

# Application of forward osmosis membrane in nanofiltration mode to treat reverse osmosis concentrate from wastewater reclamation plants

Shahzad Jamil, Sanghyun Jeong and Saravanamuthu Vigneswaran

## ABSTRACT

Reverse osmosis concentrate (ROC) from wastewater reclamation plants have high concentrations of organic and inorganic compounds, which have to be removed before its disposal. Forward osmosis (FO) and nanofiltration (NF) membranes were tested to treat the ROC for possible water reuse. This research investigated the combined and individual influence of organic and inorganic matter on the fouling of NF and FO membranes. The results revealed that the NF membrane removed most of the organic compounds and some inorganics. The study further highlighted that the FO membrane at NF mode removed the majority of the inorganic compounds and some organics from the ROC. A pretreatment of granulated activated carbon (GAC) adsorption removed 90% of the organic compounds from ROC. In addition, GAC adsorption and acid pretreatment of ROC improved the net water permeate flux by 17% when an FO membrane was used in the NF system. Acid treatment (by bringing the pH down to 5) helped to remove inorganic ions. Therefore, the resultant permeate can be recycled back to the RO water reclamation plant to improve its efficiency.

**Key words** | forward osmosis, fouling, nanofiltration, organic and inorganic compounds, reverse osmosis concentrate, scaling

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## NOMENCLATURE

ICP	internal concentration polarization
TOC	total organic carbon
GAC	granulated activated carbon
MDL	method detection limit
OMs	organic micropollutants
ROC	reverse osmosis concentrate
TIC	total inorganic carbon
J <sub>w</sub>	water flux (L/m <sup>2</sup> h)
t	membrane thickness (μm)

## INTRODUCTION

Reverse osmosis (RO) is often used to convert seawater and wastewater into potable water (Dialynas *et al.* 2008; Greenlee *et al.* 2009; Dolar *et al.* 2012; Liu *et al.* 2012; Peñate & Garcia-Rodriguez 2012; Umar *et al.* 2015). However, the real challenge for RO is the generation of the brine (or

concentrate) stream, which contains high concentrations of dissolved contaminants and salts. These are usually in the range of 10–30% of the influent for surface water and 50–70% of the influent seawater (Adham *et al.* 2007). Conventional management of RO concentrate (ROC) from wastewater or desalination plants depends on the location of the plant. In coastal plants, ROC can be discharged into the sea, while inland plants have one option: reducing the volume prior to disposal (Tang & Ng 2008). Inland facilities use traditional technologies such as surface water or sanitary sewer discharge, evaporation ponds, deep well injection and land applications (Mickley 2001). Recently there have been efforts to treat the ROC to additionally recover water at wastewater plants. Advanced oxidation processes (AOPs) have been applied to treat ROC but it has been observed that AOP alone is not enough to remove dissolved organic carbon (DOC). However, pretreatment coupled with coagulation using ferric chloride (FeCl<sub>3</sub>) can efficiently remove organics from the ROC (Zhou *et al.* 2011). In addition, pressure-assisted forward osmosis

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(PAFO) has been investigated with simple pretreatment by Jamil *et al.* (2016). The draw solution (DS) used was potassium chloride (KCl). Granular activated carbon (GAC) adsorption was used as a simple and economical pretreatment option for removing organics from wastewater and seawater (Martinetti *et al.* 2009; Zhou *et al.* 2011). GAC can efficiently remove organic matter including humics, building blocks and lower molecular weight (LMW) neutrals and acids from the wastewater as these components are easily adsorbed into GAC pores (Ciputra *et al.* 2010).

Ren & McCutcheon (2014) have indicated that the support layer of FO membranes imparts a resistance to solute diffusion and causes internal concentration polarization (ICP). Although a DS is not used in nanofiltration (NF), the permeate exiting the NF membrane can create ICP and it would help to stop solutes going into the permeate. The membrane structural contribution to this phenomenon is defined as the structural parameter,  $S$ . It is represented as the product of the thickness ( $t$ ) and pore tortuosity ( $\tau$ ) divided by the porosity ( $\epsilon$ ) (i.e.,  $S = t\tau/\epsilon$ ) of the membrane support layer.

