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Continuous dialysis of selected salts of sulphuric acid

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Abstract. The transport of selected salts of sulphuric acid (cobalt, copper, iron(II), manganese, nickel and zinc sulphate) through an anion-exchange membrane Neosepta-AFN was investigated in a counter-current continuous dialyzer at various salt concentrations and volumetric liquid flow rates. The basic transport characteristics – the rejection coefficient of salt and the permeability of the membrane – were calculated from measurements at steady state. The salt concentration in model mixtures was changed in the limits from 0.1 to 1.0 kmol m^{-3} and the volumetric liquid flow rate of the inlet streams was in the limits from 8×10^{-9} to 24×10^{-9} m^3 s^{-1}. Under the experimental conditions given, the rejection coefficient of salts tested was in the range from 65% to 94%. The lowest values were obtained for iron(II) sulphate, while the highest for copper sulphate. The maximum rejection of salt was reached at the highest volumetric liquid flow rate and the highest salt concentration in the feed. The permeability (P_A) of the Neosepta-AFN membrane for the individual salts was in the range from 0.49×10^{-7} m s^{-1} to 1.8×10^{-7} m s^{-1} and it can be described by the following series: $P_{FeSO_4}<P_{NiSO_4}<P_{ZnSO_4}<P_{CoSO_4}<P_{MnSO_4}<P_{CuSO_4}$. The permeability of the membrane was strongly affected by the salt concentration in the feed – it decreased with an increasing salt concentration.

Keywords: diffusion dialysis; anion-exchange membrane; continuous dialyzer; salt of sulphuric acid; permeability of membrane.

1. Introduction

Diffusion dialysis is one of the modern separation processes using a semi-permeable membrane – its considerable advantages are low energy consumption and a simple construction of the equipment used. Diffusion dialysis is often used to recover acids from mixtures containing acids and their salts. This separation is based on the fact that acids permeate well through the membrane, while salts are less or more rejected.

Oh *et al.* (2000) studied the effects of metal ions on diffusion dialysis of inorganic acids using a continuous dialyzer. Xu and Yang (2001, 2003) dealt with sulfuric acid recovery from titanium white waste liquor and the recovery of mixed acid ($HF+HNO_3$) from the titanium spent leaching solution using diffusion dialysis with a new series of anion-exchange membranes. In another paper, Xu and Yang (2004) aimed at simultaneous recovery of sulfuric acid and nickel from electrolysis spent liquor of relatively low acid concentration. Diffusion dialysis using an anion-exchange membrane was used to recover H_2SO_4 from waste sulfuric acid solutions in the diamond manufacturing process (Jeong *et al.* 2005). Xu *et al.* (2009a) studied the recovery of sulphuric acid from the waste anodic aluminum oxidation solution containing Al and Cu ions using the diffusion dialysis process by a

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series of batch, dynamic and pilot-scale tests. In the following study (Xu *et al.* 2009b), they employed the diffusion dialysis technique using anion-exchange membrane for recovering hydrochloric acid from the waste acid solution which contained Fe and Zn ions. Palatý and Žáková (2004a,b) investigated the separation of H_2SO_4+$CuSO_4$ and H_2SO_4+$ZnSO_4$ mixtures by diffusion dialysis in a two-compartment mixed cell with an anion-exchange membrane Neosepta-AFN. In another papers (Palatý and Žáková 2006, 2007, 2009), the same authors dealt with the separation of mixtures of HCl+$ZnCl_2$, HCl+$NiCl_2$ and HCl+$FeCl_2$ using the same apparatus. The results obtained therein showed that the Neosepta-AFN membrane can be considered a good separator for the mixtures mentioned. Besides the investigation of the separation of multicomponent inorganic mixtures by diffusion dialysis, attention has also been paid to the separation of mixtures containing organic acids and their salts (Narebska and Staniszewski 1997, 2008) and modeling the dialysis process (Bendová *et al.* 2009, Palatý *et al.* 2006, Yeh 2009, Yeh and Chang 2005, Yeh and Hsieh 2003).

In the case of diffusion dialysis, which uses an anion-exchange membrane for separation of electrolytes, the majority of cations is rejected, while the transport of anions is facilitated. Nevertheless, some penetration of cations through the membrane can be observed. However, the transport of single salts through the membrane has never been studied in detail. The aim of this paper is to obtain both the basic and quantitative information on continuous dialysis of selected salts of H_2SO_4, as these data are reference data for dialysis of mixtures containing sulphuric acid and its salt.

