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A direct method for determining chloride diffusion coefficient by using migration test

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Abstract

Calculation of the effective diffusion coefficient is proposed using an analytical model based on Nernst–Planck equation, on electroneutrality and on measurements of current intensity. To validate this approach, migration tests were carried out on different w/c mortar samples. In steady state, the resulting effective diffusion coefficients are larger than those usually obtained with models based on Nernst–Planck equation without electroneutrality. An explanation of these differences and the importance of electroneutrality are discussed in the present work.

Keywords: Analytical model; Chloride; Electroneutrality; Migration; Cement mortar

1. Introduction

During the last decade, the modelling of chloride electrodiffusion has been developed from the modified Fick's second law [1–5]. More recently, researchers [6,7] point out a limit of this approach: these models [1–5] do not take into account the concentration evolution of other ionic species located in the compartments of diffusion cell and in the pore solution of the sample. The methodology developed by these researchers [6,7], which takes into account all the ionic species, needs a set of equations involving a complex solution due to the significant number of variables. Using these models in a deterministic approach is also complicated, because the inputs are numerous and not well known.

This paper proposes a simple method based on Nernst–Planck equation and electroneutrality assumption that takes into account the multiplicity of the ionic species. In this framework, equations are simple and their solution is faster. The assessment of the effective diffusion coefficient is based on chronoamperometry, avoiding handling errors and laborious, time-consuming, chloride dosage. To validate this model, migration tests were carried out on three cement mortars. The effective diffusion coefficients obtained by the model were compared with those calculated by conventional formulation established by Andrade [8].

2. Theoretical considerations

2.1. Working assumptions

To describe properties of chloride ion transport, we have used Nernst–Planck relation, according to the following assumptions for steady state:

(a) the variation of the activity coefficients with ionic strength is not considered because its effect on the concentration profiles is negligible [6];
(b) the convection and electrochemical phenomena (electrical double layer) are neglected due to the assumptions of saturation and homogeneity of material, respectively;
(c) the electroneutrality in the sample and in the compartments of migration cell is assumed valid according to Newman [9];
(d) the diffusion is neglected because migration due to the electrical field is dominant.

Migration tests were carried out with regular renewals of the upstream solution (NaOH + KOH + NaCl) and the downstream one (NaOH + KOH) in order to keep their

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ionic compositions constant. Full details are given in the Section 3.

2.2. Change of scale and transfer equations

On microscopic scale, within a pore solution, the unidirectional flux of an ionic species k at a spatial point M is described by the following equation [9]:

$$j_k(M) = \frac{F}{RT} Z_k D_k C_{k,\mathrm{S}} E(M) \tag{1}$$

where F = Faraday's constant; R = universal gas constant; T = absolute temperature; D_k = diffusion coefficient of ion k at infinite dilution in water at 25 °C; $E(M)$ = local electrical field at a spatial point M; $C_{k,\mathrm{S}}$ = concentration of ionic species k, in the pore solution; Z_k = valence number of species k.

Considering the above working assumptions, the change from the previous microscopic scale to the sample macroscopic scale is performed by the introduction of a diffusibility ratio Q [10,11], which is the inverse of the formation factor. The average flux j_k within the sample is expressed by:

$$j_k = Q \frac{F}{RT} Z_k D_k C_{k,\mathrm{S}} E \tag{2}$$

where E is the average electrical field in the specimen; $E = U/L$, with U the potential difference applied between the two faces of the sample and L, the sample thickness.

The potential difference is assumed not to change from the initial value during the migration test.

The effective diffusion coefficient D_{ek} is obtained from the diffusion coefficient D_k of ion k by:

$$D_{\mathrm{e}k} = Q D_k. \tag{3}$$

The average current density i in the sample can be expressed by:

$$i = Q \frac{F^2}{RT} \left(\sum_k Z_k^2 D_k C_{k,\mathrm{S}} \right) E. \tag{4}$$

Before adding NaCl in the upstream cell, the two compartments of the migration cell contain Na^+, K^+ and OH^- ions. In steady state, a concentration balance is established between the sample and the two compartments. Consequently, the current density becomes:

$$i_{\mathrm{i}} = Q \frac{F^2}{RT} (D_{\mathrm{OH}} C_{\mathrm{OH}} + D_{\mathrm{K}} C_{\mathrm{K}} + D_{\mathrm{Na}} C_{\mathrm{Na}}) E \tag{5}$$

C_{OH}, C_{K} and C_{Na} are the concentrations of OH^-, K^+ and Na^+ ions in the upstream compartment, which are the same as in the sample and in the downstream compartment, before adding NaCl. We note that in the steady state, the concentrations of Na^+, K^+ and OH^- in the pore solution are not the original concentrations; in fact, $i_{\mathrm{i}} \neq i(t=0)$ (see Fig. 3).

