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Strain-controlled oedometer test for the measurement of the chemico-osmotic properties of bentonites

Essai œdométrique à déformation contrôlée pour la mesure des propriétés chimique-osmotiques des bentonites

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ABSTRACT: The possibility of relating the macroscopic transport properties and the swelling behaviour of bentonites to a limited number of physico-chemical and fabric parameters has been investigated by means of a new laboratory apparatus, which allows the reflection coefficient, which is also known as the membrane efficiency coefficient, and the swell coefficient to be simultaneously determined on the same clay specimen. The results of two multi-stage tests, which were performed on a natural sodium bentonite under fully saturated conditions, while varying both the specimen porosity and the salt concentration of the equilibrium solutions, have been interpreted through a physically-based model in which the pore-scale electro-chemical interactions between the solid skeleton, the aqueous phase and the ion species are explicitly taken into account. The efficiency of the tested bentonite in acting as a semi-permeable membrane and its swelling behaviour have been found to be accurately simulated when a single fabric parameter, referred to as the solid charge coefficient, is calibrated on the available experimental dataset, thus suggesting that the containment performance of bentonite-based barriers, used for a number of geoenvironmental applications, may be predicted on the basis of the results of a strain-controlled oedometer test.

RÉSUMÉ: La possibilité de relier les propriétés de transport macroscopiques et le comportement de gonflement des bentonites à un nombre limité de paramètres physico-chimiques et du tissu a été étudiée à l'aide d'un nouvel appareil de laboratoire, qui permet de déterminer simultanément le coefficient de réflexion, également appelé coefficient d'efficacité de la membrane, et le coefficient de gonflement sur le même échantillon d'argile. Les résultats de deux essais plusieurs-étapes, réalisés sur une bentonite sodium naturelle dans des conditions de saturation totale, tout en faisant varier la porosité de l'échantillon et la concentration en sel des solutions à l'équilibre, ont été interprétés selon un modèle physique dans lequel les interactions électrochimiques à l'échelle des pores entre le squelette solide, la phase aqueuse et l'espèce ionique sont explicitement prises en compte. On a constaté que l'efficacité de la bentonite testée en agissant comme un membrane semi-perméable et son comportement de gonflement étaient simulés avec précision lorsqu'un seul paramètre de tissu, appelé le coefficient de charge solide, est étalonné sur l'ensemble de données expérimentales disponibles, suggérant ainsi que les performances de confinement des barrières à base de bentonite, utilisées pour différentes applications en géotechnique environnementale, peuvent être prédites sur la base des résultats d'un essai œdométrique à déformation contrôlée.

Keywords: bentonite barriers; chemical osmosis; geosynthetic clay liners; swelling pressure; waste disposal.

1 INTRODUCTION

Clay soils that are characterised by a high specific surface and an electric net charge (e.g. bentonites) behave as semi-permeable membranes, since a liquid flux can occur in response not only to a gradient in the total hydraulic head, but also to gradients in the concentration of the ion species in solution: such a phenomenon, commonly known as chemical osmosis, is caused by the electrostatic repulsion of the anions by the diffuse double layers associated with adjacent clay particles. As far as the control of waste fluids in the subsoil is concerned, the rate of contaminant migration through bentonite-based liners, such as the geosynthetic clay liners that are increasingly being used as parts of landfill bottom barriers, results to be affected by the membrane behaviour, since the solute mass flux is reduced as a consequence of both the chemico-osmotic counter-advection and the decrease in the accessible porosity of the solute (Malusis et al. 2003).

The electrical interaction between the pore solution ions and the clay particles, which leads to the coupled phenomenon, is also able to influence the mechanical behaviour of bentonites. In fact, volume strains are observed when the chemical composition of the equilibrium bulk solution is changed during a conventional oedometer test (Musso et al. 2017). A change in the ion concentration of the equilibrium bulk solution induces a symmetrical variation in the macroscopic swelling pressure during a strain-controlled oedometer test (Dominijanni et al. 2018b).

In view of the advantages that may be achieved in the design of contaminant barriers constituted by bentonite, as a result of a better understanding of the physical mechanisms that govern their transport and mechanical properties, the aim of this paper is to illustrate the results of two multi-stage tests that were carried out by means of a new laboratory apparatus, which has been developed in order to allow the simultaneous measurement of the reflection co-

efficient and the swelling pressure on the same clay specimen. Furthermore, the theoretical model proposed by Dominijanni and Manassero (2005) and Dominijanni et al. (2006) is shown to be able to simulate the obtained experimental data, and a novel procedure for the interpretation of the chemico-osmotic test results is presented.

