TECHNICAL NOTE

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Calibration of a Coupled Model to Predict the Magnitude of Suction Generated by Osmotic Technique With PES Membranes and Temperature Effect

Reference

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ABSTRACT

Using the osmotic technique to control soil suction requires a reliable correlation between concentration of soluble polyethylene glycol (PEG) and suction generated under isothermal condition. Experimental data to establish such correlation can be found in the literature but they tend to be incomplete and mainly pertain to cellulose membranes. Recent research has shown that polyethersulfon membranes (PES) are a better alternative than cellulose membranes in terms of durability and reliability. However, no complete calibration dataset is available for applying the osmotic technique to account for use of PES membranes and effects of different PEG molecular weights, concentrations and temperature. This study intends to fill such a gap by providing reliable and comprehensive experimental data. The response of PES membranes was investigated over a range of temperature varying from 20°C to 40°C and the results were used to calibrate a coupled model that allows a better prediction of suction generated by the osmotic technique. The influence of PEG molecular weight has been evidenced and can be incorporated in the model.

Keywords

Suction, osmotic technique, PES membrane, polyethylene glycol (PEG), unsaturated soil, temperature

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Introduction

The osmotic technique has been used since the 1970 s (Kassiff and Ben Shalom 1971) in experimental unsaturated soil mechanics to impose suction within soil specimens. The technique is relatively easy to use and applicable over a wide range of suction. Since the 1970 s, a number of experimentalists have relied on this method to characterize the behavior of partially saturated soils (Komornik et al. 1980; Delage et al. 1987, 1998; Dineen and Burland 1995; Cui and Delage 1996; Slatter et al. 2000; Tarantino and Mongiovi 2000; Monroy et al. 2007; Nowamooz and Masrouri 2008).

Implementing the osmotic technique requires knowledge of how much suction will be generated by a certain concentration of osmotic solution (typically a solution of polyethylene glycol). Delage et al. (1998) have incorporated data from previously published studies and produced a calibration equation under isothermal condition (at 20°C) that takes the simple form $s = 11c^2$, where s is the suction in MPa and c is the polyethylene glycol (PEG) concentration in grams of PEG per gram of water. Few studies have evidenced a temperature dependence of the amount of suction generated by the osmotic technique (see Michel and Kaufmann 1973; Delage and Cui 2008; Tang et al. 2010). Such temperature dependence implies that an accurate temperature control is critical for the relationship $s = 11c^2$ to hold and, hence, obtain reliable data (Delage and Cui 2008). To date, some uncertainty remains about the exact effect of temperature fluctuations on the amount of suction generated by the osmotic technique using PEG solutions.

This paper aims to fill some critical gaps associated with the osmotic technique. So far, most semi permeable membranes used to implement the osmotic technique are cellulose based, a material prone to degradation (Tarantino and Mongiovi 2000; Tripathy et al. 2011). Polyethersulfon (PES) membranes represent a more stable alternative (Monroy 2006) but there is no comprehensive dataset relating suction, PEG concentration, and temperature for such membranes. As a matter of fact, very few studies report data pertaining to PES membranes (e.g., Cunningham 2000; Slatter et al. 2000; Monroy 2006).

The significance of temperature for the osmotic technique is the second aspect requiring more data to be better understood. Although some publications provide experimental evidence of temperature dependence (e.g., Michel and Kaufmann 1973; Tang et al. 2010), its effect has been evaluated for a limited range of PEG concentration. The tests presented in this paper provide a more complete study of the temperature effect.

Finally, although Delage et al. (1998) found that the magnitude of suction generated by the osmotic technique was solely a function of the PEG concentration. Tripathy and Rees (2013) have recently observed a slight dependence on the PEG molecular weight. However, the tests were performed at a

constant temperature of about 25°C and there exists a possibility of an exacerbated effect for higher temperatures.

This paper presents the results of a comprehensive experimental program undertaken to ascertain the influence of PEG type, concentration and temperature on the magnitude of suction generated by the osmotic method implemented with PES membranes. The paper then presents a coupled model, calibrated using the experimental data and that can better predict the suction generated by the osmotic system.

Materials and Methods

POLYETHYLENE GLYCOL AND SEMI-PERMEABLE MEMBRANES

The polyethylene glycol (PEG) used in this study was supplied by Sigma Aldrich (Munich, Germany), and four different molecular weights were tested: 6000, 12,000, 20,000, and 35,000 g/mol. The PEG solutions were prepared by mixing the appropriate mass of PEG in 60 g of distilled water with a magnetic stirrer for 30 min under room temperature (20°C ± 1°C). The polyethersulfon (PES) membranes were supplied by Sartorius Stedim Biotech GmbH, Germany, and two molecular weight cut off (MWCO) were selected: 1000 and 5000 Da, to investigate a possible effect of the PEG/membrane combination. Water permeability, thickness, and impedance (ratio of thickness over permeability) of the membranes are reported in

Table 1.