In the second stage, NaCl is added in the cathodic compartment. A concentration gradient of Na^+ and Cl^- occurs at this moment. Under the action of the electrical field, OH^- and Cl^- migrate towards the downstream compartment. Under the action of the concentration gradient, Na^+ ions tend to diffuse towards the anodic compartment, but the effect of the electrical field is greater, so Na^+ ions tend to remain in the cathodic compartment. In comparison to migration, diffusion can be neglected if the duration of the experience is adapted (see Section 4). Under these conditions, the current density is:

$$i_{\mathrm{f}} = Q \frac{F^2}{RT} (D_{\mathrm{Cl}} C_{\mathrm{Cl,S}} + D_{\mathrm{OH}} C_{\mathrm{OH,S}} + D_{\mathrm{K}} C_{\mathrm{K}} + D_{\mathrm{Na}} C_{\mathrm{Na}}) E \tag{6}$$

where $C_{\mathrm{Cl,S}}$ and $C_{\mathrm{OH,S}}$ are the concentrations of Cl^- and OH^- in the sample, respectively.

2.3. Effective diffusion coefficient of chlorides D_{eCl}

From Eq. (3) and Eq. (6), the effective diffusion coefficient of chloride is expressed as:

$$D_{\mathrm{eCl}} = \frac{i_f RT}{F^2 (D_{\mathrm{Cl}} C_{\mathrm{Cl,S}} + D_{\mathrm{OH}} C_{\mathrm{OH,S}} + D_{\mathrm{K}} C_{\mathrm{K}} + D_{\mathrm{Na}} C_{\mathrm{Na}}) E} D_{\mathrm{Cl}}. \tag{7}$$

By using the current density measurement i_{f}, in steady state, the effective diffusion coefficient D_{eCl} can be determined by Eq. (7); it can also be determined by a measurement of the chloride flux j_{Cl}. The application of Eqs. (2) and (3) to chlorides yields:

$$D_{\mathrm{eCl}} = \frac{j_{\mathrm{Cl}} RT}{C_{\mathrm{Cl,S}} FE}. \tag{8}$$

The diffusion coefficient D_{eCl} may be compared with the diffusion coefficient $D_{\mathrm{eCl,up}}$ usually calculated using Andrade's relation [8]:

$$D_{\mathrm{eCl,up}} = \frac{j_{\mathrm{Cl}} RT}{C_{\mathrm{Cl,up}} FE}. \tag{9}$$

The chloride concentration in Eq. (9) is usually the concentration in the upstream compartment $C_{\mathrm{Cl,up}}$. Indeed, it is often assumed that chlorides penetrate into the sample with the same concentration as in the upstream compartment. This would be true if the chlorides were neutral particles which migrate independently of the other ions located in the compartments and in the sample. But as chlorides are electrically charged, electroneutrality must be taken into account. In this case, the chloride concentration in the sample and in the upstream compartment will be different ($C_{\mathrm{Cl,up}} \neq C_{\mathrm{Cl,S}}$, see Eq. (13)). This is the difference between Eqs. (8) and (9).

Table 1
Chemical composition of cement used

Oxides	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	K_2O	Na_2O	SO_3	TiO_2	MnO	S_rO	P_2O_5
Composition (wt.%)	64.02	19.81	5.19	2.38	0.9	1.11	0.06	3.5	0.28	0.05	0.15	0.16

2.4. Ion concentrations in steady state

The electroneutrality in the sample implies:

−before adding NaCl

$$C_K + C_{Na} = C_{OH} \tag{10}$$

−after adding NaCl

$$C_K + C_{Na} = C_{OH,S} + C_{Cl,S}. \tag{11}$$

Eq. (11) shows that the concentration of OH^- in the specimen changes after adding NaCl, while it remains constant in the upstream compartment, because, during migration, Cl^- partially replaces OH^-. The flux conservation of Cl^- and OH^- allows us to establish that the proportion between these ions remains the same in the sample and in the cathodic compartment. Then, we have:

$$\frac{j_{Cl,S}}{j_{OH,S}} = \frac{j_{Cl,up}}{j_{OH,up}} \tag{12}$$

$j_{k,up}$ and $j_{k,S}$ are the flux of species k in the upstream compartment and in the sample, respectively.

