Iron and manganese removal in direct anoxic nanofiltration for indirect potable reuse

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Abstract. Managed aquifer recharge (MAR) systems are gaining interest as an alternative to conventional water resources. However, when the water recovered in MAR systems, dissolved iron and manganese species may easily oxidize and they cause well screen clogging or require abandonment of extraction wells. In this study, both oxic and anoxic conditions were analyzed to verify the feasibility of the membrane filtration performance under various solution chemistries. The fouling mechanisms of the metal ions under anoxic conditions were also investigated by employing synthetic wastewater. The fouled membranes were then further analyzed to verify the major causes of inorganic fouling through SEM and XPS. The newly suggested anoxic process refining existing membrane process is expected to provide more precious information about nanofiltration (NF) membrane fouling, especially for demonstrating the potential advantages to chemical-free drinking water production for indirect potable reuse.

Keywords: anoxic; managed aquifer recharge; nanofiltration; iron; indirect potable reuse

1. Introduction

Managed aquifer recharge (MAR) systems have gained increasingly gaining interest for alternate water treatment of unconventional water resources such as reclaimed water, urban storm water, or impaired surface water to augment drinking water supplies (Gorzalski *et al.* 2014, Page *et al.* 2016, Vanderzalm *et al.* 2010). Many MAR systems consist of engineered delivery of water into the subsurface and storage for an appropriate retention time in aquifer material until required. The stored water is then recovered from the same well and distributed as drinking, industrial, or irrigation water (Nowaczyk *et al.* 2010, Ward *et al.* 2009, Zuurbir *et al.* 2014). However, these systems often fail to satisfy the 0.3 mg/L iron (Fe) and 0.1 mg/L manganese (Mn) concentrations suggested by the World Health Organization (WHO) guidelines for drinking water, mainly because of deteriorated water quality during the recovery process (De Vet *et al.* 2010, Jin *et al.* 2018, Leo *et al.* 2016).

In the past few decades, MAR systems have been widely considered versatile and capable of degrading a wide range of contaminants (Page *et al.* 2010a,b, Toze *et al.* 2010). These engineered MAR systems offer several advantages over other approaches, such as advanced oxidation processes (AOPs) in terms of chemical consumption, toxic byproduct production, and capability to adjust to variations in the influent composition and flow rate (Azbar *et al.* 2004, Ratanatamskul *et al.* 2012,

Vanderzalm *et al.* 2009). On the other hand, one of the major disadvantages of MAR systems is the adverse water quality changes that occur in different phases of the process. For instance, water quality problems are often reported when oxygenated water is injected into an anoxic environment where pyrite, siderite, or other sedimentary organic materials (SOMs) are oxidized (Al-Rawajfeh *et al.* 2012, Stuyfzand, 1998). This may result in elevated concentrations of released iron, manganese, and other minerals, hence poor water quality (Nederlof *et al.* 2000). Furthermore, iron and manganese are two common trace metals that oxidize into insoluble solids that cause several problems. The most significant problems include organoleptic discomfort, well clogging, and health issues. Therefore, in order to produce proper drinkable water via MAR, the removal of certain minerals, specifically iron and manganese, is essential.

There have been many attempts to produce water using MAR. MAR systems are highly adaptable technologies that can combine with various technologies such as coagulation, dual media filtration, and disinfection (Page *et al.* 2016, Page *et al.* 2018). Previous attempts to develop chemical-free water treatment prior to providing drinking water have employed bank filtration settings. Unfortunately, these systems suffer from major limitations, including ineffective removal of pathogens, inability to remove recalcitrant compounds, and extraction well clogging by some trace metals (Djahida *et al.* 2014, Elango Lakshmanan 2012). In particular, because oxidation of iron and manganese is high near the reducing zone close to the extraction well, post treatment is compulsory. This implies the complexity of using existing MAR treatment methods. Moreover, the high cost of long-term operations and concerns about water

