# Polyethylene Glycol and Membrane Processes Applied to Suction Control in Geotechnical Osmotic Testing

**Rick Vandoorne[a♠], Petrus J. Gräbe[b], and Gerhard Heymann[c]**

**ABSTRACT**

The osmotic technique in geotechnical testing of unsaturated soils is a promising technique to control soil-water potential. This technique is sometimes used as an alternative to the axis-translation technique. In this technique an aqueous polyethylene glycol (PEG) solution is interfaced with a selectively permeable membrane. The geotechnical literature has regularly referred to membrane effects which tend to reduce the achieved matric suction in relation to that theoretically attainable by consideration of the osmotic pressure of the bulk PEG solution. In this review, the literature on membrane science is studied in order to identify potential causes for these membrane effects. It is clear that consideration of the effects of concentration polarisation, membrane fouling and reverse solute draw has not been adequately considered or discussed in the geotechnical literature. A better understanding of membrane science and its application to geotechnical osmotic testing can aid in the optimisation of osmotic testing and the systematic improvement of the technique.

**Keywords:** geotechnical; osmotic testing; polyethylene glycol; membrane; unsaturated soil; matric suction

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[a] Department of Civil Engineering, University of Pretoria, Pretoria, Gauteng, 0083, South Africa, https://orcid.org/0000-0002-5426-8569
[♠] Corresponding author: vandoorne.r@gmail.com
[b] Department of Civil Engineering, University of Pretoria, Pretoria, Gauteng, 0083, South Africa, https://orcid.org/0000-0002-1338-6075
[c] Department of Civil Engineering, University of Pretoria, Pretoria, Gauteng, 0083, South Africa, https://orcid.org/0000-0002-2338-4073

## INTRODUCTION

Laboratory testing methods to impose soil-water potential (often called soil suction in geotechnical engineering) are vital to the development of theoretical frameworks and numerical techniques for the implementation of unsaturated soil mechanics in engineering practice (Cui & Delage 1996; Houston 2019). Most laboratory techniques focus on controlling a component of soil-water potential and then measuring the change in water content, and sometimes the change in total sample volume too. These results are then interpreted to obtain unsaturated soil property functions such as the soil-water retention curve (SWRC), hydraulic conductivity function, shrinkage curve or suction-stress characteristic curve. Determination of the SWRC is often sufficient as most other unsaturated soil property functions required for engineering practice may be derived from the SWRC (Lu, Kaya, & Godt 2014; Fredlund 2019).

The necessity to measure unsaturated soil property functions has led to the development of various laboratory equipment utilising different physical techniques to impose soil-water potential in soil specimens. The various testing methods each have their own merits and demerits. These merits and demerits arise primarily due to the manner in which soil-water potential is controlled. A review of some prevalent laboratory techniques for controlling soil-water potential is provided by Masrouri, Bicalho, & Kawai (2008). The laboratory techniques differ regarding the method by which soil-water potential is imposed. These methods are characterised by:

- the range of soil-water potential over which the method is suitable
- the component of soil-water potential being controlled (capillary potential $\psi_c$, matric potential $\psi_m$ or total internal potential $\psi_t$)
- the method used to impose the soil-water potential

The different components of soil-water potential are clearly defined in the proceeding section. Some methods reviewed by Masrouri, Bicalho, & Kawai (2008) as well as others from the literature are characterised in Table 1. References to the literature to facilitate further reading on these methods are also provided.

*Table 1.Laboratory techniques for imposing fixed soil-water potential conditions*

| Technique | $\psi_t$ or $\psi_m$ or $\psi_c$ | Range | Reference |
|---|---|---|---|
| Hanging column | $\psi_m$ | 0 to -30 kPa | Vanapalli et al. (2008) |
| Axis translation | $\psi_c$ | -10 to -1 500 kPa | Vanapalli et al. (2008) |
| Osmosis | $\psi_m$ | 0 to -10 000 kPa | Delage & Cui (2008b); Tripathy & Rees (2013) |
| Vapor equilibrium | $\psi_t$ | -3 000 to -100 000 kPa | Thomas (1921); Blatz, Cui, & Oldecop (2008) |

The hanging column method is typically limited to the control of matric potential in the range of 0 to -30 kPa due to practicalities of attaining elevation differences larger than 3 m in the laboratory. With a resolution of 0.01 kPa, the method is well suited for the determination of the air-entry value of course grained soils (Vanapalli, Nicotera, & Sharma 2008).

The axis translation technique first proposed by Hilf (1956), has undoubtedly become the prevailing method for controlling soil-water potential for unsaturated soil testing in the laboratory. It is argued that this technique has paved the way for our current understanding of unsaturated soils (Vanapalli, Nicotera, & Sharma 2008). This technique has been widely used to study unsaturated soil behaviour for the development of constitutive models (Alonso, Gens, & Josa 1990; Wheeler, Sharma, & Buisson 2003; Gallipoli et al. 2003). However, the axis-translation technique is not free of criticism (Baker & Frydman 2009; Lu 2019). The technique is aimed at keeping the capillary pressure ($u_a - u_w$) constant whilst maintaining a zero or positive pore-water pressure $u_w$. This is

done by elevating the ambient air pressure of the sample $u_a$. In this way, difficulties associated with measuring negative water pressures are avoided. However, the positive hydrostatic pressure in the soil-water does not allow cavitation which would otherwise naturally occur in in-situ soils. This influences measured soil-water retention behaviour as cavitation is a known method of liquid transport in porous media (Or & Tuller 2002).

The osmotic technique controls soil-water potential by interfacing an aqueous polyethylene glycol (PEG) solution with a soil sample through the use of a selectively permeable membrane. In theory this membrane is perfectly impermeable to PEG. This method can impose greater suctions than the axis-translation technique and it can do so without preventing cavitation of the soil-water. It thus replicates field conditions within the soil-water more closely. The method is also arguably simpler and safer to use at high potentials as high air pressures would otherwise be required in the laboratory which is undesirable (Delage & Cui 2008b).

An important objective of many laboratory tests is to replicate field conditions as closely as is practically possible. To this end, it is believed that the osmotic technique of matric potential control is a promising technique and requires further investigation. Previous research has identified that processes linked to the selectively permeable membranes used in the osmotic technique are not well understood (Dineen 1997; Delage & Cui 2008b).

PEG solution chemistry, membrane science and the interaction of the PEG solution with the membrane has not been given adequate consideration within the geotechnical literature on osmotic testing. This review is aimed at providing a concise and basic framework for persons involved in the osmotic testing of unsaturated soils so that research on the topic may progress from a fundamental basis. Chemical potential equilibrium across the membrane is considered in the context of geotechnical laboratory testing. Furthermore, membranes are considered in detail with

regard to their influence on the osmotic potential generated. This review synthesises research from the polymer science and membrane science disciplines which are scattered in the literature and applies these principles to osmotic testing of unsaturated soils.

## SOIL-WATER POTENTIAL

The use of the terms matric suction, matric potential and soil suction have become ambiguous in much of the geotechnical literature. Therefore, it is necessary to clarify terminology before proceeding. It is useful to define the total internal potential of soil-water as shown in Equation 1 as the sum of the matric and osmotic potential (Frydman & Baker 2009). In order to avoid working with negative values the concept of matric suction $s$, osmotic pressure $\pi_o$ (sometimes called osmotic suction) and total suction $s_t$ are used as defined in Equation 1. The complete definition of matric suction $s$ must be understood to include both the capillary component and the adsorbed component of the matric potential (Lu and Zhang 2019).

$$\psi_t = \psi_m + \psi_o \tag{1}$$

where:

$\psi_t$ = total internal potential, $s_t = -\psi_t$

$\psi_m$ = matric potential, $s = -\psi_m$

$\psi_o$ = osmotic potential, $\pi_o = -\psi_o$

## OSMOSIS, OSMOTIC PRESSURE AND MEMBRANE PROCESSES

Consider a system similar to that shown in Figure 1(a). Two aqueous solutions with different solute concentrations are separated by a selectively permeable membrane which is permeable only to water. Osmosis is then defined as the movement of water through the membrane due to a difference in osmotic pressure. Factors influencing chemical water potential may be in the form of osmotic potential $\psi_o$, pressure potential $\psi_p$, matric potential $\psi_m$ or any combination thereof. The movement of water is governed by the total water potential and moves from the solution of higher water potential, to the solution of lower water potential until equilibrium is achieved (Cath, Childress, & Elimelech 2006). Aqueous solutions of PEG will specifically be considered herein as this is the osmotic agent of choice in geotechnical osmotic testing.

Polyethylene glycol [H-(O-$CH_2$-$CH_2$)$_n$-OH] is an inert, non-ionic polymer. The chain length can be controlled during polymerisation to produce PEG molecules with different quantities of ethylene oxide (O-$CH_2$-$CH_2$) subunits, $n$. This changes the molecular weight $M_r$ of the molecule accordingly. PEG is also known as polyethylene oxide (PEO), polyoxyethylene (POE), or colloquially as Carbowax (Harris 1992). PEG is usually manufactured using an anionic polymerisation process (Flory 1940; Harris 1992). An anionic polymerisation process produces a relatively narrow molecular weight distribution, which under favourable conditions can be approximated using a Poisson distribution (Flory 1940; Ward 1981). In reality, it appears that the molecular weight distribution is broader than that suggested by a Poisson distribution (Fischer, Borchard, & Karas 1996).

