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Efficient Modeling of Electrodialysis Process for Waste Water Treatment through Systematic Parameter Estimation

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Electrodialysis is a promising electrochemical process for separation and recovery of useful ions from waste waters. The large number of parameters involved in electrodialysis models makes their estimation challenging. This work proposes a systematic sensitivity analysis approach to identify the impact model parameter variations exert on multiple electrodialysis performance indicators, within a wide operating range. This enables the robust mapping of electrodialysis performance toward the direction of maximum variability in the multi-dimensional parametric space and the identification of operating regimes, where the input parameters exhibit the highest sensitivity. Such regimes are used to determine upper and lower limits in the parameter estimation problem. Parameters and ranges exerting insignificant changes on the output electrodialysis indicators are omitted from evaluation, which reduces the associated complexity. The approach is validated against published experimental results obtained for a lead ions removal electrodialysis recirculation batch process. Only four out of nine parameters need to be estimated, while excellent match is observed between experimental and predicted data.

1. Introduction

Electrodialysis (ED) is an electrochemical process in which ions migrate through ion-selective membranes as a result of their attraction due to two electrically charged electrodes. Although initially established for water desalination (Vargas et al., 2011), ED has been gaining attention for treatment of dilute waste water streams (Scialdone et al., 2014) due to lower capital and operational costs than methods such as ettringite precipitation (Usinowicz et al., 2006) and to efficient ion recovery for subsequent reuse (Pilat, 2001). The development of ED models is very important to scale-up and optimise the process. However, ED modelling includes significant challenges due to the complexity of the associated electrochemical and mass transfer phenomena such as ion electro-migration, ion diffusion, and water transport. The accurate prediction of ED output performance indicators such as current density and concentration profiles may require the determination of over 30 model parameters pertaining to geometrical, structural, electro-chemical and other features (Rohman et al., 2010). The values of such parameters are often not reported in experimental studies either because they are difficult to measure or the scope of the studies is to assess the separation capabilities of ED through measurement of solely output performance indicators (Campione et al., 2018). The latter could be used for the estimation of the missing model parameter values, which enabling the development of ED models for various ionic species. The large number of model parameters and the diverse operating regimes used in ED for the removal of different species make parameter estimation through non-linear optimization very challenging (Ortiz et al., 2005). Conventionally, the entire set of parameters is considered despite that the ED performance may not be affected uniformly by all of them. Parameter value ranges are defined arbitrarily through trial and error (Campione et al, 2018) with detrimental effects on estimation quality and computational effort (Ortiz et al., 2005).
To address these challenges, a systematic approach is proposed and implemented for the first time in ED systems to determine parameter value ranges which may be used as upper and lower limits in optimisation-

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based, parameter estimation methods. The proposed approach enables the identification of the impact simultaneous and multiple parameter variations exert on the ED output within a wide range of condition changes, using a non-linear sensitivity analysis method (Seferlis and Hrymak, 1996). Parameters that exert almost negligible changes on the output ED performance indicators, even under large variability of conditions, are omitted from consideration in the parameter estimation problem (Papadopoulos et al., 2013). This allows estimation algorithms to be focused on the estimation of the values for fewer parameters that significantly affect the quality of model fitting, which obtaining reliable results with reduced computational effort. The proposed approach is validated against experimental results for an ED process used in the removal of lead ions from aqueous solutions in the form of lead nitrate (Gherasim et al., 2014). Lead is a hazardous polluting metal, which is found in industrial wastewaters (dyes, batteries, vehicles, manufacture and so forth) (Ghorbani et al., 2020) and drinking water (Harvey et al., 2016), at elevated concentrations.

