

Journal of Applied Sciences

ISSN 1812-5654





Electrotransport of Molybdenum Through an Electrodialysis Membrane. Concentration and Potentiel Profiles in Aqueous Interfacial Layers

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Abstract: In the first part we have observed the electrotransport of molybdenum in carbonate media (OH^-,CO^-_3) through a commercial electrodialysis anion exchange membrane. The values of the molybdenum, hydrogen oxide and carbonate fluxes obtained in this study show that molybdenum is present as molybdate ion MoO^-_4 in carbonate solution and that the molybdyl tricarbonate ion existence hypothesis $MoO_2(CO_3)^{4-}_3$ an't no longer be maintained. In order to confirm this result we have studied the electrotransport of molybdenum and carbonate ions with different CO^-_3/MoO^-_4 ratio. The used theory is based on the fact that, the concentration ratio in the membrane equals the one of the same species within the solution. These hypothesis have to be justified given that this equality is actually usual for unstirred layers extremity. Therefore, in the second part the concentration and the potential profiles in aqueous interfacial layers were determined by a numerical resolution of the Nernst-Planck electrodiffusion equation coupled with the Poisson equation. The obtained concentration profiles from numerical resolution differ from 3 to 6% from the ones acquired by analytical resolution method assuming the constant electrical field hypothesis for OH^- , CO^-_3 ions, respectively. However the potential profiles display that constant electrical field hypothesis is strictly valid.

Key words: Electrodialysis, ion-exchange membranes, ion-transfer

INTRODUCTION

The objective of this study was to investigate the electrotransport of molybdenum in the multi-ionic system NaOH/Na₂CO₃/Na₂MoO₄. In the first part we have observed the electrotransport of molybdenum and carbonate ions through a commercial electrodialysis anion exchange membrane. We used different CO⁼₃/MoO⁼₄ ratios. In the second part the concentration and the potential profiles in aqueous interfacial layers were determined by a numerical resolution of the Nernst-Planck electrodiffusion equation coupled with the Poisson equation.

At the time of the Nernst-Planck and Poisson equations resolution several important problems are encountered: Transport in the regions where is no-zero charge density, flux of co-ion equal to zero, transport of solvent and ion-ion interaction and their impact on various transport phenomena when we studied the transport in multi-ionic systems. For the participation of the natural convection, it seems that this phenomenon is less important with the anion exchange membrane. These membranes are known for rising water splitting process

(Zabolotski et al., 1985); Transport problem treatment in multi-ionic system seems to be demanding regarding numerical techniques. Consequently several numerical methods for Nernst-Planck solution and Poisson equation system have been reported previously. As mentioned by certain authors (Choi and Moon, 2002) there is no numerical procedure applied to the description of ionic transport through both membrane and unstirred layers that takes into consideration the mentioned problems. The resolutions of boundary problems are done with considerable mathematical difficulties, especially if the transport involved more then two sorts of ions (Lebedev, 1999). Other problems arise from the mathematical complexity of the coupled differential equation ruling transport. Many researchers have solved the governing equations using simplifying assumptions such as electroneutrality ($\rho = 0$), constant field condition (Lim, 2007). His goal is to develop a Finite Element Method (FEM) to solve the NPP equations (Nernst-Planck-Poisson).

In this present study we propose an interpretative model. This model is based on the one hand on electrodiffusion equations and on the other hand on the

fact that concentration amount ratio of both species in the membrane equals the one of the species it selves within the solution. This theory will be employed to elucidate the independence or the interdependence of CO3 ion and molybdenum. However this hypothesis have to be justified given that this equality is actually usual for unstirred layers extremity. That's why, the concentration and the potential profiles in aqueous interfacial layers (multi-ionic system NaOH/Na₂CO₃/Na₂MoO₄) were determined by a numerical resolution of the Nernst-Planck electrodiffusion equation coupled with the Poisson equation for. We consider two cases, the constant and variable electrical fields. The non-conformity of the electric field in the unstirred layers incites to take into consideration the space charges in this layer, that can't be neglected. The density of space charges is proportional to $\overset{n}{\sum} z_i c_i$. Concerning the flux of co-ion we have used the

radiotracers to underline an eventual leak. Unidirectional ions flux values are calculated from sample radioactivity variation, measured by spectrometry γ (Packard Auto Gamma 500C). The acquired results display that the co-ion Na * leak through the anions exchange membrane is a negligible phenomenon.

