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## Fouling reduction by formation of iron oxide cake layer on the pretreatment membrane in an SWRO plant

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### ABSTRACT

There has been a recent increase in the interest for desalination technologies, and this has also brought attention to the importance of pretreatment processes. Applying the appropriate pretreatment processes makes it possible to improve the efficiency of reverse osmosis, as the primary treatment, as well to improve the output of the entire desalination process. Although pretreatment methods using microfiltration and ultrafiltration, which are able to produce a permeate with stable qualities, have been actively considered, fouling inevitably occurs in these membranes, so it is important to actively investigate methods to reduce fouling. In this study, iron oxide was used to apply a pretreatment process in order to reduce membrane fouling in a seawater reverse osmosis plant since iron oxide has a high removal efficiency for dissolved organic carbons in seawater which has a high ionic strength. Powdered iron oxide was used to form a cake layer on the surface of the dynamic membrane in order to improve the membrane performance, and as a result, an additional, temporary intensive process will not be necessary when there is an algal bloom. This study investigated whether the dynamic membrane filtration system could operate in a stable manner without additional injection by forming a cake layer with iron oxide on the surface of the membrane at the beginning of the operation. The results indicate that stable operation was in fact achieved, even when the backwashing flow rate was applied at 0.67 Q compared with filtration flow rate (Q) without aeration. This is because the membrane fouling loading was reduced by the presence of the iron oxide, and the membrane performance improved as a result. This method is thus expected to increase the recovery rate of the pretreatment processes and to reduce maintenance costs.

Keywords: Iron oxide; Dynamic membrane filtration; Pretreatment; Fouling; Desalination

### 1. Introduction

The output of a seawater reverse osmosis (SWRO) process is directly related to the quality of the seawater that flows into the SWRO membrane. In other words, fouling also occurs in reverse osmosis (RO) membranes, as has been shown in low-pressure microfiltration (MF) and ultrafiltration (UF) membranes. In particular, fouling is one of the most important issues in the SWRO process because it can reduce the flux and increase the operational energy and cost. Regarding foulants, organic matter is adsorbed on the membrane surface, even at low concentrations, because it has particular physicochemical properties to cause severe fouling [1], and furthermore fouling be accelerated by the effects of changes in the pH levels and by the presence of divalent cations under certain conditions [2].

Although such fouling in the SWRO process can be reduced by implementing a variety of methods, effective and suitable pretreatment technologies still need to be developed. Some examples of pretreatment processes in the SWRO process include dual media filtration with precipitation and coagulation, dissolved air floatation, and low-pressure membrane filtration (such as MF and UF mentioned above) [3–6]. However,

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membrane processes using low-pressure membranes such as MF and UF have recently gained widespread consideration for use as pretreatment processes for desalination. Low-pressure membranes have a pore size of approximately 0.01–0.1 µm and can effectively remove particulate matter, colloidal matter, organic matter, and microorganisms present in seawater. In addition, low-pressure membranes are available for use with various materials and conditions, including MF/ UF, submerged/pressurized, polyvinylidene fluoride (PVDF), and polyethersulfone (PES), and have been implemented with various methods for each of these conditions [7–11].

However, fouling is an inevitable phenomenon for low-pressure membranes that are applied as pretreatment for desalination. The accumulation of foulants on the surface clog the pores of the membrane, and the resistance to the permeate water flow through the pores increases as fouling further develops, causing a decrease in permeate flux or an increase in transmembrane pressure [12]. In particular, fouling was recently reported to cause severe problems in low-pressure membranes for pretreatment and in SWRO membranes by releasing a large amount of transparent exopolymer particles, polysaccharides, and toxic substances discharged by algae from red tides or harmful algae blooms [3,21–23].

An increasing number of studies have investigated the effects of membrane fouling caused by algae or algal by-products. According to a study by van den Brink et al. [13], fouling rate by alginate increased due to the cake layer formation that was promoted on the membrane surface when the calcium concentration of the inflow water increased by introducing sodium alginate to the model foulant. On the other hand, an increase in the ionic strength resulted in a tendency for alginate fouling to decrease at high calcium concentrations. This was because calcium reduced the relative effect on alginate fouling as the competition to occupy the binding position in the alginate molecules between monovalent and divalent cations (calcium) increased due to the high ionic strength. Ladner et al. [14] observed the effect that a shear force on the surface of MF and UF membranes had on fouling by seawater algae. Their results indicated that membrane fouling increased rapidly due to the destruction of algal cells under a high shear force. In addition, the removal efficiency of the organic matter decreased because the algal by-products that were released were smaller than the pore size of the applied membranes. Studies on hybrid systems combining coagulation and membranes have also increased, but there remains a need to derive the optimum coagulation conditions in order to apply MF and UF membranes as pretreatment for desalination [15]. This is because inorganic fouling can take place on the posterior RO membrane when an excessive amount of coagulant is injected.