2. Theory

In order to study diffusion dialysis, a counter-current continuous dialyzer with two identical compartments can be used – its scheme is shown in Fig. 1. If the concentration of component A (salt) in compartment I is higher than that in compartment II, then mass transfer from compartment I to compartment II exists. For a preliminary evaluation of continuous dialysis, the rejection coefficient of salt, R_A, can be used

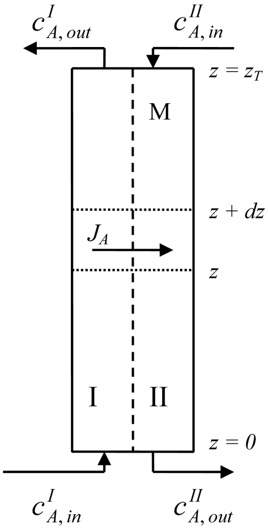

Fig. 1 Scheme of continuous dialyzer: M - membrane, I, II - compartments

$$R_A = \left(1 - \frac{\dot{V}_{out}^{II} c_{A,out}^{II}}{\dot{V}_{in}^{I} c_{A,in}^{I}}\right) \times 100 \tag{1}$$

where $\dot{V}$ is the volumetric flow rate of liquid, c_A is the molar concentration of component A, superscripts *I* and *II* mean compartment I and compartment II and subscripts *in* and *out* mean inlet and outlet.

Moreover, the transport of component A through the membrane can be characterized by the permeability coefficient of the membrane, P_A. At steady state and assuming the plug flow, the balance of component A over the differential volume of compartment I ($j = I$) and compartment II ($j = II$) can be written as

$$Su^j c_A^j\big|_z = Su^j c_A^j\big|_{z+dz} + J_A \frac{A}{z_T} dz\,, \quad j = I, II \tag{2}$$

In Eq. (2), S is the cross-section of the compartment, u is the flow rate, J_A is the flux of component A, A is the membrane area, z_T is the total height of the compartment and z is the length coordinate. Using the definition of derivation and further arrangement, one can obtain Eqs. (3) describing the concentration profiles of component A in compartments I and II

$$\frac{\mathrm{d}c_A^j}{\mathrm{d}z} = -\frac{1}{Su^j}\frac{A}{z_T}J_A - \frac{c_A^j}{u^j}\frac{\mathrm{d}u^j}{\mathrm{d}z}, \quad j = I, II \tag{3}$$

The initial conditions for Eqs. (3) are

$$\mathrm{z} = 0, \quad c_A^I = c_{A,in}^I, \quad c_A^{II} = c_{A,out}^{II} \tag{4}$$

The flux of component A through the membrane is interrelated with the permeability of the membrane by the following equation

$$J_A = P_A(c_{Ai}^I - c_{Ai}^{II}) \tag{5}$$

where c_{Ai}^I and c_{Ai}^{II} are the concentrations in liquid at the solution/membrane interfaces. Due to the low volumetric flow rates of liquid in both the compartments of the continuous dialyzer, besides the transport of component A through the membrane also the transport of component A through liquid films (Eqs. (6) and (7)) on both sides of the membrane must be solved

$$J_A = k_L^I(c_A^I - c_{Ai}^I) \tag{6}$$

$$J_A = k_L^{II}(c_{Ai}^{II} - c_A^{II}) \tag{7}$$

In Eqs. (6) and (7), k_L^I and k_L^{II} are the liquid mass transfer coefficients, which can be estimated from the following equation

$$Sh = CRe^{1/2}Sc^{1/3} \tag{8}$$

If the concentrations of component A in the streams entering and leaving the dialyzer on both sides of the membrane are known, it is possible to numerically integrate the set of Eqs. (3) with the initial conditions (4). If this procedure is combined with a suitable optimizing procedure, then the basic transport characteristic of the membrane/solution system, *i.e.*, the permeability of the membrane, P_A, can be calculated.

