of concern because of the soil texture, other reasons must be found to justify the observed experimental results. One possible explanation for such behavior is that diesel is a mixture of compounds with varying vapor pressures. Therefore the evaporation process is selective and only the compounds

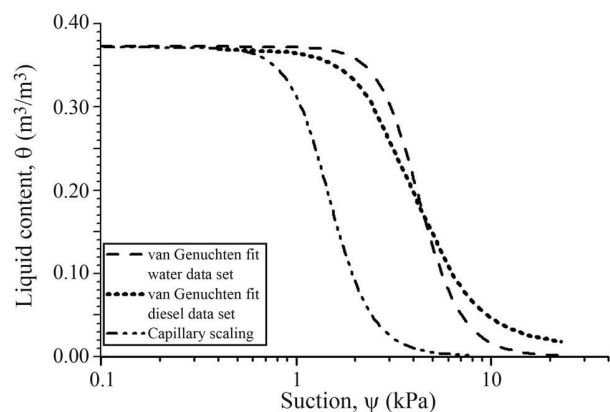


Figure 6 - Fitted soil-liquid retention curves and soil-Diesel capillary scaling curve (use of Eq. (5)).

with very low vapor pressures remain in the soil. After about a 30% reduction in the initial degree of saturation, the interstitial fluid of the soil becomes much more viscous than the original diesel used for sample saturation and its color is clearly changed. These findings are in accordance with previous observations made by Bradford & Leij (1995 and 1996) concerning the use of capillary scaling.

Table 3 illustrates how the diesel interfacial tension changes as a function of the amount of liquid already evaporated (ESTC, 1997). All the presented values refer to a temperature of 25 °C. As can be observed from Table 3, air-liquid interfacial tension tends to increase with the evaporation process, the opposite occurring with the NAPL-water interfacial tension. Furthermore, according to (ESTC, 1997), diesel viscosity remained almost constant for 25 °C. For values of temperature of 0 °C and 15 °C however, viscosity increased with the evaporation process. Volatile Organic Compounds decreased from 17793 ppm (about 1.8%) to 272 ppm when the diesel evaporated 14%.

The data presented in Tables 1 and 3 help to understand why capillary scaling seems to fail in the case of diesel. Not only is Eq. (6) not satisfied, considering the fluid with its original characteristics, but the liquid also changes its interfacial tensions as the evaporation process progresses.

Despite these experimental aspects the evaporation technique proved to be very useful and repeatable and reduced the time required to carry out the experiment accomplishment. This is particularly true in the case of fluids having high vapor pressures. In the first set of tests, the average time required to carry out the tests was about 5 days (soil water retention curve). In the second set of tests, the experiment performed keeping the room closed lasted about 90 h while the experiment performed with better ventilation conditions required about 24 h to complete. In the case of clayey soils, however, due its lower fluid conductivity, the time required to finish the tests must be higher and the replacement of the used tensiometers for micro-tensiometers, which are able to sustain values of suction higher than 70 kPa without cavitation is mandatory. Figures 7 and 8 present the performance of the tensiometers used in the second set of tests. As mentioned before, in this case samples were first saturated with water and then submitted to continuous vaporization until the desired value of suction was reached. After that, samples were saturated with diesel.

Table 3 - Changes in some diesel properties in function of the progress of the evaporation process.

Diesel evaporation (%)	Air-liquid interfacial tension (dynes/cm)	NAPL-water interfacial tension (dynes/cm)
0	26.9	22.3
8	27.2	20.9
14	27.4	19.8

Source: ESTC (1997).

In the case of Fig. 7, the tensiometer used had a standard porous tip. As can be observed, in the first phase of the test (water evaporation), the oil tensiometer measured similar results of suction compared with the tensiometer saturated with water (the observed differences in the first part of the experiment can be credited to the fact that tensiometers are located in different parts of the samples and that the vaporization and sample compaction are not completely uniform). In other words, the tensiometer tip behaved hydrophilically. After oil injection in the system, the suction values in the oil tensiometer drop immediately while the suction recorded in the water tensiometer presented a decrease over time.

When the tensiometer with the silanized tip (saturated with diesel) is used (Fig. 8), it is almost insensitive to water suction, presenting only a slight increase in the suction (less than 10% of the water suction) as the water evaporation process progressed. This indicates that the silanization process used was not 100% efficient.

After diesel injection the suction in the silanized tensiometer reduced to zero, while the water suction reduced

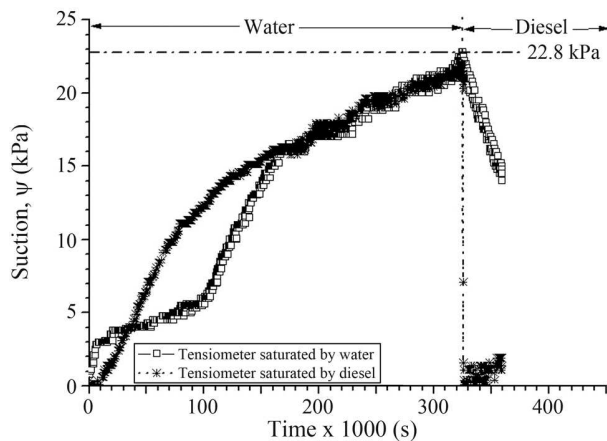


Figure 7 - Performance of the tensiometers with standard porous tip.

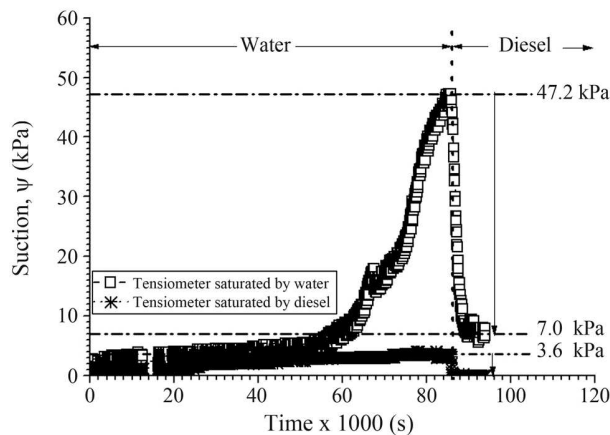


Figure 8 - Performance of the tensiometers with the silanized tip.

from 47.2 kPa to 7 kPa. This reduction was higher than predicted by the use of Eq. (5) (NAPL-water interfacial tension is about 22.3 dynes/cm, which should produce a NAPL-water suction of about 14.64 kPa after sample saturation with oil).

Analyzing the results presented in Figs. 5, 6 and 8 and considering the capillary scaling rule valid for this soil, the diesel used in the tests behaves as if it has a higher superficial tension (air-diesel interfacial tension) and a lower diesel-water interfacial tension than values presented in Table 3. These differences can be related to the selective nature of the diesel vaporization process which changes its interfacial tensions over time or simply to a different characteristic of Brazilian diesel.

4. Final Remarks

This technical note presents some preliminary results obtained with the use of the vaporization technique to obtain soil-liquid retention curves. Despite some problems due to the diesel selective evaporation process, the evaporation technique proved to be very useful and repeatable and reduced the required time for SLRC determination. This technique can be easily adapted for use in clayey soils, replacing the used tensiometers for micro-tensiometers, which are able to sustain values of suction higher than 70 kPa without cavitation.

In the second set of tests, the silanized tensiometers suffer little influence of the water suction, the contrary occurring in the case of the standard tips. Due to the water preferential wettability, water tensiometers presented satisfactory hydrophilic behavior in measuring water suction.

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