This study aimed to apply iron oxide, instead of coagulant, to improve the efficiency of the removal of organic matter and to mitigate the fouling of low-pressure membranes in pretreatment processes for desalination. Several studies have demonstrated the adsorption capacity of iron oxide for organic matter under natural conditions [16,17]. The main adsorption mechanism between organic substances and iron oxide is known as a surface complexation–ligand exchange reaction [16–19].

It is difficult to apply activated carbon, which is mainly used as an adsorbent in water purification processes, to seawater pretreatment processes because its removal efficiency for organic matter greatly decreases in seawater. Conversely, iron oxide is the most characteristic in that its adsorption capacity increases by the screening effect of the electrostatic interaction in seawater with a high ionic strength [18,20–22].

Therefore, this study selected powdered iron oxide using adsorption isotherm experiments in seawater, with red tides as a severe condition, the performance was evaluated for the membranes combined with iron oxide. Moreover, this study used a dynamic membrane filtration system with powdered iron oxide to show its excellent responsiveness to red tides that temporarily occur in SWRO plants, with a consideration of the economic feasibility of its use, and aimed to derive the application method for iron oxide and the operating conditions in normal seawater by conducting a continuous operation.

### 2. Methods and materials

### 2.1. Adsorption isotherm experiments

This study selected iron oxide from among three types of iron oxides by conducting adsorption isotherm experiments. Goethite (FeOOH), hematite (Fe<sub>2</sub>O<sub>3</sub>), and magnetite  $(Fe_3O_4)$  were purchased from Ewic (Korea) as industrial iron oxide samples and were then tested. This study used artificial seawater total dissolved solids (TDS) 35,000 mg/L and deionized water as the feedwater. The dissolved organic carbon (DOC) was maintained at 10.0 mg/L by injecting sodium alginate (99%, Sigma-Aldrich, USA), which was applied as the model foulant of assimilable organic matter (AOM). Only 35 g/L of NaCl (99.9%, Sigma-Aldrich) was utilized for preparation of artificial seawater since alginate molecules are cross-linked and settled down with the divalent ions like Ca2+. Iron oxide was injected into 150 mL of solution at concentrations of 0.1, 0.3, 0.5, 1, 2, 5, 10, 20, 30, and 50 g/L, and it was adsorbed using a shaking incubator at 150 rpm at a temperature of 25°C for 2 h. After the adsorption, the DOC in the solution was filtered using a 0.45  $\mu$ m syringe filter and was then analyzed using a total organic carbon (TOC) analyzer (TOC-LCPH Shimadzu, Kyoto, Japan).

### 2.2. Stirred cell test

As shown in Fig. 1, this study evaluated the effect of the selected iron oxide on the membrane process by conducting the stirred cell test. A PVDF membrane with a pore size



Fig. 1. Schematic diagram of the stirred cell test.

of 0.1  $\mu$ m was applied, artificial seawater TDS 35,000 mg/L using NaCl was prepared as feedwater, and DOC was maintained at 12 mg/L by injecting sodium alginate. Iron oxide was adsorbed at a concentration of 2 g/L for 10, 30, 60, 120, and 180 min, and it was rapidly stirred using a jar tester at 180 rpm for 1 min and then slowly stirred at 70 rpm for each adsorption period. After agitation, the feedwater and the iron oxide were fed into stirred cells. 150 mL of the introduced feedwater was filtered at a constant pressure of 0.5 bar using N<sub>2</sub> gas, and the evaluation was carried out by comparing the flux decline rate. In addition, this study aimed to evaluate how iron oxide had coated the membrane surface by conducting an scanning electron microscope (SEM) analysis after drying the applied membranes.