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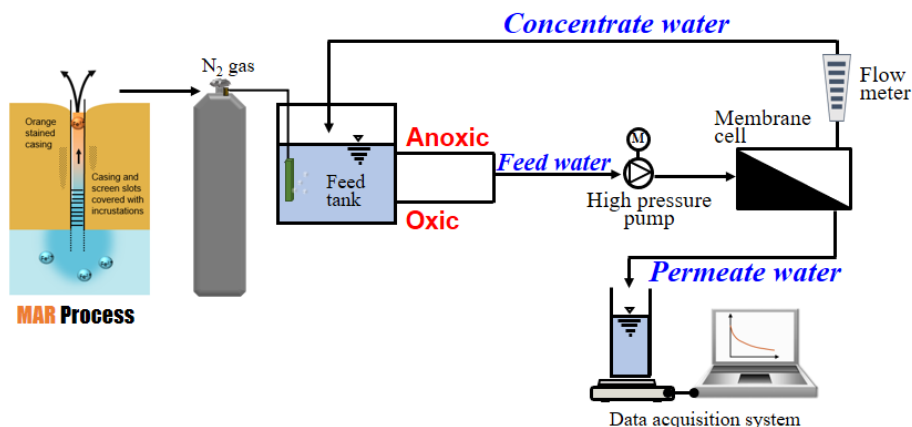 E-mail: chp@ewha.ac.kr

Fig. 1 Scheme description of the NF filtration system

quality are common issues for former MAR methods in general. Considering that former methods are insufficient for safe production of drinking water, there is a need for new technology that is both easy and inexpensive to manage.

In this research, an anoxic nanofiltration (NF) membrane process was preliminary tested as an innovative means for treating iron and manganese released in the reducing zone for drinking water purpose. In particular, the fouling mechanisms of the anoxic fouling behavior based on various operating conditions were studied. In addition, the feasibility of the anoxic process was compared with the oxic condition under various operating conditions and further confirmed through scanning electron microscope (SEM) and X-ray photoelectron spectroscopy (XPS) analysis.

2. Material and methods

2.1 Feed water preparation

The specific chemical composition of the feed water used in this study was designed to represent the groundwater from MAR extraction wells. Typical iron concentrations in the recovered water were ranged from 0.1 to 10.6 mg/L (Hahn *et al*. 2016, Yi *et al*. 2015), thus the 1, 5 and 10 mg/L of total iron concentration were chosen to investigate the fouling potential in both oxic and anoxic filtration experiments. Iron ($FeCl_2 \cdot 6H_2O$, Sigma-Aldrich, USA) was selected and used as a model foulant. Deionized (DI) water with a quality of 18 μs/cm was used for all solution preparation. Specifically, the synthetic recovered water was prepared by dissolving 0.2 ± 0.14 mM iron in DI water. To make an anoxic condition, dissolved oxygen was purged by nitrogen gas. Finally, the pH of the feed waters was adjusted by using hydrochloric acid (HCl) and sodium hydroxide (NaOH).

2.2 Experimental set-up and operational procedure

Bench-scale NF experiments were carried out for 4 hours to observe membrane fouling caused by iron and

Table 1 Specific properties of the NF membrane used in this research.

Parameter	NF-4040
Material	Polyamide TFC
MWCO	200 Da
Roughness	5.8 nm
Contact angle	35.1º
Active area	7.6 m²
Rejection ($MgSO_4$)	> 99 %

manganese under crossflow operating conditions (Fig. 1). Specifically, the feed water was pumped into the NF unit to gain NF softening permeate water. A crossflow membrane cell with an effective membrane filtration area of 21.56 cm² (7.7 cm in length, 2.8 cm in width, and 1 mm in depth) was used for the NF experiment. The operating conditions of the NF unit were kept at a transmembrane pressure of 10 bar by an outlet valve and high-pressure pump (G03E, Hydra-cell). The feed temperature was kept constant at 20 °C by a cooling/heating circulator (DRC8, CPT Inc.). Before the experiment, all membranes were rinsed with DI water several times and compacted with DI water over 12 hours until the flux reaches stable. The permeate water reservoir was placed on a weighing balance and mass changes were automatically recorded by a computer to calculate the flux. The concentration of the DO under feed tank was constantly monitored using a digital optical sensor (HQ40d, Hach).