The numerical resolution used requires the following steps: discretization, linearization by the Newton method then the resolution by a direct method.

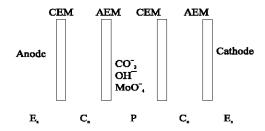
This theme of research which concerns the purification of the carbonate leach solutions of the uranium ores by electrodialysis process has been investigated since 1994 in the laboratory of analysis (Nuclear Research Center of Draria.

The first results have been presented during the international congress on membranes and membrane process (Lounis, 2002).

MATERIALS AND METHODS

Laboratory electrodialysis cells, as well as the experiment procedure and titration method of OH⁻, CO⁼₃ and HCO⁼₃ have been previously described (Lounis, 1997). The effective area of each membrane and each electrode is 10 cm². An ASTI pump provides the solution dilution flow; its initial volume is 100 mL. The current density is 20 mA cm⁻². The cell, schematically represented in Fig. 1, is composed of five compartments, that are separated by ion exchange membranes. In the central compartment P, a solution containing OH⁻, CO⁼₃ and MoO⁼₄ anions was circulating.

The membranes used are Selemion AMV (anion exchange membrane) and CMV (cation exchange membrane) produced by Asahi Glass. Sampling is made in



E, and E, : Electrode compartments
P : Central compartments
C, and C, : Concentration compartments
CEM : Cation exchange membrane
AEM : Antion exchange membrane

Fig. 1: Electrodialysis cell schematic

the central and anodic compartment at regular intervals of time. Molybdenum is titrated by spectrophotometric method using thiocyanate (Gordon and Parker, 1983).

RESULTS AND DISCUSSION

On Fig. 2 are represented the number variations of meq of $\mathrm{OH^-}$ and $\mathrm{CO^-_3}$ ions transferred from the diluate of which initial compositions are NaOH 0,125 eq $\mathrm{L^{-1}}$ and Na₂CO₃ 0,13 eq $\mathrm{L^{-1}}$ to the anodic concentrate. From the obtained straight lines, we can deduce that during the two hours the flux represent 88% of the total current that is carried by these two ions. The flux of electrical charges carried by $\mathrm{OH^-}$ ions is almost two times higher than the one carried by $\mathrm{CO^-_3}$ ions.

It results that in the central compartment, after two hours of ED, the concentration of OH⁻ ions is 10 times lower than that of CO⁼₃ while at initial time the concentration of these two ions was practically identical.

The Fig. 3 demonstrates that the transfer of hydroxyl ions (OH⁻) is not modified by the presence of molybdenum ions. However, the electrotransport of carbonate ions is altered. During 90 min of ED we have obtained similar result with the former case, the concentration of OH⁻ ions is 10 times smaller than that of CO⁻₃. The percentage of ions recovery are 72, 33 and 13% for OH⁻, MoO⁻₄ and CO⁻₃, respectively.

These results allow us to claim the following conclusion: The molybdenum is present in alkaline solution as the molybdate ion (MoO_4^-) form, in case of the existence of molybdyl ion $MoO_2(CO_3)^{4}$, we should observe a higher transfer of CO_3^- ions. This result is similar to that of Pascal (1959). The action of an excess hard base on the molybdenum gives in the solution a molybdate ion when the ratio of $MOH/MoO_3 > 2$, in our case this ratio is as high as 6.