### 2.3. Dynamic membrane filtration

A UF process combined with iron oxide was constructed, as shown in Fig. 2. In order to determine the injection method and the conditions of the iron oxide in normal seawater, the feedwater was constantly maintained at a TDS of 35,000 mg/L and a DOC of 1.3–1.5 mg/L using bay salt and NaCl. This study used an outside-in type pressurized hollow fiber membrane made from PVDF. It has a normal pore size of 0.1  $\mu$ m and an effective surface area of 0.1 m<sup>2</sup>.

The feedwater was input into the adsorption reactor using a pump controlled by a level switch and was reacted with iron oxide at room temperature ( $20^{\circ}C \pm 2^{\circ}C$ ) for 30 min of hydraulic retention time. Powdered iron oxide (goethite) was purchased from Ewic (Korea) and was then introduced from the concentrated iron oxide tank into the adsorption reactor according to the experimental conditions. The iron oxide was injected into the adsorption rector at concentrations of 0.1, 0.5, 1.0, and 2.0 g/L, and the iron oxide and feedwater were flown to the membrane module after being stirred with seawater using an agitator in the adsorption reactor.

Filtration was performed for a certain period of time (0, 30, 60, and 120 min) to coat the iron oxide on the membrane



Fig. 2. Schematic diagram of the dynamic membrane filtration process combined with the iron oxide adsorption process.

surface only at the beginning of the operation. The process was constructed so that continuous operation could be performed with filtration, backwashing, and draining. The membrane system was operated in the dead-end filtration mode with permeate flux of  $1.5 \text{ m}^3/\text{m}^2$ ·d, and the filtration was performed for 30 min followed by backwashing without air injection to prevent the iron oxide coated on the membrane surface from desorbing.

The membrane recovered to its initial state by cleaning in place (CIP) after finishing the experiment for one condition. CIP was performed by using 0.5% sulfuric acid and 0.5% sodium hypochlorite for 3 h each, and then sufficiently rinsed. The experimental apparatus was automatically operated using a programmable logic controller.

### 3. Results and discussion

### 3.1. Adsorption isotherm experiments

# 3.1.1. DOC removal efficiency according to the concentration of iron oxide

Fig. 3 shows the DOC removal efficiency according to the type of iron oxide and the injection concentration in seawater and deionized water. Iron oxide reached equilibrium in 30 min because it was rapidly adsorbed, and then it was further adsorbed for 2 h to ensure a sufficient reaction.

As shown in Figs. 3(a) and (b), the three types of iron oxides showed a higher DOC removal efficiency in seawater than in deionized water. Goethite had the largest specific surface area of  $37.5 \text{ m}^2/\text{g}$  and showed a similar DOC removal



Fig. 3. DOC removal efficiency according to injection concentrations by iron oxide: (a) seawater and (b) deionized water.

rate as hematite in deionized water, but it showed the highest organic matter removal efficiency in seawater because iron oxides are frequently adsorbed in the presence of a high ionic strength. Adsorption frequently occurs at a high ionic strength due to the decrease in repulsion between the alginate molecules and the repulsion between the iron oxide and alginate molecules. This stands in contrast with activated carbon, which has a decreasing organic matter removal efficiency in seawater due to the high ionic strength [22].

### 3.1.2. Freundlich isotherm

The Freundlich isotherm is determined by the heat of adsorption that exponentially decreases with the degree of surface cover to the Langmuir isotherm [23]. The relationship between the amount of adsorbent and the solution concentration is defined as  $q_e = K_F C_e^{1/n}$ , and it can be transformed into a linear equation by taking the logs of both sides.

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \tag{1}$$

In this case,  $q_e$  is the amount of adsorbate per unit weight of adsorbent (mg/g),  $K_F$  and 1/n are the Freundlich constants, and  $C_e$  means the equilibrium concentration in the solution (mg/L). The Freundlich isotherm constant ( $K_F$ ), as a scale mg<sup>1-(1/n)</sup>L<sup>1/n</sup>g<sup>-1</sup> of adsorption capacity, indicates a favorable adsorption capacity as it increases, and *n* indicates the size of the adsorption kinetics. In general, adsorption occurs easily



Fig. 4. Freundlich isotherm plots of DOC by powdered iron oxide: (a) seawater and (b) deionized water.

when n is larger than 2, and adsorption hardly occurs when n is smaller than 1 [24].