**Osmotic Pressure and Soil-Water potential**

In Figure 1(a) an aqueous solution of PEG is separated from pure water by a selectively permeable membrane in a u-tube manometer. The membrane allows the passage of water (solvent) molecules and not the dissolved PEG (solute) molecules. Osmotic pressure $\pi_o$ arises due to the presence of a solute and is a measure of the reduction in the chemical water potential of the solution with reference to the chemical potential of pure water at the same temperature and pressure. The total chemical water potential is lowered by the participation of water molecules in the solvation of the solute. The difference in water potential drives the movement of pure water through the membrane towards the solution side.

An additional hydrostatic pressure can be applied to the solution such that the flow of water across the membrane is halted. This pressure is defined as the osmotic pressure $\pi_o$ and is shown in Figure 1(b) (Robinson & Stokes 1959). If the system is left in the configuration shown in Figure 1(a), water will be drawn from the pure water side to the solution side. This will decrease the osmotic pressure of the solution by dilution, whilst lowering the level of the pure water side and also creating an additional hydrostatic pressure by raising the solution level. This will continue until the osmotic potential of the diluted solution is balanced by the elevation difference (pressure potential) between the pure water and solution.

Let us now consider a system as shown in Figure 1(c) where a soil with its soil-water is separated from a solution by a selectively permeable membrane. At this stage, for simplicity we shall assume that the soil-water is continuous and in perfect contact with the membrane. If the membrane is impermeable to the PEG in the solution, the solution will have a corresponding osmotic pressure $\pi_o^{sol}$. If solutes are present within the soil-water and the membrane is impermeable to these solutes, the soil-water will have a corresponding osmotic pressure $\pi_o^{soil}$.

However, if there are no solutes within the soil-water or if the membrane is permeable to the soil-water solutes, the soil-water will have no osmotic potential $\pi_o^{soil} = 0$. In the first case, equilibrium is attained when $\pi_o^{sol} = \pi_o^{soil} + s$. In the second case, equilibrium is attained when $\pi_o^{sol} = s$.

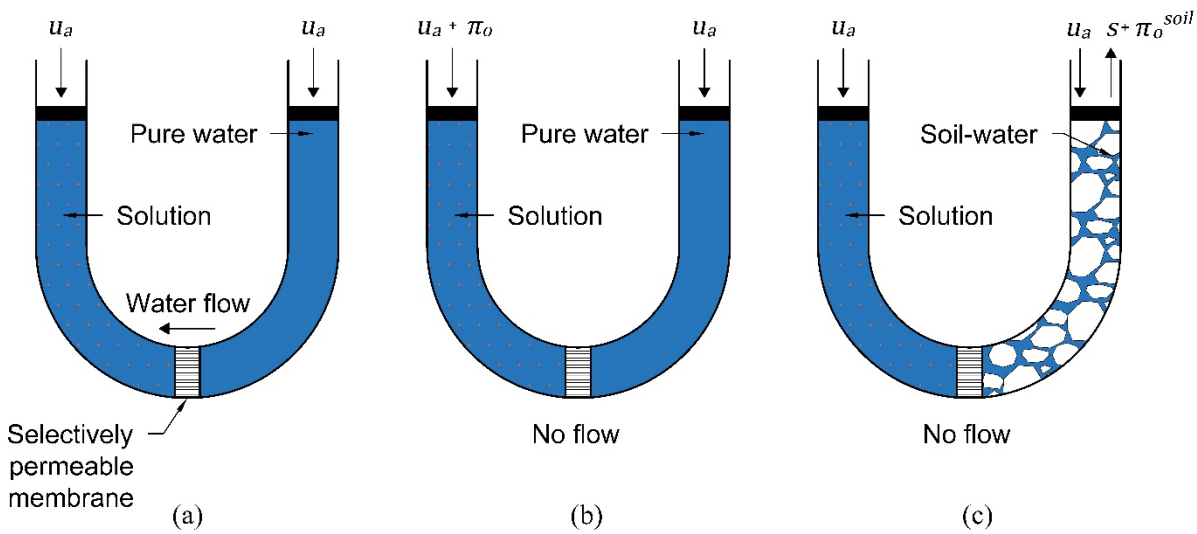


*Figure 1. Osmosis and the concept of osmotic pressure $\pi_o$. (a) Initial state of the system. Pure water will tend to flow from right (feed side) to left (draw side) through the selectively permeable membrane. (b) The system at equilibrium due to additional pressure $\pi_o$ on the solution side. (c) The osmotic pressure of the solution is balanced by the total internal soil-water suction ($s + \pi_o^{soil}$)*

Through proper selection of the membrane, one can control either the total soil-water potential or the matric soil-water potential. Total potential is controlled when the membrane is impermeable to the soil-water solutes whereas matric potential is controlled when the membrane is permeable to the soil-water solutes. In general, the membranes used for geotechnical osmotic testing are permeable to dissolved salts within the soil-water and thus the matric potential and not the total potential is controlled by the osmotic technique.

Under the assumptions of an ideal and infinitely-dilute solution Van’t Hoff's (1887) equation may be used to estimate the osmotic pressure $\pi_o$ of a solution:

$$\pi_o = RTM \qquad 2$$

where

$R$ = universal gas constant (8.314 L·kPa/K·mol)

$T$ = temperature (K)

$M$ = molarity of the solution (mol/L)

An ideal solution is one in which the volume change of mixing and change in enthalpy during mixing are zero (Rudin 1999). In an ideal solution solute-solute, solvent-solvent and solute-solvent interactions are indistinguishable from one another, obeying Raoult's law. Thus, ideality is generally approached for very dilute solutions and for solutions where the solvent and solute molecules are of a similar size and nature (Brown et al. 2018). For solutions of PEG as are relevant to this paper, Equation 2 deviates rapidly from reality as the concentration increases (Flory 1953; Manohar 1966; Money 1989). PEG molecules are much larger than water molecules and form strong hydrogen bonds in aqueous solution (Eliassi, Modarress, & Mansoori 1998). This compromises the assumptions of Raoult's law. The presence of hydrogen bonds lowers the water activity more than would be calculated by Equation 2. Therefore, application of Van't Hoff's (1887) equation to determine the osmotic pressure of aqueous PEG will result in gross errors.

The theoretically correct expression for $\pi_o$ for a real solution with a non-volatile solute is shown in Equation 3 (Feher 2017):

$$\pi_o = -\frac{RT}{\overline{V_m^w}} \ln a_w \qquad 3$$

Where:

$\overline{V_m^w}$ = partial molar volume of water (L/mol)

$a_w$ = chemical water activity (dimensionless)

The partial molar volume of water $\overline{V_m^w}$ is dependent on the concentration-density relationship of the solution which is also a function of temperature. With prior knowledge of $\overline{V_m^w}$ application of Equation 3 is trivial as $a_w$ may be determined from the equilibrium relative humidity of the water vapour in the head space above the solution (Feistel & Lovell-Smith 2017). The partial molar volume of water in an aqueous PEG solution may be approximated by the molar volume of pure water without introducing significant errors. Therefore, Equation 3 may be readily used given knowledge of $a_w$.

Without knowledge of $\overline{V_m^w}$, a virial expansion is commonly used to express the osmotic pressure of a real solution. The virial expansion aims at capturing deviations from ideality of real solutions using the empirical virial coefficients ($A_2$, $A_3$). Virial expansions for osmotic pressure in polymer science have taken many forms. However, the form shown in Equation 4 is used as given by Rudin (1999) and Flory (1953). Generally, determination of the second and third virial coefficients are sufficient (Yokozeki 2006).

$$\frac{\pi_o}{c} = RT\left[\frac{1}{\overline{M_n}} + A_2 c + A_3 c^2 + \cdots\right] \qquad 4$$

where:

$c$ = concentration of solute (g/L)

$A_2$, $A_3$ = second and third virial coefficients respectively

$\overline{M_n}$ = number-averaged molar mass of solute (g/mol)

### *Determination of Osmotic Pressure*

Osmotic pressure is a colligative property of a solution and is thus related to the other colligative properties: freezing-point depression, boiling-point elevation and vapour pressure depression (Rudin 1999). Boiling-point elevation and freezing-point depression techniques are not well suited for use with aqueous PEG solutions (Flory 1953; Sweeney & Beuchat 1993). The presence of a solute stabilises the solvent molecules decreasing their tendency to escape into the vapour phase. This manifests as vapour pressure depression. As noted by Feher (2017), vapour pressure depression and osmotic pressure are perfect predictors of each other as they are essentially the same phenomenon. Membrane osmometry or vapour pressure osmometry are thus regularly used to measure either the osmotic pressure directly or the vapour pressure depression respectively. At low concentrations, membrane osmometry is better suited than vapour pressure osmometry techniques to determine osmotic pressure (Rudin 1999).

Membrane osmometry involves the direct measurement of the osmotic pressure of the solution across a selectively permeable membrane. This may involve either the measurement of the equilibrium head difference between solutions on either side of a membrane or measurement of the solution pressure via an electronic pressure transducer at equilibrium. Membrane osmometry is limited by the permeability of solutes to the membrane being used, equilibration time and the accuracy of the pressure measurement. Membrane osmometry techniques have been successfully used by Slatter et al. (2000) and Ng et al. (2007) for calibration of the osmotic technique for geotechnical testing. Tensiometers have also been used to measure the osmotic pressure directly across the membrane (Dineen 1997; Tarantino & Mongiovì 2000; Monroy 2006; Tang et al. 2010a; Yuan, Liu, & Buzzi 2017). This is a form of membrane osmometry unique to geotechnical testing.