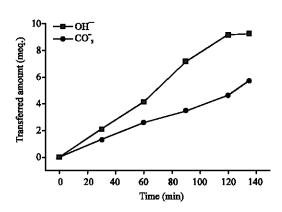


Fig. 2: Variation vs. time of the OH⁻, CO⁻₃ ions leaving the central compartment

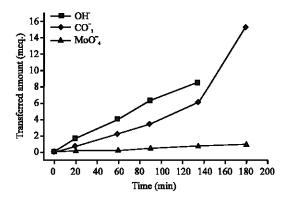


Fig. 3: Variation vs. time of the OH⁻, CO⁻₃ and MoO⁻₄ ions leaving the central compartment

In order to confirm this result we have studied the electrotransport of molybdenum and carbonate ions with different [CO⁻₃]/[MoO⁻₄] ratio. The ratios are 1.16, 3.5, 7.33, 10, 14.5 and 18.29. The Fig. 4-9 assert these results.

The following assumptions are made to analyze the obtained data:

- Electro neutrality holds in all part of the membranesolution system.
- The two layers on both sides of the solutionmembrane interface are in quasi equilibrium data.
- The diffusion term is neglected.

The Nernst-Planck electrodiffusion equation is

$$J_{i} = -D_{i} \left[\frac{dC_{i}}{dx} + z_{i} \frac{F}{RT} C_{i} \frac{d\Phi}{dx} \right]$$
 (1)

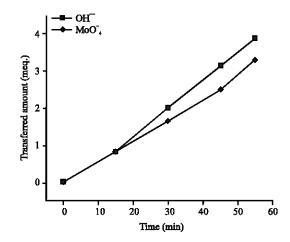


Fig. 4: Variation vs. time of the $CO_3^=$ and $MoO_4^=$ ions leaving the central compartment $[CO_3^=]/[MoO_4^=]$ = 1.16

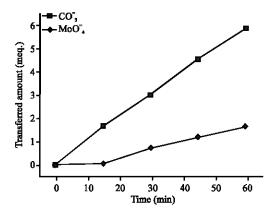


Fig. 5: Variation vs. time of the ${\rm CO}_3^=$ and ${\rm MoO}_4^=$ ions leaving the central compartment $[{\rm CO}_3^=]/[{\rm MoO}_4^=]$ = 3.5

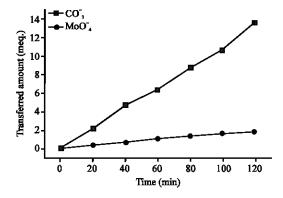


Fig. 6: Variation vs. time of the $CO_3^=$ and $MoO_4^=$ ions leaving the central compartment $[CO_3^=]/[MoO_4^=]$ = 7.19

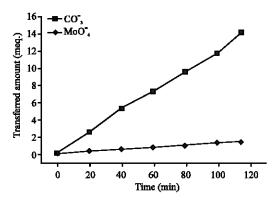


Fig. 7: Variation vs. time of the $CO_3^=$ and $MoO_4^=$ ions leaving the central compartment $[CO_3^=]/[MoO_4^=]$ = 10

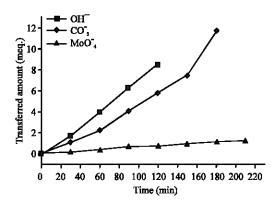


Fig. 8: Variation vs. time of the CO⁼₃, OH and MoO⁼₄ ions leaving the central compartment [CO⁼₃]/[MoO⁼₄] = 14.5

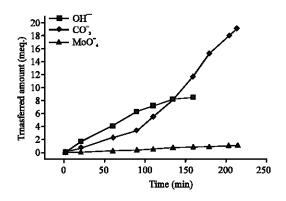


Fig. 9: Variation vs. time of the $CO_{3}^{=}$, OH^{-} and $MoO_{4}^{=}$ g.ions leaving the central compartment. $[CO_{3}^{=}]/[MoO_{4}^{=}] = 18.3$

 J_i = Flux of I ion across the exchange membrane (g eq cm⁻² s⁻¹)

 D_i = Diffusion coefficient of ion (cm² s⁻¹)

 C_i = Concentration of i ion (g eq L^{-1})

F = The Faraday constant (c)