3. Experimental

Dialysis of selected salts of sulphuric acid was investigated in a two-compartment counter-current continuous dialyzer. The experimental set-up used is presented elsewhere (Bendová *et al.* 2009). In this study, an anion-exchange membrane Neosepta-AFN was used. The membrane area was 3.31×10^{-2} m^2. The dimensions of each compartment of dialyzer used were 0.92 m×0.036 m×0.0011 m (height× width×thickness). In both the compartments, there were net-type spacers made of PVC also acting as turbulence promoters. The dialyzer was placed into a box, where the temperature was kept constant.

In the experiments, aqueous solutions of salts of sulphuric acid were used. The feed entered the bottom of compartment I, while distilled water entered the top of compartment II. In all the experiments, liquid flow rate of the feed was always equal to that of water ($\dot{V}_{in}^{I} = \dot{V}_{in}^{II}$). After the steady state has been reached (a time period from 2 to 10 hours), three samples were taken from each stream leaving the dialyzer and mass flow rates of both these streams were determined. Before each experiment, also mass flow rates of both feed and water were measured. All the experiments were carried out at a temperature of 25±0.5°C.

The solutions of $CoSO_4$, $CuSO_4$, $FeSO_4$, $MnSO_4$, $NiSO_4$ and $ZnSO_4$ were tested. The salt concentration of model mixtures was changed in the limits from 0.1 to 1.0 kmol m^{-3}. The volumetric liquid flow rate of the inlet streams was in the limits from 8×10^{-9} to 24×10^{-9} m^3 s^{-1} (*i.e.*, liquid flow rate was in the limits from 2.0×10^{-4} to 6.1×10^{-4} m s^{-1}). The concentration of Cu^{2+}, Co^{2+}, Mn^{2+}, Ni^{2+} and Zn^{2+} ions was determined by titration with 0.02 M EDTA, while the concentration of Fe^{2+} ions was determined by titration with a standard $KMnO_4$ solution.

4. Data treatment and discussion

4.1 Rejection coefficient of salt, R_A

The rejection coefficients of salt, R_A, were calculated from Eq. (1). Under the experimental conditions given, the rejection coefficients are found to be in the range from 65% to 88% for $CuSO_4$, from 69% to 91% for $MnSO_4$, from 68% to 92% for $CoSO_4$, from 70% to 92% for $ZnSO_4$, from 72% to 93% for $NiSO_4$ and from 75% to 94% for $FeSO_4$. In all the cases, the experiments prove that rejection coefficients always increase with an increasing volumetric liquid flow rate and the salt concentration in the feed. The maximum of rejection coefficients can be found at the end of the volumetric liquid flow rate and the salt concentration intervals.

For illustration, Figs. 2 and 3 present the dependences of rejection coefficients for copper sulphate (Fig. 2) and nickel sulphate (Fig. 3) upon the volumetric liquid flow rate. The salt concentration in the feed is a parameter of the individual lines. The corresponding dependences for the remaining salts are very similar.

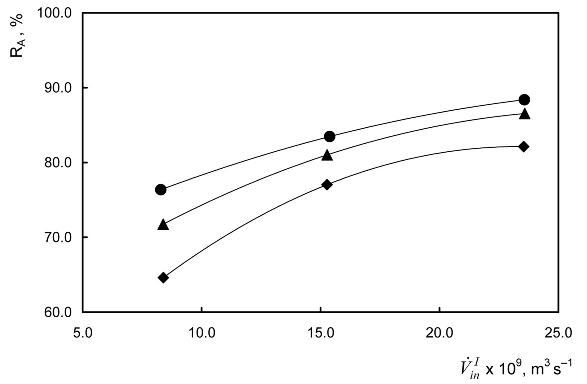

Fig. 2 Dependence of rejection coefficient upon volumetric liquid flow rate for CuSO4. $c^{I}_{A,\,in}$ [kmol m–3]: ◆-0.1, ▲-0.5, ●-1.0

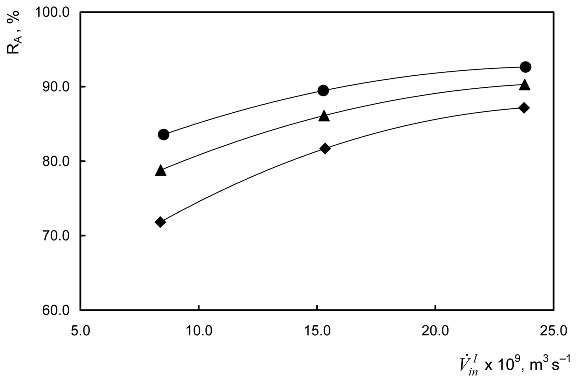

Fig. 3 Dependence of rejection coefficient upon volumetric liquid flow rate for $NiSO_4$: $c^{I}_{A,\,in}$ [kmol m^{-3}]: ◆-0.1, ▲-0.5, ●-1.0

