



Simulating the separation performance of nanofiltration membranes for salt lake brine

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ABSTRACT

An improved semi-empirical model used to predict the separation performance of salt lake brine by nanofiltration (NF) membrane was obtained. The transmission of the eight single salts is in the order of NaCl > LiCl > KCl > MgCl₂ > Li₂SO₄ > K₂SO₄ > Na₂SO₄ > MgSO₄. In the double salts permeation experiment, the regulation coefficient was revised by introducing an adjustment equation A in this model, because the calculated values deviated from the experimental data. The modified semi-empirical model and the experimental data under different concentration conditions has a good consistency. It shows that the semi-empirical model can be used to predict the transmission of salt lake brine.

Keywords: Nanofiltration; Membrane separation; Salt lake brine; Model; Regulation coefficient

1. Introduction

Nanofiltration (NF) [1], a press-driven process, which can be used to separate multivalent ions and small organic molecular due to its charges and nano-pores of membrane surface, thus it has been used in various areas, such as desalination of seawater and brine, drinking water production. In the latest few years, more attention was paid to the separation performance of an NF membrane for single salt [2,3] and mixed salts solution such as three and more than three kinds of ions [4–8], but it is still difficult to evaluate the separation performance of an NF membrane for the mixed salts solution.

Wang et al. [9,10] developed two models to evaluate the separation performance of NF membrane for the mixed salts solution, more than three species of ions, without and with multivalent cations (Mg²⁺, Ca²⁺), respectively. In the two models, the observed transmission of an ion was applied to express the separation performance of NF membranes

(ESNA 1-LF, ESNA 1, and LES 90) in the mixed salts solution, which had a relationship with the total concentration, the equivalent fraction and the species of each ion in the mixed salts solution. There are four assumptions in the former model: (a) According to the electroneutrality principle [11], the total concentration of mixed salts is equal to that of cations (or anions) in the mixed salts solution. (b) The volume membrane charge is considered as an increasing empirical function with the feed salt concentration, which is independent of the salt type [12]. In the mixed salts solution, the effective fixed charge density of NF membrane is constant and has nothing to do with the component of mixed salts solution, if the total concentration of mixed salts does not change. (c) The mixed salts solution with n kinds of cations and m kinds of anions was equivalent to the mixed solution composed of n multiply m kinds of single salts. (d) The competitive effect between two cations (or anions) through NF membrane was considered and the competition

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coefficients of ions were obtained through the permeation experiments of NF membranes for some binary salts solutions. The agreement between the model evaluation results and the experimental data indicated that the model is suitable for evaluating the separation performance of ESNA 1-LF, ESNA 1, and LES 90 membranes for the mixed salts solution. The latter model for the mixed salts solution with Mg^{2+} and Ca^{2+} was developed base on the former one. In this model, a regulation coefficient was added into it because the effective fixed charge density of NF membrane will be changed due to the counter-ion adsorption of multivalent cations [13]. The competition coefficient is a function of total salt concentration and ion equivalent fraction only. The model evaluation result of the observed transmission of each ion in the mixed salts solution with Mg^{2+} and Ca^{2+} agreed quite well with the experimental data. The deviation between the model evaluation results and the experimental data of most ions was less than 10% for the case of ESNA 1, which of ESNA 1-LF and LES 90 membrane was less than 20%.

There are a variety of mineral resources in salt lake brine, such as Li, Mg, Na, and K. Lots of methods have been developed to obtain these mineral resources. Some researchers have made attempts to separate divalent ions from salt lake brine using NF membranes. Wen et al. [14] first showed the recovery of Li^+ from diluted salt lake brine using NF membrane, and the result showed that the separation factor (SF) of Li^+ over Mg^{2+} was about 3.5. Yang et al. [15] reported that the SF was 2.6 for simulated brine with an Mg/Li ratio of 24 at 1.0 MPa using a spiral-wound Desal DK membrane. Somrani et al. [16] showed that NF90 was better than the XLE membrane (a kind of low pressure reverse osmosis membrane of DOW FilmTec) in separating Mg^{2+} and Li^+ from diluted Tunisian salt lake brine. Sun et al. [17] reported that the rejection of Mg^{2+} was 65% and the SF was about 3.3 for the simulated West Taijiner brine with a Mg/Li ratio of 64 at 3.0 MPa using Desal DL-2540 model. However, there are no models have been developed to predict the separation performance of NF membrane in high concentration, multiple ions system, especially in the salt lake brine system. This is because ion association will affect ion concentration and equivalent fraction in solution, it is difficult to predict the factors affecting the competitive coefficient accurately. A large number of solution thermodynamic data and NF membrane model are needed to analyze the competitive coefficient. So, it is very necessary to develop a model including a theoretical model, empirical model, or semi-empirical model to predict the separation performance of NF membranes for salt lake brine.