A recent study (Xie *et al.* 2012) investigated a FO membrane (HTI-type CTA-ES), which is cellulose triacetate (CTA) embedded in a polyester mesh for mechanical support. They estimated the mean effective pore diameter through a membrane transport model as 0.74 nm. However, using the same transport model the average pore diameter of 'tight' NF membrane (type NF 90) was determined to be 0.68 and 0.78 nm according to Nghiem & Coleman (2008) and López-Muñoz *et al.* (2009), respectively. In comparison, the FO membrane has a considerably smaller pore diameter than 'loose' NF membranes like NF 270 with a pore diameter of 0.88 and 0.84 nm (Nghiem & Coleman 2008; López-Muñoz *et al.* 2009) and BQ01 with a pore diameter of 1.6 nm (Seidel *et al.* 2001).

Xie *et al.* (2012) stated that the active layer of the HTI FO membrane is mostly made of CTA; however, the skin layer of most commercially available NF and RO membranes is made of polyamide (PA) or its derivatives. So the basic properties of FO membranes differ from those of NF membranes. It was further noted that HTI FO membranes have lower permeability but higher NaCl rejection compared to most NF membranes. The pure water permeability and NaCl salt rejection of the HTI FO membrane in RO mode were 1.1 L/m<sup>2</sup>·h·bar and 92.8%, respectively. However, it was reported that pure water permeability and NaCl salt rejection by NF membrane NF90, which is a tight NF membrane, were 6.4 L/m<sup>2</sup>·h·bar and 85%, respectively (Xie *et al.* 2012). Therefore, it can be concluded that the rejection of inorganics by FO membranes would be better than by NF membranes.

The objectives of this study are (i) to analyse the factors affecting the NF and FO membranes in an NF system for wastewater ROC treatment and (ii) to study the behaviour of NF and FO membranes with ROC, and pretreated ROC as feed solution (FS) for permeate water recovery.

## MATERIALS AND METHODS

### ROC characteristics

ROC was collected from a water reclamation plant in Sydney, Australia. RO was operated at a water recovery of about 72%, which generated an ROC stream of about 28%. The water quality of the ROC is summarised in Table 1.

### Membranes

Three types of membranes, one NF type NP 030 Microdyn-Nadir (Xiamen Co., Ltd, Germany), and two FO membranes, one an HTI-type CTA-ES and the other thin-film composite with embedded polyester screen support (TFC-ES), were used in an NF apparatus to compare their effectiveness in

Table 1 | Characteristics of the ROC

Parameters	Units	Value	Sample for the present study
RO recovery	%	–	72
Total organic carbon (TOC)	mg/L	18.0–45.0	44.3
Total inorganic carbon (TIC)	mg/L	30.0–95.0	33.0
UV <sub>254</sub>	1/m	0.7	0.7
pH		7.8	7.8
Conductivity	mS/cm	2.6–3.6	2.5
Alkalinity as CaCO <sub>3</sub>	mg/L	380	–
Ca <sup>2+</sup>	mg/L	70.0–108	70.7
Mg <sup>2+</sup>	mg/L	55.0–84.0	57.5
K <sup>+</sup>	mg/L	40.0–75.0	44.0
Na <sup>+</sup>	mg/L	376.0–563.0	359.0
Silica as Si	mg/L	0.6–0.9	0.7
Br <sup>–</sup>	mg/L	<5.0	<5.0
Cl <sup>–</sup>	mg/L	478.0–819.0	491.0
F <sup>–</sup>	mg/L	4.8	<5.0
SO <sub>4</sub> <sup>2–</sup> as S	mg/L	5.0–200	186.0
NO <sub>3</sub> <sup>–</sup> as N	mg/L	5.5–17.0	9.4
Total P	mg/L	6.5–6.9	6.8

removing organic and inorganic compounds. The properties of these membranes are given in Table 2.

