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Prediction of Salt Rejection in Seawater Nanofiltration Membrane Process

[1]Gh. Al Bazedi, [1]Shadia R. Tewfik, [2]Reem S. Ettouney
[1]Mohamed H. Sorour and [2]Mahmoud A. El-Rifai

[1]Department of Chemical Engineering and Pilot Plant, National Research Center, Dokki, Egypt
[2]Chemical Engineering Department, Cairo University, Giza, Egypt

Abstract: Estimation of salt rejection in nanofiltration and reverse osmosis desalination processes is crucial for accurate process design of the membrane desalination schemes and for planning of salt recovery processes. Observed rejection values in current desalination and pilot plant units reflect the interaction among seawater composition, membrane characteristics and operating parameters. This paper is concerned with the development of simplified correlations to estimate ion rejection values under different sets of conditions from seawater desalination plants involving pretreatment nanofiltration steps. The empirically developed models are based on compiled data pertinent to some full scale nanofiltration plants and also some nanofiltration pilot plant units. Two alternative approaches have been developed to generate the simplified correlations expressing rejection in terms of feed TDS, ion hydration energy, estimated ion Stokes radius, membrane pore size diameter, as well as in terms of the operating conditions of feed pressure, recovery and temperature. The calculated ion rejections manifest varying degrees of accuracy for specified target ions. On validating the developed models using experimental reported data, the estimated ranges of percentage errors are (0 to 8), (0 to 7), (0 to 11), (0 to10), (10 to 22) for Cl, Na, Ca, Mg and TDS, respectively. The proposed model has been verified for a newly proposed NF/NF/RO seawater system developed and simulated in this work. Results indicated the validity of the model with a percentage error of (9 to 17), (15 to 22), (16 to 17), (49-63) for Ca, Mg, Cl and Na respectively. The results indicate that the developed models can be used for preliminary prediction of the performance of combined NF/RO systems.

Key words: Nanofiltration separation • Desalination • Seawater • Quantitative predictive model: NF/NF/RO system

INTRODUCTION

Nanofiltration (NF) membrane is an emerging type of pressure-driven membrane with properties in between reverse osmosis (RO) and ultrafiltration (UF) membranes. NF membranes are still sometimes denoted as ‘‘softening’’membranes [1, 2]. At the present time, nanofiltration is capable of removing hardness and a wide range of other components [3]. NF has recently gained wide interest in R&D and other applications. Due to their unique separation properties, NF membranes are widely used in industry. This includes treatment of pulp-bleaching effluents from the textile industry [4, 5], separation/demineralization in the dairy industry [6-8], virus removal and pharmaceuticals [9] and wastewater treatment [10]. NF membranes offer several advantages such as low operating pressure, high flux, high retention of multivalent anion salts and organic molecular size above 300 Da, in addition to relatively low investment and low operation and maintenance costs [11]. The main advantage of NF membranes, in addition to the lower osmotic pressures compared to reverse osmosis, is the proportionally lower energy consumption [12]. NF membranes are charged and partially reject multivalent ions. Most NF and RO membranes have lower rejection at low pH, or after acid rinse [13, 14]. They have been recently used in pretreatment of seawater in the reverse osmosis desalination process [15]. Different types of integrated membranes were used in the pretreatment of seawater prior to desalination [16, 17]. This enhanced the recovery factor of RO. Using the newly integrated MF/UF+RO+MD; the preliminary experimental results confirmed the possibility of reaching a seawater recovery factor of 87%. Another integrated system (NF+RO+MC)

Corresponding Author: Shadia R. Tewfik, Department of Chemical Engineering and Pilot Plant, National Research Center, Dokki, Egypt.