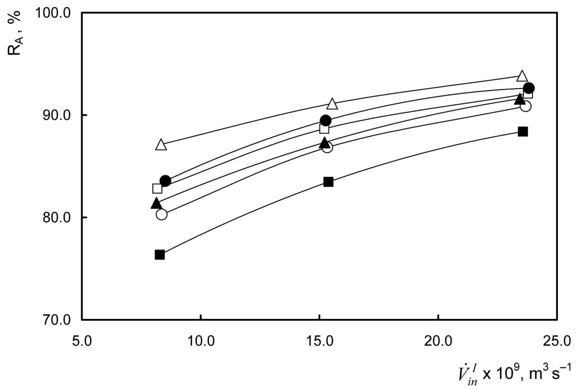

Fig. 4 Dependence of rejection coefficient upon volumetric liquid flow rate for salt concentration of 1.0 kmol m^{-3}: ■-$CuSO_4$, ○-$MnSO_4$, ▲-$CoSO_4$, □-$ZnSO_4$, ●-$NiSO_4$, △-$FeSO_4$

For the highest salt concentration in the feed, the rejection coefficients of the membrane for all salts are compared in Fig. 4. A detailed inspection of these dependences reveals that the anion-exchange membrane Neosepta-AFN exhibits the lowest rejection for copper sulphate, while the highest one for $FeSO_4$. Moreover, all rejection coefficients follow the series: $R_{\mathrm{CuSO_4}}<R_{\mathrm{MnSO_4}}<R_{\mathrm{CoSO_4}}<R_{\mathrm{ZnSO_4}}<R_{\mathrm{NiSO_4}}<R_{\mathrm{FeSO_4}}$.

4.2. Permeability of membrane, P_A

The permeability of the membrane was determined by the numerical integration of the set of Eqs. (3), where J_A is expressed by Eq. (5), using the Runge-Kutta 4th order method with the integration step $h = 0.001$ m. First, the set of Eqs. (3) was integrated in the positive direction of the coordinate z, so that the calculated values of the concentrations $c^{I,calc}_{A,out}$ and $c^{II,calc}_{A,in}$ were obtained. Then the integration in the opposite direction was applied and the calculated values of the concentrations $c^{I,calc}_{A,in}$ and $c^{II,calc}_{A,out}$ were determined – in this case, the first term on the right-hand side of Eq. (3) is positive and the initial conditions are: $z = z_T$, $c^{I}_{A} = c^{I}_{A,out}$, $c^{II}_{A} = c^{II}_{A,in} = 0$. In each integration step, it was necessary to calculate the concentration of acid in liquid at the solution/membrane interface by solving Eqs. (5)-(7). In order to estimate mass transfer coefficients, kinematic viscosity and diffusivity of acid were needed. As viscosity of solution and diffusivity of acid depend upon its concentration, it was necessary to determine these parameters in both liquid films by an iterative procedure using literature data (Landolt-Börnstein 1969). Using an optimizing procedure (Golden Section Search) such values of P_A were searched, at which the objective function (9) reached a minimum

$$F(P_A) = (c_{A,out}^{I,\exp} - c_{A,out}^{I,calc})^2 + (c_{A,in}^{II,\exp} - c_{A,in}^{II,calc})^2 + (c_{A,in}^{I,\exp} - c_{A,in}^{I,calc})^2 + (c_{A,out}^{II,\exp} - c_{A,out}^{II,calc})^2 \qquad (9)$$

The procedure for obtaining permeability of the membrane given above needs the value of the constant C in Eq. (8). Its determination was based on the following considerations: Permeability of the membrane is a membrane/solution parameter, which is not affected by the flow of liquid. This means that the values of the permeability obtained at various liquid flow rates must not be highly different from each other. For this reason, we defined a criterion $\overline{S}$ as a sum of variances of P_A at a constant $c_{A,in}^{I}$ and searched for its minimum (for details, see *e.g.*, Bendová *et al.* 2009).