## Analytical methods

The pH and electrical conductivity of the FS and DS were measured at the beginning and end of experiment using a manual Multi Portable pH and conductivity meter (HQ 40d, HACH USA). For the analysis of a wider range of anions and cations in the ROC, an inductively coupled plasma optical emission spectrometry instrument (ICP-OES, Perkin Elmer OPTIMA 7300 DV ICPOES Instruments, USA) was used. The samples were diluted with deionised (DI) water by a dilution factor of 1:10 and 1:20. Total organic carbon (TOC) and total inorganic carbon (TIC) were measured using a TOC analyser (multi N/C 3100, Analytic Jena AG, Jena, Germany). Concentrations of TIC and TOC in the initial and final solutions were analysed to determine the amount of these compounds retained on the membrane surface and hence to identify the compounds causing fouling. In other words, the amount of TIC and TOC retained on the membrane was assumed to be the difference between the product of their respective initial and final concentrations in the FS and permeate. A spectrophotometer was used for the UV measurement.

## Bench-scale nanofiltration unit

A bench-scale NF unit was used (Figure 1). The NF filtration unit was equipped with rectangular (108 mm length ×

63 mm width) cross-flow cell having a membrane effective surface area of 68 cm<sup>2</sup>. The tests were conducted on a laboratory-scale membrane cell using flat sheet NF and FO membranes. In this study, 4–5 bar pressure was applied. Pressure on the FS side of the membrane was regulated manually using the valve located at the outlet of the membrane cell. The flow rate of the FS was maintained at 500 cm<sup>3</sup>/min across the membrane by adjusting knobs manually using a variable speed drive (Magnetic drive pump 316 SS, Cole-Parmer, USA). The flow rate was monitored using rota-meters (Cole-Parmer, USA), which were installed in series with pumps along the FS streams in the circuit. The temperature of the FS was kept constant at 25.0 ± 0.2 °C using a water bath, in which stainless steel coiled pipes were immersed. The water bath was connected to a temperature control system (Thermoline BL-30) to ensure the temperature remained constant. The water flux was calculated by measuring the mass of permeate over time, recording the data with a computer fitted with software. Readings were recorded at 3 min intervals and the permeate flux was calculated every 2 hours. Experiments were started with 2 L of FS.

## Organics removal using fixed-bed GAC column as pretreatment

ROC is a complex mixture of organic compounds consisting of humic substances and LMW acids and neutrals, biopolymers and organic micropollutants (OMs). A GAC column

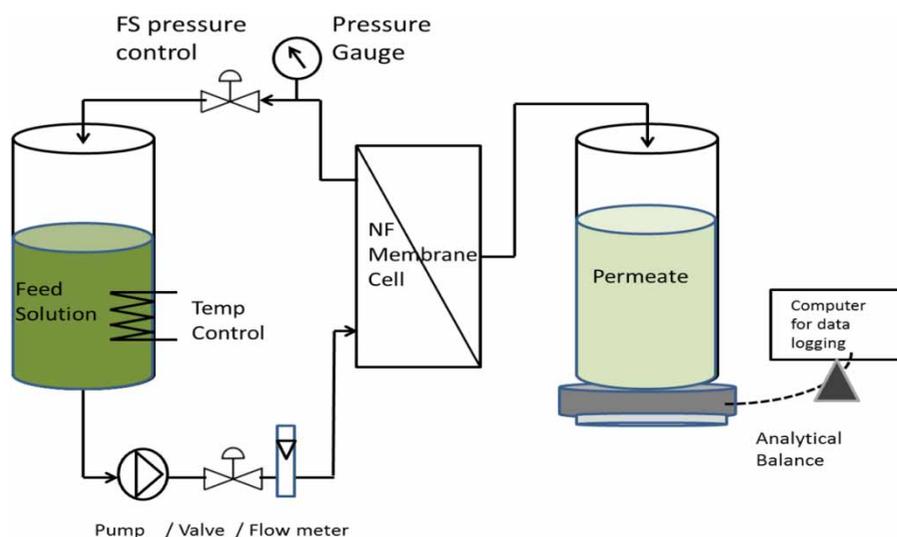
**Table 2** | Properties of NF and FO membranes (provided by vendor, otherwise specified)

	CTA-ES	TFC-ES	NP 030
Membrane supplier	HTI 97322 USA	HTI 97322 USA	Microdyn-Nadir (Xiamen) Co., Ltd
Membrane type	HTI OsMem CTA-ES membrane (FO)	HTI OsMem TFC-ES membrane (FO)	NP 030 (NF)
Membrane material	Cellulose triacetate (CTA) embedded polyester screen support	Polyamide thin-film composite (TFC) embedded polyester screen support	Polyethersulfone (PES)
Average Pore diameter	0.74 nm <sup>a</sup> 0.5–0.6 nm <sup>c</sup>		
pH range	3–8	2–11	
Membrane property	Hydrophilic	Hydrophilic <sup>b</sup>	Hydrophilic
Thickness inclusive support layer	210 μm	126 μm	
MWCO	–	–	400 Da

<sup>a</sup>Xie et al. (2012).