[2] used a membrane crystallizer (MC) to achieve the total recovery of desalted water. Using nanofiltration membrane NF90 in desalination processes for pretreatment of seawater led to reduction of the concentration of monovalent salts by 40% and the overall TDS by 57.7% (18). The permeate thus obtained was far superior to seawater as a feed to SWRO or MSF. These results have been obtained for Indian Ocean seawater collected from the coast of Oman [19]. The characteristics of some commercially available NF membranes regarding flux and percent rejection coefficients of Ca^{++}, Mg^{++} and Cl^- have been also reported by Turek *et al.*[20] and Wilf [21].

Pre-Transport by Nanofiltration Membranes: Despite the extensive use of NF, the mechanism of transport through NF membrane has not been yet explained. To date, a large amount of papers describing mass transport in NF systems can be found in the literature. These models are divided into two main categories; irreversible thermodynamic models and transport mechanism models. The models derived from irreversible thermodynamics include Kedem-Kastchalsky and Speigler-Kedem models. This approach treats the membrane as a black box, ignoring the mechanism of transport and the structure of the membrane. Transport mechanism models utilize Nernst-Plank equation for the prediction of ion fluxes. They include Solution-Diffusion Model, Homogenous Solution Diffusion Model (HSDM), Solution-Diffusion Imperfection Model, Film Theory Diffusion Model (FTM) and Modified FTM, Preferential Sorption-Capillary Flow Model (Kimura and Sourirajan) [22] and Donnan-Steric-Pore model DSPM-DE [23]. In the state-of-the-art models, the membrane charge characteristics are either determined by fitting a model to membrane separation (i.e. retention) data [24] or they are measured independently [25]. In most of these models the variation of charge and potential is directly related to the solution properties, that is, they do not consider the underlying surface adsorption chemistry that directs the charging behavior. Combined theories such as the combined film theory/solution diffusion model (CFSD) or the combined film theory/Spiegler-Kedem model (CFSK) have been recently used for the prediction of NF performance [26]. Recent studies regarding nanofiltration modelling and simulation describe the process ranging from the atomic microscopy studies to pilot studies. Studies of the properties of NF membranes [27] were also used in the development of a model for industrial applications. Different electrolyte solutions have been investigated by Avlonitis *et al.* [28] to study nanofiltration process modelling. Geraldes *et al.* [29] developed a simulation program for nanofiltration mass transfer using tertiary electrolyte system. The rejections of salt mixtures from highly saline waters by nanofiltration membranes were also studied by Hassan *et al.* [15] and Al-Zoubi [18]. The most important operating parameters affecting the performance of NF membranes are pressure difference, recovery rate, temperature and salinity. Increased pressure and temperature increase rejection while the increase in the recovery rate or salinity reduces the rejection of mono-valent ions. Di-valent ions are affected by salinity to a lower extent.

This paper is concerned with the development of empirical correlations for the prediction of NF rejection in the pretreatment of seawater for desalination. The principal objective is to develop predictive models to quantify the selectivity of nanofiltration membranes operated at high recoveries and to validate these models.

Nanofiltration Membrane Transport Model: Spiegler Kedam model was an improvement to Spiegler Katchalsky model, by applying linear relationship between fluxes and the forces on local level rather than on the whole membrane [30].

$$Jv = Lp\,(\Delta P - \sigma\Delta\Pi) \quad (1)$$

$$R = 1 - C_p/C_f \quad (2)$$

$$R = 1 - (1-\sigma)/(1-\sigma\exp(-J_v*(1-\sigma)/P_s) \quad (3)$$

The black box approach allows the membranes to be characterized in terms of salt permeability P_s and the reflection coefficient σ. Koyuncu and Yzgan [31] found that this model was able to fit experimental data for different salt mixtures using TFC-S nanofiltration membranes. However many authors used this model for measurement of retention of electrolytes in charged nanofiltration membranes [30, 32]. It is clear from equation (3) that the retention increases by increasing the water flux and reaches a limiting value of σ at high water flux. Reflection coefficient σ is a characteristic of convective transport of the solute and it is a parameter that measures the degree of semi-permeability of the membrane [33]. Reflection coefficient can be calculated using different models such as steric hinderance pore (SHP) model or Verniory model [34]. Salt permeability can be calculated using Teorell-Meyer-Sievers (TMS) model [35] where:

$$P_s = D_s*(1-\sigma)*A_k/\Delta X \quad (4)$$

According to film theory, the relation between the observed rejection rate and the true rejection [36-38] may be expressed as:

$$\ln(1-R_{obs})/R_{obs} = \ln(1-R)/R + J_v/K \quad (5)$$

$$\sigma = 1-\{(1+(16/9)\lambda^2)(1-\lambda^2)(2-(1-\lambda^2)\} \quad (6)$$

$$\lambda = r_s/r_p \quad (7)$$

Modelling and Simulation Approach: Multiple regression methods are used to predict the rejection of permeate water ions. Correlations expressing the rejection in terms of membrane characteristics and/or operating conditions are developed based on published data of operating plants and pilot plants. The rejection calculated may be assumed to be the average rejection obtained across the module. Two approaches have been investigated. The first approach is the Operating Conditions/Membrane Characteristics (OC/MC) approach which depends on the structure of the membrane used, pore size diameter, in addition to hydration energy of the ions and operating parameters (pressure, temp., recovery). The membranes used in this approach are NF-90, NF-70, Desal HL, Desal DL, Trisep TS, Desal DK and Desal DK5L. The membrane pore size diameter is a comprehensive pore size with variation of about 20% according to the pore size investigation method [18, 25, 34, 39, 40, 41, 42]. Cited data is compiled in table A1 in Annex A. The developed regression models are presented in table 1 in the next section.

Annex A

Table A1: Compiled data for Pore size approach model.*

Ion	Ion conc./TDS$_f$	Pressure Bar	Recovery %	Ion hydration energy Kj.mol^{-1}	Membrane pore size nm	Ion diameter nm	Ion rejection ratio
NF-90							
Ca	0.01090	24.1	15	1548	0.55[a]	0.311	0.96
Mg	0.03659	24.1	15	2018	0.55	0.341	0.98
Cl	0.51772	24.1	15	376	0.55	0.121	0.63
NF-70							
Ca	0.01090	19.3	43.2	1548	0.28[b]	0.311	0.81
Mg	0.03659	19.3	43.2	2018	0.28	0.341	0.892
Cl	0.51772	19.3	43.2	376	0.28	0.121	0.247
Desal HL 4040							
Ca	0.01090	17.2	64.6	1548	0.48[e]	0.311	0.57
Mg	0.03659	17.2	64.6	2018	0.48	0.341	0.83
Cl	0.51772	17.2	64.6	376	0.48	0.121	0.146
Desal DL4040							
Ca	0.01090	17.2	50.6	1548	0.55[f]	0.311	0.66
Mg	0.03659	17.2	50.6	2018	0.55	0.341	0.86
Cl	0.51772	17.2	50.6	376	0.55	0.121	0.11
Trisep TS 80							
Ca	0.01078	25	41	1548	0.25[g]	0.311	0.84
Mg	0.03313	25	41	2018	0.25	0.341	0.956
Cl	0.29898	25	41	376	0.25	0.121	0.378
Ca	0.01056	25	43	1548	0.25	0.311	0.814
Mg	0.03549	25	43	2018	0.25	0.341	0.957
Cl	0.52207	25	43	376	0.25	0.121	0.37
Ca	0.01045	25	53.3	1548	0.25	0.311	0.8
Mg	0.03301	25	53.3	2018	0.25	0.341	0.956
Cl	0.50758	25	53.3	376	0.25	0.121	0.3
Desal DK8040							
Ca	0.01075	30	60	1548	0.55[d]	0.311	0.91
Mg	0.03359	30	60	2018	0.55	0.341	0.98
Cl	0.47485	30	60	376	0.55	0.121	0.24
Desal DK5L							
Ca	0.01048	24	62.57	1548	0.45[c]	0.311	0.9
Mg	0.03286	24	62.57	2018	0.45	0.341	0.96
Cl	0.51572	24	62.57	376	0.45	0.121	0.227
Ca	0.01042	24	64.09	1548	0.45	0.311	0.89
Mg	0.03266	24	64.09	2018	0.45	0.341	0.966
Cl	0.52236	24	64.09	376	0.45	0.121	0.18
Ca	0.01046	24	62.83	1548	0.45	0.311	0.87
Mg	0.03278	24	62.83	2018	0.45	0.341	0.966
Cl	0.50850	24	62.83	376	0.45	0.121	0.166