In this paper, an improved model to evaluate the separation performance of NF membranes for salt lake brine was developed based on the work of Wang et al. [9,10]. In this model, the competition coefficient is a function of total salt concentration, ion equivalent fraction, and system composition. And a new regulation coefficient was obtained. Firstly, the permeation experiments of eight single salt solutions through NT201 membrane were carried out under different concentrations. NT201 is a suitable membrane used in salt lake brine separation process according to our preceding research [18]. Then, the model parameters were obtained from the permeation experiments of binary mixed salts solutions (Na^+ , K^+ , and Cl^- ; Na^+ , Li^+ , and Cl^- ; Na^+ , Cl^- , and

SO_4^{2-} ; Na^+ , Mg^{2+} , and Cl^- ; K^+ , Mg^{2+} , and Cl^- ; Li^+ , Mg^{2+} , and Cl^-). Finally, the model was verified with the permeation experiments of simulated salt lake brine with Na^+ , K^+ , Li^+ , Mg^{2+} , SO_4^{2-} , and Cl^- .

2. Model and methods

In this study, the separation performance of NF membrane is represented by the observed transmission (Tr_{obs}). The rejection of most salts by NF membrane is more than 50%, the Tr_{obs} will make the comparison between the calculated results and the experimental results more remarkable [9,10]. The operating pressure is high and the effect of concentration polarization is neglected, thus, the Tr_{obs} is approximately equal to the real one [19]. The transmission of cation (Tr_{obs,C_i}), anion (Tr_{obs,A_j}), and mixed salts ($Tr_{obs,salts}$) was defined as follows:

$$Tr_{obs,salts} = \frac{C_{p,salts}}{C_{b,salts}} = 1 - R_{obs,salts} \quad (1)$$

$$Tr_{obs,C_i} = \frac{C_{p,C_i}}{C_{b,C_i}} \quad (2)$$

$$Tr_{obs,A_j} = \frac{C_{p,A_j}}{C_{b,A_j}} \quad (3)$$

where $C_{p,salts}$ and $C_{b,salts}$ are the total concentrations of mixed salts in permeate and feed, respectively. C_{p,C_i} and C_{b,C_i} are the concentration of cation C_i in permeate and feed, respectively. C_{p,A_j} and C_{b,A_j} are the concentration of anion A_j in permeate and feed, respectively. $R_{obs,salts}$ is the observed rejection of mixed salts.

According to the model proposed in references [9,10], the mixed salts solution with n kinds of cations (C_1, C_2, \dots, C_n) and m kinds of anions (A_1, A_2, \dots, A_m) corresponds to the mixed solution composed of n multiply m kinds of single salts. All concentrations are expressed by equivalent concentration and the unit is mN, equivalent concentration means the gram-equivalent number of the solute in 1 L solution. The cation equivalent fraction (x_{b,C_i}) is defined as the ratio of cation C_i to total cations concentration. Similarly, the anion equivalent fraction (x_{b,A_j}) can be obtained.

In order to calculate the separation performance of NF membranes for mixed salts solution, the assumptions and some corresponding calculation equations (Eq. (4) and (5)) were made as reference [10] described:

The total salt transmission of mixed salt solution is equal to the sum of the transmissions of various single salts, as shown in Eq. (4). For mixed salt solutions containing Mg^{2+} , $Tr_{obs,salts}$ is obtained by Eq. (5), and regulation coefficient (Φ) is calculated by Eq. (6) based on reference [10].

$$Tr_{obs,salts} = \sum_{i=1}^n \sum_{j=1}^m x_{b,C_i} x_{b,A_j} Tr_{obs,C_i A_j} \quad (4)$$

$$\text{Tr}_{\text{obs,salts}} = \sum_{i=1}^n \sum_{j=1}^m x_{b,C_i} x_{b,A_j} \Phi_{C_i A_j} \text{Tr}_{\text{obs},C_i A_j} \quad (5)$$

$$\Phi_{\text{Na}A_j} = \Phi_{\text{K}A_j} = \Phi_{\text{Li}A_j} = A \times \left(1 + x_{b,\text{Mg}^{2+}} + \frac{\tilde{C}_{\text{Mg}^{2+}}}{C_{b,\text{salts}}} \right) \quad (6)$$

where x_b is the equivalent fraction, $\tilde{C}_{\text{Mg}^{2+}}$ is the empirical concentration parameters of Mg^{2+} , A is a coefficient which is the function of the concentration of salts.