2. Models and methods

2.1 Modeling of an ED system

A schematic diagram of an ED process for batch recirculation is presented in Figure 1. Briefly, a salt solution is pumped through an ED cell from a feed tank, while an electric voltage potential is applied by the power supply across the stack resulting to two distinct groups of compartments-cells. The first solution is called concentrate (indicated as C) and includes the brine solution. The second solution is called diluate (indicated as D) and is related to the feed solution. The cations move towards the cathode, and the anions towards the anode. The cations pass through the negatively charged CEM (Cation Exchange Membranes), and the anions pass through the positively charged AEM (Anion Exchange Membranes). Consequently, a rise of ion concentration is observed in the concentrate compartment and a decrease in the dilute compartment.

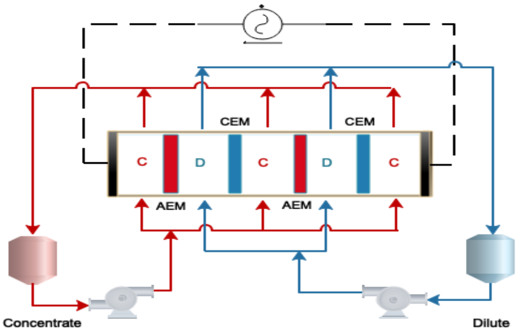


Figure 1: ED batch recirculation process

The mathematical model that describes the ED process is given by the following equations (Ortiz et al., 2005):

$$\frac{dC_{conc}}{dt} = \frac{Q(C_{conc}^{tank} - C_{conc}) + \frac{N\phi jA}{zF} - \frac{NAD_{AEM}\left(C_{conc}^{AEM} - C_{dil}^{AEM}\right)}{l_{AEM}} - \frac{NAD_{CEM}\left(C_{conc}^{CEM} - C_{dil}^{CEM}\right)}{l_{CEM}}}{NV_{comp}} \tag{1}$$

$$\frac{dC_{dil}}{dt} = \frac{Q\left(C_{dil}^{tank} - C_{dil}\right) - \frac{N\phi jA}{zF} + \frac{NAD_{AEM}\left(C_{conc}^{AEM} - C_{dil}^{AEM}\right)}{l_{AEM}} + \frac{NAD_{CEM}\left(C_{conc}^{CEM} - C_{dil}^{CEM}\right)}{l_{CEM}}}{NV_{comp}} \tag{2}$$

$$\frac{dC_{conc}^{tank}}{dt} = \frac{Q(C_{conc} - C_{conc}^{tank})}{V_{conc}^{tank}}, \quad \frac{dC_{dil}^{tank}}{dt} = \frac{Q\left(C_{dil} - C_{dil}^{tank}\right)}{V_{dil}^{tank}} \tag{3}$$

$$C_{conc}^{AEM} = C_{conc} + \frac{\phi j}{zFk_m}(t^{AEM} - t^-), \quad C_{dil}^{AEM} = C_{dil} - \frac{\phi j}{zFk_m}(t^{AEM} - t^-) \tag{4}$$

$$C_{conc}^{CEM} = C_{conc} + \frac{\phi j}{zFk_m}(t^{CEM} - t^+), \quad C_{dil}^{CEM} = C_{dil} - \frac{\phi j}{zFk_m}(t^{CEM} - t^+) \tag{5}$$

It is composed of the mass balances in the concentrate, Eq(1), and diluate, Eq(2), compartments of the ED stack, and the mass balances in the two tanks shown in Eq(3). N is the number of cell pairs, V_{comp} the compartment volume, Q the overall flow rate of the solutions, $C_{conc}, C_{dil}, C_{conc}^{tank}, C_{dil}^{tank}$ the concentration of the concentrate and diluate solutions at the outlet and inlet of the ED stack, ϕ the current efficiency, j the current density, A the membrane area, z the ion's charge, F Faraday's constant, D_{AEM} and D_{CEM} the diffusion coefficients through AEM and CEM, l_{AEM} and l_{CEM} the membrane thickness, and $C_{conc}^{AEM}, C_{dil}^{AEM}$, $C_{conc}^{CEM}, C_{dil}^{CEM}$ the concentrations on the surfaces of the AEM and CEM in concentrate and dilute compartments. The latter are calculated with Eq(4) and Eq(5), where t^- and t^+ are the solution's transport numbers, t^{AEM} and t^{CEM} are the transport numbers in AEM and CEM, and k_m the mass transfer coefficient. The first three modes of mass transport are derived mainly from fundamental continuity and Nernst-Planck equations. In the selected model, it is assumed that the water volume transferred from the diluate to the concentrate tank is negligible (Wright et al., 2018). ED highly relies on the applied voltage and on the corresponding current density. The relationship describing the electrical current flow is obtained from Kirchhoff's 2nd law and Ohm's law (Wright et al., 2018).