*[39], [a] [18], [b][40], [c] [48], [d][41], [e][42], [f] [34], [g][43]

Table 1: Simulation results using OC/MC model

Ions	Feed Ion conc. mg/l	Actual Rejection %	OC/MC approach simulation Rejection%	% Deviation
NF-70 membrane, Pressure =19.3 bar, Recovery=43.2%				
Cl	22780	24	25	4
Na	12860	38	34	11
Ca	480	81	80	1
Mg	1610	89	91	2
TDS	44000	32	35	9
Trisep TS 80 membrane, Pressure =25 bar, Recovery=43%				
Cl	23768	37	31	19
Ca	481	81	86	6
Mg	1616	95	98	3
TDS	45526	47	50	6
Desal DK 5L membrane, Pressure =24 bar, Recovery=62%				
Cl	23651	22	23	4
Ca	481	90	80	12
Mg	1507	96	90	7
TDS	45860	32	49	35
Desal DL membrane, Pressure =17 bar, Recovery=50%				
Cl	22780	11	17	35
Ca	480	66	72	8
Mg	1610	86	84	2
TDS	44000	22	42	47

Table A2: Compiled data for Pore size approach model

TDS ppm	Pressure bar	Recovery %	Na mg/l	Ca mg/l	Mg mg/l	Cl mg/l
Deep sea water data*						
34000	3.4	15.7	11160	416	1234	--
34000	6.8	15.6	11160	416	1234	--
34000	10.3	17.3	11160	416	1234	--
long beach Desalination **						
37480	39	40	11912	546	1532	19737
37480	22	55	12860	481	1608	22780
33000	31	60	12860	481	1608	22780
Umm-Lujj Desalination***						
38000	18	50	12860	481	1608	22780
38000	22	55	12860	481	1608	22780
38000	31	60	12860	481	1608	22780
44046*a	22	44	12860	481	1608	22780

* [44], ** [45], ***[46], [47], *a [15]

The second approach is the Operating Conditions (OC) approach where parameters such as temperature and pH are assumed constant during the process and fouling factor is not taken into account (annex A). Flux through the membrane is a function of the operating pressure. Molecular cut off ranges are assumed from 150-300 Da. Divalent and multivalent anions are preferentially rejected by the membrane while monovalent ion rejection is dependent upon feed concentration and composition. Since monovalent ions pass through the membrane, they do not contribute to the osmotic pressure, thus enabling nanofiltration membrane systems to operate at feed pressures below those of RO systems. Cited data is compiled in Table A2 in Annex A and developed regression models are presented in the next section.

A proposed Novel System for Model Validation: Scheme 1 presented in Fig. 1 has been simulated using OC/MC approach. Results have been validated using outputs from IMS Design (Hydranutics) tool for seawater desalination system design. The results are shown in Table 4.