2 THEORETICAL FRAMEWORK

On the basis of pioneering models that were developed with the aim of simulating transport phenomena in fine-porous charged diaphragms, such as track-etched membranes and polyelectrolyte gels (Yaroshchuk 1995), Dominijanni et al. (2006) were able to identify the pore-scale interactions that govern the behaviour of chemically active clays at the macroscopic scale, thus allowing a complete characterisation of the osmotic properties of bentonites to be obtained in the frame of a purely mechanistic approach. In their capillary space-charge model, where the most suitable pore geometry is recognised to be the slit-like one, the phenomenological coefficients that are introduced within the scope of the thermodynamics of irreversible processes (Dominijanni and Manassero 2005) are expressed in terms of a few physico-chemical and fabric parameters, under the assumption of an infinitely diluted solution and a constant distribution of the electric potential in the cross-section of the idealised pore.

2.1 Membrane behaviour

The extent to which bentonites behave as semi-permeable membranes can be evaluated through the laboratory measurement of the reflection coefficient, ω , which is also known as the membrane or chemico-osmotic efficiency coefficient (Malusis et al. 2003). Among the possible test configurations that can be used to measure ω , the one that is usually referred to as the “closed hydraulic control system” has been found to provide the most suitable results for a theoretical interpretation, due to the ease of control of the

chemical and hydraulic boundary conditions (Dominijanni et al. 2018a). As illustrated in Figure 1, the clay specimen is interposed between two porous stones through which salt solutions with different ion concentrations are circulated, while the volumetric flux of water is hindered across the soil. When these conditions are maintained over time, a hydraulic head difference arises between the specimen boundaries under steady state conditions. The measured global value of the reflection coefficient, ω_g , which has been demonstrated to be equal to the integral mean value of ω that is calculated with respect to the boundary salt concentrations (Dominijanni et al. 2018b), is given by:

$$\omega_g = \left(\frac{\Delta h \cdot \gamma_w}{\Delta \Pi} \right)_{q=0} = \frac{1}{\Delta c_s} \int_{c_b}^{c_t} \omega \cdot dc_s \quad (1)$$

where Δh is the difference in hydraulic head across the porous medium, γ_w is the water unit weight (9.81 kN/m³), $\Delta \Pi$ is the difference in osmotic pressure across the porous medium, q is the volumetric flux of the salt solution, Δc_s is the difference in salt concentration across the porous medium ($= c_t - c_b$), and c_t and c_b are the salt concentrations of the external bulk solutions in contact with the top and bottom boundaries of the porous medium, respectively.

Provided that the testing solutions are sufficiently diluted (i.e. an electrolyte concentration of less than 1.0 mol/L), the osmotic pressure difference for a solution containing a single salt is obtained according to the van't Hoff expression as follows:

$$\Delta \Pi = \nu RT \Delta c_s \quad (2)$$

where ν is the number of ions per molecule of salt (e.g. $\nu = 2$ for NaCl or KCl, $\nu = 3$ for CaCl₂), R is the universal gas constant (8.314 J/mol·K) and T is the absolute temperature.

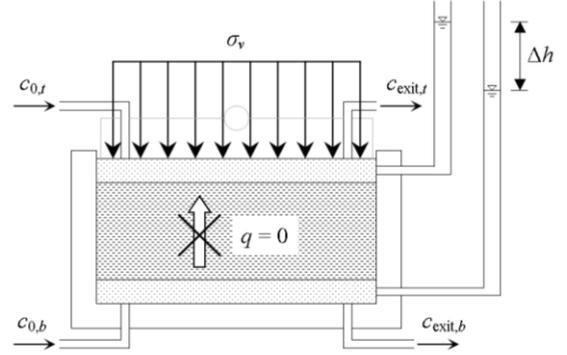


Figure 1. Schematic of the modified strain-controlled oedometer cell and the associated circulation system used to measure the transport properties and the mechanical behaviour of active clays. $c_{0,t}$ and $c_{0,b}$, salt concentrations of the solutions injected into the top and bottom porous stones, respectively; $c_{exit,t}$ and $c_{exit,b}$, salt concentrations of the solutions withdrawn from the top and bottom porous stones, respectively; q , volumetric flux of the solution; Δh , hydraulic head difference between the specimen boundaries; σ_v , total vertical stress acting on the specimen.