<sup>b</sup>Ren & McCutcheon (2014).

<sup>c</sup>Fang et al. (2014).



**Figure 1** | Flow diagram of a bench-scale NF set-up used in this study.

was found to be a suitable and practical pretreatment for removing TOC to minimise organic fouling on the membranes (Jamil *et al.* 2016). In this particular test, a GAC fixed-bed column (35 cm height, 2.2 cm internal diameter) at an ROC flow of 40 ml/min was used. The GAC fixed-bed column was able to reduce TOC by 90%. The properties of GAC and flow diagram of the GAC column are given in Jamil *et al.* 2015.

### Acid pretreatment through pH adjustment

In our previous paper (Jamil *et al.* 2016), the ROC solution was softened by the addition of HCl and the softened solution was used as the feed to the FO experiment. The initial TIC of the ROC was 78.4 mg/L and with the addition of HCl it was reduced to 5.8 mg/L by the release of CO<sub>2</sub>. This increases the permeate conversion significantly (Jamil *et al.* 2016).

## RESULTS AND DISCUSSION

### Operation time and feed recoveries

As shown in Table 4, the NF operation time was in the range of 6–90 hours for different membranes. The NF operation time is selected on the basis of feed recovery. The feed recoveries for the three selected membranes (NP 030, TFC-ES and CTA-ES) were 37, 32 and 58% respectively. The feed

recovery was calculated by using following equation.