R = The gas constant $(J \text{ mole } K^{-1})$

T = The absolute temperature (K) $d\phi/dx$ = The gradient of electrical potential

Under this assumptions the Nernst-Planck electrodiffusion equation becomes:

$$\mathbf{J}_{i} = \mathbf{u}_{i}^{m} \mathbf{z}_{i} \mathbf{C}_{i}^{m} \frac{d\Phi^{m}}{d\mathbf{x}} \tag{2}$$

The Nernst Einstein relation relates the mobility of the species i to its diffusion coefficient:

$$\mathrm{D}_{i}^{m} = \frac{\mathrm{RT}}{\mathrm{E}} \mathrm{u}_{i}^{m} \tag{3}$$

Equation 1 and 2 give

$$J_{i} = -\frac{F}{RT} D_{i}^{m} z_{i} C_{i}^{m} \frac{d\Phi^{m}}{dx}$$

$$\tag{4}$$

From the Eq. (3) the expressions of ${}^{J}_{MoO_4^{2-}}$ and ${}^{J}_{CO_3^{2-}}$ are:

$$J_{MoO_{4}^{=}} = \frac{F}{RT} D_{MoO_{4}^{=}}^{m} 2C_{MoO_{4}^{=}}^{m} \frac{d\Phi^{m}}{dx}$$
 (5)

$$J_{CO_3^{=}} = \frac{F}{RT} D_{CO_3^{=}}^{m} 2 C_{CO_3^{=}}^{m} \frac{d\Phi}{dx}^{m}$$
 (6)

The ratio of these fluxes gives

$$\frac{J_{MoO_{4}^{=}}}{J_{CO_{3}^{=}}} = \frac{D_{MoO_{4}^{=}}^{m} C_{MoO_{4}^{=}}^{m}}{D_{CO_{3}^{=}}^{m} C_{CO_{3}^{=}}^{m}}$$
(7)

In which the concentration ratio inside the membrane can be replaced by the concentration ratio in the bulk.

$$\frac{C_{MoO_{4}^{-}}^{m}}{C_{CO_{2}^{-}}^{m}} = \frac{C_{MoO_{4}^{-}}}{C_{CO_{3}^{-}}}$$
(8)

Thus

$$K = \frac{D_{MOO_{4}^{\pm}}^{m}}{D_{CO_{3}^{\pm}}^{m}} = \frac{J_{MoO_{4}^{\pm}}^{-}C_{MoO_{4}^{\pm}}^{-}}{J_{CO_{3}^{\pm}}^{-}C_{CO_{3}^{\pm}}}$$
(9)

$$\frac{J_{MOO_{4}^{-}}}{J_{CO_{3}^{-}}} = K \frac{C_{MoO_{4}^{-}}}{C_{CO_{3}^{-}}}$$
(10)

On the Fig. 10 the value of $^{-J}_{MoO_4^+}/^{J}_{CO_3^-}$ were plotted as a function of $^{C}_{MoO_4^-}/^{C}_{CO_3^-}$. The values show that the

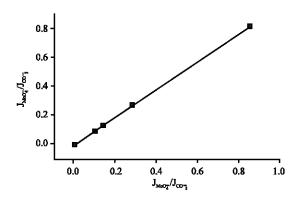


Fig. 10: Variation vs. $C_{MoO_4^{-}}/C_{CO_3^{-}}$ of $J_{MoO_4^{-}}/J_{CO_3^{-}}$

transport theoretical model used is satisfied at different concentration ratios. A proportional variation is obtained as might be expected from Eq. 10.

Concentration and potential profiles: Since necessary factors to resolve the Nernst-Planck equation within the membrane are being unknown, we will only study the ions electrotransport in the unstirred layers (Nernst-Planck film) close to the membrane.

The Nernst Planck equation is resolved keeping in mind, on one hand that the electrical field is constant and on the other hand using the Poisson equation, which explains the distribution of electrical charges.