The results for the Freundlich isotherm are shown in Fig. 4 and Table 1.  $K_F$  indicates a favorable adsorption capacity as its value increases. As shown in Table 1, all three types of iron oxides show larger  $K_F$  values in seawater than in deionized (DI) water. In addition, goethite has a larger  $K_F$  value and shows superior adsorption strength to the other iron oxides. This occurs because the adsorption capacity conversely increased by the screening effect of the electrostatic interaction in seawater with a high ionic strength [18,20–22].

It is known that adsorption occurs well when 1/n values representing the adsorption strength in the Freundlich isotherm range from 0.1 to 0.5 and that it hardly occurs when they are larger than 2 [25]. Except for goethite, all 1/n values obtained from the adsorption experiments of this study are greater than 2 in seawater. Although adsorption does not easily occur at these values, among the three types of iron oxides, goethite showed the highest adsorption strength because it had relatively low 1/n values.

### 3.2. Stirred cell test

The graph in Fig. 5 shows a decrease in flux according to the adsorption time by injecting goethite as an adsorbent, which was selected in the adsorption isotherm experiments at a concentration of 2 g/L. Concerning the non-adsorbed

#### Table 1

Parameters calculated from the Freundlich isotherm model

	Seawater			DI water			
	K <sub>F</sub>	1/n	$R^2$	K <sub>F</sub>	1/n	$R^2$	
Goethite	0.1313	1.9235	0.9947	7.917E-05	4.4643	0.9971	
Hematite	0.0010	2.6350	0.9361	4.487E-04	3.5079	0.9581	
Magnetite	0.0017	3.4302	0.9910	4.578E-16	14.6000	0.7107	

 $J/J_0$ 



Fig. 5. Flux reduction trends for the samples with different adsorption times (12 mg/L initial alginate and 2.0 g/L iron oxide loading).

feedwater, the flux rapidly decreased at the beginning of the filtration because organic matter exists in a coil shape or spherical shape in seawater with high ionic strength, and the accumulation of organic matter on the membrane surface is caused by a decrease in the electrostatic repulsion force between the organic matter and membranes [2]. The relatively thick and compact organic layers increased the hydraulic resistance and then rapidly reduced the permeate flux. On the other hand, regarding the feedwater adsorbed with iron oxides for 10 and 30 min, the flux decline rate was considerably mitigated. When adsorbing for more than 60 min, over 80% of the initial flux could be maintained.

Table 2 shows the DOC concentrations before and after filtration according to the adsorption time. The iron oxide was quickly adsorbed because the DOC removal efficiency rapidly increased to 30 min and then did not show clear differences when adsorbing goethite at a concentration of 2 g/L. Also, removal by the iron oxides accounted for a large portion of DOC removal because there was little difference between the DOC before and after filtration. These results indicate that the foulants were effectively removed by the iron oxides, and thus the membrane fouling potential decreased.

SEM images of the membrane surface were taken after filtration to examine how the iron oxides had been coated on the membrane surface. As shown in Fig. 6, the membrane was fouled by organic matter after filtering alginate (as compared with a pristine membrane), and this resulted in a flux decline. When filtering with iron oxides, thin and long iron oxides form cake layers on the membrane. Such cake layers physically prevent organic matter from fouling the membrane by working as a filtration layer on the surface, and some organic matter can be adsorbed into the iron oxides. Such effects rapidly reduce fouling due to the dynamic membrane filtration of iron oxides.

### 3.3. Dynamic membrane filtration

### 3.3.1. Influence by the initial cake formation

Based on results of the adsorption isotherm experiments presented above, this study selected goethite as an adsorbent and aimed to derive a method to combine goethite with a pretreatment of UF membrane in normal seawater conditions.

As shown in Fig. 6, the decline in fouling was first evaluated by coating iron oxides on the membrane surface before starting operation because iron oxides decrease the fouling potential by forming cake layers on the membrane surface. Moreover, when coating iron oxides, excluding the control group, aeration was not applied to backwashing to preventing these from being desorbed. Fig. 7 shows the trans-membrane pressure (TMP) graph of the UF membrane when forming cake layers for each iron oxide concentration for the first 30 min and injecting iron oxides at the concentration for the first 1 min during the filtration cycle. When forming cake layers at a concentration of 0.1 g/L for 30 min, the results are similar to those without injecting iron oxides, and a decline in fouling was not observed. However, the TMP increasing rate decreased at a concentration of more than 0.5 g/L when forming cake layers and injecting iron oxides at the beginning of the filtration.