$$\text{Feed recovery} = \frac{V_i - V_f}{V_i} \times 100$$

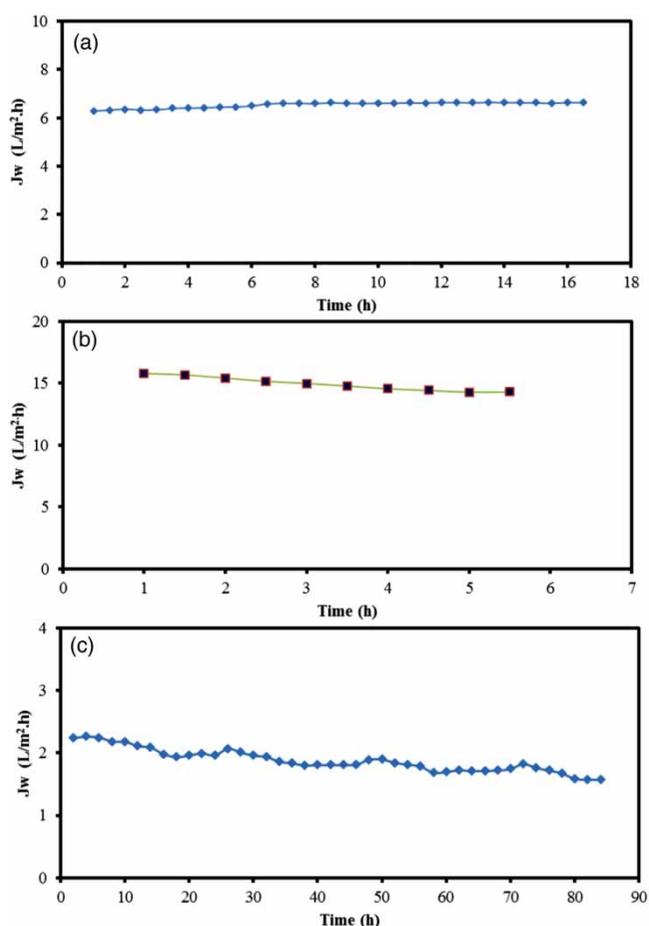
where

$V_i$  = initial volume of FS

$V_f$  = final volume of FS

### Flux and fouling study with ROC with NF membrane

NF was tested at 4 bar applied pressure. The average flux was about  $6.5 \pm 0.1$  L/m<sup>2</sup>·h as shown in Figure 2(a). It can be seen that flux remained constant during the 17.5 hours of operation. The initial conductivity of the FS was 2.5 mS/cm and final conductivity of the permeate was 2.15 mS/cm; which shows that most of the ionic species passed through the membrane. Analysis of the NF permeate showed that main cations, calcium (Ca<sup>2+</sup>), magnesium (Mg<sup>2+</sup>) and sodium (Na<sup>+</sup>) were rejected by only less than 10% (Table 3). However, among anions only phosphate (PO<sub>4</sub><sup>3-</sup>) with initial concentration 6.8 mg/L, was rejected to the method detection limit (MDL = 5) and the rest of anions passed through the membrane. As shown in Table 2, the molecular weight cut-off (MWCO) of this membrane is high (400 Da) which indicates the main reason for penetration of inorganics through the membrane (Nghiem & Coleman 2008). On the other hand, most TOC (about 92%) was retained by this membrane (Table 4). Thus, this membrane was efficient in restricting organic compounds but the rejections of inorganic species were not good.



**Figure 2** | Water permeate flux ( $J_w$ ) profile of (a) NP 030, (b) TFC-ES and (c) CTA-ES at 4 bar applied pressure (feed solution = ROC from water reclamation plant).

## Flux and fouling study with FO membranes

### TFC-ES

The FO membrane from HTI (TFC-ES) was tested in NF operation. The average flux was about  $14.9 \pm 0.4$  L/m<sup>2</sup>·h as shown in Figure 2(b). This flux was higher than that of NF membrane NP 030. The flux decreased only marginally

from 15.6 to 15.1 L/m<sup>2</sup>·h during 6 hours of operation. The initial concentration of TOC in the FS was 43 mg/L and the final concentration of permeate was about 10 mg/L (Table 4). Similarly, the initial conductivity of the FS was 2.34 mg/L and final conductivity of permeate was 1.94 mg/L. This indicates that about 83% ionic species passed through the membrane during the 6 hours of operation.

Table 3 shows that it rejected about 28% of cation species and 14–24% of anions. On the other hand, it rejected about 77% of organic compounds (Table 4). This indicates that the membrane is good at rejecting organic compounds but the rejection of inorganic species was poor: only 14–28%. The rejection by FO membranes was higher than by NF. As mentioned in the Introduction, there are two reasons why FO membranes could behave better in NF systems: firstly, most FO membranes are tight compared to NF membranes; secondly, FO membranes have a support layer that can restrict solutes due to ICP.

### CTA-ES

CTA-ES was also tested with real ROC as the FS at 4 bar applied pressure. The experiment with this FO membrane was conducted for 90 hours in NF mode. The initial flux was low at about 2.3 L/m<sup>2</sup>·h and after 90 hours of operation it was reduced to about 1.5 L/m<sup>2</sup>·h (Figure 2(c)). The initial concentrations of TOC and TIC were 43.2 and 32.9 mg/L, respectively (Table 4). The decrease in flux was due to organic fouling and inorganic scaling of the membrane. The organic fouling was 0.32 mg/cm<sup>2</sup> during the 90 hours of operation. The initial conductivity of the FS was 2.5 mS/cm and it was increased to 4.5 mS/cm during 90 hours of operation. This indicated about 55% of ionic species were rejected by this membrane. It rejected about 80% of organic compounds (Table 4) and the rejection of the ionic species were 20–65% of the initial concentrations

**Table 3** | Rejection behaviour of NF and FO membranes with respect to ionic species with ROC as the FS

	Ca <sup>2+</sup>	K <sup>+</sup>	Mg <sup>2+</sup>	Na <sup>+</sup>	Cl <sup>-</sup>	NO <sub>3</sub> <sup>-</sup>	PO <sub>4</sub> <sup>3-</sup>	SO <sub>4</sub> <sup>2-</sup>
ROC (mg/L)	70.7	44.0	57.5	359.0	491.0	41.7	6.8	186.0
% removed by NP 030	8.8	2.2	6.0	6.0	0.0	0.0	<sup>a</sup>	3.0
% removed by TFC-ES	27.9	27.9	26.9	26.7	20.0	14.2	<sup>a</sup>	24.4
% removed by CTA-ES	46.6	29.7	40.8	29.8	20.0	25.5	<sup>a</sup>	65.0

<sup>a</sup>Method detection limit (MDL = 5).