TESTING SETUP AND METHOD

The testing setup is shown in Fig. 1a. The setup includes a cell containing some PEG solution with a semi permeable membrane at its base and a high-capacity tensiometer (HCT) (Mendes and Buzzi 2013) placed below the membrane. Such setup allows the direct measurement of suction generated by the osmotic solution. A conical plug prevents evaporation and allows condensed water (if any) to fall back into the solution, avoiding concentration changes. All experiments were conducted in a refrigerated incubator (temperature control range: −10°C to 40°C, ± 0.1°C, supplied by Laboratory Equipment) to maintain constant temperature, which was checked via a thermocouple immersed in the PEG solution (accuracy of ± 0.1°C). Finally, the Brix index of all PEG solutions was measured, at the beginning and the end of each test, using a hand held refractometer, to check occurrence of evaporation.

TABLE 1 Permeability and thickness of semi-permeable membranes.

Membrane	Thickness, e (μm)	Permeability, k (m/s)	Impedance, e/k (s)
PES, 1000 MWCO	124	7.6×10^{-13}	1.6×10^8
PES, 5000 MWCO	135	11×10^{-13}	1.2×10^8

FIG. 1

(a) Setup for osmotic technique calibration: HCT, high-capacity tensiometer; and (b) temperature dependence of the HCT response: relative change of pressure measured at different temperatures, pressure measured at 20°C is used as a reference.

The response of the high-capacity tensiometer was checked under the temperature range considered in this study and the temperature influence was found to be marginal. Indeed, **Fig. 1b** shows that the relative change is around 6 % at the low end of pressure (0–200 kPa, in the positive range) and falls below 2 % when the pressure exceeds 200 kPa.

TESTING PROGRAM

As a preamble, please note that not all possible combinations of membranes and PEG types have been tested in this study. A selection of combinations was made to limit the testing time. Three common PEG types were selected (6000, 20,000, and 35,000) and the possible combination of PEG and membrane molecular weight cut off (MWCO) that would not lead to diffusion of PEG across the membrane. That is why PEG6000/PES5000 was not tested. An additional PEG type (PEG12000) was considered as preliminary results revealed an influence of PEG molecular weight. Because results of the series of tests showed no influence of the membrane MWCO, only one membrane type (PES5000) was combined to PEG12000. The results presented here demonstrate that the selection of combinations tested is sufficient to draw meaningful conclusions.

Two series of tests were performed. The first series, conducted at 20°C, focus on the relationship between suction and PEG concentration. Five concentrations were tested (0.15, 0.2, 0.25, 0.3, and 0.35 g PEG/g water) on two membranes and three PEG types. These PEG concentrations were selected to cover the measurement range of the high-capacity tensiometer (up to 1.5 MPa). The PEG molecular weights considered herein are those typically used in unsaturated soil mechanics (e.g., [Delage and Cui 2008](#); [Tripathy and Rees 2013](#)). The exact combinations used are PEG6000/PES1000, PEG20000/PES1000, PEG35000/PES1000, PEG20000/PES5000, and PEG35000/PES5000.

The second series intends to provide comprehensive data on the temperature dependence of the suction generated by the

osmotic technique. These results will constitute a database to calibrate a coupled predictive model. Three PEG concentrations (0.15, 0.25, and 0.35 g PEG/g water) and four additional temperatures (25°C, 30°C, 35°C, and 40°C) were considered. In addition, several studies have investigated thermo-mechanical couplings for which higher temperatures are imposed (e.g., up to 80°C; [Cui et al. 2009](#)). Herein, the temperature range was limited to 40°C because of technical limitations of the equipment. However, the temperature range here considered is sufficient to highlight the effect of temperature on the magnitude of suction imposed by the osmotic technique. PEG types and membranes were combined as follows: PEG6000/PES1000, PEG20000/PES1000, PEG20000/PES5000, PEG12000/PES5000, and PEG35000/PES5000.

Experimental Results

Fig. 2 shows the typical evolution of suction measured with a high-capacity tensiometer during a test. It can be seen that suction rapidly reaches a plateau, which can be sustained for as long as 500 h. Note that the cavitation often occurs during a long-term suction measurement, which may be triggered by the air bubbles diffused from PEG solution toward a water reservoir of the HCT. The response visible in **Fig. 2** is an indication of the stability of PES membranes and the absence of degradation, at least during the period tested. The integrity of the membrane was corroborated by the fact that no PEG was found on the tensiometer (i.e., on the other side of the membrane). Some incremental swelling tests that were performed for another component of the research ([Yuan et al. 2016](#), not reported here) confirmed that PES membranes could satisfactorily be used for over 100 days, which is in agreement with observations by [Monroy \(2006\)](#) and [Monroy et al. \(2007\)](#).