The NF membrane used in this research was purchased from Dow Chemical Company, USA. The characteristics of the selected membrane are shown in Table 1. The molecular weight cut-off (MWCO) for the membrane was 200 Da. The membrane was cut to fit into the cell size of the NF unit, then immersed in DI water and stored in the 4°C refrigerator.

2.3 Analytical methods

To characterize the NF membranes after the experiments, field emission scanning electron microscope (FE-SEM, Inspect F50, FEI), energy dispersive spectrometer (EDS, Apollo XL, EDAX Ametek) and X-ray photoelectron spectroscopy (XPS, PHI 5000 VersaProbe,
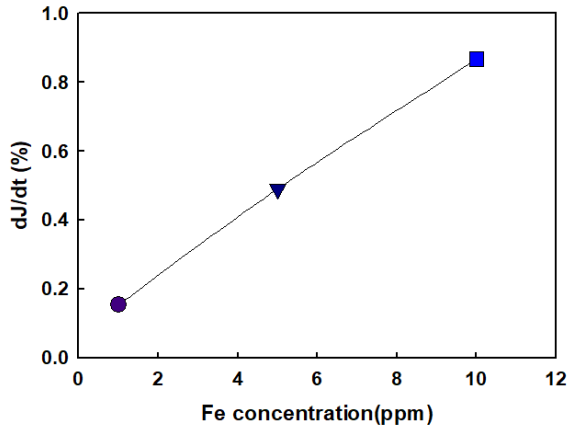
Fig. 2 Evaluation of the NF fouling by FDR increment as the iron concentration increased
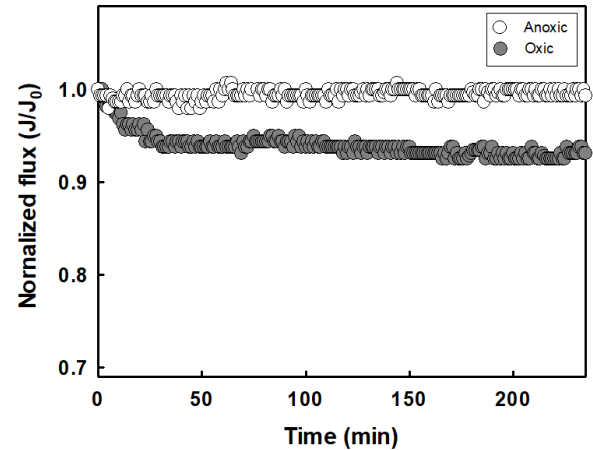

Fig. 3 Comparison of the NF flux decline under anoxic and oxic conditions. The fouling experiments were conducted as follows: feed water = 3 L, Fe concentration = 10 mg/L. The anoxic condition was maintained by purging N_2 gas during NF filtration. The total Fe concentration was estimated by the difference in values as measured by the Hach device before and after filtration by each NF membrane

ULVAC PHI) were utilized to take measurements. FE-SEM provided qualitative information by visualizing the surface and cross-section of the membrane. EDS provided semi-quantitative and qualitative elemental analysis of unknown foulants. After the NF fouling experiments under anoxic and oxic conditions, we confirmed differences in the active surface layer of the NF membranes and identified the major components of the foulants by employing these two methods. XPS identified surface elements and their chemical state, and then analyzed them quantitatively by determining the photoelectron energy of the chemical binding state of Fe in the foulants.

During the lab-scale experiment, pH, dissolved oxygen (DO), oxidation-reduction potential (ORP), and concentration of inorganic ions in each NF unit were detected. DO, pH value, and ORP were analyzed by using probes with a portable multi-meter (HQ40d, Hach). Cation and anion concentrations were analyzed using an inductively coupled plasma-optical emission spectrometer (730 Seires, Agilent), ion chromatography (883 Basic IC Plus, Metrohm), and UV/VIS spectrophotometer (DR/3900, Hach).