The obtained values of constant C are summarized in Table 1 together with the values of the permeability of the membrane for selected salts of sulphuric acid. The values of P_A are also presented graphically in Fig. 5. From this figure, where the permeability of the membrane is plotted against the salt concentration in the feed, it is evident that this membrane characteristic is strongly

Table 1 Constants C in Eq. (8) and permeability of Neosepta-AFN membrane

Salt	$c_{A,in}^{I}$ [kmol m^{-3}]	C	$P_A \times 10^7$ [m s^{-1}]
$CoSO_4$	0.1-1.0	0.74	0.687-1.34
$CuSO_4$	0.1-1.0	0.70	1.03-1.81
$FeSO_4$	0.1-1.0	0.39	0.492-0.990
$MnSO_4$	0.1-1.0	0.39	0.819-1.47
$NiSO_4$	0.1-1.0	1.21	0.597-1.12
$ZnSO_4$	0.1-1.0	0.43	0.635-1.46

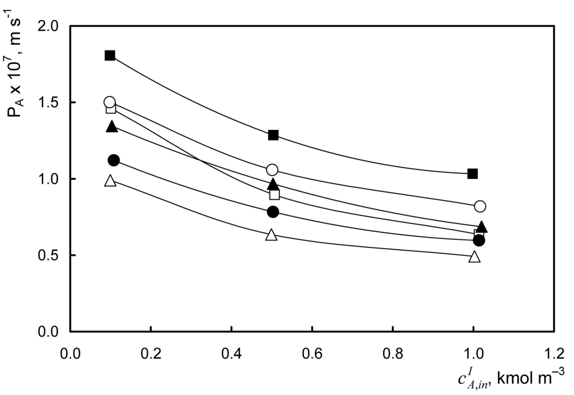

Fig. 5 Dependence of permeability of membrane upon salt concentration in feed: ■-$CuSO_4$, ○-$MnSO_4$, ▲-$CoSO_4$, □-$ZnSO_4$, ●-$NiSO_4$, △-$FeSO_4$

affected by the salt concentration in the feed – it decreases with an increasing salt concentration. From Table 1, where the lower and upper limits of P_A are presented, it can be seen that the permeability of the Neosepta-AFN membrane for the selected salts lies in the range from 0.49×10^{-7} m s^{-1} to 1.8×10^{-7} m s^{-1}. The membrane studied exhibits the highest permeability for copper sulphate, while the lowest one for iron(II) sulphate. Based on the graphical presentation in Fig. 5 it can be stated that the permeability of the membrane obeys the following series: $P_{\mathrm{FeSO_4}}<P_{\mathrm{NiSO_4}}<P_{\mathrm{ZnSO_4}}<P_{\mathrm{CoSO_4}}<P_{\mathrm{MnSO_4}}<P_{\mathrm{CuSO_4}}$. This is in accordance with findings concerning the rejection coefficients. The comparison with the permeability of the Neosepta-AFN membrane for sulphuric acid (Palatý *et al.* 2006) shows that the pemeability of the membrane for the selected salts of sulphuric acid is about one order of magnitude lower than that for H_2SO_4.

The Neosepta-AFN membrane is an anion-exchange membrane, which has in its structure positively charged sites, so that the transport of counter-ions through the membrane is facilitated while co-ions are rejected. The transport of the salts through the membrane could be affected by the presence of negatively charged complexes and non-dissociated forms. The concentration of these species can be found by solving the set of equations describing ionic equilibria, the electroneutrality condition and balance of the metal. Examples are given elsewhere (Palatý and Žáková 2004a,b). Using the complexity constants taken from the literature (Kotrlý and Šůcha 1985), the set of these equations was solved by the Newton-Raphson method for low concentration and zero ionic strength (the reason was that limited data have been published). The sum of concentrations of the negative charged complexes and non-dissociated forms for the individual salts, c^-, corresponds to the following series: $c^-_{\mathrm{FeSO_4}}<c^-_{\mathrm{MnSO_4}}<c^-_{\mathrm{NiSO_4}}<c^-_{\mathrm{ZnSO_4}}<c^-_{\mathrm{CuSO_4}}\approx c^-_{\mathrm{CoSO_4}}$ which differs from the series of the permeability found experimentally. Moreover, we did not succeed in the correlation of the permeability coefficient with neither the molar mass ($M_{\mathrm{MnSO_4}}<M_{\mathrm{FeSO_4}}<M_{\mathrm{NiSO_4}}<M_{\mathrm{CoSO_4}}<M_{\mathrm{CuSO_4}}<M_{\mathrm{ZnSO_4}}$) nor ionic radius ($r_{\mathrm{Fe}^{2+}}<r_{\mathrm{Co}^{2+}}<r_{\mathrm{Mn}^{2+}}<r_{\mathrm{Cu}^{2+}}<r_{\mathrm{Fe}^{2+}}<r_{\mathrm{Zn}^{2+}}$). The transport of the salts through the membrane Neosepta-AFN is probably affected by a combination of all the effects mentioned.