For the NaOH/Na₂CO₃/Na₂MoO₄ system, the Nernst-Planck electrodiffusion equation in general feature gives:

$$J_{OH^{-}} = -D_{OH^{-}} \left[\frac{dC_{OH^{-}}}{dx} - \frac{C_{OH^{-}}}{RT} .F. \frac{d\Phi}{dx} \right]$$
 (11)

$$J_{CO_{\overline{3}}^{=}} = -D_{CO_{\overline{3}}^{=}} \left[\frac{dC_{CO_{\overline{3}}^{=}}}{dx} - \frac{2C_{CO_{\overline{3}}^{=}}}{RT} F. \frac{d\Phi}{dx} \right]$$
 (12)

$${\rm J}_{MoO_{4}^{=}} = -{\rm D}_{MoO_{4}^{=}} [\frac{{\rm dC}_{MoO_{4}^{=}}}{{\rm dx}} - \frac{{\rm 2C}_{MoO_{4}^{=}}}{{\rm RT}}.{\rm F}.\frac{{\rm d\Phi}}{{\rm dx}}]^{-(13)}$$

$$J_{Na^{+}} = -D_{Na^{+}} \left[\frac{dC_{Na^{+}}}{dx} + \frac{C_{Na^{+}}}{RT} .F. \frac{d\Phi}{dx} \right]$$
 (14)

(It can be checked that $J_{Na^+} = 0$)

The relation between potential and distribution of electrical charges is ruled by the Poisson equation.

$$\frac{\mathrm{d}^2\Phi}{\mathrm{dx}^2} = -\frac{\rho}{\varepsilon_0\varepsilon_\mathrm{r}} \tag{15}$$

Where, ε_0 is the permittivity of vacuum, ε_r the relative permittivity, Φ the electrical potential and ρ the local charge density defined as:

$$\rho(x) = F\left(\sum_{i}^{n} z_{i} C_{i}(x) - N(x)\right)$$
 (16)

N(x) is the concentration of fixed charges

$$\rho = ({\rm C}_{Na}^+ - 2{\rm C}_{{\rm CO}_3^-}^- - 2{\rm C}_{{\rm MoO}_4^-} - {\rm C}_{OH}^-) F \ \ \text{in a medium with}$$

no fixed charge distribution

Reduced variables are defined to resolve the different equations.

$$\begin{split} \xi &= \frac{x}{\delta}; \phi = \frac{F.\Phi}{RT}; \sigma = \frac{\epsilon_{O}.\epsilon_{T}.RT}{N_{O}..F^{2}.\delta^{2}} \\ OH &= \frac{C_{OH}^{-}(x)}{N_{O}}; CO = 2\frac{C_{CO_{\overline{\mathbf{3}}}}(x)}{N_{O}}; MO = 2\frac{C_{MoO_{\overline{\mathbf{4}}}}(x)}{N_{O}}; \\ NA &= \frac{C_{Na^{+}}(x)}{N_{O}} \end{split}$$

Where, N_0 is the normality of the bulk solution expressed in mol cm⁻³.

The membrane solution interfaces are given in Fig. 11.

With the reduced variables the above system of equation becomes:

$$OH + CO + MO - NA + \sigma \frac{d^2\phi}{d^2} = 0$$
 (17)

$$\frac{dOH}{d\xi} - OH\frac{d\phi}{d\xi} + \alpha = 0 \tag{18}$$

$$\frac{1}{2}\frac{dCO}{d\xi} - CO\frac{d\phi}{d\xi} + \beta = 0 \tag{19}$$

$$\frac{1}{2}\frac{dMO}{d\xi} - MO\frac{d\phi}{d\xi} + \gamma = 0 \tag{20}$$

$$\frac{dNA}{d\xi} + NA\frac{d\phi}{d\xi} = 0 \tag{21}$$

Where the unknown factors are OH, CO, MO, NA and ϕ and the boundary conditions are:

$$\begin{split} & \mathrm{OH}_0 = \mathrm{OH}_{\xi=0}\,;\, \mathrm{CO}_0 = \mathrm{CO}_{\xi=0}\,;\, \mathrm{MO}_0 = \mathrm{MO}_{\xi=0} \\ & \mathrm{NA}_0 = \mathrm{NA}_{\xi=0}\,;\, \phi_{\xi=0} = 0;\, \phi_{\xi=1} = \mathrm{V} \end{split}$$