2.2 Proposed sensitivity analysis approach

This work employs sensitivity analysis in order a) to identify the ED process operating parameters that affect the process performance significantly and b) to determine the upper and lower bounds of those parameter values, where high sensitivity is observed. Firstly, for every time step t, out of a total N_t steps, a sensitivity matrix $P(t)$ is developed which incorporates the derivatives of multiple output ED operating indicators in vector $F(t)$ (e.g., concentrations and current density) with respect to input parameters in vector $\varepsilon(t)$, as follows:

$$P(t) = \begin{bmatrix} \frac{\partial lnF_1(t)}{\partial ln\varepsilon_1(t)} & \cdots & \frac{\partial lnF_1(t)}{\partial ln\varepsilon_{N_p}(t)} \\ \vdots & \ddots & \vdots \\ \frac{\partial lnF_{N_F}(t)}{\partial ln\varepsilon_1(t)} & \cdots & \frac{\partial lnF_{N_F}(t)}{\partial ln\varepsilon_{N_p}(t)} \end{bmatrix} \tag{6}$$

In Eq(6), N_p is the total number of operating parameters and N_f is the total number of ED operating indicators. The matrix derivatives are calculated by propagating through the ED model a vector of infinitesimal changes $\Delta\boldsymbol{\varepsilon}$ on vector ε^{nom}, which includes the nominal values for the ED model parameters. The nominal values are obtained either from experimental data when available, or from prior knowledge of similar systems. The decomposition of $P^T(t)P(t)$ provides the matrix eigenvectors $\Theta^k(t)$ for each parameter in vector ε, where $k = [1, N_e]$ and N_e is the total number of eigenvectors. The eigenvector $\Theta^1(t)$ that corresponds to the largest in magnitude eigenvalue of $P^T(t)P(t)$ indicates the dominant direction of variability in the multi-parametric space which results from the simultaneous consideration of the combined effects of all parameters in vector $\varepsilon(t)$ on all ED performance indicators in $F(t)$. Individual entries in the eigenvector $\Theta^1(t)$ direction indicate the contribution of the corresponding to the entry parameter in the dominant direction. A relatively few eigenvectors can explain a large percentage of the system variability and the dimensionality of the problem can be reduced. Subsequently, the parameter estimation problem can focus on the parameters involved in the few dominant eigenvector directions. For each such parameter, the range in which they exert the highest effect on the ED performance indicators can be identified through the following transformation:

$$\varepsilon(t,\zeta_t) = \varepsilon^{nom} \cdot \zeta_t \cdot \Theta^1(t) + \varepsilon^{nom}, \qquad \zeta_t \in [\zeta_t^L, \zeta_t^U] \tag{7}$$

In Eq(7), ζ_t represents the parameter variation magnitude coordinate. The same ζ_t value is imposed on all parameters to move $\varepsilon(t,\zeta_t)$ in the direction of $\Theta^1(t)$, accounting for the direction of maximum variability with respect to $F(t,\zeta_t)$. The values of $\varepsilon(t,\zeta_t)$ are propagated through the ED model to obtain $F(t,\zeta_t)$. The values of $F^{exp}(t)$ are available from experimental results. By moving ζ_t within $[\zeta_t^L, \zeta_t^U]$, so that $F(t,\zeta_t)$ approaches $F^{exp}(t)$ to the closest possible proximity, $\varepsilon(t,\zeta_t^L)$ and $\varepsilon(t,\zeta_t^U)$ are obtained. These values are used as upper and lower limits in parameter estimation. The method is implemented for every time step t. In this respect, the parameter estimation problem is limited systematically within a reduced range that captures the combined effect of all parameters on the ED performance indicators, as opposed to the arbitrary determination of such ranges otherwise. Having estimated the upper and lower limits, parameter estimation is implemented as follows:

$$\begin{aligned} &\min_{\varepsilon(t)} J(t) = \sum_{l=1}^{N_f} \left(W_l \cdot \sum_{t=1}^{N_t} \frac{F_l(\varepsilon(t),t) - F_l^{exp}(t)}{F_l^{exp}(t)} \right)^2 \\ &s.t.\ Eq(1) - Eq(5) \\ &\quad \varepsilon^L(t) \le \varepsilon(t) \le \varepsilon^U(\mathrm{t}) \end{aligned} \tag{8}$$

$$t^L \leq t \leq t^U$$

The objective function depicts the changes of the performance indicators of the ED system, caused by changes in parameter values between the upper and lower limits as set above, compared to the experimental data points. Vector t represents the time with t^L and t^U being lower and upper limits and W_l is a vector of weights in case it is desired to differentiate the impact of the objective functions. $\varepsilon^L(t)$ and $\varepsilon^U(\mathrm{t})$ correspond to the previously determined $\varepsilon(\mathrm{t}, \zeta_t^L)$ and $\varepsilon(t, \zeta_t^U)$ at a specific ζ, and so the lack of notation.

3. Implementation

The proposed approach is implemented to estimate parameters required for the simulation of an ED process used for the separation of Pb^{2+} and NO_3^- ions from waste water (Gherasim et al., 2014). These authors examined the impact flow rate, applied voltage, temperature, and feed concentration have on lead nitrate removal by ED in a batch recirculation mode. The parameters used within the current work include V_{comp}, k_m, D_{CEM}, D_{AEM}, R_{CEM}, R_{AEM}, t^-, t^+ and E_{el} (electrode potential difference). After calculation of the eigenvectors, it is observed that k_m, R_{CEM} and R_{AEM} are not involved in the dominant direction, their values are considered fixed at the values suggested by Ortiz et al. (2005). Non-dominant behaviour is also observed for t^- and t^+; their values are also considered fixed and calculated based on Gao et al. (2009). The relationship between t^-, t^+ and Λ_0 (infinite molar conductivity) is used with the infinite ion conductivities for Pb^{2+}, NO_3^- (Haynes et al., 2012). Eventually, the parameters involved in the dominant direction of variability are V_{comp}, D_{CEM}, D_{AEM} and E_{el}.Their initial (nominal) values for the parameter estimation were also obtained from Ortiz et al. (2005). Except for the latter four parameters, the values of all others are shown in Table 1. The parameters used as performance criteria in vector F are the concentrate and diluate concentrations and the current density. In parameter estimation, the concentrate and diluate concentrations are assumed to have a significant effect on the system performance, so their weights in Eq(8) are higher than that of the current density. The applied source voltage is 10 V, the flow rate is 0.070 m^3/h, and the geometrical features of the ED stack are from Gherasim et al. (2014).