Vapour pressure osmometry measures the vapour pressure depression of the vapour in the

head space above the solution at equilibrium. There are several vapour pressure osmometry techniques available. These include but are not limited to (Greenspan 1977):

- Direct measurement of vapour pressure
- The dew-point method
- Isopiestic methods
- Measurement with a calibrated relative humidity sensor
- Gravimetric determination

The dew-point method has been successfully used by Steuter, Mozafar, & Goodin (1981) and Tripathy & Rees (2013) to measure the osmotic pressure of aqueous PEG over a wide concentration range. The isopiestic method has also been successfully used by McClendon (1981) and Delage, Howat, & Cui (1998). Other authors used calibrated psychrometers to determine the relative humidity of the vapour phase in equilibrium with PEG solutions (Williams & Shaykewich 1969; Michel & Kaufmann 1973; Money 1989).

**Membrane Processes**

A membrane is defined as an interphase between two bulk phases. For the current discussion, a membrane may take on the nature of a:

- Nonporous dense solid
- Microporous solid with liquid or gas in its pores

Membranes may also take on the form of liquids and gels but these are not relevant to membranes used in geotechnical osmotic testing (Ho & Sirkar 1992).

The filtration spectrum for membranes is illustrated in Figure 2 (Kubota, Hashimoto, & Mori 2008). Different filtration types are defined according to the filtered particle size. The

molecular weight scale and particle size scale as shown in Figure 2 are only an approximate relation because the true relationship between particle size and molecular weight depends on the solution chemistry, molecular configuration, molecular orientation and operating conditions of the membrane (Kulkarni, Funk, & Li 1992; Mehta & Zydney 2005).

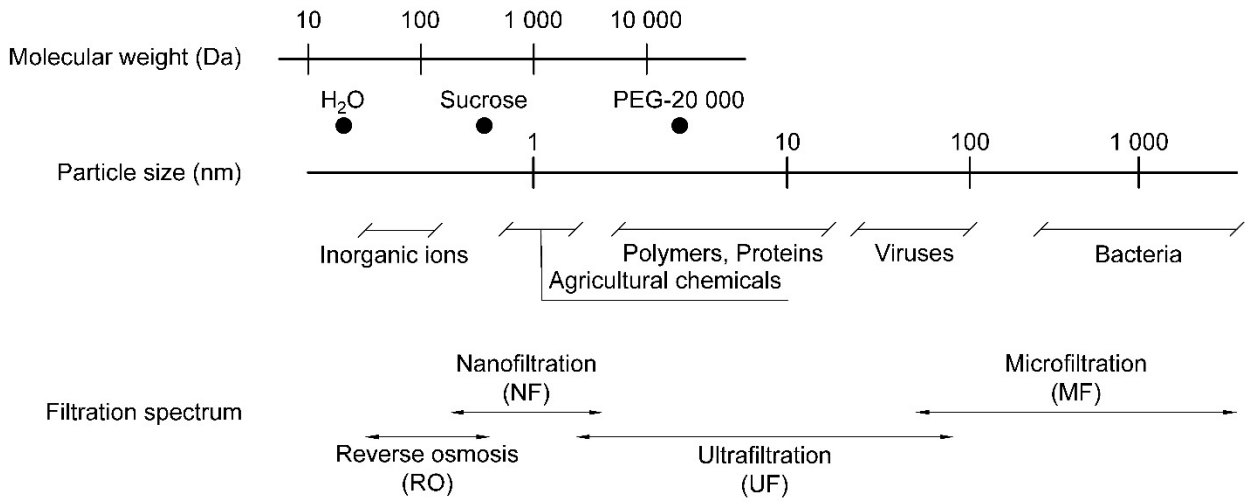


*Figure 2. The filtration spectrum (adapted from Kubota, Hashimoto, & Mori (2008))*

In geotechnical osmotic testing PEG has been exclusively used as the osmotic agent (Delage & Cui 2008b). PEG of varying molecular weight between 1 500 – 35 000 Da has been used. However, molecular weights of 6 000, 20 000 and 35 000 Da appear most common (Yuan, Liu, & Buzzi 2017). The molecular weight of the PEG is denoted using a hyphenated suffix indicating $M_r$ in Da e.g. PEG-6 000 denotes PEG with $M_r$ = 6 000 Da. PEG with molecular weights of current interest fall within the ultrafiltration (UF) range of the filtration spectrum. Theoretically, nanofiltration (NF) and reverse osmosis (RO) membranes may be used but this is not optimal as equilibrium will be slower due to the reduced water flux through the more selective membranes. Dissolved salt ions in the soil-water can easily pass through the pores of UF membranes, therefore, UF membranes

control the matric suction of the soil-water and not the total suction. If total suction control is desired, RO membranes must be used which will unfortunately also result in longer equilibrium times.

Different osmosis modes may be defined with regard to the forces driving the osmosis process (Roest 2018). These driving forces are transmembrane osmotic pressure $\Delta\pi_o$, transmembrane hydrostatic pressure $\Delta p$ and the matric suction $s$ of the soil sample. Figure 3(a) illustrates and defines the four osmosis modes in the context of geotechnical osmotic testing, namely: forward osmosis (FO), reverse osmosis (RO), pressure-assisted osmosis (PAO) and pressure-retarded osmosis (PRO). In the absence of a transmembrane pressure difference $\Delta p = 0$, FO occurs and is governed by matric suction and osmotic pressure. FO between two liquid bulk phases is equivalent to dialysis where the osmotic pressure difference $\Delta\pi_o$ is the sole driving force (Baker 2004).

It should be noted that the term RO is applied to the filtration spectrum (Figure 2) as well as to the osmosis modes (Figure 3(a)). The term RO *mode* will be used when referring to RO in the context of Figure 3(a). This ambiguity arose due to the popularity of RO filtration for the desalination of sea water which lead to the ubiquity of the term with both the membranes and the osmosis mode (Voutchkov & Semiat 2008). RO membranes differ from FO membranes in that they typically need to withstand higher transmembrane pressures $\Delta p$ of up to 100 bar (Zhang et al. 2015).

The general equation describing water transport in membrane processes (Cath, Childress, & Elimelech 2006) may be extended to include matric suction as shown in Equation 5. Figure 3(b) defines the variables of Equation 5 in the context of geotechnical osmotic testing.

$$J_w = A_w(\sigma_r \Delta\pi_o + s - \Delta p) \quad (5)$$

where:

$J_w$ = water flux ($m^3/m^2/s$)

$A_w$ = water permeability coefficient (m/s/kPa)

$\sigma_r$ = the reflection coefficient (dimensionless)

$\Delta\pi_o$ = transmembrane osmotic pressure difference, $\pi_o^{soil} - \pi_o^{PEG}$ (kPa)

$s$ = matric suction (kPa)

$\Delta p$ = transmembrane hydrostatic pressure difference, $p^{soil} - p^{PEG}$ (kPa)

Water flux $J_w$ is positive when water flows towards the soil sample as indicated in Figure 3(b). Positive values of $\Delta p$ and $\Delta\pi_o$ (as defined in Equation 5) drive water out of and into the soil sample respectively. The reflection coefficient $\sigma_r$ corrects for any permeable solutes which would tend to reduce the transmembrane osmotic pressure difference. $\sigma_r$ is a value between zero and unity and is the ratio of the actual osmotic pressure difference to the theoretical osmotic pressure difference of a perfect membrane.

When the hydrostatic pressure on both sides of the membrane is equal $\Delta p = 0$, then FO exists. Pumping the PEG solution across the membrane at an elevated pressure $p^{PEG} > 0$ whilst maintaining the soil-water side at atmospheric pressure $p^{soil} = 0$ would create a PRO or RO mode scenario depending on whether $p^{PEG} + \Delta\pi_o + s > 0$ or $p^{PEG} + \Delta\pi_o + s < 0$ respectively. Alternatively, if the PEG solution was "sucked" across the membrane surface using sub atmospheric pressure, a PAO mode process would result. Hydrostatic pressure may be an important consideration as PEG is usually circulated using a peristaltic pump in osmotic testing. Some authors prefer using negative pressures and others prefer using positive pressures. If

significant hydrostatic pressures build up under the membrane due to the high viscosity of the solution, fouling or blocking of the circulation tubes, the hydrostatic pressures could change the equilibrium matric suction of the soil sample.