From Eqs. (11) and (12), we deduce the chloride concentration in the sample:

$$C_{Cl,S} = \frac{C_{Cl,up}}{C_{Cl,up} + C_{OH}} C_{OH}. \tag{13}$$

The chloride flux can also be determined by current measurement. In fact, the combination of Eqs. (5) and (6) and Eqs. (11) and (13) gives:

$$j_{Cl,S} = \frac{i_i - i_f}{F\left(\dfrac{D_{OH}}{D_{Cl}} - 1\right)}. \tag{14}$$

Then, Eq. (8) becomes:

$$D_{eCl} = \frac{(i_i - i_f)RT}{C_{Cl,S}EF^2\left(\dfrac{D_{OH}}{D_{Cl}} - 1\right)}. \tag{15}$$

3. Experimental study

3.1. Materials and specimens

Three types of cement mortar samples with different w/c (0.35, 0.50, 0.70) and the same s/c (1:3) have been manufactured without workability agent. Consequently, the mortar with w/c = 0.35 needs to be more compacted. The cement used is of type CPA-CEM I 52.5 according to the European Norms (NF ENV 197-1). Its chemical composition is given in Table 1.

The specimens had a prismatic form (12 × 12 × 20 cm^3). After being demoulded (24 h after manufacturing), the specimens have been cured during 28 days in an alkaline solution which is identical to the solution of the electrodiffusion test (0.083 mol/l of KOH and 0.025 mol/l of NaOH), in order to avoid leaching phenomena. To

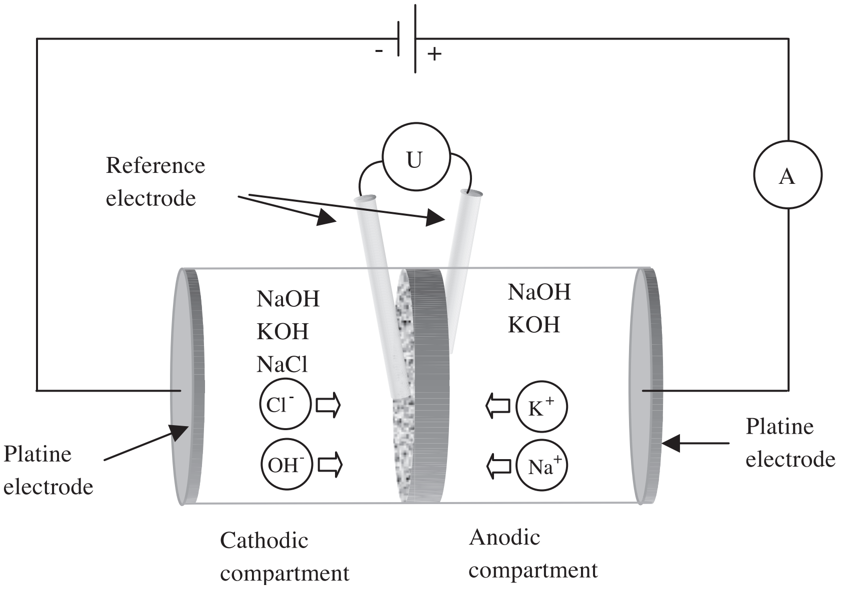

Fig. 1. Experimental device used.

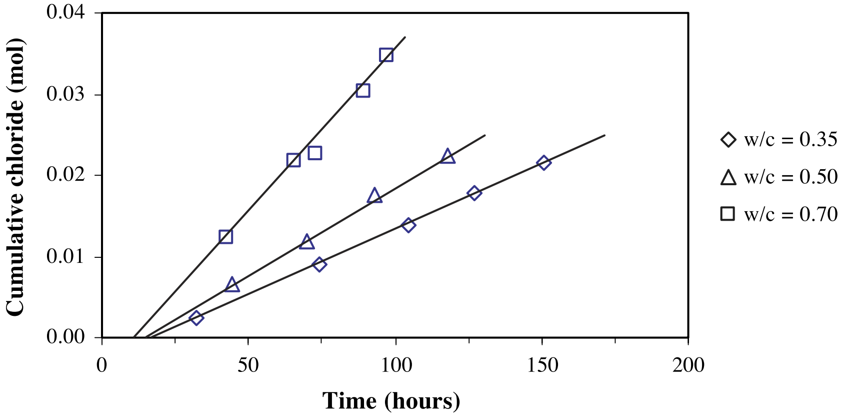

Fig. 2. Time evolution of chloride concentration in the downstream compartment.

avoid wall effects, samples tested in the electrodiffusion cell have been obtained by a technique of core sampling and cutting to a disc of 6.5 cm in diameter and 1 cm in thickness. The lateral surface was covered with a resin to ensure a unidirectional transfer.