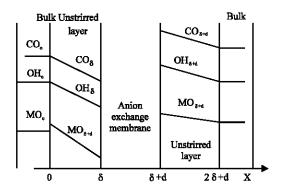


Fig. 11: Concentration profiles in unstirred layers (schematic)

 $^{J}{\rm Na^{+}}, ^{J}{\rm OH^{-}}, ^{J}{\rm CO_{3}^{=}}$ and δ being constant the same for $\alpha,$ β and $\gamma.$

With
$$\alpha = \frac{J_{OH}^{-}.\delta}{N_{o}.D_{OH}^{-}}$$
; $\beta = \frac{J_{CO_3^{=}.\delta}}{N_{o}.D_{CO_3^{=}}}$; $\gamma = \frac{J_{MoO_4^{=}.\delta}}{N_{o}.D_{MoO_4^{=}}}$

Case where the electrical field is constant: The concentration profiles are obtained by resolving the system of equations differential in first order with constant coefficients, which solutions are:

$$OH = (OH_0 - \frac{\alpha}{\theta}) \exp(\theta \xi) + \frac{\alpha}{\theta}$$
 (22)

$$CO = (CO_0 - \frac{\beta}{\rho}) exp(2\theta\xi) + \frac{\beta}{\rho}$$
 (23)

$$MO = (MO_0 - \frac{\gamma}{\theta}) exp(2\theta\xi) + \frac{\gamma}{\theta}$$
 (24)

$$NA = NA_0 \exp(-\theta \xi) \tag{25}$$

With
$$\theta = \frac{d\phi}{d\xi}$$

Replacing the expressions OH; CO; MO and NA in the electroneutrality Eq. 17 we get a third degree equation in which the unknown θ appears both in the exponential term and in the quotient.

$$\begin{split} &(\mathrm{OH}_{o}-\frac{\alpha}{\theta})\exp(2\theta\xi)+(\mathrm{CO}_{o}+\mathrm{MO}_{o}-\frac{\beta}{\theta}-\frac{\gamma}{\theta})\exp(3\theta\xi)+\\ &(\frac{\alpha+\beta+\gamma}{\theta})\exp(\theta\xi)-\mathrm{NA}_{o}=0 \end{split}$$

This equation presents a non-linearity. It is solved by the dichotomy method (Press *et al.*, 1992) for a given value of ξ . Then, we obtain the concentration profiles. The potential profiles are obtained from:

$$\frac{d\Phi}{dx} = -\theta \frac{RT}{FS} \tag{27}$$

The following parameters were used for the calculations:

D_{OH} =
$$5.31.10^{-5}$$
cm² s⁻¹; δ = $2.25.10^{-3}$ cm
D_{MoO}⁼ = $0.995.10^{-5}$ cm². s⁻¹; R = 8.3144 Jmol⁻¹K⁻¹;
D_{CO}⁼ = $0.925.10^{-5}$ cm² s⁻¹; T = 294 K
F = $9.6485.10^{4}$ Cmol⁻¹:

Case where the electrical field is not constant: The numerical resolution used requires the following steps:

- Discretization with the finite difference method
- Linearization by the Newton method
- Equation system is solved using a direct method

We note a slight discrepancy between the profiles obtained from the Nernst-Planck-Poisson equation solution and those obtained with the resolution of equation using the constant electrical field hypothesis.

The result accords with those proposed by Taky (1991) and Chapotot (1994) where the difference appears from a current density over or equal to 20 mA cm⁻².