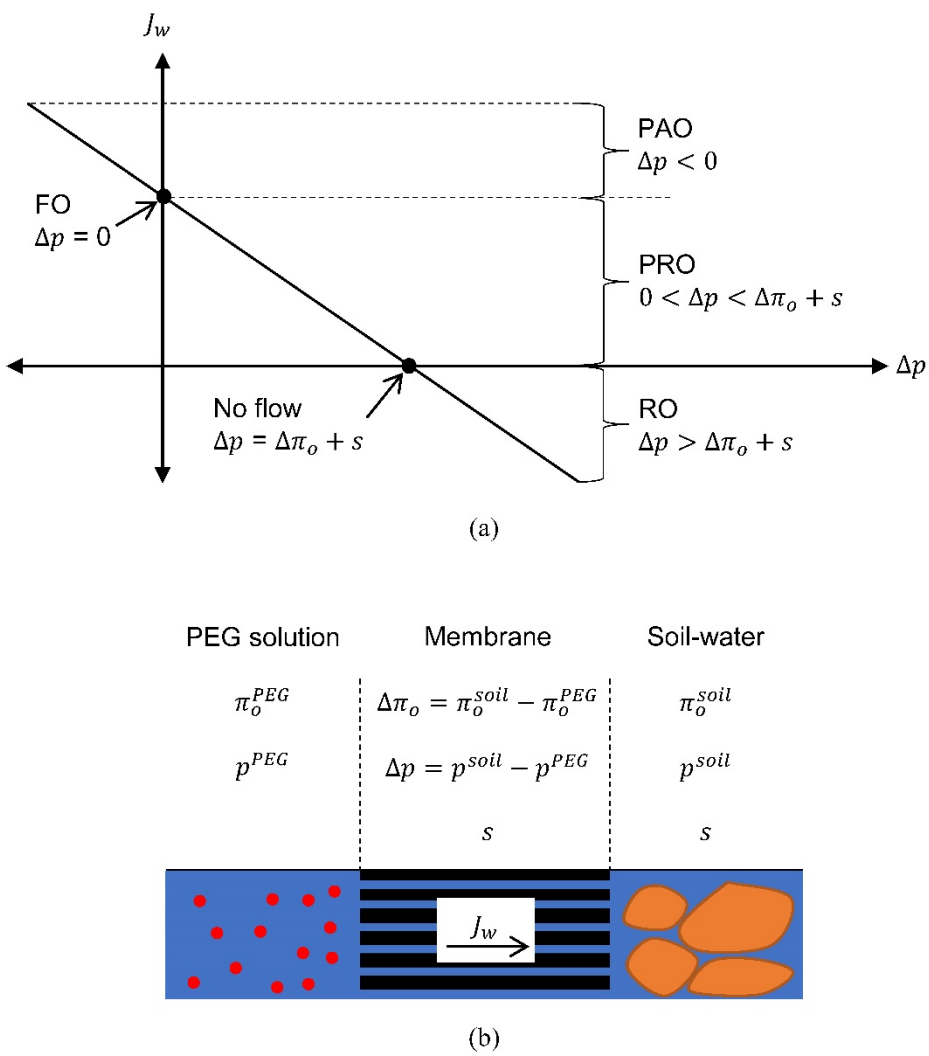


*Figure 3. (a) Definition of forward osmosis (FO), reverse osmosis (RO), pressure-retarded osmosis (PRO) and pressure-assisted osmosis (PAO) in terms of transmembrane pressure difference $\Delta p$, transmembrane osmotic pressure difference $\Delta\pi_o$ and matric suction s (b) Definition of transmembrane driving forces for water flux between a PEG solution and a soil sample*

UF and MF membranes are used in RO mode for the separation, concentration and preparation of protein, food and beverage products (Mehta & Zydney 2005). There has been renewed impetus over the last two decades in the development of membranes for use in FO mode for desalination and PRO mode for power generation from brine (Zhao et al. 2012). However, these processes generally involve particle sizes in the RO and NF range of the filtration spectrum. This means that membranes currently being developed for use in FO mode have an active layer which is not optimal for use with PEG-6 000, PEG-20 000 or PEG-35 000 as an osmotic agent. It is clear that membranes that would be optimal for use in geotechnical testing applications do not have significant economic potential currently. This has restrained research and development in this niche.

Nonetheless, the ongoing development of improved FO membranes is important to geotechnical osmotic testing as improved membrane performance will undoubtedly improve experimental performance. Previous geotechnical researchers have already shown that developments in membrane technology have allowed for tests of longer duration to be performed due to the improved bacterial resistance of newer synthetic membranes such as PES UF membranes (Monroy et al. 2007).

## MEMBRANES

Common membrane types are illustrated in Figure 4. Membranes can be divided into symmetrical and asymmetrical membranes. Generally, symmetrical membranes are isotropic in nature and asymmetrical membranes are anisotropic in nature along their cross section. A unique situation occurs for double-skinned membranes which are symmetrical but anisotropic, however, these are not common. Membranes can further be divided according to their internal structure namely microporous or nonporous and dense. Microporous membranes act similarly to a common filter and permeation can be described using pore-flow models. Membranes in the UF spectrum range are considered microporous. Nonporous dense membranes facilitate permeate flow according to the solution-diffusion model. Membranes in the RO filtration spectrum range are considered nonporous and dense (Baker 2004).

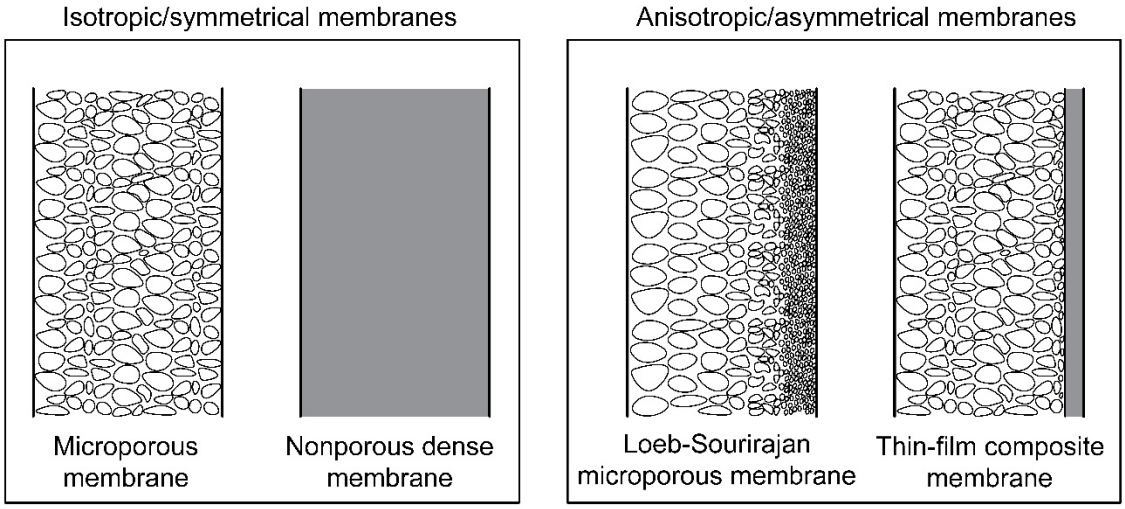


*Figure 4. Common membrane types* (adapted from Baker (2004))

Commercial membranes which operate under RO mode generally consist of an active layer and a porous support layer. The active layer is responsible for filtering of the desired solute or colloid whilst the support layer protects the active layer from damage due to any pressures associated with the RO mode filtration process. The support layer allows the active layer to be only as thick as necessary to fulfil its separation function. The support layer, however, creates asymmetry and therefore the orientation of the membrane becomes an important consideration. The combination of a thin active layer and a porous support layer has drastically improved flux in commercial processes. Using a RO membrane designed for use in RO mode in FO mode will result in poor permeate flux across the membrane as the support layer is thicker than required and creates excessive impedance (Loeb et al. 1997).

Loeb-Sourirajan type membranes consist of a porous support and a filtering active layer cast from a single material in a process known as phase inversion (Loeb & Sourirajan 1964). These membranes are also called integrally-skinned membranes as the support layer and active layer (skin) are a single material (Zhang et al. 2015). Thin-film composite membranes are constructed in two or more steps. First, the support layer is fabricated and then the active layer is bonded to the support layer. Additional layers are sometimes used. This enables the layers to be engineered independently with properties to match the layer function. Thin-film composite membranes commonly have a dense nonporous active layer bonded to a porous support layer. These membranes are currently common in commercial RO processes. The scope of the remaining discussion will focus on UF membranes as used in geotechnical osmotic testing to-date. The reader is referred to Ho & Sirkar (1992) and Baker (2004) for additional reading if so desired.

**Membrane Characterisation**

UF membranes are usually classified according to a nominal molecular weight cut-off (MWCO) rather than a pore size distribution. The latter is difficult if not impossible to define through direct methods due to the inherently heterogenous nature of membrane pores. The MWCO is typically defined as the molecular weight of a solute with a rejection coefficient (Equation 6) $R$ = 90 % (Mehta & Zydney 2005). However, this is not standardised. Different manufacturers use different solutes in determination of the MWCO. Some typical solutes used include polydextran, PEG, polyvinylpyrrolidone and certain proteins. The shape of the molecule in solution will affect how easily it may permeate through the membrane, with linear molecules permeating easier than globular (coil-like) molecules with the same molecular weight (Baker 2004). PEG molecules appear to be rigid and straight at lower concentrations and transition towards a more coiled structure as concentration increases (Michel & Kaufmann 1973).

$$R = \frac{c_f - c_d}{c_f} \qquad 6$$

Where:

$R$ = rejection coefficient (%)

$c_f$ = concentration of solute on feed side

$c_d$ = concentration of solute on draw side

Figure 5 shows the relationship between the average molecular weight $M_r$ of a solute and the average membrane pore diameter $d$ for different rejection coefficients $R$. The relationship shown in Figure 5 was established using data assembled by Sarbolouki (1982) from various researchers relating measured molecular diameter in solution to molecular weight. This was done for a number of solutes typically used in the determination of the MWCO of membranes, including PEG.

Figure 5 may be used to convert a MWCO rating of a membrane to an average pore diameter rating. However, this must be understood to be a nominal relationship.