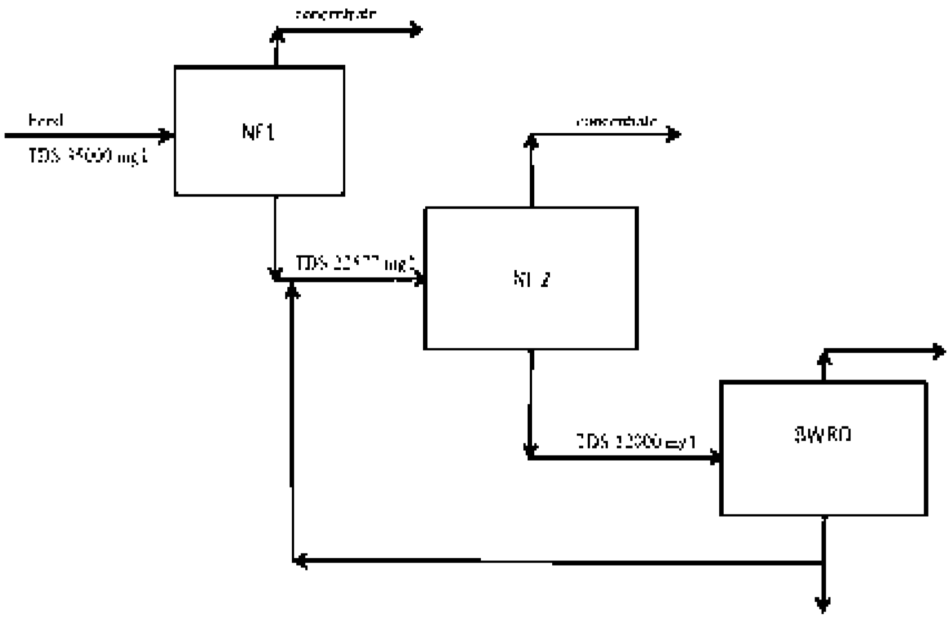

Fig. 1: Scheme 1

RESULTS AND DISCUSSION

Developed Models for the Operating Conditions/ membrane Characteristics (OC/MC) Approach: The developed equations for calculating ion rejection, concentration and permeate TDS.

Operating Conditions/membrane Characteristics (OC/MC) Approach Model Equation:

$$R_i = a_8 - b_8(I_r) + c_8(P_f) - d_8(R_f) + e_8(H_e) + f_8(r_p) + g_8(r_i) + h_8(T) \quad (8)$$

$$I_r = \text{Ion concentration}/ TDS_f \quad (9)$$

$$TDS = (a_{10}+(b_{10}* TDS_f)+(c_{10}* P_f)+(d_{10}* R_f)) \quad (10)$$

Where numerical values of the coefficients (a-h) are presented in Table A3

Developed Models for the OC Approach Operating Conditions (OC) Approach Model Equations:

$$TDS = (a_{11}+(b_{11}* TDS_f)+(c_{11}* P_f)+(d11* R_f)) \quad (11)$$

$$Cl = a_{12}+(b_{12}* TDS_f)+(c_{12}* P_f)+(d_{12}* R_f) \quad (12)$$

$$Mg = a_{13}+(b_{13}* TDS_f)+(c_{13}* P_f)+(d_{13}* R_f)+(e_{13}*Mg_f) \quad (13)$$

$$Ca = a_{14}+(b_{14}* TDS_f)+(c_{14}* P_f)+(d_{14}* R_f)+(e_{14}*Ca_f) \quad (14)$$

$$Na = (a_{15}+(b_{15}*TDS_f)+(c_{15}* P_f)+(d_{15}* R_f)+(e_{15}*Na_f) \quad (15)$$

Where numerical values of the coefficients (a-h) are presented in table A3

Table A3: Equations constants

Equation Number	A	b	C	D	e	f	g
8	-0.28	0.15	0.02	0.0045	0.00016	0.016	0.005
10	27861.63	0.050411	-330.673	16.88763			
11	27861.63	0.050411	-330.673	16.88763			
12	139490.6	-1.72622	579.9509	-1352.82			
13	3639.24	0.054327	-26.2772	29.4727	-4.0767		
14	2669.979	0.018122	-9.19885	10.18359	-7.5278		

Table 2: Simulation results using operating conditions (OC) approach model.