Fig. 8 shows the TMP according to the cake formation time at the beginning of operation. The iron oxides were injected at a concentration of 2 g/L and were not injected further after forming the cake layer. Although TMP reached 1 bar after 120 h without forming cake layers, the TMP increase rate was alleviated, and the system was operated for more than 150 h when forming cake layers for 10 min. However, when forming cake layers over 30 min, the TMP increasing rate was substantially alleviated, and the system was then operated for more than 170 h. This was because the iron oxide cake layers that formed at the beginning of operation not only adsorbed and removed organic matter but also reduced foulants flown to the membrane surface or membrane supply.

### 3.3.2. Effect according to backwash conditions

This study evaluated how the membrane surface coated with iron oxides was affected by the backwash conditions, including backwash flux and time (Fig. 9). When draining without backwashing, the TMP reached 1 bar faster than the control condition without the iron oxide coating. When backwash was performed for 15, 30, and 60 s at a backwash flux of 1 m/d, the membrane system was operated for 130 h. When performing backwash for 15 s, the TMP increase rate was not greatly alleviated compared with the control condition (120 h). However, when backwashing for more than 30 s, the membrane system was operated for over 180 h, and then the fouling rate greatly decreased. Thus, the need to remove foulants was prevented by the iron oxide cake layers formed at the beginning of the operation through backwashing at more than a certain strength.

When performing backwash for 60 s at backwash volume of 1.5 m/d, the TMP was lower than that at backwash volume of 1.0 m/d, but the TMP increased after 120 h. Although the TMP increase rate is low at the beginning of the operation with a strong backwash, it took a shorter amount of time to reach the inflection point, which means that the fouling rate increased. This is considered to be the case as a result of the iron oxides that had formed on the membrane surface being desorbed.

Table 2

DOC before and after filtration in the stirred cell test

	Raw water DOC	Hydraulic retention time (min)						
		10	30	60	120	180		
DOC (mg/L) before filtration (removal, %)	11.8 mg/L	5.191 (56.0%)	3.182 (73.0%)	2.745 (76.7%)	2.632 (77.7%)	2.586 (78.1%)		
DOC (mg/L) after filtration (removal, %)		5.24 (-0.4%)	3.222 (-0.3%)	2.74 (0%)	2.583 (0.4%)	2.592 (-0.1%)		



Fig. 6. SEM image for membrane surface with and without iron oxide adsorption. (a) Virgin membrane (×5,000), (b) virgin membrane (×20,000), (c) fouled membrane with alginate (×5,000), (d) fouled membrane with alginate (×2,000), (e) fouled membrane with alginate and iron oxide (×5,000), and (f) fouled membrane with alginate and iron oxide (×50,000).

### 4. Conclusions

The pretreatment processes are very important in that they can determine the efficiency and economic feasibility of the entire desalination process. In particular, red tides occur frequently and cause serious damage to desalination equipment. Thus, pretreatment processes have become more important. This study aimed to improve the efficiency of the pretreatment processes by evaluating the performance of the dynamic membrane filtration combined with iron oxides for pretreatment and then deriving the corresponding operating plans.

Goethite was selected in this study since it had the best adsorption capacity of the three types of iron oxides tested in the adsorption isotherm experiments. Then, a stirred cell test was conducted. According to the flux decline rate and SEM images of the membrane surface, fouling was alleviated. Iron oxides particularly show a high removal efficiency of the adsorption to organic matter, causing irreversible fouling in the MF/UF membrane processes because they are capable of easily adsorbing polymer alginate [26]. In addition, the TMP increase rate of the MF/UF membranes could be alleviated by forming iron oxide cake layers on the membrane surface. This made it possible to improve the energy efficiency of the pretreatment processes and to reduce the maintenance costs by postponing the CIP cycle. Furthermore, this made it possible to operate the membrane system to backwash without aeration and to obtain a high recovery rate through stable operation, even in backwashing with 0.67 *Q* compared with the permeate flux *Q*.



Fig. 7. Changes in TMP according to injection concentration of iron oxides in the initial cake formation.



Fig. 8. Effect of TMP according to the initial cake formation time.



Fig. 9. Changes in TMP according to the backwashing conditions.

Therefore, it will be possible to improve the overall efficiency of the desalination processes by reducing the fouling of not only the pretreatment of the MF/UF membranes but also that of the RO membranes.

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