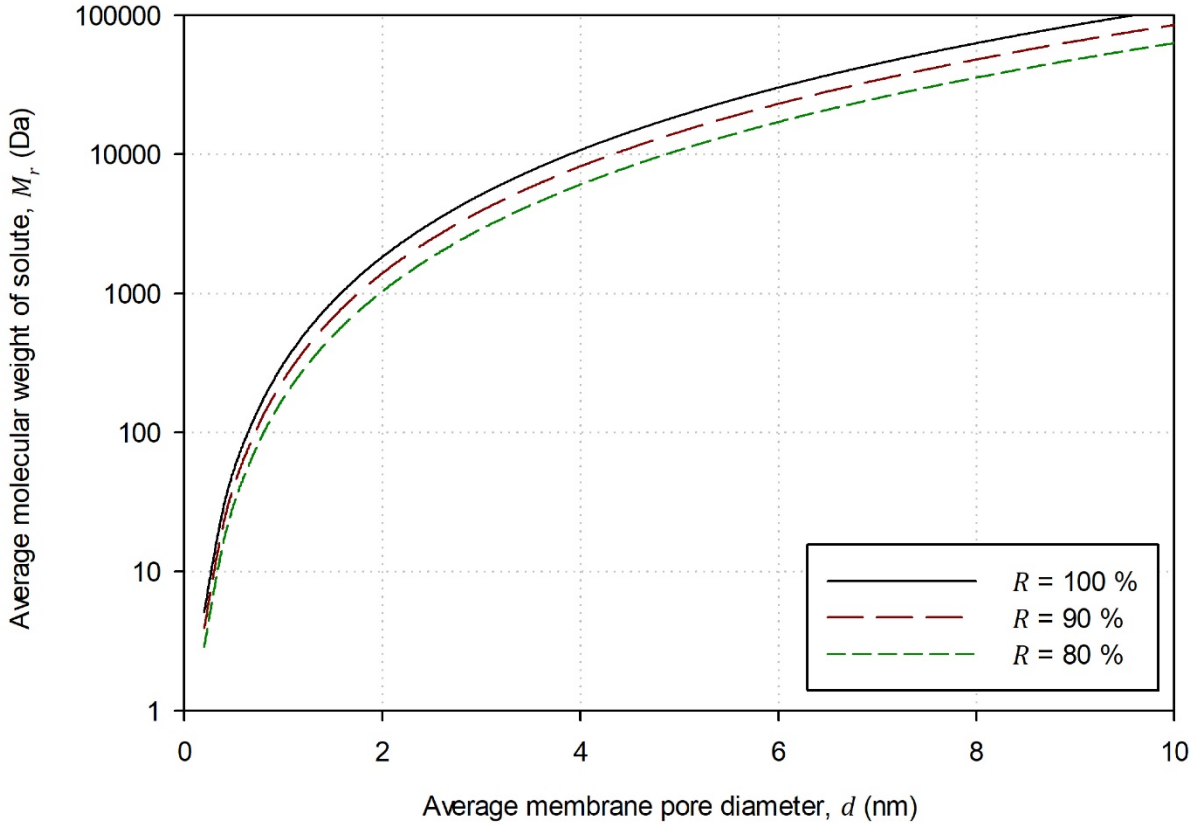


*Figure 5. Typical relationship between average membrane pore diameter and molecular weight of solute at different rejection coefficients*

## Membrane Materials

Table 28-2 in Kulkarni, Funk, & Li (1992) lists 27 different commercial manufacturers of UF membranes. Most of these manufacturers produce more than one type of UF membrane. It is reasonable to expect that the number of UF membrane products available on the market has since increased. Therefore, the number of potential UF membrane options available for use in geotechnical testing is significant. In reality, however, only a few membranes have actually been

used.

Table 2 shows details of membranes used in the geotechnical testing literature. Tripathy, Tadza, & Thomas (2011) state that membranes made from cellulose acetate (CA) are the most common membranes used in geotechnical testing. However, Table 2 shows that membranes made from regenerated cellulose (RC) are clearly the most common membrane used to-date. In order to dispel ambiguity and avoid confusion between different membrane materials, a list of the most common polymeric UF membrane materials is listed below (Kulkarni, Funk, & Li 1992):

- Regenerated cellulose (RC)
- Cellulose acetate (CA)
- Cellulose triacetate (CTA)
- Polysulfone (PS)
- Polyethersulfone (PES)
- Polyamides
- Polyvinylidenefluoride
- Polyacrylonitrile

Information pertaining to the membranes used in geotechnical testing has often been poorly documented in the literature:

- The manufacturer or supplier of the membrane used is not always stated.
- The membrane model/name or MWCO is occasionally not stated.
- There are very few hydraulic conductivity data available for the membranes used.
- The thickness of the membrane is often not reported.
- Membrane symmetry/asymmetry is rarely mentioned or considered.

- There are very few scanning electron microscope images of the membranes which could assist in understanding membrane structure and performance.

This lack of information makes comparison of membranes difficult if not impossible and hinders the systematic progression of osmotic testing. It is believed that the lack of adequate information is due to the proprietary nature of commercial membranes, a lack of understanding of the different membrane materials available, a lack of consideration of the literature on membrane science and the complexity in general of membrane science.

RC membranes (cellophane) were the first to be used for the application of soil-water potential using aqueous PEG by Zur (1966). This paved the way for the predominant use of RC membranes. However, RC membranes lack bacterial resistance. In this regard, researchers have used antibiotics such as penicillin to extend the life of the membrane (Dineen 1997; Delage & Cui 2008a). Monroy (2006), following initial trials by Colmenares (2002), showed that PES UF membranes could resist bacterial attack and maintain significant osmotic pressure for up to 20 weeks. This is an order of magnitude improvement over the maximum recommended period of two weeks by Delage & Cui (2008b) for RC membranes with the use of antibiotics.

Literature references are provided in Table 2 as well as the MWCO, manufacturer, material, hydraulic conductivity and thickness of the membranes where available. Inconsistencies are evident in the water hydraulic conductivity data provided for RC membranes. Dineen (1997) reported a hydraulic conductivity of approximately $2.2 \times 10^{-13}$ m/s for an RC membrane with MWCO 12 000-14 000 Da. However, Delage, Suraj de Silva, & Vicol (1992) and Delage, Howat, & Cui (1998) reported values of $5 \times 10^{-9}$ m/s and $1 \times 10^{-11}$ m/s respectively for RC membranes with the same MWCO. Monroy et al. (2007) reported a hydraulic conductivity of $8 \times 10^{-11}$ m/s for a PES membrane with MWCO 15 000 Da. Depending on which data are considered, the hydraulic

conductivity of the PES membrane is either two orders of magnitude greater or two orders of magnitude less than the RC membranes of similar MWCO. This may have significant implications regarding the selection of membranes for future testing. The RC membrane used by Dineen (1997) is made by a different manufacturer than the RC membranes used by Delage and co-workers. This may be partly responsible for the significant difference in measured permeabilities.

The manner in which the hydraulic conductivity data were collected may be a further reason for the observed scatter in the hydraulic conductivity data in Table 2. A range of methods were used: falling head hydraulic conductivity tests, constant head hydraulic conductivity tests and Slatter, Allman, & Smith (2000) used the osmotic pressure of the solution to determine the hydraulic conductivity rather than a hydrostatic pressure. This method introduces additional error due to concentration polarisation and related phenomena discussed in the proceeding section. It is also worth mentioning that compaction of the membranes due to transmembrane pressures can influence the thickness and pore characteristics of the membrane. Thus, in membrane science it is customary to preload a membrane with transmembrane hydrostatic pressure before testing its hydraulic conductivity to mimic testing conditions (Bhattacharjee & Bhattacharya 1992). Due to lack of adequate data, no strong conclusions can be made regarding the hydraulic conductivity of RC membranes versus the PES membranes shown in Table 2.

When selecting a membrane for testing it is more prudent to compare impedance $I$ rather than hydraulic conductivity $k_s$, as impedance accounts for the thickness $t$ of the membrane ($I = t/k_s$). Monroy (2006) notes that his PES membrane was thinner than the RC membrane used by Dineen (1997). Furthermore, the hydraulic conductivity values presented by these two authors show that the PES membrane is more permeable than the RC membrane. This would indicate that the PES membrane is superior to the RC membrane as the thinner membrane and higher hydraulic

conductivity both positively contribute to lower impedance. Unfortunately, actual thickness nor impedance values are presented in their work. These inconsistencies in the available hydraulic conductivity/thickness/impedance data could significantly influence the attractiveness of the use of PES membranes over RC membranes. Further systematic research is required in this regard.