3.2. Migration test

The experimental setup is depicted in Fig. 1.

The sample is placed between two 1-L compartments. The potential difference is applied between two platine electrodes. Two reference electrodes are used to accurately monitor a constant electrical field (300 V/m) between the faces of the sample. The concentrations of NaOH and KOH were chosen to simulate the pore solution of the mortar [12]: NaOH (0.025 mol/l), KOH (0.083 mol/l).

Two types of measurements were carried out during the test: chloride concentrations in the downstream compartment of the cell by using potentiometric titration and current measurements in time. This migration test is carried out in two stages:

(a) measurement of steady electrical current without NaCl;
(b) measurement of steady electrical current after adding NaCl (0.5 mol/l) in the cathodic solution.

3.3. Results

The chlorides flux through the sample is calculated by:

$$j_{\mathrm{Cl}} = \frac{n}{S\Delta t} \tag{16}$$

where n = increase in number of chloride moles in the downstream compartment in time interval Δt; S = sample area.

Fig. 2 shows the evolution of the chloride concentration measurements in time.

After adding NaCl, the current decreases and becomes temporarily stable. The experimental values of current

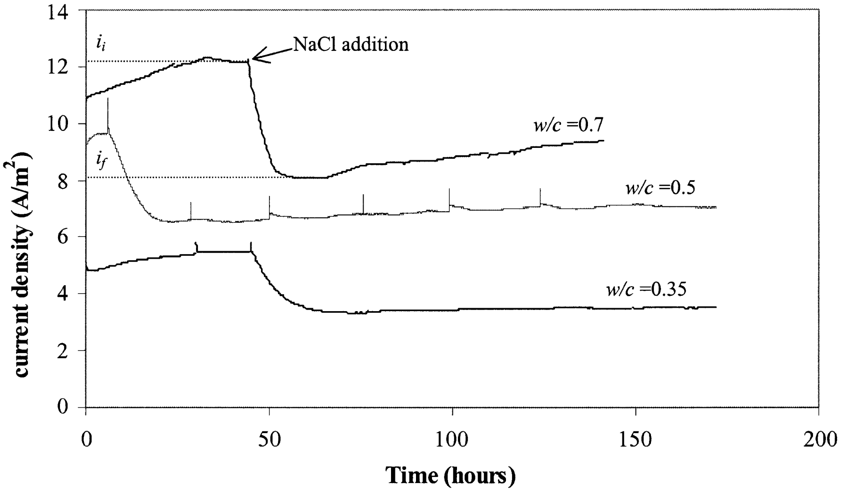

Fig. 3. Chronoamperometry of the migration test for mortar with different w/c ratios.

Table 2
Diffusibility Q calculation for different concentrations of chloride $C_{Cl,up}$, in experiments by Arsenault [13]

$C_{Cl,up}$ (mol/l)	$D_{eCl,up}$ (10^{-12} m^2 s^{-1})	Q for mortar M2[a]
0.248	7.10	0.0116
0.592	3.70	0.0119
0.917	2.30	0.0108
1.015	2.30	0.0119

[a] The mortar M2 used by Arsenault [13] has the same characteristics (w/c = 0.5) as the mortar used in this work.

densities i_i and i_f, corresponding to pseudo-steady state before and after adding NaCl, respectively, are given in the (Table 4). Fig. 3 shows the time evolution of current densities for different mortars.

4. Model validation and discussion

The proposed model is based on Eq. (13). To validate this equation, we try to show that it gives a constant diffusibility, Q, which is an intrinsic characteristic of the material, independently of experimental parameters. We chose to study the $C_{Cl,up}$ effect on the diffusibility Q, from Arsenault's [13] experimental results, which are obtained with the same solution (NaOH and KOH) as the one used in this work. From her measurements of j_{Cl} for different concentrations $C_{Cl,up}$ and from Eqs. (3), (8) and (13), we obtain the diffusibility Q. The calculated values of Q are presented in Table 2.

As can be seen in Table 2, the diffusibility Q is practically constant on the range of concentration considered.