3. Results and discussion

3.1 Comparison of NF fouling under oxic and anoxic conditions in post MAR process

Bench-scale NF filtration tests were first conducted under oxic conditions. The flux decline rate (FDR) was employed, which is a common method of quantifying the fouling potential of feed water (Choi *et al.* 2009; Yu *et al.* 2010). FDR was defined as the following equation:

$$\varphi = -\frac{1}{J_0^2} \cdot \left.\frac{dJ}{dt}\right|_{t=0} \tag{1}$$

where φ is the FDR, J_0 is the initial flux ($m^3 \cdot m^{-2} \cdot s^{-1}$), and t is the filtration time (min).

To validate the usefulness of NF measurement in MAR post-treatment, we performed NF oxic filtration tests using

synthetic iron. Fig. 2 compares the variation of FDR as the iron concentration increased. The experimental results provided reasonable fits between the iron concentration and flux decline caused by iron under oxic conditions (R^2=0.99), which implies the cake layer resistance had an effect on the membrane surface (Fig. 2).

Bench-scale NF filtration tests were also conducted under anoxic conditions. In these experiments, the anoxic condition was controlled by purging N_2 gas to maintain the DO concentration lower than 0.3 mg/L. Both feed waters were set at the same concentration (Fe_{total} = 10 mg/L) by considering the practical operating condition in the post MAR process. While a more rapid flux decline was observed in the oxic condition, there was almost no flux decline for the anoxic condition, which means less membrane fouling occurred (Fig. 3).

There are two possible reasons for the difference. First, the filtration tests were conducted under a crossflow filtration system, which significantly affected the selective deposition of particles. Generally, the larger particles moved away from the membrane surface due to their greater back transport, and the smaller particles tended to be deposited on the membrane surface in crossflow mode (Sim *et al.* 2010, Sim *et al.* 2011). In the case of iron, foulants can be defined as the summation of all dissolved iron species, as shown in Eq. (2). In this case, the larger particles such as iron oxides may be moved away from the membrane surface due to the effect of inertial lift or shear-induced diffusion. However, the smaller foulants such as divalent ions (Fe^{2+}) may be easily adsorbed or passed through the membrane due to the negative charge of the NF membrane active layer or the Brownian diffusion (Romero *et al.* 1988, Chellam *et al.* 1997, Sim *et al.* 2011). Additionally, the water quality analysis confirmed that the concentration of Fe^{2+} in concentrate water was much higher in anoxic than oxic conditions (Table 2).
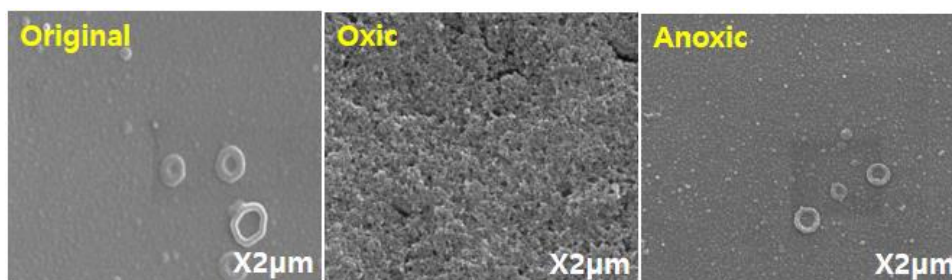
Fig. 4 SEM analysis of membrane surface after the NF filtration process. (a) Virgin, (b) Oxic condition and (c) anoxic condition. Experimental conditions: $FeCl_2$=10 ppm, temperature = 15 ℃
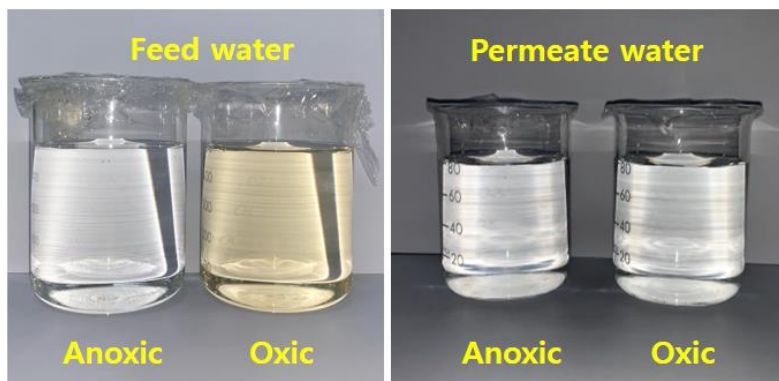