*Table 2. Membranes and PEGs used to-date in geotechnical osmotic testing*

| ***Reference*** | ***Manufacturer/ Supplier*** | ***Membrane name/model*** | ***Material*** | ***MWCO*** ***kDa*** | ***PEG $M_r$*** ***kDa*** | ***Hydraulic conductivity*** ***$\times 10^{-12}$ m/s*** | ***Thickness*** ***µm*** | ***Impedance*** ***$\times 10^{6}$ s*** |
|---|---|---|---|---|---|---|---|---|
| Kassiff & Ben Shalom (1971) | - | - | RC | - | 6 | - | - | - |
| Suraj de Silva (1987) | Repligen | Spectra/Por® 2 | RC | 12-14 | 20 | 1.0 | 47.5 | 48 |
| Delage, Suraj de Silva, & Vicol (1992) | - | - | RC | 12-14 | 20 | 5000 | - | - |
| Cui & Delage (1996) | Repligen | Spectra/Por® 4 | RC | 12-14 | 20 | 10 | - | - |
| Dineen (1997) | Medicell Ltd | Visking dialysis tubing | RC | 12-14 | 20 | 0.15 - 0.29 | - | - |
| Delage, Howat, & Cui (1998) | - | - | RC | 14<br>4 | 20<br>6 | - | - | - |
| Slatter et al. (2000) | - | - | CA<br>PES | -<br>- | 20<br>20 | - | - | - |
| Slatter, Allman, & Smith (2000) | - | - | CA | 14 | 20 | 0.10 – 0.33 | 85 | 258 - 850 |
| | | | PES | - | - | 0.10 – 0.20 | - | - |
| Tarantino & Mongiovì (2000) | Repligen | Spectra/Por® 4 | RC | 12-14 | 20 | - | - | - |
| | | Spectra/Por® 5 | RC | 12-14 | 20 | - | - | - |
| | Viskase | Membra-cel® dialysis tubing | RC | 14 | 20 | - | - | - |
| Cuisinier & Masrouri (2005) | Repligen | Spectra/Por® 4 | RC | 12-14 | 20 | - | - | - |
| Monroy et al. (2007) | Fluid Systems® | - | PES | 15 | 35 | 80 | - | - |
| Ng et al. (2007) | Repligen | Spectra/Por® 2 | RC | 12-14 | 20 | - | - | - |
| Ghembaza et al. (2007) | Repligen | Spectra/Por® 3 | RC | 3.5 | 20 | - | - | - |
| Delage & Cui (2008a) | - | - | RC | 3.5 | 6 | - | - | - |
| Tang et al. (2010a) | Repligen | - | RC | 1 | 20 | - | - | - |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | | 3.5 | 20 | | | |
| | | | | 12-14 | 20 | | | |
| | | | | 1 | 6 | | | |
| | | | | 3.5 | 6 | | | |
| Ajdari et al. (2016) | Repligen | Spectra/Por® 4 | RC | 12-14 | 20 | - | - | |
| Yuan et al. (2017) | Sartorius Stedim Biotech | - | PES | 1000 | 6<br>20 | 0.76 | 124 | 160 |
| | | | PES | 5000 | 12<br>20<br>35 | 1.1 | 135 | 120 |

The following three membranes were imaged using a Zeiss Crossbeam 540 scanning electron microscope (SEM) in order to visualise their structure:

- Figure 6)
- a TRISEP® UF10 PES membrane with a MWCO of 10 000 Da (Figure 7)
- an OsmoF$_2$O™ FO CTA membrane from Fluid Technology Solutions with an unknown MWCO (Figure 8)

The first two membranes were selected as they are believed to be representative of typical membranes used in geotechnical osmotic testing. The third membrane is relatively new and is designed specifically for FO mode applications. Cross sectional images were taken of each membrane. The membranes were placed in a desiccator over desiccant crystals whilst under vacuum for 24 h to thoroughly dry. The membranes were then placed in liquid nitrogen in an attempt to make them brittle enough to form a clean cross section through fracturing. Only the regenerated cellulose (RC) membrane snapped successfully. The polyethersulfone (PES) and cellulose triacetate (CTA) membranes were cut with a blade. This may create some smear on the cross section.

The membrane thicknesses vary significantly. The active layer of the RC, PES and CTA membranes are approximately 75, 50 and 15 μm respectively. The RC membrane has no support layer and the cross section appears isotropic and symmetrical. The PES membrane has a support layer made from a non-woven polyester. This gives the PES membrane the largest overall thickness of approximately 150 μm. The CTA membrane has the thinnest active layer. A woven

support can be seen embedded within the membrane assisting with the structural support of the thin active layer. In geotechnical osmotic testing a woven steel or nylon mesh is sometimes used to facilitate adequate circulation of the PEG solution below the membrane. It may be worthwhile investigating whether the embedded mesh in the CTA membrane could serve this circulation function.

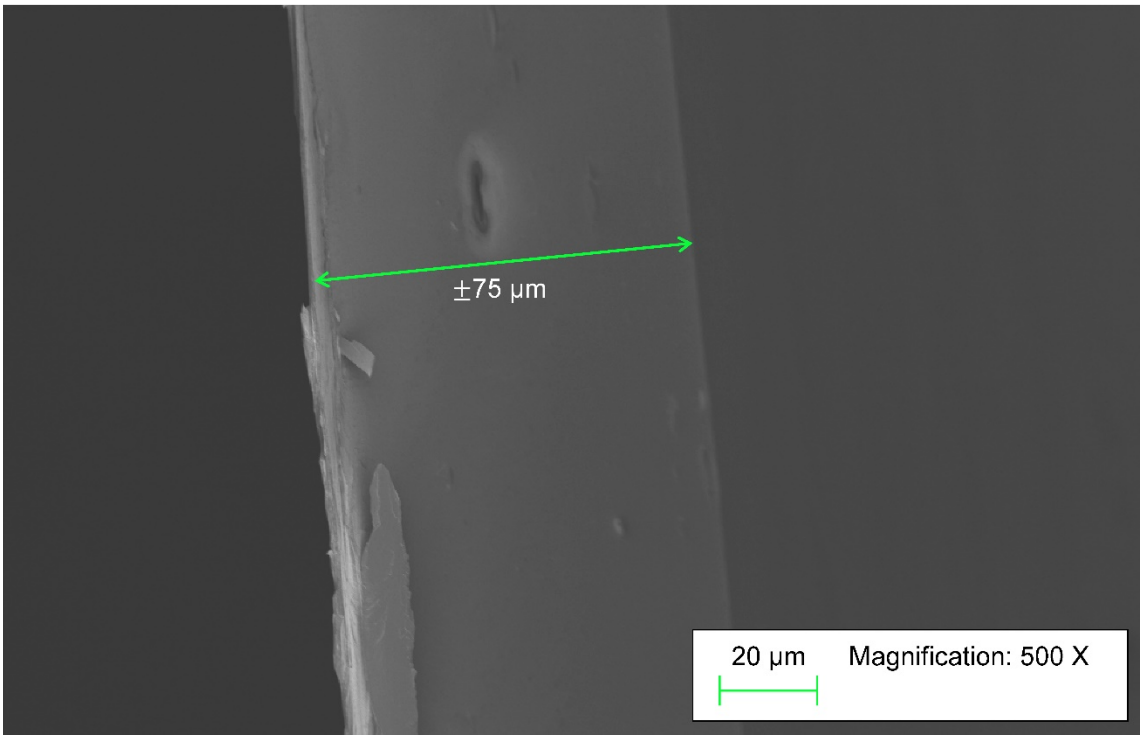


*Figure 6. SEM image of a cross section of a RC Visking dialysis membrane from Medicell Membranes Ltd with MWCO 12 000 – 14 000 Da. The membrane thickness is in the order of 75 µm. This membrane has no support layer and consists of only a single homogenous isotropic active layer.*

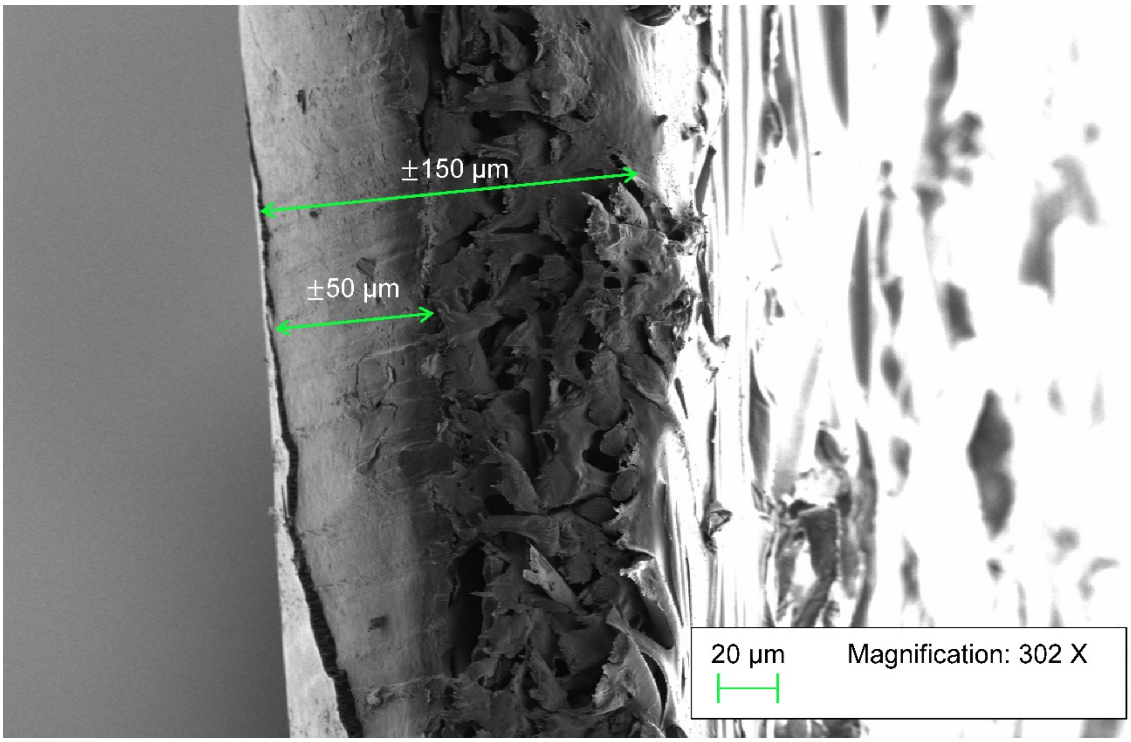


*Figure 7. SEM image of a cross section of a TRISEP® UF10 PES membrane with MWCO of 10 000. The active layer is in the order of 50 µm thick and the total membrane thickness is approximately 150 µm. The support layer is made from a non-woven polyester.*