By considering the experimental procedure presented in Section 3.2, concentrations of OH^- and Cl^- ions in the specimen are calculated with Eqs. (11) and (13). Concentrations of the different ions are given in Table 3. It should be noted that the penetration of chlorides involves a significant diminution of the OH^- concentration in the sample, in accordance with previous studies [6,14].

The currents i_i and i_f are deduced from the chronoamperometry (see Fig. 3). These values allow us to calculate the diffusibility Q and the effective diffusion coefficient D_{eCl} with Eqs. (6) and (7), respectively. These calculations are carried out by using experimental value of i_i, i_f and ionic diffusion coefficients at infinite dilution in water at 25 °C: $D_{Cl} = 2.032 \times 10^{-9}$ m^2 s^{-1}; $D_{OH} = 5.26 \times 10^{-9}$ m^2 s^{-1}; $D_{Na} = 1.334 \times 10^{-9}$ m^2 s^{-1}; $D_K = 1.957 \times 10^{-9}$ m^2 s^{-1}.

Table 4
Experimental and theoretical results

	w/c	0.35	0.5	0.7
Experimental results	i_i (A m^{-2})	5.49	9.64	12.15
	i_f (A m^{-2})	3.35	6.54	8.08
	j_{Cl} (10^{-5} mol m^{-2} s^{-1})	1.35	1.81	3.35
Model	Q (10^{-2})	0.62	1.21	1.50
	j_{Cl} (10^{-5} mol m^{-2} s^{-1}), Eq. (14)	1.40	2.02	2.65
	$D_{e,Cl}$ (10^{-11} m^2 s^{-1}), Eq. (8)	1.30	1.74	3.23
	$D_{e,Cl}$ (10^{-11} m^2 s^{-1}), Eq. (7)	1.26	2.47	3.05
	$D_{e,Cl}$ (10^{-11} m^2 s^{-1}), Eq. (15)	1.34	1.95	2.56
From Eq. (9)	$D_{eCl,up}$ (10^{-12} m^2 s^{-1})	2.31	3.10	5.73

Furthermore, the experimental chlorides flux is obtained from Eq. (16) by using chloride titration (Fig. 2). It serves to calculate effective diffusion coefficients D_{eCl} and $D_{eCl,up}$ by using Eqs. (8) and (9), respectively. The diffusion coefficient D_{eCl} can also be calculated by current measurements (i_i, i_f) by using Eq. (7) or Eq. (15). Table 4 summarises these results for the three mortars used in this work.

Based on conductivity measurements, Streicher and Alexander [15] have found Q values between 1 and 1.5×10^{-2} for a mortar w/c = 0.5. The values presented in Table 4 show similar results.

As can be seen in Fig. 3, for all samples, the current density decreases from an initial value i_i to a much lower end value i_f when NaCl is added in the upstream compartment. This current becomes stable then for a few hours (pseudo-steady state) before increasing very slowly. Arsenault [13] and Tong and Gjørv [14] have noted a similar phenomenon. The current decrease can be explained by a modification of the pore conductivity. Indeed, while penetrating in the sample, chlorides replace hydroxides [6,14]. Because of the significant difference between the diffusion coefficient of these ions, both the conductivity and the current decrease.

During the current decrease and the pseudo-steady state, the migration can be considered as the dominating transport phenomenon. But after a long enough time, the ions (Na^+, Cl^-) penetrate in the sample also by diffusion. This phenomenon produces perceptible effects: conductivity increases very slowly and induces a slight rise of the current in time.

For all samples, it must be noted that diffusion coefficients obtained with the model, are about 10^{-11} m^2 s^{-1}, whereas those obtained by using Andrade's relation (Eq. (9)) are about 10^{-12} m^2 s^{-1}, when chloride

Table 3
Concentrations of anions and cations

	Upstream (mol/l)				Sample (mol/l)				Downstream (mol/l)			
	C_{Na}	C_K	C_{OH}	C_{Cl}	C_{Na}	C_K	C_{OH}	C_{Cl}	C_{Na}	C_K	C_{OH}	C_{Cl}
Before adding NaCl	0.025	0.083	0.108	0	0.025	0.083	0.108	0	0.025	0.083	0.108	0
After adding NaCl	0.525	0.083	0.108	0.500	0.025	0.083	0.019	0.089	0.025	0.083	0.108	0