Ions	Feed Ion conc. mg/l	Actual Rejection %	Simulated Product ion mg/l	Simulated Rejection%	% Deviation
Water characteristics at Pressure 18 bar and 50% recovery					
Cl	22780	27	16692	27	0
Na	12860	27	9214	28	6
Ca	481	81	81	83	3
Mg	1608	88	149	91	3
TDS	38000	32	24670	35	10
Water characteristics at Pressure 22 bar and 55% recovery					
Cl	22780	46	12248	46	1
Na	12860	46	7332	43	7
Ca	481	90	95	80	11
Mg	1608	94	191	88	6
TDS	38000	48	23431	38	-20
Water characteristics at Pressure 22 bar and 44% recovery					
Cl	22780	27	16692	27	0
Na	12860	27	9426	27	0
Ca	481	81	93	81	0
Mg	1608	88	195	88	0
TDS	44000	35	23550	38	7
Water characteristics at Pressure 31 bar and 60% recovery					
Cl	22780	58	10703	53	8
Na	12860	58	5226	59	3
Ca	481	89	63	87	3
Mg	1608	91	102	94	3
TDS	38000	59	20539	46	22

Table 3: Thermodynamic results vs. OC/MC model approach simulation results

Ion	Reflection coefficient	Solute permeability m/s	Calculated rejection %[a]	Calculated rejection[b]%	Actual rejection%
Cl	0.1	1.26E-06	3	35	24
Na	0.2	7.25E-06	9	49	nm
Mg	0.77	1.10E-07	67	99.9	98
Ca	0.65	1.847E-07	51	90	91

[a]Simulation using SK model, [b] Simulation using empirical OC/MC model, nm: not mentioned

Table 4: Simulation results of the proposed scheme NF/NF/RO

Water Ions	Feed Ion mg/l	Predicted permeate conc. from IMS Design mg/l	Simulated Rejection mg/l	% Deviation
NF Stage 1, Pressure: 22.5 bar, Recovery: 65%				
Cl	19516	13416	16015	16
Na	10800	8025	4899	63
Ca	411	132	113	17
Mg	1290	250	204	22
TDS	35000	22577	23118	2
NF Stage 2, Pressure: 25 bar, Recovery:75%				
Cl	12220	8224	9965	17
Na	7309	4900	3278	49
Ca	505	148	136	9
Mg	227	40	34	15

Application of these equations for predicting ion rejection across NF-70, TriSep TS 80, Desal DK 5L, Desal DL membranes, shows different maximum error ranges of 11%, 19%, 35% and 47%, respectively. The minimum errors were observed for Ca and Mg (with exception of Desal DK 5L), when the model equations are applied. The highest predicted error is manifested by TDS rejection for Desal DK 5L and Desal DL membranes. The absolute minimum errors are shown for Ca (NF-70), Mg (TriSep TS80), Cl (Desal DK 5L) and Mg (Desal DL). In general the deviation ranges as shown in Table 2 are reasonable for preliminary process design application and seawater pretreatment applications. These equations are specific for each ion and require minimum data that could be obtained easily from current operating schemes. Applications of this approach as shown in Table 2 manifests minimum deviation as compared with data presented in Table 1. Percentage error at 18 bar and 50% recovery are associated with 10% error for TDS and zero error for monovalent ions.

The error slightly increased at 22 bar and 55% recovery, approaching 20%. A slight decrease in recovery to 44% reflects the best prediction within the entire operating range. Relatively, higher deviation is observed at 31 bar and 60% recovery. The later approach enables efficient prediction of ion rejection within 18-31 bar and a recovery range of 44-60%. It is thus possible to predict fairly accurate permeate composition and consequently the likely performance of subsequent operating stages. The economics of salt recovery especially for divalent ions could also be estimated for preliminary feasibility studies.