At the beginning, the competition coefficient (β) of two ions is defined as the ratio of ion transmission. For cations and anions, the Eqs. (7) and (8) are shown, respectively. β depends on the types of ions and NF membrane, but is independent of the concentration of ions in mixed salt solution. Then revise it according to the experiment results.

$$\beta_{C_i} = \frac{\text{Tr}_{\text{obs},C_i}}{\text{Tr}_{\text{obs},C_1}} \quad (7)$$

$$\beta_{A_j} = \frac{\text{Tr}_{\text{obs},A_j}}{\text{Tr}_{\text{obs},A_1}} \quad (8)$$

where C_1 and A_1 are the selected benchmark cations and anions, respectively.

$\text{Tr}_{\text{obs},C_i}$ and $\text{Tr}_{\text{obs},A_j}$ in a mixed salt solution can be calculated by the following Eqs. (9) and (10):

$$\text{Tr}_{\text{obs},C_i} = \frac{\sum_{i=1}^n \sum_{j=1}^m x_{b,C_i} x_{b,A_j} \Phi_{C_i A_j} \text{Tr}_{\text{obs},C_i A_j}}{x_{b,C_1} \beta_{C_1} + \dots + x_{b,C_n} \beta_{C_n}} \beta_{C_i} \quad (9)$$

$$\text{Tr}_{\text{obs},A_j} = \frac{\sum_{i=1}^n \sum_{j=1}^m x_{b,C_i} x_{b,A_j} \Phi_{C_i A_j} \text{Tr}_{\text{obs},C_i A_j}}{x_{b,A_1} \beta_{A_1} + \dots + x_{b,A_m} \beta_{A_m}} \beta_{A_j} \quad (10)$$

3. Experimental section

3.1. Materials

A commercial NF membrane (NT201) with an active area of 0.00353 m² was purchased from Microdyn-Nadir Co., Ltd. (Xiamen, China). Potassium chloride (KCl, AR), potassium sulfate (K₂SO₄, AR), sodium sulfate (Na₂SO₄, AR), sodium chloride (NaCl, AR), magnesium sulfate (MgSO₄, AR), magnesium chloride hexahydrate (MgCl₂·6H₂O, AR), lithium sulfate monohydrate (Li₂SO₄·H₂O), and lithium

chloride (LiCl, AR) were purchased from Aladdin Co., Ltd. (Shanghai, China). The solution was prepared with pure water obtained from Milli-Q pure water/ultrapure water machine (Germany).

In this study, a prepared low-lithium brine of West Tajinar salt lake (simulated brine) in Qinghai Province was used as the simulation object, and its composition was shown in Table 1.

3.2. Experimental procedure

Firstly, the ion concentration is converted to equivalent concentration according to the composition of simulated brine in Table 1, and the total equivalent concentration is 10,400 mN. Because of the high concentration of the solution, which was necessary to be diluted when NF membrane separation was carried out. The dilution multiples were 10, 20, 40, and 80 times, respectively. The corresponding equivalent concentrations were 1,040; 520; 260; and 130 mN, respectively. The Tr_{obs} of single salts were obtained by permeation experiments.

Secondly, in order to calculate β and Φ , the double salts permeation experiments of (1) NaCl and KCl; (2) NaCl and LiCl; (3) NaCl and MgCl₂; (4) LiCl and MgCl₂; (5) KCl and MgCl₂; and (6) NaCl and Na₂SO₄ were carried out.

Finally, compared the calculated values with the experimental data of the simulated brine.

In this experiment, the operating pressure was 2.0 MPa, the temperature of the feed solution was 18°C ± 0.5°C and the pH value of the solution was not adjusted. The experiment flow chart diagram was shown in Fig. 1.

3.3. Analysis method

The concentration of cations was determined by atomic absorption spectrophotometer (AA-6300C, Shimadzu, Japan), and the concentration of anions was measured by anion chromatography (ICS-1100, Thermo Fisher Scientific, USA).

4. Results and discussion

4.1. Effect of concentration of single salts on observed transmission

The observed transmission of eight kinds of single salts at different concentrations is shown in Fig. 2. The curves in the figure are obtained by least square method. The observed transmission of all single salts except for MgSO₄ increased with the increase of single salts concentration, while the observed transmission of MgSO₄ changed slightly. For 1–1 type single salts, the observed transmission is obviously higher than that of salt solution with divalent ions, the observed transmission of MgSO₄ which is 2–2 type single salt is the lowest. The reason is that the radius of the hydrated

Table 1
Ion concentration of the simulated brine

Ions	Na ⁺	K ⁺	Li ⁺	Mg ²⁺	Cl ⁻	SO ₄ ²⁻
Mass concentration (g/L)	1.9515	1.0415	1.7227	120.0000	340.0718	35.1660

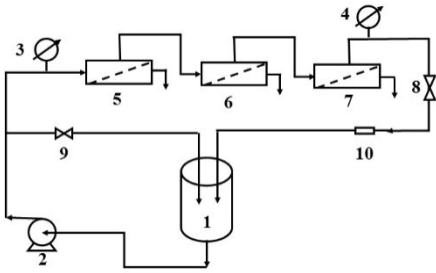


Fig. 1. Experiment flow chart diagram. (1) Feed tank, (2) Pump, (3, 4) Pressure gauge, (5–7) Membrane module, (8, 9) Valve, and (10) Flow meter.