Fig. 5 Color changes of both feed and permeate water under anoxic and oxic conditions

$$Fe_T = [Fe^{2+}] + [FeOH^+] + [Fe(OH)_2^0] + [Fe(OH)_3^-]$$
$$+[FeHCO_3^+] + [Fe(CO)_3^0] + [Fe(SO)_4^0] + [FeCl^+] \quad (2)$$

When the iron contacts with oxygen, it is easily oxidized with dissolved oxygen, forming a compound with low solubility (Bottino *et al*. 2011, Jährig, J. *et al*. 2018). The higher solubility of reduced iron under anoxic conditions results in lower fouling potential of NF feed water as well as less iron fouling.

Second, iron oxidates may be strongly affected by solution chemistry such as pH and ionic strength. In this study, all initial pH was 7.0. However, pH reduced much more severely in the anoxic condition during the filtration process.

3.2 Membrane surface analysis

To explain the difference in anoxic and oxic conditions mechanistically, the cake layer characteristics were further investigated using SEM after each experiment. It is evident from SEM imaging (Fig. 4) that there are many more iron particles under the oxic than anoxic condition. The micrographs of the cake layer support the above conclusion that the oxic condition of NF filtration is influenced by the size and shape of the accumulated iron particles.

The iron concentration under anoxic and oxic conditions from the feed water indicated that, in all installations, only half of the iron was present in the concentrate water under oxic conditions. Adding the irons to the feed water, it is accompanied with sensitive colour change to yellowish under oxic condition because they precipitate in the

Table 2 Water quality analysis under oxic and anoxic conditions

	Anoxic					Oxic				
	**Fe_{total}	Fe^{2+}	***DO	pH	Cond.	Fe_{total}	Fe^{2+}	DO	pH	Cond.
	(ppm)	(ppm)	(ppm)	-	(μs/cm)	(ppm)	(ppm)	(ppm)	-	(μs/cm)
*Feed	10.10	8.30	0.08	7.00	44.10	9.75	0.25	8.87	7.00	45.10
*Conc.	10.00	6.90	0.06	5.60	49.40	4.40	0.05	9.69	6.13	50.50
*Perm.	0.85	0.60	6.27	5.87	13.60	0.02	0.02	9.56	6.42	7.03

* Feed, Conc., and Perm. are the feed water, concentrate water, and permeate water.
** The concentration of Fe_{total} was measured instead of Fe^{3+} by referring the following references (Sullivan *et al*. 1976).
***Feed and concentrate were measured by a DO meter as an in-line, but permeate was collected in a sample vial of 50 mL, and then measured the DO concentrations.