A physical identification of the reflection coefficient can be derived from the macroscopic transport equations of the solvent and solute fluxes in a semi-permeable porous medium, together with the condition of null electric current density (Dominijanni et al. 2006). Using such an approach, the reflection coefficient for a 1:1 electrolyte can be related to the bentonite void ratio, e , the equilibrium salt concentration, c_s , and the solid charge coefficient, $\vec{c}_{sk,0}$, through the following equation:

$$\omega = 1 - \frac{1}{\sqrt{\left(\frac{\vec{c}_{sk,0}}{2c_s e} \right)^2 + 1} + (2t_1 - 1) \left(\frac{\vec{c}_{sk,0}}{2c_s e} \right)} \quad (3)$$

where

$$t_1 = \frac{D_{1,0}}{D_{1,0} + D_{2,0}} \quad (4)$$

with t_1 being the cation transport number, $D_{1,0}$ the free-solution or aqueous-phase diffusion coefficient of the cation and $D_{2,0}$ the free-solution or aqueous-phase diffusion coefficient of the anion.

The corresponding theoretical value of the global reflection coefficient, ω_g , which is obtained by integrating Equation 3 between the two boundary concentrations c_t and c_b , is given by:

$$\omega_g = 1 + \frac{\bar{c}'_{sk,0}}{2\Delta c_s e} \cdot \left[Z_2 - Z_1 - (2t_1 - 1) \cdot \ln \left(\frac{Z_2 + 2t_1 - 1}{Z_1 + 2t_1 - 1} \right) \right] \quad (5)$$

where:

$$Z_1 = \sqrt{1 + \left(\frac{2c_t e}{\bar{c}'_{sk,0}} \right)^2} ; \quad Z_2 = \sqrt{1 + \left(\frac{2c_b e}{\bar{c}'_{sk,0}} \right)^2} . \quad (6)$$

The previously introduced solid charge coefficient, $\bar{c}'_{sk,0}$, can be expressed as a function of a given number of intrinsic soil properties (i.e. the cation exchange capacity, the specific gravity and the fraction of exchangeable cations specifically adsorbed in the Stern layer) and of a single fabric parameter, which is referred to as the average number of montmorillonite lamellae per clay particle. Manassero (2017) showed that the latter fabric parameter is susceptible to variations in response to the possible evolutions of the bentonite microstructure and, as a result, the solid charge coefficient, $\bar{c}'_{sk,0}$, should be regarded as a function of the ion concentration in the equilibrium solution and of the total soil porosity, which in turn is related to the history of the effective isotropic stress. However, the parameter $\bar{c}'_{sk,0}$ can be assumed constant, as a first approximation, for relatively low values of the salt concentration (i.e. $c_s < 0.1$ mol/L) and it can

be derived from the interpretation of macroscopic laboratory tests.

2.2 Mechanical behaviour

The swell coefficient, ϖ , was introduced by Dominijanni et al. (2018b) to quantify how efficient a change in the salt concentration of the equilibrium solution is at producing a variation in the chemico-osmotic swelling pressure that develops in response to the ion partition mechanisms which occur within the semi-permeable porous medium. The coefficient ϖ , which may more accurately be referred to as the “chemico-osmotic swelling pressure efficiency coefficient”, can be determined by means of the modified strain-controlled oedometer cell that is illustrated in Figure 1, in which the variation in the total vertical stress, caused by a change in the equilibrium salt concentration, is measured under constant volume conditions.

The measured global value of the swell coefficient, ϖ_g , which has been demonstrated to be equal to the integral mean value of ϖ that is calculated with respect to the equilibrium salt concentrations at two different time instants (Dominijanni et al. 2018b), is given by:

$$\varpi_g = - \left(\frac{\delta \sigma}{\delta \Pi} \right)_{d\varepsilon_v=0; \delta u=0} = \frac{1}{\delta c_s} \int_{c_i}^{c_f} \varpi \cdot dc_s \quad (7)$$

where $\delta \sigma$ is the change in the total vertical stress over time, $\delta \Pi$ is the change in the osmotic pressure over time, $d\varepsilon_v$ is the volumetric strain increment, δu is the change in the hydraulic pressure over time, δc_s is the change in the equilibrium salt concentration over time ($= c_f - c_i$), and c_i and c_f are the salt concentrations of the external bulk solution in equilibrium with the porous medium at two different time instants.