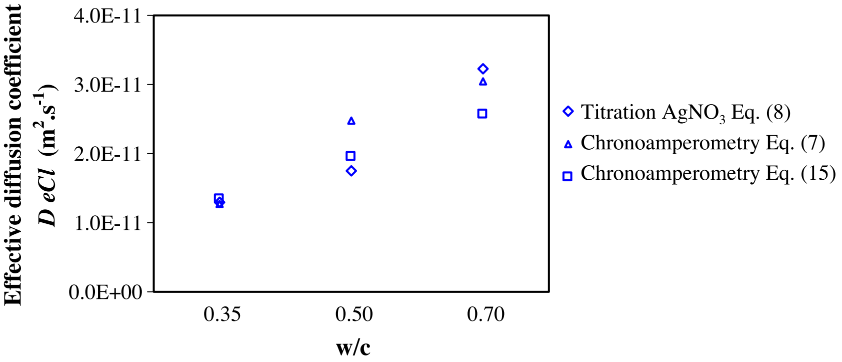

Fig. 4. Comparison between diffusion coefficients (chloride dosage–chronoamperometry).

is dosed in downstream compartment of the migration cell. Relying on the work of Samson and Marchand [7], Arsenault [13] noticed the same phenomenon. The difference between the two values (D_{eCl} and $D_{\mathrm{eCl,up}}$) is due to the limits of the relation (Eq. (9)) because by using this relation for chloride transfer modelling, electroneutrality is obviously neglected. However, in the present work, electroneutrality is considered and explains the difference in the values of diffusion coefficients. With this consideration, it is important to notice that titration (Eq. (8)) or current measurements (Eqs. (7) and (15)) give diffusion coefficients D_{eCl} of the same order of magnitude, as shown in Fig. 4.

As other authors mentioned [16,17], transfer of charged particles involves interfacial phenomena between pore solution and capillary walls (electrical double layer). The geometrical effect (parameter Q) would not be the only factor connecting the effective diffusion coefficient D_{eCl} to the ionic diffusion coefficient D_{Cl}. The model would acquire greater depth in taking into account these interfacial phenomena [18,19].

5. Conclusion

A phenomenological model of the chloride migration in steady state was developed. Based on the electroneutrality and on the Nernst–Planck equation, the model leads to simple equations which are easy to solve. We showed that chronoamperometry is enough to calculate the chloride diffusion coefficient. Thus, chloride titration, a heavy and time-consuming operation, may be avoided.

The chloride diffusion coefficients obtained by using the model D_{eCl}, are higher than those usually found in literature $D_{\mathrm{eCl,up}}$. This work shows the limits of the Nernst–Planck equation without electroneutrality in describing chlorides transfer in cement-based materials. However, as chlorides are charged particles, the model would be highly improved by taking into account electrical double layer occurring at the interface between pore walls and pore solution.

As a first step, this study is limited to steady state. A continuation of this work would consider the nonsteady state in order to shorten the duration of the migration test and to further validate the model.

Nomenclature

C_k	Concentration of the ionic species k in the compartments (mol m^{-3})
$C_{k,\mathrm{S}}$	Concentration of the ionic species k in the pore solution (mol m^{-3})
$C_{k,\mathrm{up}}$	Concentration of the ionic species k in the upstream compartment (mol m^{-3})
$D_{\mathrm{e}k}$	Effective diffusion coefficient of the ionic species k (m^2 s^{-1})
D_k	Diffusion coefficient of ion k at infinite dilution in water at 25 °C (m^2 s^{-1})
$D_{\mathrm{eCl,up}}$	Effective diffusion coefficient of chloride calculated with $C_{\mathrm{Cl,up}}$ (m^2 s^{-1})
L	The thickness of the sample (m)
$E(M)$	Local electrical field at a spatial point M in the pore solution (V m^{-1})
E	Average of electrical field (V m^{-1})
F	Faraday's constant (96,500 C mol^{-1})
i	Average current density (A m^{-2})
i_{i}	Average current density before NaCl addition (A m^{-2})
i_{f}	Average current density after NaCl addition (A m^{-2})
$j_k(M)$	Flux of an ionic species k in the pore solution at a spatial point M (mol m^{-2} s^{-1})
$j_{k,\mathrm{up}}$	Average flux of an ionic species k in the upstream compartment (mol m^{-2} s^{-1})
$j_{k,\mathrm{S}}$	Average flux of an ionic species k in the sample (mol m^{-2} s^{-1})
j_k	Average flux of an ionic species k (mol m^{-2} s^{-1})
Q	Diffusibility
R	Universal gas constant (8.31 J mol^{-1} K^{-1})
T	Absolute temperature (K)
U	Electrical potential difference applied between the two faces of the sample (V)
Z_k	Valence number of species k

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