**Table 4** | Behaviour of NF and FO membranes when ROC is used as the FS

Membrane type	NP 030	TFC-ES	CTA-ES
Applied pressure (bar)	4	4	4
Initial/final volume of FS (mL)	2000/1260	2000/1370	2000/850
Permeate (mL)	740	630	1150
Feed recovery (%)	37.0	32.0	58.0
Initial conc. of TOC in FS (mg/L)	43.9	43.2	43.2
Final conc. of TOC in permeate (mg/L)	3.7	9.9	8.9
TOC restricted by the membrane (%)	92.0	77.0	80.0
Initial conc. of TIC in FS (mg/L)	33.2	32.9	32.9
Total TOC in the system (mg)	87.7	86.4	86.4
Total TOC on the membrane surface (mg)	32.6	21.6	21.6
TOC on the membrane surface (mg/cm <sup>2</sup> )	0.40	0.32	0.32
Initial cond. of FS (mS/cm)	2.5	2.3	2.5
Final cond. of FS (mS/cm)	2.1	2.6	4.5
Final cond. permeate (mS/cm)	2.2	1.9	1.1
Membrane surface area (cm <sup>2</sup> )	68	68	68
Operation time (h)	17.5	6.0	90.0

(Table 3). This agrees with the findings of previous studies (Xie *et al.* 2012; Fang *et al.* 2014). The rejection of inorganics was better than NP 030 and TFC-ES membrane (Table 3).

It was revealed that without any pretreatment, NF membrane (NP 030) rejected less than 10% of ionic species. The FO membrane (TFC-ES) rejected about 17% of ionic species, while about 77% of organic compounds were rejected. Finally, the third tested membrane CTA-ES, rejected 55% of inorganics, and 80% of organic compounds. As CTA-ES gave the best results in terms of inorganic removal, it was decided to check the performance of this membrane with GAC pretreatment so that the resultant permeate flux can be recycled.

### Performance of FO membrane in NF operation ROC with pretreatment

Two different pretreatments, namely (i) removal of organics with GAC adsorption column and (ii) pH adjustment to 5 to precipitate inorganic ions were tried individually and

**Table 5** | Behaviour of FO membrane (CTA-ES) in NF operation: (i) ROC without any treatment; (ii) ROC (GAC pretreatment) and (iii) ROC (GAC pretreated and softening)

	ROC	ROC-GAC	ROC-GAC and acid treatment
Applied pressure (bar)	5	5	5
Initial/final volume of FS (mL)	2000/850	2000/825	2000/610
Permeate (mL)	1150	1175	1390
Feed recovery (%)	57.0	59.0	70.0
Initial conc. of TOC in FS (mg/L)	43.22	4.3	0.553
Final conc. of TOC in permeate (mg/L)	9.91	0.12	0.45
Initial cond. of FS (mS/cm)	2.54	2.44	2.97
Final cond. of FS (mS/cm)	4.48	4.27	6.82
Final cond. permeate (mS/cm)	1.14	1.57	1.25
Membrane surface area (cm <sup>2</sup> )	68	68	68
Operation time (h)	90	90	76

together before the treatment with FO membranes in NF operation mode. As shown in Table 5, 90% of the organics were removed by the GAC pretreatment. The GAC pretreatment of ROC increased the water permeate flux by only 2%. However, the permeate flux was increased by 17% with GAC adsorption followed by acid pretreatment. This indicates that inorganic scaling was the main cause for the reduction of membrane performance. Another parameter to check for the membrane performance is the conductivity of the FS. If the conductivity of the FS increases with membrane operation, it means that inorganic components of the FS are rejected by the membrane. Thus, the conductivity of the FS was increased from 2.5 to 4.5 mS/cm without GAC pretreatment. When the pH of the ROC was reduced from 8.9 to 5.0 with acid treatment, the rejection of ionic species was increased from 43 to 56%. They may have followed other mechanisms like such as electrostatic interactions between ions and with the membrane surface, which is based on the Donnan exclusion. Here, the cations are repulsed by the membrane surface and equal number of counter-ions are retained, which results in salt retention (Teixeira *et al.* 2005).