presence of oxygen (Fig. 5). This means that iron accumulated on the membrane surface, confirming the previous discussion, regardless of feed water composition (Table 2). On the contrary, the iron concentration in both feed water and concentrate were similar in the anoxic condition. The rejection rate of the anoxic (93.04%) was slightly lower than that of the oxic condition (99.79%). This may be due to most iron particles being positively charged, thus the electrostatic repulsion between evenly charged iron and the negatively surface charged membrane was reduced during the filtration process (Brant *et al*. 2006, Yang *et al*. 2009). In addition, it should be mentioned that in a mass balance of the feed, concentrate, and permeate water, such low deposition rates were hard to detect due to fluctuation of the feed water quality.
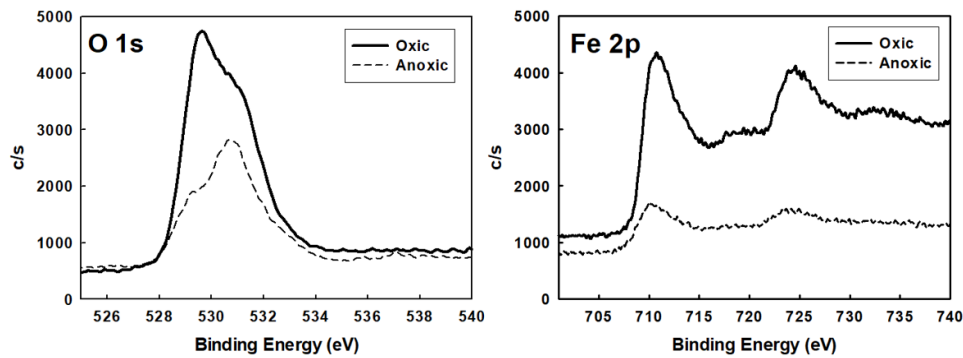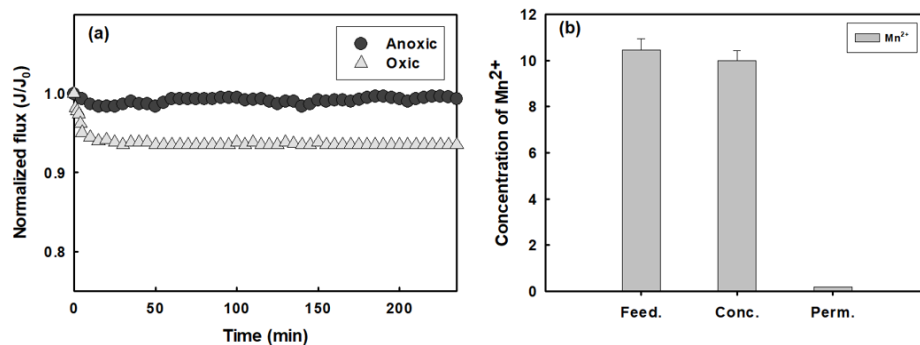
Fig. 6 Comparison of XPS spectra of the iron oxide products under O 1s and Fe 2p states

(a) Comparison of NF flux decline caused by manganese under anoxic and oxic conditions

(b) Mn^{2+} concentrations in feed water, concentrate water, and permeate water

Fig. 7 The fouling experiments were conducted as follows: feed water = 3 L, Mn concentration = 10 mg/L. The anoxic condition was maintained by purging N_2 gas during the NF filtration process. The concentrations of total Mn (Mn_{Total}) and Mn^{2+} were estimated by the difference in values as measured by the Hach device before and after filtration by each NF membrane

The surface electronic states and the chemical composition of the iron products were further examined by XPS (Hallam *et al.* 2012). The XPS analysis revealed the presence of Fe, C, and O with the binding energies of the O 1s and Fe 2p states as showed in Fig. 6. The O 1s spectrum of the oxic condition exhibits peaks at 529.33 eV, which is attributed to oxygen atoms binding with the iron. Compared with the anoxic spectra, the energy dispersive XPS analysis of the working surface shows a significant increase in oxygen content. Specifically, the main Fe 2p peak, including two satellite peaks on the higher binding energy side (710.5 eV and 724.8 eV), indicate the Fe ion is in a divalent state. Moreover, the area of the Fe 2p spectrum in oxic condition is much wider than in anoxic, which means the filtration after oxic filtration process has more iron on the membrane surface.

3.3 Anoxic filtration under various operating conditions

The anoxic filtration process has been suggested as a novel filtration process to prevent membrane fouling and scaling in the MAR post-treatment process. In this section, the applicability of the anoxic filtration process was further investigated through comparison of anoxic filtration with other foulants (i.e., manganese) and various operating conditions (i.e., applied pressure).