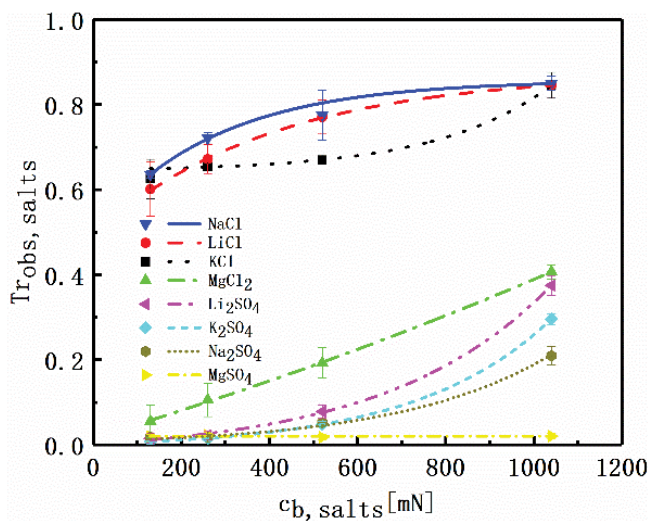


Fig. 2. Effects of concentration of single salts on observed transmission.

ion of divalent ion is larger [20]. The steric hindrance of NF membrane is remarkable, and there are both Donnan and dielectric exclusion effect which reject the divalent ion. Therefore, the observed transmission of single salts with divalent ions is lower than that of 1–1 type single salts. The sequence order of the observed transmission of the eight single salts is: NaCl > LiCl > KCl > MgCl₂ > Li₂SO₄ > K₂SO₄ > Na₂SO₄ > MgSO₄. The observed transmission of KCl is higher than that of NaCl and LiCl at lower concentration, the results are consistent with reference [21]. This can be explained by the electrostatic and steric-hindrance (ES) model [22], which considers both the charge and space effects. It can be concluded that the retention mechanism of NF membrane to inorganic salts depends on the charge effect at low concentration, while the charge effect and screening effect need to be considered at high concentration. At high concentration, the retention of 1–1 type inorganic salt tends to a certain value, which can be attributed to the size of ions.

4.2. Double salts permeation experiments without Mg²⁺

The double salts permeation experiments without Mg²⁺ results are shown in Fig. 3. The curves in the figure are obtained by the experimental curves of single salts. It is

obvious that the curves calculated by Eq. (4) of single salts data have deviated from the actual double salts observed transmission, and the calculated observed transmission of double salts is much lower than the real one. The reason for the deviation is that the competition coefficient between ions in high concentration solution is greatly influenced by the composition of solution and the concentration of total salt. But the calculated curve changes in a similar trend of the actual one. After obtaining the competition coefficient, the simulation calculation is carried out, and it is uniformly revised when simulating the real brine. The assumption of ion competition coefficient originally defined in the semi-empirical model is no longer valid. So a revised β is obtained, which is the function of total salt concentration, ion equivalent fraction and system composition:

$$\beta = f(C_{b,\text{salts}}, x_{b,\text{salts}}, \text{composing system}) \quad (11)$$

In high concentration solution, the properties of solution such as the interaction between ions and the association degree of different ions will change, so the composition of high concentration solution system becomes more complex [14]. A large number of solution thermodynamic data and NF membrane model are needed to analyze the competitive coefficient. Therefore, semi-empirical method is used to simulate the simulation.