Fig. 3 presents the results obtained under a temperature of 20°C, and it can clearly be observed that all data points fall on

FIG. 2 Evolution of suction (negative pressure) in time.

the calibration line proposed by Delage et al. (1998). Also, consistent with the conclusions by Delage et al. (1998), the suction generated by the osmotic solution (<1.5 MPa) is independent from the PEG molecular weight. However, it has been found that the molecular effect can be more pronounced at suctions higher than 1.5 MPa (Tripathy and Rees 2013). Interestingly, it is also found that the membrane molecular weight cut off plays no role, as long as no diffusion of PEG takes place across the membrane.

Tests performed under different temperatures revealed a clear and consistent trend; raising the temperature yields a progressive reduction of suction, at constant PEG concentration (Fig. 4). Interestingly, this effect is more pronounced for higher

FIG. 3 Experimental data of PEG concentration and measured suction for different combination of PEG and PES, obtained at 20°C. In the equation $s = 11000c^2$, c is in g PEG/g water, and s is in kPa. **FIG. 4** Evolution of suction for different temperatures, different PEG molecular weights, and three different concentrations, c (in g PEG/g water).

values of concentration. The other remarkable feature is that the suction generated also appears to depend on the PEG molecular weight. The effect is exacerbated under high concentrations and high temperatures. Note that no data are provided in Fig. 4 for the combination of PEG35000/PES5000 at a concentration of 0.35 g PEG/g water because it is impractical to prepare such PEG solution at the high concentration.

These results show that a comprehensive calibration equation for the osmotic method should account for three key parameters: PEG concentration, PEG molecular weight, and temperature. Such calibration will be achieved via the coupled equation proposed by Michel and Kaufmann (1973):

$$s = A_1c + A_2c^2 + A_3cT + A_4c^2T \quad (1)$$

where:

s = suction in kPa,

c = the PEG concentration in g PEG/g water,

T = the temperature in °C, and

A_1, A_2, A_3, A_4 = fitting coefficients.

The coefficients A_1 to A_4 were inferred from the data plotted in Fig. 4 for different molecular weights and are presented, in a rounded form, in Table 2. Note that the coefficients are similar for molecular weights of 20,000 and 35,000 g/mol and those data were combined for the calibration.

TABLE 2 Fitting coefficients of the coupled model (Eq 1), back calculated from the experimental data of Fig. 4.

PEG MW	A_1	A_2	A_3	A_4
6000	-19	15,000	5	-110
12,000	-1200	20,000	42	-292
20,000/35,000	-1500	21,000	48	-365

Note that data pertaining to the combination PEG20000/PES5000 (19 data points covering five concentrations and five temperatures) were not used to calibrate the coefficients A_1 to A_4 , but were reserved for the assessment of the predictive capability of the coupled model, which will be presented in Fig. 5.

In addition, data obtained on PEG8000 by Michel et al. (1983; Fig. 2) were also used in a prediction exercise. The model coefficients for PEG8000 were obtained by linear interpolation between PEG6000 and PEG12000 of Table 2, which yields $A_1 = -413$, $A_2 = 16666$, $A_3 = 17$, and $A_4 = -171$.

Fig. 5 shows that all predicted values fall very close to the experimental ones, which demonstrate that the combined

effects of PEG molecular weight, concentration, and temperature are well captured by the coupled model (Eq 1).

Conclusions

This study presents comprehensive experimental data on the suction generated by the osmotic method where a semi-permeable PES membrane is combined to PEG solutions of various concentrations. Most studies of the literature have focused on cellulose membranes and data on PES membranes are relatively scarce. In addition, although some publications have evidenced a temperature dependence, no calibration equation accounts for temperature effect and some uncertainty remain about the exact extent of the temperature influence.

A wide selection of PEG type and PEG concentration were here tested at temperatures varying from 20°C to 40°C. The results clearly show that a robust and comprehensive calibration equation for the osmotic method should account for PEG concentration, PEG molecular weight, and temperature. Consequently, a coupled model has been calibrated and its predictive capability has been tested against present data as well as existing data from the literature. It was found that the model captures the reduction of suction as the temperature increases and the dependence on the PEG molecular weight in a very satisfactory manner. Such a model can be used to adjust the expected value of suction generated during experimental testing should temperature fluctuation occur.

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FIG. 5 Comparison of the suction predicted by the calibrated model (Eq 1) and the suction measured: (a) data obtained for PEG20000/PES5000, and (b) data pertaining to PEG8000 published in Michel et al. (1983).

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