In order to derive a physical identification of the swell coefficient, the mechanical constitutive equation of a saturated semi-permeable porous medium has to be coupled with Donnan's equations, which allow the pore solution pressure to

be related to the bentonite void ratio and the ion concentrations of the equilibrium solution. Thus, Dominijanni et al. (2018b) were able to obtain the following relation in the case of a 1:1 electrolyte:

$$\varpi = 1 - \frac{1}{\sqrt{\left(\frac{\bar{c}'_{sk,0}}{2c_s e}\right)^2 + 1}} \quad (8)$$

The corresponding theoretical value of the global swell coefficient, ϖ_g , which is the integral mean value of ϖ calculated between the two salt concentrations c_i and c_f , is given by:

$$\varpi_g = 1 + \frac{\bar{c}'_{sk,0}}{2\delta c_s e} \cdot \left[\sqrt{1 + \left(\frac{2c_i e}{\bar{c}'_{sk,0}}\right)^2} - \sqrt{1 + \left(\frac{2c_f e}{\bar{c}'_{sk,0}}\right)^2} \right] \quad (9)$$

2.3 Reference salt concentrations

The measured values of ω_g and ϖ_g are “global” coefficients that represent the integral mean values of the corresponding “local” coefficients ω and ϖ , respectively, with regard to the salt concentration variable. Using the theoretical relationships given by Equations 3 and 8, the local coefficients can be evaluated for a given range of salt concentrations when e is known and a value of $\bar{c}'_{sk,0}$ is assumed. As a result, the theoretical mean value can be calculated for any salt concentration range by integrating the local coefficient, as done in Equation 5 for ω and in Equation 9 for ϖ .

The salt concentration value for which the function that represents the local coefficient is equal to its integral mean value can be identified with the reference salt concentration for the specified testing stage. The determination of the reference salt concentration, $c_{s,\omega}$, for the coefficient

ω can be attained analytically, by imposing the following condition:

$$\omega_g(c_b, c_i) = \omega(c_{s,\omega}) \quad (10)$$

where the global value of the reflection coefficient is given by Equation 5, once the external salt concentrations at the specimen boundaries (i.e. c_i and c_b) and the void ratio are known, and a value of $\bar{c}'_{sk,0}$ is hypothesized. Substitution of Equation 3 in Equation 10 yields the following expression for the reference salt concentration, which is valid when ω_g is non-negative:

$$c_{s,\omega} = -\frac{\bar{c}'_{sk,0}}{e} \cdot \frac{2\alpha}{\beta + \sqrt{\beta^2 - 4\alpha\gamma}} \quad (11)$$

where:

$$\alpha = t_1(t_1 - 1); \beta = \frac{1 - 2t_1}{1 - \omega_g}; \gamma = \left(\frac{1}{1 - \omega_g}\right)^2 - 1. \quad (12)$$

Analogously, the reference salt concentration, $c_{s,\varpi}$, can also be derived analytically for the coefficient ϖ by imposing the following condition:

$$\varpi_g(c_i, c_f) = \varpi(c_{s,\varpi}) \quad (13)$$

where c_i and c_f are the equilibrium salt concentrations at two different testing stages. The substitution of Equation 8 in Equation 13 yields the following expression for the reference salt concentration:

$$c_{s,\varpi} = \frac{\bar{c}'_{sk,0}}{2e \sqrt{\left(\frac{1}{1 - \varpi_g}\right)^2 - 1}} \quad (14)$$

The theoretical definition of the swell coefficient is based on the assumption of a homoge-

neous salt concentration profile within the bentonite specimen during each testing stage. When this condition is not fulfilled, as in the case of testing stages aimed at measuring the reflection coefficient, a rigorous experimental determination of ω_g and a theoretical derivation of the corresponding $c_{s,\omega}$ are not possible. However, as a first approximation, the estimation of these parameters can be attempted by assuming the arithmetic average salt concentration across the specimen, c_{avg} , as a representative value of the equilibrium salt concentration:

$$c_{avg} = \frac{c_b + c_t}{2}. \quad (15)$$

3 EXPERIMENTAL EVIDENCE

3.1 Materials and laboratory apparatus

The powdered bentonite tested in this study was an Indian sodium bentonite, whose physico-chemical properties have already been described by Dominijanni et al. (2018b). The salt solutions were prepared with sodium chloride and deionized water at molar concentrations ranging from 5.01 to 89.20 mmol/L.