4.3. Double salts permeation experiments with Mg²⁺

The double salts permeation experiments with Mg²⁺ results are shown in Fig. 4. The curves in the figure are calculated by Eq. (4). The experimental data have obvious divalent from the calculated permeation. Under different equivalent fractions, the experimental data of double salts permeation and the simulated values are in good agreement. With the increase of the Mg²⁺ concentration of mixed salts solution, the permeation of total salt decreased. This phenomenon is due to the change of surface charge properties of NF membrane after the adsorption of Mg²⁺ on the membrane surface [7,12,23–26]. Because of the high valence of Mg²⁺, the surface charge of NF membranes will be shielded after Mg²⁺ being adsorbed by NF membranes. Therefore, the effective charge of the membranes will be neutralized or changed. At the same time, due to the existence of Mg²⁺, the dielectric exclusion effect can also cause this phenomenon, which is an important factor [27–29]. According to the experimental data of Fig. 4a, the competition coefficient between Na⁺ and Mg²⁺ can be obtained. It is proved that the competition coefficient is a function of total salts concentration, ion equivalent fraction and system composition. The model assumes that the ion competition coefficient is a constant, and the ion competition coefficient in this study is shown in Table 2. Under high concentration conditions, the trend of total salt transmission is simulated by empirical Eq. (6) similar to real salts transmission, but there is a deviation between simulated and real salts transmission values.

4.4. Comparisons between simulated and experimental values

Under the condition of high concentration solution, the simulated values of total salts transmission without and with

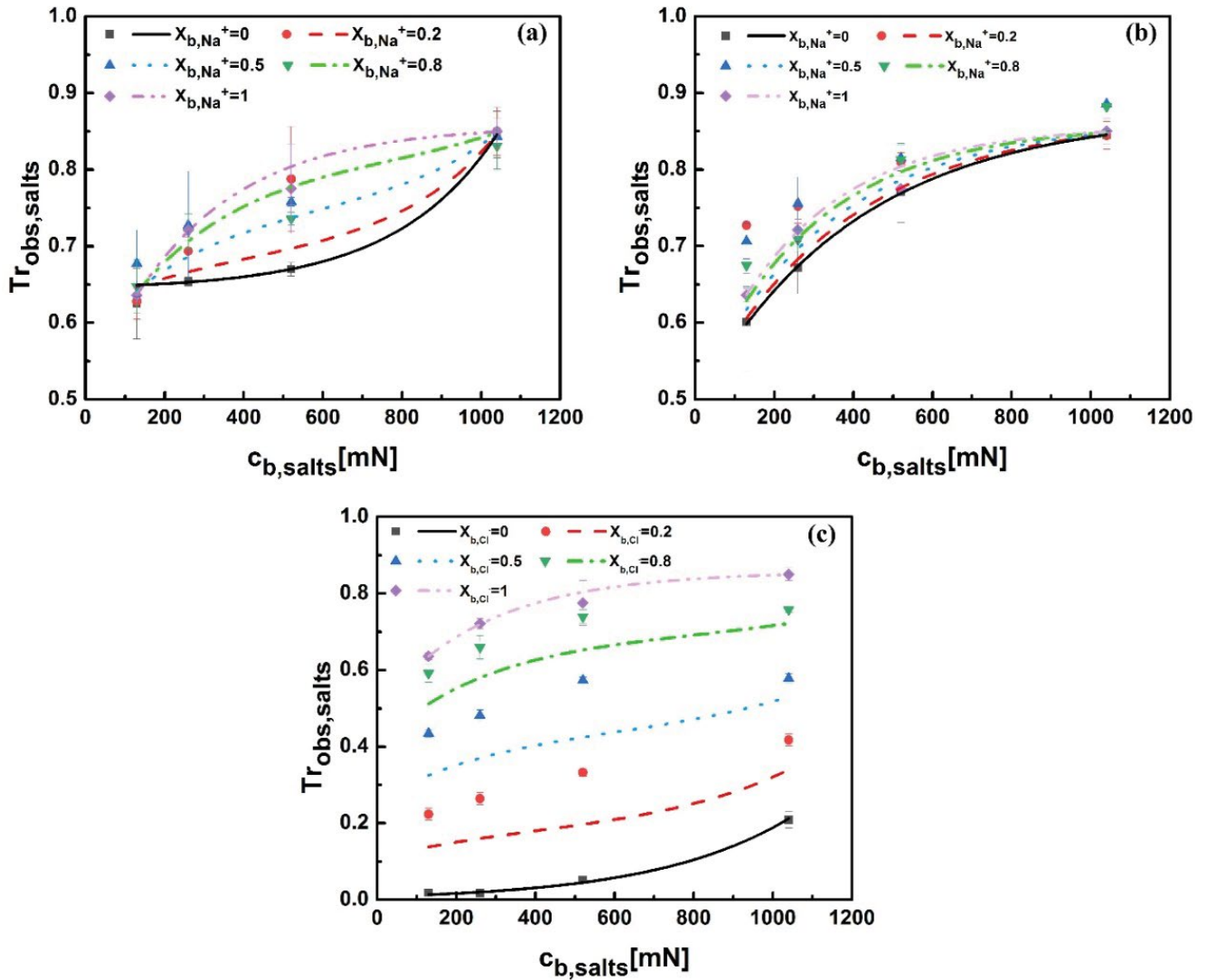


Fig. 3. Effects of equivalent concentration and fraction on observed transmission of (a) NaCl and KCl, (b) NaCl and LiCl, and (c) NaCl and Na_2SO_4 .