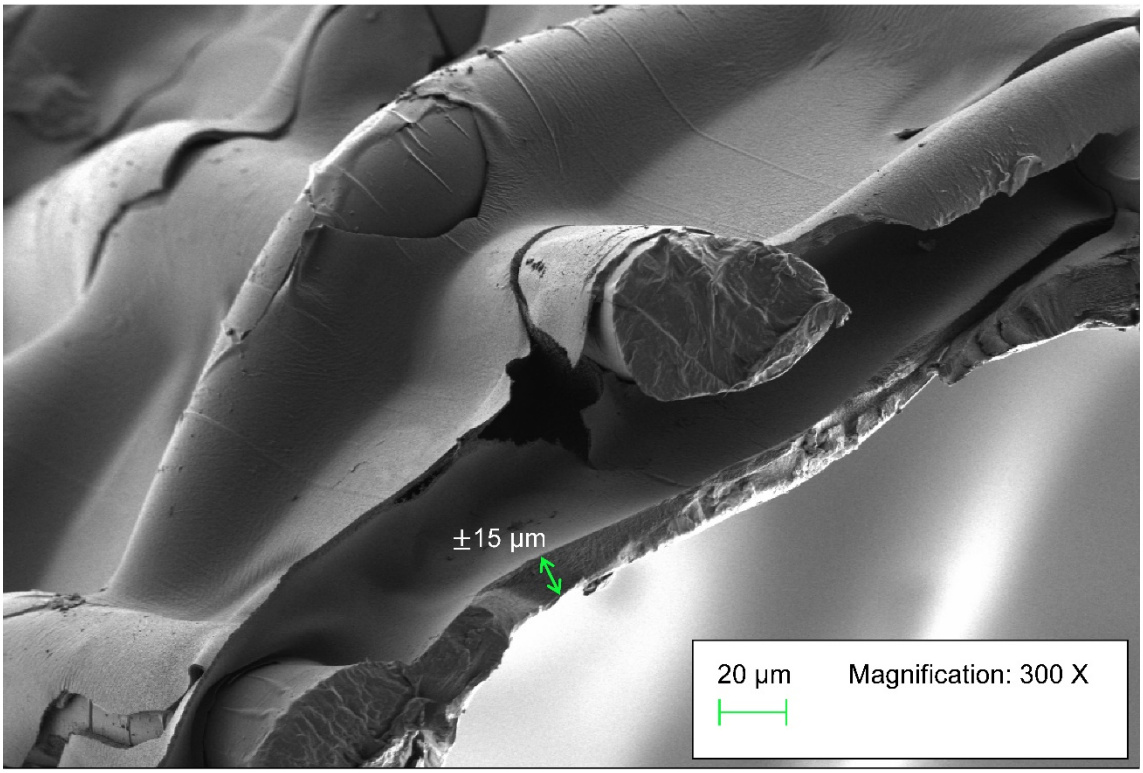


*Figure 8. SEM image of a cross section of an OsmoF$_2$O™ FO CTA membrane from Fluid Technology Solutions. The active layer is in the order of 15 µm thick. The embedded support mesh is clearly visible. MWCO unknown.*

## Concentration Polarisation and Fouling

During FO mode processes it is often found that the actual permeate water flux is far lower than the theoretical flux (McCutcheon & Elimelech 2006). This is predominantly due to a phenomenon termed concentration polarisation (CP). CP is an important consideration in FO mode process efficiency (Zhao et al. 2012). Membrane fouling, whilst less severe in FO mode than RO mode processes, is another important factor influencing the efficiency of membranes in FO mode.

### *Concentration Polarisation*

As the feed solution moves towards the membrane, it transports solute molecules (if present) via convection. This solute accumulates, increasing the concentration of solute on the feed side, thus

lowering the water potential relative to the bulk feed water potential. A concentration gradient develops due to the increased solute concentration at the feed side. This creates a diffusive solute flux $J_s$ opposite in direction to the water flux $J_w$ (Figure 9). Equilibrium will eventually be achieved between the convective solute flow and the diffusion in the reverse direction. However, at equilibrium the solute concentration at the feed side membrane boundary will be higher than that of the bulk feed solution. Furthermore, as the water permeates through the membrane it will dilute the draw solution at the membrane. This increases the water potential on the draw side of the membrane. The combined effect is a reduction in the transmembrane osmotic pressure difference across the active layer of the membrane $\Delta\pi_o^{eff}$ (Gray, McCutcheon, & Elimelech 2006; McCutcheon & Elimelech 2006; Tang et al. 2010b).

CP can be categorised according to where it occurs relative to the membrane; internal concentration polarisation (ICP) and external concentration polarisation (ECP); and according to whether it dilutes or concentrates the solution. Thus, four types of CP may be defined (Cath, Childress, & Elimelech 2006):

- Concentrative ECP
- Dilutive ECP
- Concentrative ICP
- Dilutive ICP

ECP can be mitigated by ensuring adequate flow of solution across the membrane to encourage hydrodynamic mixing. However, ICP is confined to the internal structure of the membrane and hydrodynamic mixing of the solution cannot mitigate it (McCutcheon & Elimelech 2006). ICP occurs within the porous support layer of asymmetrical membranes. It is dependent on membrane porous support layer geometric properties, membrane orientation and the diffusivity of the solute

in contact with the support layer. With reference to Figure 9, when the active layer of a membrane faces the draw side (AL-DS configuration), concentrative ICP occurs. When the active layer faces the feed side (AL-FS configuration), dilutive ICP occurs.

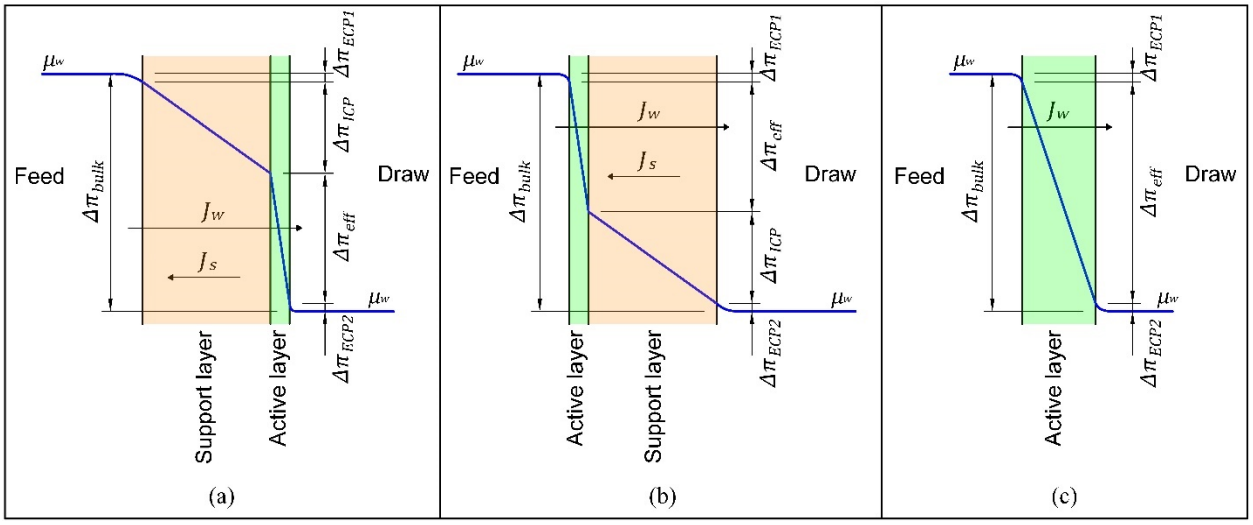


*Figure 9. Concentration polarisation (CP) (a) Active layer – draw side (AL-DS) configuration gives rise to concentrative ICP (b) Active layer - feed side (AL-FS) configuration gives rise to dilutive ICP (c) No support layer creates no ICP but generally a much thicker active layer is required for mechanical strength*

### *Membrane Fouling*

Membrane fouling occurs when molecules are deposited on and within the membrane, thence changing its diffusivity and effective pore size distribution. Decreased diffusivity of the membrane increases the potential for ICP and a smaller effective pore size acts to decrease water flux (Tang et al. 2010b). Membrane fouling is influenced by the hydrophilicity of the membrane in aqueous solutions, the surface charge of the membrane in relation to solutes in solution and the relative size of the solutes and the membrane pores (Musale & Kulkarni 1998). The propensity for fouling increases with higher transmembrane pressures, lower crossflow velocities and higher solute concentration.

Transmembrane pressures are relatively low in geotechnical osmotic testing and the crossflow velocities are also low, but high concentrations of PEG are typically used. This may create a high propensity for fouling if the PEG solution is in contact with the support layer of the membrane. If the support layer is in contact with the soil sample, fouling will occur due to the soil particles. However, the soil sample will naturally behave as a large fouling layer on one side of the membrane. Therefore, the fouling of the support by soil material acts only to increase the effective thickness of the soil sample by a few microns. Figure 10 illustrates the fouling of the support layer of a TRISEP® UF10 membrane after being used in a geotechnical osmotic test. The membrane was washed with water after the test, then dried in a vacuum desiccator before being imaged using a SEM.