Two multi-stage chemico-osmotic tests were performed by means of a new laboratory apparatus, which has been illustrated in detail in Dominijanni et al. (2018b). Briefly, the apparatus is made up of the stainless steel oedometer depicted in Figure 1, which is endowed with drainage lines that allow different solutions to circulate in the porous stones and, hence, to establish a constant concentration gradient across the clay specimen. The difference in hydraulic head that develops between the specimen boundaries is measured by means of a differential pressure transducer, and a load cell detects the vertical pressure that is applied by the top piston of the oedometer in order to hinder any volumetric strain of the specimen. Finally, a flow pump system prevents the volumetric flux

through the clay soil by simultaneously injecting the same volume of solution into the porous stones and removing it.

3.2 Testing procedures and results

Prior to the tests, the sodium bentonite was subjected to the conditioning process that is known as “squeezing” (Dominijanni et al. 2018b) to remove the soluble salts that are naturally present inside the powdered material. A known amount of squeezed, oven-dried sodium bentonite, sifted through an ASTM No. 200 mesh sieve, was dusted inside the oedometer ring and a NaCl solution was supplied from the bottom pedestal to saturate the specimen, which was allowed to swell freely, in the axial direction, to a specified void ratio (i.e. $e = 3.33$ and $e = 4.18$ during the first and second chemico-osmotic tests, respectively). At the end of the hydration stage, the top piston was locked in place and the chemico-osmotic test was started.

The differential hydraulic head induced across the specimen, Δh , the total vertical stress acting on the specimen, σ , and the salt concentration of the solutions injected into the top and bottom boundaries (i.e. $c_{0,t}$ and $c_{0,b}$), as well as of the solutions withdrawn from the top and bottom boundaries (i.e. $c_{exit,t}$ and $c_{exit,b}$), were measured during each stage of the two chemico-osmotic tests until steady state conditions were reached (Table 1).

Because of the condition of null volumetric flux through the specimen, the global reflection coefficient, ω_g , was calculated using Equation 1, in which c_t and c_b were assessed as follows:

$$c_t = \frac{c_{0,t} + c_{exit,t}}{2}; \quad c_b = \frac{c_{0,b} + c_{exit,b}}{2}. \quad (16)$$

Under the conditions of constant hydraulic pressure and absence of volumetric strains, the determination of the global swell coefficient, ω_g , was based on Equation 7.

Table 1. Results of reflection coefficient and swell coefficient measurements of the two multi-stage chemico-osmotic tests. The arithmetic average concentration, c_{avg} , was considered as the equilibrium salt concentration.

e (-)	Steady state values											
	$c_{0,t}$ (mM)	$c_{0,b}$ (mM)	$c_{exit,t}$ (mM)	$c_{exit,b}$ (mM)	$\Delta\Pi$ (kPa)	Δh (m)	ω_g (-)	$c_{s,\omega}$ (mM) ^a	$\delta\Pi$ (kPa)	$\delta\sigma$ (kPa)	ω_g (-)	$c_{s,\sigma}$ (mM) ^a
3.33	10.07	10.07	10.25	10.25	-	-	-	-	-	-	-	-
	29.66	10.07	29.38	12.21	90.51	1.199	0.13	18.80	50.08	-15	0.300	14.86
	59.89	10.07	57.35	14.25	228.79	1.399	0.06	27.35	74.16	-7	0.094	27.11
	89.20	10.07	81.56	19.80	346.87	1.238	0.035	34.51	72.73	-	-	42.22
4.18	5.01	5.01	5.65	5.73	-	-	-	-	-	-	-	-
	19.71	5.01	19.00	6.65	66.58	0.814	0.12	11.56	35.65	-13	0.365	8.74
	19.71	19.71	20.25	19.44	-	-	-	-	35.41	-8	0.226	15.95
	34.97	19.71	34.87	22.32	68.50	0.419	0.06	27.00	40.31	-8	0.198	23.60
	34.97	34.97	35.42	35.33	-	-	-	-	35.48	-4	0.113	31.39
	50.52	34.97	49.03	36.90	68.20	0.209	0.03	41.93	37.84	-2	0.053	38.84

^a The reference salt concentration values $c_{s,\omega}$ and $c_{s,\sigma}$ were calculated assuming $\bar{c}_{sk,0}^* = 110$ mmol/L.