Table 1: List of parameters

Parameters	Units	Values	Source	Parameters	Units	Values	Source
A	cm^2	64	Gherasim et al. (2014)	V_{conc}^{tank}	L	5	Gherasim et al. (2014)
B_0	Å mol^{-1}	0.327	Kortum et al. (1965)	V_{dil}^{tank}	L	20	Gherasim et al. (2014)
B_1	$mol^{-1/2}$	0.227	Kortum et al. (1965)	R_{AEM}	Ω cm^2	29	Ortiz et al. (2005)
B_2	$Ω^{-1}$ m^2 $mol^{-3/2}$	54.16	Kortum et al. (1965)	R_{CEM}	Ω cm^2	24	Ortiz et al. (2005)
l_{AEM}, l_{CEM}	mm	0.45	Gherasim et al. (2014)	$\gamma_{conc}^{AEM}, \gamma_{conc}^{CEM}$	-	0.9	Wright et al. (2018)
L	mm	0.8	Gherasim et al. (2014)	$\gamma_{dil}^{AEM}, \gamma_{dil}^{CEM}$	-	0.8	Wright et al. (2018)
N	-	10	Gherasim et al (2014)	F	C mol^{-1}	96485	-
α	Å	4	Kortum et al. (1965)	T	K	297	Gherasim et al. (2014)
ϕ	-	0.92	Ortiz et al. (2005)	R	J mol^{-1} K^{-1}	8.314	-
t^{AEM}, t^{CEM}	-	1	Ortiz et al. (2005)	Λ_0	S m^2 mol^{-1}	$213 \cdot 10^{-4}$	Gao et al. (2009)
z	-	2	-	t^+	-	0.66	Gao et al. (2009)
k_m	m s^{-1}	$0.77 \cdot 10^{-3}$	Ortiz et al. (2005)	t^-	-	0.33	Gao et al. (2009)

4. Results and Discussion

4.1. Sensitivity analysis

Table 2 shows the ranking of parameter eigenvalues with respect to different time instants. For each instant, the parameters' impact on the ED system decreases from left to right in the table. Parameters V_{comp}, D_{CEM}, D_{AEM} and E_{el} are clearly influential on ED performance at different instants, which they are all selected.

Table 2: Parameters ordered from left to right based on eigenvalues for different time instants

t (h)	Parameters ordered based on eigenvalue magnitude		
0.01	V_{comp}	E_{el}	D_{CEM}, D_{AEM}
0.28	E_{el}	V_{comp}	D_{CEM}, D_{AEM}
0.50	E_{el}	V_{comp}	D_{CEM}, D_{AEM}
0.83	D_{CEM}, D_{AEM}	E_{el}	V_{comp}
1.00	D_{CEM}, D_{AEM}	V_{comp}	E_{el}

Figure 2 illustrates the results of the proposed method with respect to the determination of upper and lower limits for the parameters of vector ε. Note that the results with respect to vector F are reported as an average for all time instants, i.e. $\bar{F}(\zeta)$ and $\bar{F}^{exp}$, with $\bar{F}(0)$= $\bar{F}^{exp}$. By initially selecting a range of [-5, 0.5] as $[\zeta_t^L, \zeta_t^U]$, it is observed that the diluate concentration and the current density approach closest to the experimental values around ζ=-3. It further appears that these two performance indicators are affected more intensely by the simultaneous variation of V_{comp}, D_{CEM}, D_{AEM} and E_{el} than the concentrate concentration. The latter remains very close to its experimental values, exhibiting very little sensitivity to changes. The upper and lower limits are determined within a range of [-3.5, -2.5], i.e. at ±16% from ζ=-3. The corresponding values for V_{comp}, D_{CEM}, D_{AEM} and E_{el} are shown in Table 3. Different ranges can also be considered and tested.

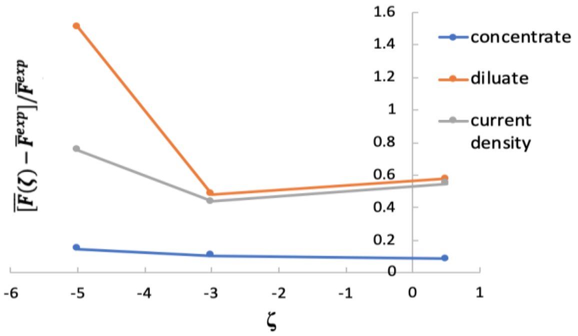


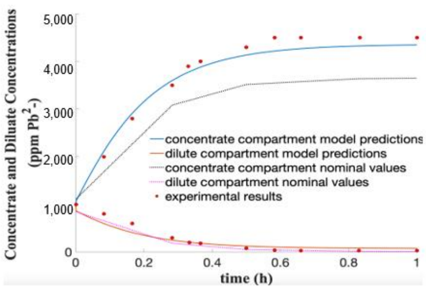
Figure 2: Combined effect of parameters on performance indicators