Mg^{2+} are deviated from the experimental values, therefore, through a large number of experiments, the semi-empirical adjustment equations are obtained as follows:

$$A = \left(-\frac{33436.56}{C_{b,salts}^2} + \frac{505.74}{C_{b,salts}} + 0.08333 \right) \quad (12)$$

So the new regulating coefficient is obtained:

$$\Phi_{NaA_j} = \Phi_{KA_j} = \Phi_{LiA_j} = \left(-\frac{33436.56}{C_{b,salts}^2} + \frac{505.74}{C_{b,salts}} + 0.08333 \right) \times \left(1 + x_{b,Mg^{2+}} + \frac{\tilde{C}_{Mg^{2+}}}{C_{b,salts}} \right) \quad (13)$$

The simulated salt lake brine was prepared according to the composition in Table 1. The separation performance

data were obtained by the experiment of simulated salt lake brine with dilution to 1,040; 520; 260; and 130 mN, respectively.

The comparison between the simulated and experimental values is shown in Fig. 5. It can be seen that under different concentration conditions, the deviation of results between the modified model and the experimental data is slightly, and there is a good consistency, while the deviation of results between the unmodified model and the experimental data is large. It shows that the modified semi-empirical model can be used to predict the transmissivity of brine in salt lakes. It is worth noting that with the increase of concentration, the change of regulation coefficients cannot be ignored.

Table 3 lists the calculated values (Mod), the experimental data (Exp), and the deviation (Dev). It can be seen that the deviation between the calculated values and the experimental data is less than 29%, most ions is less than 20%, which of K^+ and Li^+ is less than 15%. Only at 1,040 mN, the