3.3.1 Mn fouling under anoxic condition

Manganese is another major concern in the composition of the post-treatment MAR process since it widely exists in soluble forms (Hasan *et al.* 2018). In order to verify the applicability of anoxic filtration on manganese fouling, NF fouling tests were also conducted by considering the actual condition of manganese in MAR. In these tests, the concentration of manganese was set to approximately 10 mg/L under 10 bar. As shown in Fig. 7(a), anoxic filtration was maintained in a stable condition with lower fouling than oxic. Similar to the iron filtration process, the rapid flux decline at the initial stage and slow flux decline after 50 min were showed. This might be mainly attributed to foulant-foulant interaction rather than foulant-membrane interaction on the surface.

The chemistry of manganese in water is complex. Generally, dissolved manganese in water is in the form of Mn^{2+}. In oxygenated waters, manganese probably exists in its higher oxidation states with Mn^{3+} or Mn^{4+} and its solubility is limited by equilibria with MnO_2 (Thamdrup and Dalsgaard, 2000). Water quality analysis of dissolved manganese confirmed that the concentration of Mn^{2+} was similar in both feed and concentrate waters (Fig. 7 (b)). This implies that the anoxic condition increased the solubility of manganese with lower fouling than the oxic condition. Therefore, future research should evaluate the filtration performance caused by both manganese oxide and
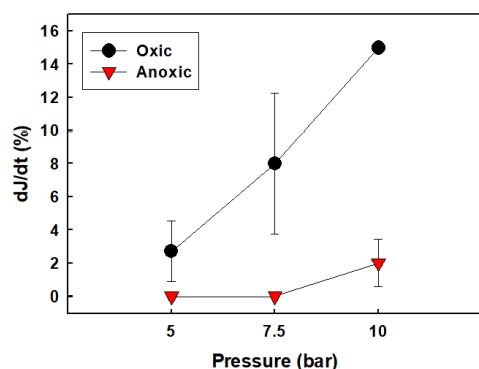
Fig. 8 Comparison of the flux decline rate (FDR) under oxic and anoxic conditions in various operating pressures. Iron concentration was maintained at 10 mg/L. The compaction was over 6 hours, and each FDR was calculated after 30 min of filtration

dissolved ions at similar operating conditions in MAR systems.

3.3.2 Operating under different pressures

Anoxic NF filtration showed lower membrane fouling for applications in the post MAR process since the NF showed a lower flux decline rate. Therefore, anoxic NF filtration under different pressures was applied to evaluate the influence of applied pressure on anoxic NF filtration. By producing low membrane fouling, a more precise simulation of reverse osmosis (RO) performance was expected.

In this section, the FDRs of both anoxic and oxic conditions were determined for each fouling run. Both oxic and anoxic filtration conditions increased linearly with an R^2 of 0.99 and 0.75, respectively (Fig. 8). Specifically, FDR increased sharply as applied pressure increased in the oxic condition, while it increased only slightly in the anoxic condition. The FDRs under different operating pressures were in very good agreement with experimental observations, which demonstrated that the anoxic condition could have lower membrane fouling due to less cake resistance on the membrane surface.

4. Conclusions

Membrane technology is widely used as the main post-treatment to remove major dissolved ions, but also particles such as iron oxides. An oxic condition during direct NF filtration showed much higher flux decline than for the anoxic condition due to deposited foulants on the membrane surface as iron oxides. In this research, we suggested anoxic NF membrane filtration as a feasible new protocol tool to alleviate membrane fouling. The primary findings from this study are summarized as follows:

- NF fouling under anoxic filtration conditions showed a more stable filtration performance than oxic conditions due to the NF crossflow filtration mode having different impacts on iron oxides and ions.
- Under cross-flow filtration mode, most of the larger

iron particles passed away from the membrane surface, and the smaller particles or ions were deposited or passed through the membrane surface.
- Analysis of membrane surface characteristics by employing SEM and XPS confirmed that the oxic filtration condition formed many more iron oxide particles and significant increased oxygen content, which caused significant membrane fouling.
- The anoxic filtration mode is also useful for other foulants such as manganese or different operating conditions.

Future improvements to the direct anoxic NF filtration in MAR system for indirect potable reuse, such as cost-effectiveness evaluation, feasibility test in real systems and optimizing operation, would make it competitive with the rapid-oxidation and filtration processes as a post-treatment in MAR processes.

Acknowledgments

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