5. Conclusion

Using a counter-current continuous dialyzer, the transport of selected salts of sulphuric acid (cobalt, copper, iron(II), manganese, nickel and zinc sulphate) through an anion-exchange membrane Neosepta-AFN was investigated at steady state. The dialysis process was characterized by the rejection coefficient of salt and the permeability of the membrane. The permeability was determined by the numerical integration of a set of differential equations, which describes the concentration profile of the salt in both compartments of the dialyzer, combined with an optimizing procedure. It was found that the rejection coefficients increased with an increasing volumetric liquid flow rate and the salt concentration in the feed. The lowest values were obtained for copper sulphate, while the highest for iron sulphate. The permeability coefficient of the membrane decreased with an increasing salt concentration in the feed. It follows the series: $P_{\mathrm{FeSO_4}}<P_{\mathrm{NiSO_4}}<P_{\mathrm{ZnSO_4}}<P_{\mathrm{CoSO_4}}<P_{\mathrm{MnSO_4}}<P_{\mathrm{CuSO_4}}$.

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Nomenclature

A	membrane area, m^2
C	constant in Eq. (8)
c	molar concentration, $\mathrm{kmol\ m}^{-3}$
c^-	sum of concentrations of negatively charged complexes and non-dissociated forms, $\mathrm{kmol\ m}^{-3}$
D	diffusivity, $\mathrm{m}^2\ \mathrm{s}^{-1}$
d_e	equivalent diameter, m
F	objective function, $\mathrm{kmol}^2\ \mathrm{m}^{-6}$
J	molar flux, $\mathrm{kmol\ m}^{-2}\ \mathrm{s}^{-1}$
h	integration step, m
k_L	liquid mass transfer coefficient, $\mathrm{m\ s}^{-1}$
M	molar mass, $\mathrm{kg\ kmol}^{-1}$
P	permeability of membrane, $\mathrm{m\ s}^{-1}$
R	rejection coefficient, %
r	ionic radius, m
Re	$\left(=\frac{ud_e}{\nu}\right)$ Reynolds number
S	cross-section of compartment, m^2
$\bar{S}$	sum of variances, $\mathrm{m}^2\ \mathrm{s}^{-2}$
Sc	$\left(=\frac{\nu}{D}\right)$ Schmidt number
Sh	$\left(=\frac{kd_e}{D}\right)$ Sherwood number
u	liquid flow rate, $\mathrm{m\ s}^{-1}$
$\dot{V}$	volumetric flow rate, $\mathrm{m}^3\ \mathrm{s}^{-1}$
z	length coordinate, m
z_T	height of compartment, m
ν	kinematic viscosity, $\mathrm{m}^2\ \mathrm{s}^{-1}$

Superscripts

calc	calculated value
exp	experimental value
I	referred to compartment I
II	referred to compartment II

Subscripts

A	referred to component A (salt)
Co^{2+}	referred to Co^{2+} ions
$CoSO_4$	referred to cobalt sulphate
Cu^{2+}	referred to Cu^{2+} ions
$CuSO_4$	referred to copper sulphate
Fe^{2+}	referred to Fe^{2+} ions
$FeSO_4$	referred to iron (II) sulphate
i	referred to solution/membrane interface
in	inlet
M	referred to membrane
Mn^{2+}	referred to Mn^{2+} ions
$MnSO_4$	referred to manganese sulphate
Ni^{2+}	referred to Ni^{2+} ions
$NiSO_4$	referred to nickel sulphate
out	outlet
Zn^{2+}	referred to Zn^{2+} ions
$ZnSO_4$	referred to zinc sulphate