



Impedance spectroscopy and membrane potential analysis of microfiltration membranes. The influence of surface fractality

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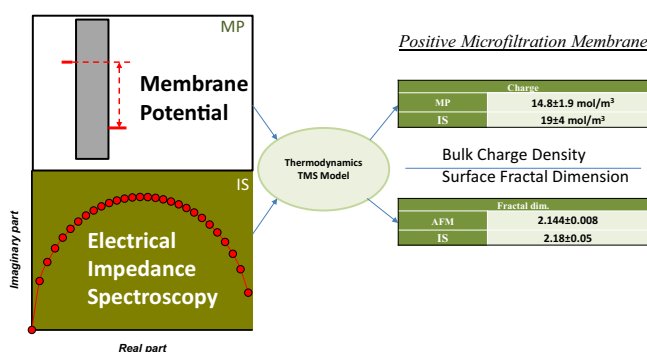
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HIGHLIGHTS

- The transport properties of a charged microfiltration membrane were studied.
- Membrane potential (MP) allowed a calculation of the transport numbers.
- Impedance Spectroscopy (IS) allowed to get electric conductivity inside the pores.
- Similar electrical proper charge was obtained from MP and IS methods.
- The roughness fractal dimension of the membrane surface was correlated with IS results.

GRAPHICAL ABSTRACT



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ABSTRACT

In this work, the proper charge density of a microfiltration membrane has been determined by using two different methods. Firstly, the ionic transport of a KCl solution has been investigated by simultaneous measurements of saline flux and membrane potential (MP) resulting from a concentration gradient through the membrane. A simple model, including all the relevant contributions to the global electrical potential drop, allowed a calculation of transport numbers and membrane charge density.

The response of ions inside the membrane to an oscillating electrical potential has been analyzed by impedance spectroscopy (or electrical impedance spectroscopy EIS). A quite simple experimental EIS design allowed, by taking into account MP measurements too, an easy assignment of an equivalent circuit. After a careful analysis of EIS results, it was possible to evaluate the electrical conductivity inside the pores and the charge density. Both were found to be quite similar to the values obtained from MP alone. This agreement of EIS results with the MP ones, that are much simpler to deal with, confirms the accuracy of EIS to study the electrical properties of microfiltration membranes.

The influence of electrode roughness and, in our EIS cell, the membrane roughness, on the constant phase element (CPE) of the equivalent circuit has been proved. Within this frame, the roughness fractal dimension of the membrane surface could be determined from EIS measurements. It resulted in fair agreement with the atomic force microscopy (AFM) determination.

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Nomenclature

Symbol list

A	membrane area exposed to flux
$(a_j)'$	activity at the corresponding interface offside the membrane of the j th species
$(\bar{a}_j)'$	activity at the corresponding interface inside the membrane of the j th species
(a_{\pm})	average activity of the electrolyte
$(\bar{a}_{\pm})'$	average activity of the electrolyte at the corresponding interface offside the membrane
$(\bar{a}_{-})'$	activity at the corresponding interface inside the membrane of anion.
C_{pore}	Capacitance of the solution inside the pores
C	salt concentration
C'	salt concentration at the corresponding interface offside the membrane
C_j	concentration of ion j outside the membrane
\bar{C}_j	concentration of ion j inside the membrane
D_d	diffusivities through the diffusion layers
\bar{D}_m	membrane diffusion coefficient
D_f	fractal dimension
d_p	pore diameter
E_{cell}	cell potential
E_m	electric potential drop
E'_m	proper membrane potential
\bar{E}_{diff}	diffusion potential inside the pores
E_{lj}	liquid junction potential
E_{Nernst}	Nernstian contribution potential.
F	Faraday constant
G_{pore}	conductance inside the pores
I, I', II', II	successive interfaces encountered along the membrane system
j	saline flux; Imaginary number $\sqrt{-1}$
j_0	initial flux
K	constant in Eq. (25)
L	channel length
n	exponent relating impedance of CPE (Eqs. (22) and (23))
n'	slope of the function $\gamma(\sigma)$ in a double logarithmic scale
P_{ms}	permeability
Q	constant (measured in $F \cdot s^{n-1}$) of Eq. (23)
R	universal constant of gases
R_{pore}	resistance of the solution inside the pores
$r_{j,Stokes}$	Stokes radius of the j th species
r_{pore}	pore radius
T	temperature
t	time
t_j	transport numbers outside the membrane of the j th species
\bar{t}_j	transport numbers inside the membrane of the j th species
U	mobility outside the membrane
\bar{U}	mobility inside the membrane

V	volume of high and low concentration compartments (assumed equal to each other)
v	average tangential speed.
x	coordinate
X_m	proper membrane charge
Z	impedance
Z'	real component of the system impedance
Z''	imaginary component of the system impedance
z_j	ion valence of the j th species

Greeks letters

α	fraction of the pore section defined in Eq. (18)
δ	diffusion layer thicknesses
$\Delta W'_{j,Born}$	free energy difference due to Born effects
$\Delta W'_{j,im}$	free energy difference due to image
ΔX	membrane thickness
$\Delta \Psi'$	Donnan's potential at the corresponding interface
$\Delta \Psi$	normalized Donnan's potential
ε	permittivity
ε_0	vacuum permittivity
ϕ_j	steric factor of the ion j
γ_j	activity coefficient of de ion j outside the membrane
$\bar{\gamma}_j$	ionic activity coefficient inside the membrane
γ_{\pm}	average activity coefficient of the electrolyte
$\gamma(\sigma)$	power spectra density function
η	viscosity of the solution
κ	conductivity
κ_{total}	total conductivity
μ_j	chemical potential of the j th species
$(\mu_j)'$	chemical potential at the corresponding interface outside the membrane of the j th species
$(\bar{\mu}_j)'$	chemical potential at the corresponding interface inside the membrane of the j th species
θ	membrane porosity
ρ	density of solution.
ω	angular frequency
ξ	sign (+1 or -1) of the membrane charge

Sub indexes

bulk	outside the membrane
high	high concentrations side in MP measurements
j	numeral/counter of species
low	low concentrations side in MP measurements
m	membrane
pore	pore
surface	surface
\pm	average
+	cation
-	anion

1. Introduction

The charge of a membrane controls its separation properties especially when dealing with electrolytes or any other charged species (Pinelo et al., 2013; Schaep et al., 1998; Tanninen et al., 2007). This is particularly true for ion exchange membranes. The charge, or its volume or surface density, and its sign play a key role in electrodialysis, with only secondary role for pore sizes, and its selectivity (Mikhaylin and Bazinet, 2016). In many other situations, the charge of the membrane, but also its dielectric properties, play

a significant enough role as to justify a careful investigation of these properties. This would help to predict the adequacy of a membrane for the desired application requirements (Xu, 2005).

Many experimental methods can be used to study the transport properties with proper charge or showing a certain charge when immersed in a charged solution, due for example to adsorption. Among others, we can mention: ionic permeability, streaming potential, streaming current, membrane potential, impedance spectroscopy, etc. (Benavente and Fernandez-Pineda, 1985; Hernandez et al., 1995; Lakshminarayanaiah, 1965; Martínez et al., 2002).

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