Current values of reflection coefficient are related to bulk concentration, which should be reevaluated in case of concentration polarization. For instance, increasing current values for Mg and Ca by 15% will increase Mg and Ca rejection to 74 and 57.7%.The proposed scheme depicted in Fig. 1 has been verified using IMS Design software developed by Hydranutics, to design desalination systems including UF,NF and RO. The results are shown in Fig. 2 and Table 4.

Predicted permeate concentrations obtained by IMS Design and simulated rejection procedures indicate comparable percentage error for the two stages in spite of permeate and recovery variations. The highest percentage deviation is manifested by Na ions. Calcium and magnesium rejection differ from IMS Design by 17%, 22% and 9% and 15% respectively between the first and second stage. The feed concentration decreased from 35,000 mg/l to almost 22,000 mg/l in the second stage which is a significant advantage for all downstream processes. The feed concentration to the proposed brackish water reverse osmosis unit in Fig. 1 approaches 13,000 mg/l according to IMS Design and simulated procedures.

In general, there is reasonable agreement between the results obtained from IMS Design software and the simulated results. Thus both methods could be adopted for preliminary anticipation of the performance of new NF/RO separator. Further work is still needed to explore the effect of high operating pressure on the rejection of specific ions for the pretreatment of seawater using NF. Simulation results tend to provide reasonable rejection of divalent cations. Additional data are still needed to improve predictability of the simulation model for high concentration water (Red sea and Arabian Gulf area).

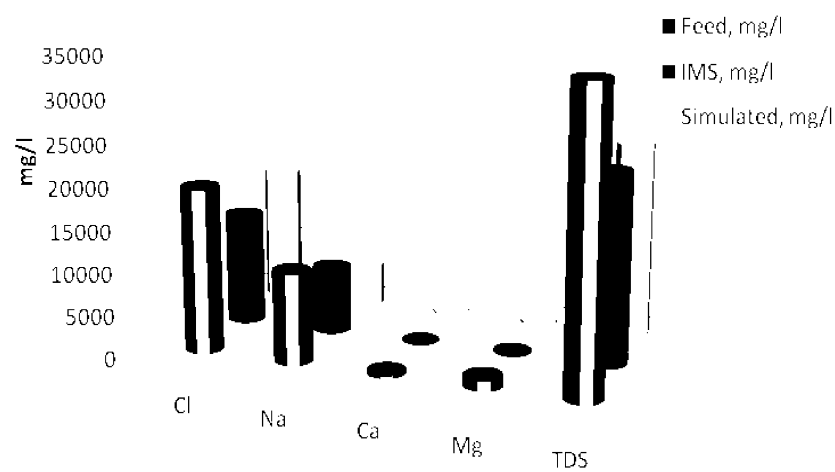

Fig. 2: Nanofiltration Stage 1 of scheme 1 results

CONCLUSION

The growing importance of NF as a pretreatment for SWRO desalting systems is related to its technical and economic merits. However, capital outlay manifested by NF pretreatment mandates reliable prediction methodology to carefully balance additional cost versus obtained benefits. Two empirical prediction schemes have been developed and tested for estimating rejection of mono and multivalent ions. Incorporation of pore diameter and hydration radius enables practical estimation of the divalent ion rejection. Results are in acceptable agreement with available simulation software. Additional work is still needed to correlate reflection coefficient with prevailing concentration polarization under high pressure and conversion in seawater nanofiltration pre-treatment system.

Nomenclature:

Cp	:	Permeate concentration (mgL^{-1})
C_f	:	Feed concentration
H_e	:	Hydration energy ($kJ\ mol^{-1}$)
J_v	:	Flux (m/s)
P_f	:	Pressure (bar)
Ps	:	Solute permeability (m/s)
R	:	Rejection%
R_f	:	Recovery %
R_i	:	Ion diameter (nm)
r_p	:	Pore size diameter(nm)
r_s	:	Stokes radii
T	:	Temperature (°c)
TDS_f	:	Feed TDS (mg/l)
σ	:	Reflection coefficient
λ	:	r_s/r_p

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