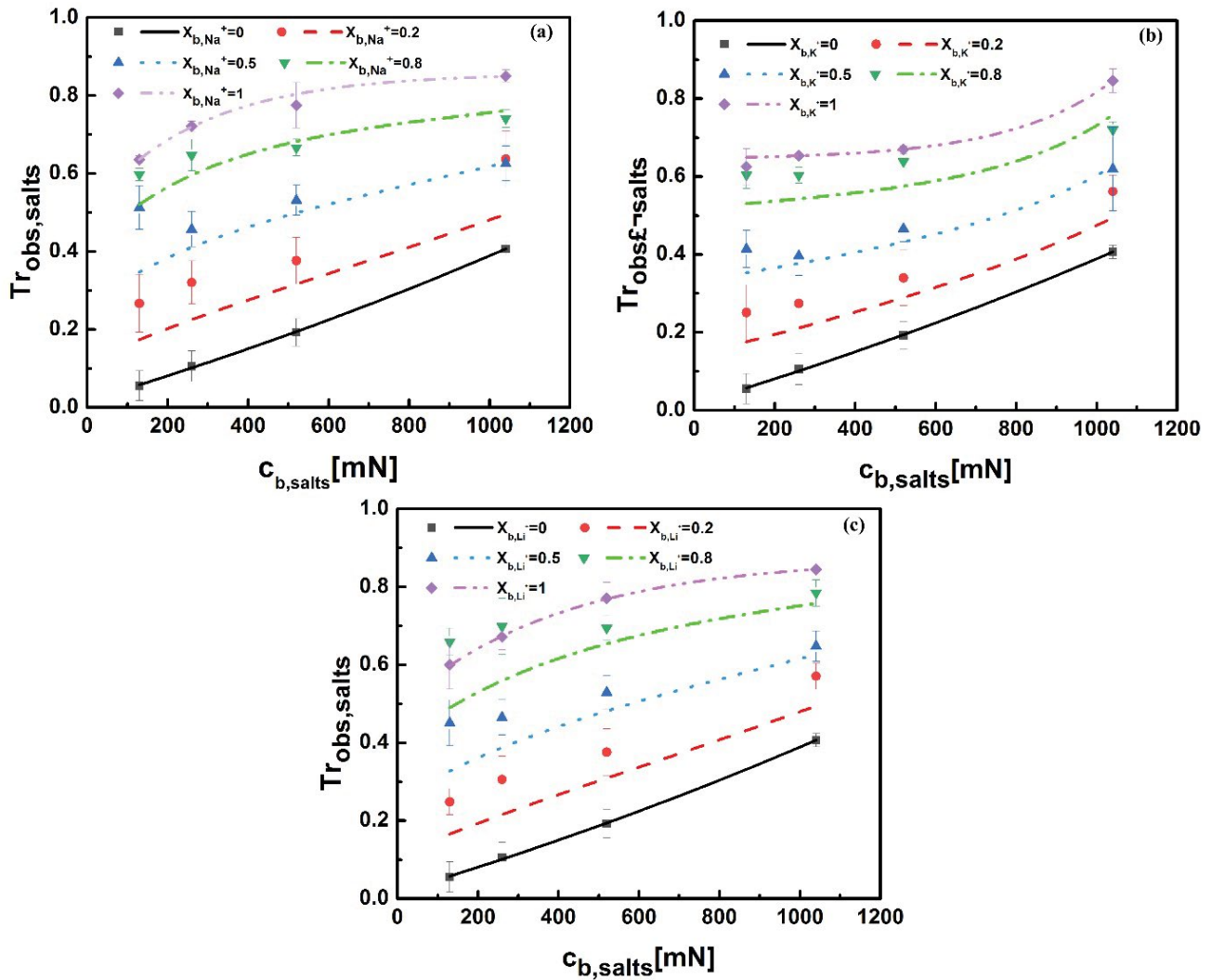


Fig. 4. Effects of equivalent concentration and fraction on observed transmission of (a) NaCl and MgCl₂, (b) KCl and MgCl₂, and (c) LiCl and MgCl₂.

Table 2
Competition coefficients of ions

Ions	Competition coefficient
Na ⁺	1
K ⁺	0.94
Li ⁺	0.99
Mg ²⁺	0.28
Cl ⁻	1
SO ₄ ²⁻	0.08

Cl⁻ transmission deviation is reaching 47.3%. At 520 mN, the calculated values of the transmission of each ion fit well with the experimental data. The maximum deviation occurs in Cl⁻, with a deviation of 22.4%. The deviation of Cl⁻ under high concentration is related to the calculation method of the competition coefficient. Under high concentration, the

competition coefficient of Cl⁻ and SO₄²⁻ will change greatly. Under the condition of 260 mN concentration, the simulation deviation of Na⁺ is the largest, which is 27.4%. At 130 mN concentration, the simulation deviation of Mg²⁺ is the largest, which is 28.9%. Generally speaking, this semi-empirical model simulation can accurately reflect the actual separation process. Therefore, the modified semi-empirical model could effectively simulate the separation performance of the simulated salt lake brine by NF membrane.

5. Conclusions

The observed transmission of the eight single salts is in the order of NaCl > LiCl > KCl > MgCl₂ > Li₂SO₄ > K₂SO₄ > Na₂SO₄ > MgSO₄. The transmission of KCl is higher than NaCl and LiCl at lower concentration. In the double salts permeation experiment, it was found that the simulated values deviated from the experimental data. Therefore, the adjustment equation A is introduced to modify simulation for brine. The modified semi-empirical model and the experimental