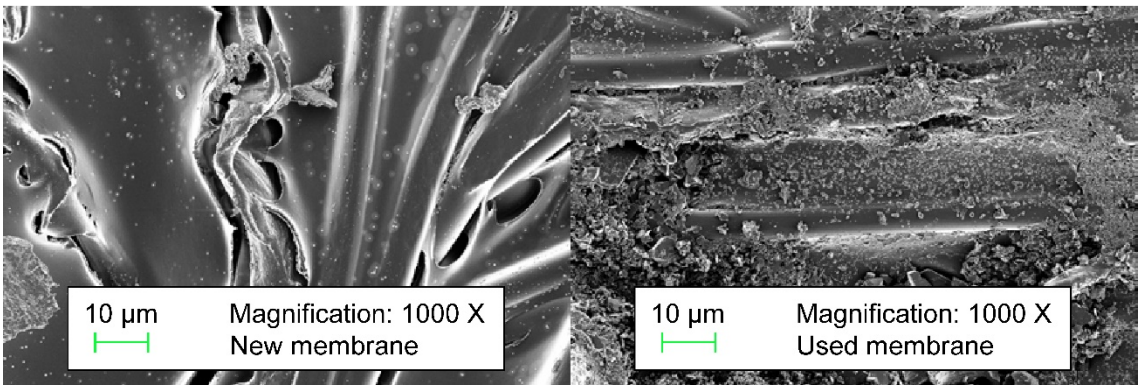


*Figure 10. Fouling of a TRISEP® UF10 membrane support layer with soil material.*

### *Reverse Solute Draw*

Reverse solute draw occurs when PEG moves through the membrane pores in the opposite direction to the water permeate. This occurs via diffusion in FO mode processes and via convection in RO mode processes. Significant reverse solute draw occurred in the work of Vela et al. (2008b)

despite the large difference of 20 000 Da between the MWCO of 15 000 Da and the PEG-35 000 used. Evidence of reverse solute draw in geotechnical osmotic testing was also seen by Delage & Cui (2008a) who suggested prefiltering the PEG before use in tests in order to minimise reverse solute draw. This shows evidence that the molecular weight distribution of PEG may not be as narrow as the polymerisation theory suggests. Flory (1953) and Lieske et al. (2020) show that one can determine the average molecular mass of a PEG sample using the osmotic pressure data. This could be useful to determine the extent of reverse solute draw one may expect. However, these methods should be applied with caution as they are only applicable at low concentrations. In addition, the average molecular weight does not give an indication of the molecular weight distribution within the sample; this can only be determined using size exclusion chromatography or ultracentrifugation (Ward 1981).

## MEMBRANES IN THE CONTEXT OF GEOTECHNICAL TESTING

Numerous geotechnical researchers have eluded to the presence of a "membrane effect" without providing a proven root cause (e.g. Slatter et al. (2000); Dineen & Burland (1995)). Other researchers have shown that osmotic pressure calibrations are membrane dependent (Tarantino & Mongiovì 2000; Monroy et al. 2007). This membrane effect has been described as acting to reduce the osmotic pressure measured across a membrane when compared to that measured using vapour pressure depression techniques over the free surface of the solution. Monroy (2006) has also reported hysteresis in the transmembrane osmotic pressure measured using tensiometers. These membrane effects mean that the soil suction must be verified during testing as the imposed osmotic pressure may not be equal to the matric suction in the sample at equilibrium due to these effects. The need to verify the matric suction in the soil negates the benefit of being able to impose higher levels of suction than the axis-translation technique. There may be multiple reasons for these membrane effects including but not limited to:

- ICP in the porous support of the membrane
- ECP due to inadequate solution circulation
- Reverse solute draw of PEG through the active layer of the membrane
- Membrane punctures caused by the soil or supporting mesh
- Potential measurement errors

Conventional membrane processes separate two bulk liquid phases and generally occur under a constant transmembrane water potential gradient. Geotechnical osmotic testing is unique in the fact that the membrane separates a liquid and a soil. Furthermore, with each new suction target, the transmembrane water potential gradually declines until equilibrium is achieved. This means that the osmotic driving force diminishes as equilibrium is approached. At low transmembrane

potentials, it is important to ensure that the membrane is working as efficiently as possible. This means that one must be cognisant of the factors that influence transmembrane osmotic potential.

ECP may be mitigated by ensuring adequate hydrodynamic mixing at the membrane surface. However, hydrodynamic mixing of the soil-water is not possible. Therefore, water transport within the soil is still governed by the soil-water potential differences within the sample and the soil properties. The membrane imposes the boundary conditions that influence the internal equilibration of soil-water potential within the sample. Behaviour on the soil side of the membrane is largely dictated by the soil properties rather than the membrane properties.

Water flux in geotechnical testing is comparatively low but PEG concentrations are comparatively high in comparison to commercial FO mode processes. These two factors compete against each other in determining the extent of CP on the PEG solution side of the membrane. The high concentrations of PEG used creates a viscous PEG solution that is difficult to circulate adequately. It remains to be seen to what extent CP (both ICP and ECP) influences equilibrium times and effective transmembrane osmotic pressures $\Delta\pi_o^{eff}$ within the constraints of geotechnical osmotic testing. This field is largely unexplored and research in this area is encouraged.

Commercial membranes are designed to be operated in a fixed orientation for their entire life as the feed side and the draw side remain the same in commercial osmotic processes. In geotechnical testing the situation is more complex. When decreasing the soil suction, the feed solution is the PEG solution and the draw solution is the soil-water. When increasing the soil suction, the feed solution is the soil-water and the draw solution is the PEG solution. Ideally, the membrane should remain in AL-FS configuration as this creates a lower propensity for ICP by PEG as there is less solute on the draw side (Zhao & Zou 2011). However, geotechnical testing is often concerned with the hysteretic behaviour of soil during drying-wetting cycles and this

complicates choosing a membrane orientation as it cannot be changed during the test.

In order to overcome CP effects which could increase equilibrium times and result in lower soil suctions than targeted, researchers could consider changing the pumping scheme of the PEG solution. Some researchers have used negative relative pressure and others have used positive relative pressures to move the PEG solution beneath the membrane. This creates a small transmembrane hydrostatic pressure difference. The placement of the peristaltic pump can be changed during the test in order to assist with either “pulling” or “pushing” water through the membrane to overcome CP effects according to Equation 5.

In the context of membrane fouling, the situation is again unique in geotechnical testing. In conventional UF processes, the solution concentrations are well below saturation to maintain a low fluid viscosity for easy pumping. However, in geotechnical testing, solutions are much more concentrated in order to achieve the desired soil-water potentials. In UF fouling tests on PEG-35 000 conducted by Vela et al. (2008b) and Vela et al. (2008a) the highest PEG concentration considered was 15 g/L. This equates to approximately 0.015 g PEG/g water, significantly lower than typical concentrations used in geotechnical osmotic testing. Therefore, conclusions regarding membrane fouling from these tests is only partly applicable to geotechnical testing and the results should be extrapolated with caution. It is necessary that fouling tests be conducted at the concentrations used in geotechnical testing.

In order to mitigate fouling, it is believed that membranes should be oriented with their active layer facing the PEG solution. This is because hydrodynamic mixing can be controlled on the PEG side of the membrane. This will mitigate ECP and negate ICP caused by the PEG solution. The effect of fouling of the support layer by the soil in this orientation is believed to be less severe. Water, which permeates to or from the soil sample, will need to move through the soil structure

and “fouling” of the support layer would be analogous to increasing the soil sample drainage path length by a few microns.

## CONCLUDING REMARKS AND RECOMMENDATIONS

In their review, Delage & Cui (2008b) made it clear that so called "membrane effects" were evident and not well understood in the geotechnical osmotic testing literature. It is proposed that an understanding of fundamental concepts relevant to membrane science will aid in mitigating these membrane effects and progressing the state-of-the-art of geotechnical osmotic testing.

In this review, concepts from membrane science have been summarised and applied to geotechnical osmotic testing. A number of references to the literature have also been provided for additional reading. Common membrane materials and membrane structures were discussed in order to contextualise the use of membranes in geotechnical testing. The concepts of concentration polarisation, membrane fouling and reverse solute draw were considered as potential causes for the membrane effects previously identified in geotechnical literature.

It is clear that the use of osmotic membranes for geotechnical testing falls within a largely unexplored niche. It will be relevant and useful to promote further research to characterise different membranes within the geotechnical osmotic testing context. Research in this regard should consider multiple types of membranes, different membrane orientations, different polyethylene glycol solution concentrations as well as different circulation environments.

Membranes should be characterised using their pure water flux before being characterised when in contact with a soil sample. This will provide comparable data between different membranes. Geotechnical researchers are encouraged to provide additional information relevant to their membranes and to do additional characterisation such as SEM imaging to determine membrane structure and thicknesses.

Despite the challenges that membrane effects bring to geotechnical osmotic testing, the technique shows much promise to become a routine method to quantify unsaturated soil behaviour

in geotechnical practice. This paper provides some guidance on avoiding errors due to membrane effects and provides protocols that may improve the standardisation of geotechnical osmotic testing.

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## DISCLOSURE

The authors have no conflict of interest to report.

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