In the anodic interfacial layer the concentration and potential profiles are given in the Fig. 12-15 for the studied

ratio
$$\frac{[CO_3^{\pm}]}{[MoO_4^{\pm}]} = 14.5 \text{ and } \frac{[CO_3^{\pm}]}{[MoO_4^{\pm}]} = 18.3,$$

respectively with the AMV Selemion membrane.

We observe that in the present conditions (current density 20 mA cm $^{-2}$ and complete concentration equal 0.265 mol $\rm L^{-1}$) the concentration profiles obtained from numerical resolution differ about 3 to 6% from to the values obtained by the analytical resolution method assuming the constant electrical field hypothesis for OH ,CO $^{-}_{3}$ ions, respectively. However, the potential profiles show that the constant electrical field hypothesis is strictly valid.

The concentration of OH⁻ in the diffusion layer decreases near the interface.

The concentration diminution of CO₃ and MoO₄ ions near the membrane surface shows that those ions transfer across the membrane in spite of their

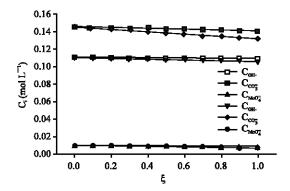


Fig. 12: Concentration profiles for the case $N^{\circ}1$ [CO $^{-}_{3}$]/[MoO $^{-}_{4}$] = 14.5, $\blacksquare \infty$ Constant electrical field hypothesis. $+\Delta+$ Nernst-Planck-Poisson resolution

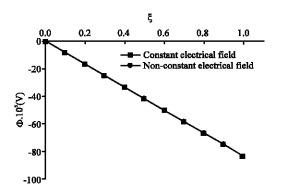


Fig. 13: Potential profiles for the case $N^{\circ}1$ [CO $_{3}$]/[MoO $_{4}$] = 14.5

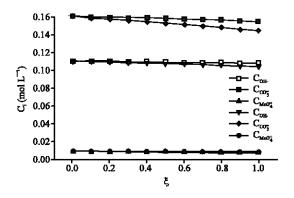


Fig. 14: Concentration profiles for the case $N^{\circ}2$ [CO $^{\circ}_{3}$]/[MoO $^{\circ}_{4}$] = 18.3, $\blacksquare \infty$ Constant electrical field hypothesis. $+\Delta+$ Nernst-Planck-Poisson resolution

electrotransport mobility is less fast then that of the hydroxyl ions. The values of the transfer show that 97, 25 and 48% for the OH, CO⁼₃ and moO⁼₄ ions, respectively are reached.

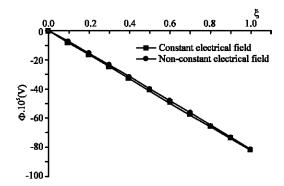


Fig. 15: Potentiel profiles for the case $N^{\circ}2$ [$CO^{=}_{3}$]/[$MoO^{=}_{4}$] = 18.3

We also note that when the ratio

$$\frac{[CO_3^{=}]}{[MoO_4^{=}]}$$

ions in the solution increases the CO⁼₃ ions it contributes more to current transport and the concentration of CO⁼₃ decreases in the interface layer. This result is also confirmed with the percentage recovery of CO⁼₃ ions which is 51 and 60.05%, respectively for both cases

$$\frac{[\text{CO}_3^{=}]}{[\text{MoO}_4^{=}]}$$
 = 14.5 and 18.3.

CONCLUSION

- The elimination of carbonate and molybdenum ions from aqueous solution can be easily performed by electrodialysis.
- The electrotransfer of molybdenum ion shows that molybdate ion is present in alkaline solution.
- The high values of the molybdenum flux obtained in this study show that electrodialysis can be used as a separation technique for molybdenum extraction.
- The results reported here indicate that the best value of the ratio CO[■]₃ for molybdenum extraction through an anion exchange membrane is 1.16.
- The concentration profiles obtained from numerical resolution differ in 3 and 6% from to the values obtained by the analytical resolution method assuming the constant electrical field hypothesis for OH⁻, CO⁻₃, respectively. Meanwhile the potential profiles show that the constant electrical field hypothesis is strictly valid.

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