3.3 Discussion

The introduction of the reference salt concentration concept allows the results of tests referring to any combination of extreme salt concentration values to be related to a single concentration value. The reference salt concentration $c_{s,\omega}$ replaces the salt concentrations c_t and c_b of the solutions circulating at the top and the bottom specimen boundaries, respectively. Similarly, the reference salt concentration $c_{s,\sigma}$ condenses the salt concentrations c_i and c_f of the solution that is in equilibrium with the bentonite at two different temporal states.

The results of the reflection coefficient measurements obtained from the multi-stage tests were interpreted according to an iterative procedure, where a value of $\bar{c}_{sk,0}^*$ was assumed as a first attempt and a corresponding theoretical value of ω_g was calculated for each testing stage using Equation 5. The reference salt concentrations, $c_{s,\omega}$, were successively determined from the theoretical values of ω_g using Equation 11, and the experimental values of ω_g (Table 1) were plotted as a function of the calculated values of $c_{s,\omega}$, in order to compare them with the theoretical curve given by Equation 3. The previously outlined procedure was repeated until a

best-fitting value of $\bar{c}_{sk,0}^*$, which resulted to be equal to 110 mmol/L for both of the tested specimens, was reached. Although the measured ω range was narrow, a satisfactory agreement was found with the behaviour predicted by the theoretical model, as shown in Figure 2.

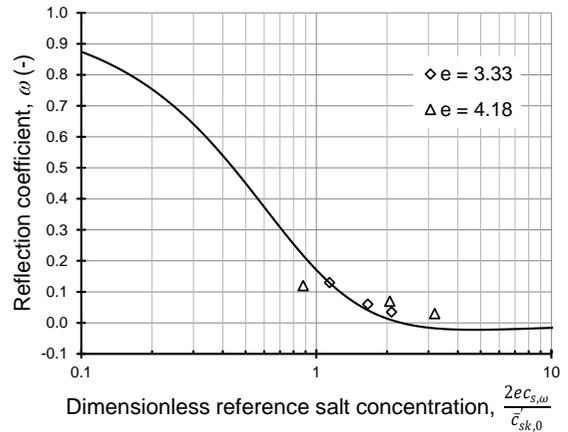


Figure 2. Reflection coefficient values, ω obtained from the two chemico-osmotic tests (open symbols), and theoretical interpretation based on Equation 3 with $\bar{c}_{sk,0}^* = 110$ mmol/L (continuous line).

An even better agreement with the proposed model was observed for the swell coefficient data, as illustrated in Figure 3. It should be stressed that the latter data were interpreted according to

an analogous procedure to the one adopted for the reflection coefficient, and a best-fitting value of $\bar{c}'_{sk,0}$ equal to 110 mmol/L was obtained.

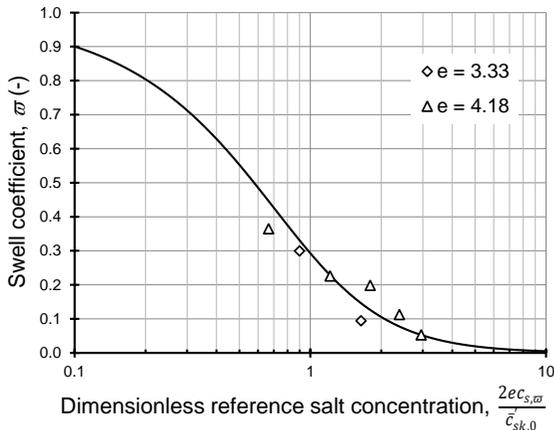


Figure 3. Swell coefficient values, ϖ , obtained from the two chemico-osmotic tests (open symbols), and theoretical interpretation based on Equation 8 with $\bar{c}'_{sk,0} = 110 \text{ mmol} / \text{L}$ (continuous line).

4 CONCLUSIONS

The results of two multi-stage chemico-osmotic tests, which were conducted on a natural sodium bentonite by means of a new laboratory apparatus, have been interpreted in light of a mechanistic model, in which the macroscopic effect of the pore-scale electrical interactions is taken into account via a single fabric parameter, $\bar{c}'_{sk,0}$. The possibility of using the same value of $\bar{c}'_{sk,0}$ in order to interpret both the reflection coefficient and the swell coefficient data suggests that information on a transport property may be obtained through the measurement of a mechanical parameter, and vice versa.

5 ACKNOWLEDGEMENTS

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