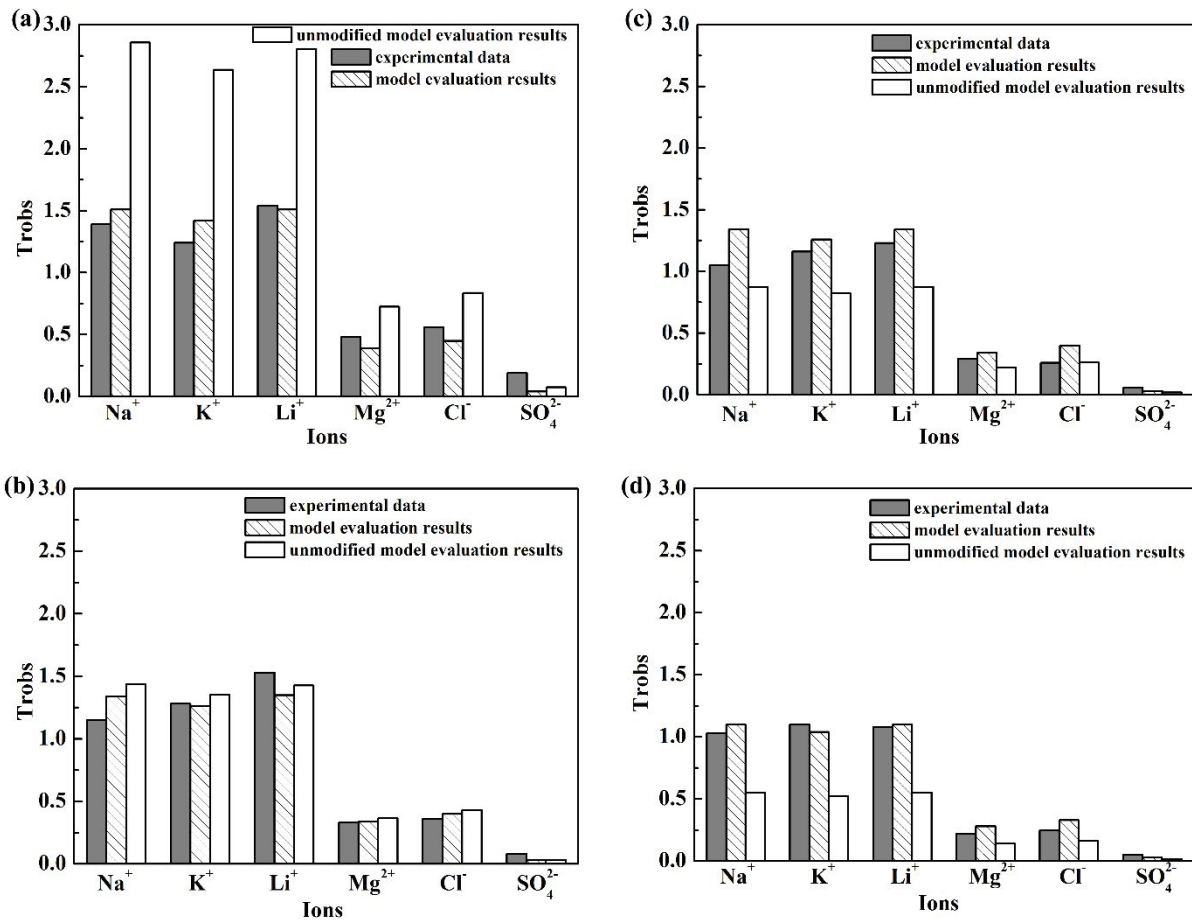


Fig. 5. Observed transmission of ions in (a) 1,040 mN; (b) 520 mN; (c) 260 mN; and (d) 130 mN, respectively.

Table 3
Comparison of simulated values and experimental data

Con. (mN)	Tr_{obs,Na^+}			Tr_{obs,K^+}			Tr_{obs,Li^+}			$Tr_{obs,Mg^{2+}}$			Tr_{obs,Cl^-}			$Tr_{obs,SO_4^{2-}}$		
	Mod.	Exp.	Dev. (%)	Mod.	Exp.	Dev. (%)	Mod.	Exp.	Dev. (%)	Mod.	Exp.	Dev. (%)	Mod.	Exp.	Dev. (%)	Mod.	Exp.	Dev. (%)
1,040	1.51	1.39	8.6	1.42	1.24	14.8	1.51	1.54	1.9	0.39	0.48	19.1	0.45	0.56	47.3	0.04	0.19	22.5
520	1.34	1.15	16.8	1.26	1.28	2	1.33	1.53	13.1	0.34	0.33	4.7	0.4	0.36	22.4	0.03	0.08	10.3
260	1.34	1.05	27.4	1.26	1.16	9	1.34	1.23	8.4	0.34	0.29	17.8	0.4	0.26	11	0.03	0.06	8.1
130	1.1	1.03	6.7	1.04	1.1	5.6	1.1	1.08	1.3	0.28	0.22	28.9	0.33	0.25	8	0.03	0.05	2.8

data under different concentration conditions has a good consistency. Thus, this semi-empirical model can be used to predict the transmission of salt lake brine. However, the model parameters and a more accurate models to predict the separation performance of NF membrane for salt lake brine should be discussed and studied in the future.

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Symbols

A	—	Coefficient
A_1	—	Selected benchmark anions
C_1	—	Selected benchmark cations
C_{b,A_j}	—	Concentration of anion j of feed
C_{b,C_i}	—	Concentration of cation i of feed
$C_{b,salts}$	—	Total concentrations of mixed salts of feed
C_{p,A_j}	—	Concentration of anion j of permeate
C_{p,C_i}	—	Concentration of cation i of permeate
$C_{p,salts}$	—	Total concentrations of mixed salts of permeate
$\tilde{C}_{Mg^{2+}}$	—	Empirical concentration parameters of Mg^{2+}
$R_{obs,salts}$	—	Observed rejection of mixed salts

Tr_{obs,A_i}	– Transmission of anion
Tr_{obs,C_i}	– Transmission of cation
$Tr_{obs,salts}$	– Transmission of mixed salts
x_b	– Equivalent fraction
x_{b,A_j}	– Anion equivalent fraction
x_{b,C_i}	– Cation equivalent fraction
Φ	– Regulating coefficient
β	– Competition coefficient of two ions
β_{A_j}	– Competition coefficient of two anions
β_{C_i}	– Competition coefficient of two cations
i	– Cation or anion

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