Table 6 shows that there was an increase in rejection of Ca, K, Mg, Na Cl, NO<sub>3</sub>, and SO<sub>4</sub> with acid pretreatment followed by GAC pretreatment.

**Table 6** | Rejection of inorganics by FO membrane (CTA-ES) in NF operation with GAC pretreatment and GAC along with acid pretreatment

		Ca <sup>2+</sup>	K <sup>+</sup>	Mg <sup>2+</sup>	Na <sup>+</sup>	Cl <sup>-</sup>	NO <sub>3</sub> <sup>-</sup>	PO <sub>4</sub> <sup>3-</sup>	SO <sub>4</sub> <sup>2-</sup>
ROC (mg/L)		70.7	44.0	57.5	359.0	491.0	41.7	6.8	186.0
CTA-ES	% removed	46.0	29.7	38.8	28.8	19.0	24.5	<sup>a</sup>	65.0
CTA-ES (GAC and acid treatment)	% removed	89.6	32.01	90.2	52.1	33.9	79.1	<sup>a</sup>	94.9

<sup>a</sup>Method detection limit (MDL = 5).

## WHY FO MEMBRANE IS PREFERRED OVER RO MEMBRANE IN NF SYSTEM

The membrane properties are adjudged on the basis of permeability, pore size and applied pressure. FO membranes are better in terms of pore size and lower applied pressure. RO membranes are operated at high pressure as compared to FO or NF membranes. In NF system the applied pressure is 5–30 bars only, which why FO membranes were preferred over RO membranes (Table 7).

When FO membranes are operated at NF or RO mode, the property of the membrane is affected by the pressure applied. In particular, membrane thickness and porosity of the CTA-FO membrane may be reduced by applied pressure indicating that the FO membrane has become denser. Therefore, both rejection and water flux of FO membranes can be improved in NF or RO mode. Another reason for the increase in rejection in the NF or RO mode is as follows: when FO is operated in FO mode, there will be a reverse salt flux from the DS side, decreasing rejection efficiency of the FO membrane. However, when FO membranes are used in NF or RO mode, there is only water flux from the FS side without any need for DS.

## CONCLUSIONS

- This study showed that an NF membrane is a good option for pretreatment for organic compounds from wastewater however, most inorganic compounds passed through the membrane.
- This study further investigated FO membranes which have smaller pore sizes than most NF membranes. Thus they performed better in terms of inorganic compound removal than most NF membranes.
- CTA-ES has lower flux as compared to NP 030 and TFC-ES membrane. However, CTA-ES (FO membrane) removed inorganic compounds better than the other membranes, although the operation was longer (90 hours).
- Acid pretreatment (by lowering the pH to 5) increased the inorganic ion removal from 43 to 56%, by electrostatic interactions between ions and with the membrane surface, which is based on the Donnan exclusion (Table 6).
- GAC pretreatment removed 90% of organic compounds from the ROC, which helped to reduce the organic fouling of the membranes.

**Table 7** | Overview of pressure-driven membrane processes and their characteristics – adopted and modified from van der Bruggen et al. (2003)

	FO/PAFO	NF	RO
Permeability (L/m <sup>2</sup> ·h·bar)	0.7–1.16	1.5–30	0.05–1.5
Pressure (bar)	0–10	5–30	20–120
Pore size (nm)	0.5–1	0.5–2	<0.5
Rejection			
1. Monovalent ions	–	–	+
2. Multivalent ions	+	+	+
3. Small organic compounds	– / +	– / +	+
4. Macromolecules	+	+	+
5. Particles	+	+	+
Separation mechanism	Sieving/charge effects	Sieving/charge effects	Solution/diffusion
Applications	Removal of (multivalent) ions and relatively small organics	Removal of (multivalent) ions and relatively small organics	Ultrapure water; desalination

Note: '+' denotes significant removal and '–' is insignificant removal.

- GAC pretreatment followed by NF operation with FO membrane removed most of the inorganic compounds from the ROC.

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