4.2. Parameter estimation and validation

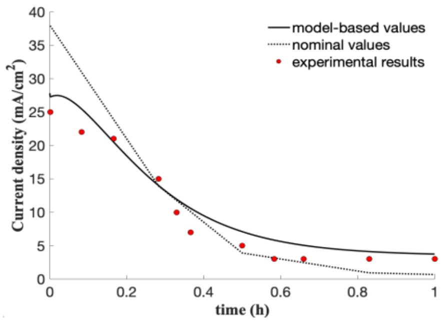
Using the limits of Table 3, the values for V_{comp}, D_{CEM}, D_{AEM} and E_{el} are obtained from parameter estimation, also in Table 3 (last column). The values used as nominal from Ortiz et al. (2005) are selected initially due to the lack of data. Despite similarities in the species, that system has different features and is used for a different purpose (desalination). Results in Table 3 indicate that the estimated parameter values are considerably different. The ED process investigated here requires a smaller compartment volume, and higher electrode potentials and diffusion coefficients. Smaller volumes and higher diffusion coefficients account for a faster ion contact to the membrane surfaces, while the electrode potential values are proportional to the system kinetics.

Table 3: Upper and lower limits and parameter estimation results

Parameter	Units	Nominal value (Ortiz et al., 2005)	Upper limit	Lower limit	Value from parameter estimation
V_{comp}	m^3	$33 \cdot 10^{-6}$	$40.8 \cdot 10^{-6}$	$3.34 \cdot 10^{-6}$	$3.56 \cdot 10^{-6}$
E_{el}	V	1.5	5.57	0.91	3.89
D_{CEM}, D_{AEM}	m^2s^{-1}	$3.28 \cdot 10^{-11}$	$19.1 \cdot 10^{-11}$	$1.37 \cdot 10^{-11}$	$18.5 \cdot 10^{-11}$



(a)



(b)

Figure 3: Model validation vs. experimental results of a) concentration profiles and b) the current density profile

The results of the concentration and current density profiles are verified against experimental data in Figure 3. The latter illustrates experimental and predicted results for all performance indicators, using both the nominal and estimated values of Table 3. It appears that the estimated values provide a very close match with the experimental results, as opposed to the nominal values in the cases of the concentrate and of the current density. This indicates that the values of Ortiz et al. (2005) are not suitable for the ED process case investigated in this work and highlights the necessity of the proposed approach. Regarding the ED results, the system's fast kinetics is noticeable. The lead's remaining concentrations (1- 2 mg/L) are estimated to be below the toxicity limits for irrigation water (5 mg/L, WHO/FAO, 2007). Also, the decreasing current density observed in Figure 3b is due to the increase of the diluate's resistance. The latter is likely due to a white precipitate formed on the AEM surface, possibly consisting of $Pb(OH)_2$, based on experimental observations reported by Gherasim et al. (2014).

5. Conclusions

The proposed approach unveiled the sensitivity of each one of 9 parameters with respect to ED performance indicators. It was shown that only 4 of them are necessary for parameter estimation, which the use of additional parameters would only complicate the estimation problem. The sensitivity analysis results are then used to determine the upper and lower limits in parameter estimation. The narrow range indicated by these limits enabled very good fit of the performance indicators with respect to experimental results from literature. Apart from its common use for desalination purposes, ED can provide a promising solution for wastewater treatment, applicable to the removal of several different types of heavy metal ions. In the present case, the concentration of lead ions is reduced to levels acceptable for plant irrigation. From the total of 140 g of lead entering the ED process, 139.72 g are removed, indicating a reduction of 99.8 % in what is released to the environment.

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