



Evaluation of direct osmosis at high salinities method by renewable lactate salts as draw solutes in RO membrane cleaning

Mostafa Narimani^a, Mehdi Ardjmand^{b,*}, Seyed Mostafa Tabatabaee Ghomsheh^c,
Ali Akbar Safekordi^a

^aDepartment of Chemical Engineering, Science and Research Branch, Islamic Azad University, Tehran, Iran,
emails: mostafa_narimani2005@yahoo.com (M. Narimani), safekordi@sharif.edu (A.A. Safekordi)

^bDepartment of Chemical Engineering, South Tehran Branch, Islamic Azad University, Tehran, Iran, email: m_arjmand@azad.ac.ir

^cDepartment of Chemical Engineering, Mahshahr Branch, Islamic Azad University, Mahshahr, Iran, email: m.mahshahruni@gmail.com

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ABSTRACT

The objective of the study was to develop the cleaning performance of the RO membrane with direct osmosis at high salinities (DO-HS) method. Lactate salts were selected as new draw solutions in order to create the required osmotic pressure. This technique is a new backwash method by intermittent injection of the high salinity solution without stopping of high pressure pump. DO-HS trials were carried out with an RO pilot system which was operated on site with the synthetic groundwater as the raw feed. Different operating conditions for DO-HS treatment in the actual process were investigated. A short injection of feed water with increased salt concentrations (1.5 M) with an associated osmotic pressure of 105.32 bar overcomes feed pump gauge pressure and reverse osmosis shifts to direct osmosis, leading to a permeate backwash stream through the RO membrane. The results showed that the fouling could be almost fully reversible and the membrane needed to be cleaned for about 2 min (12 pulse injection) because of the appearance of all salts on membrane surface at the fouling time of 10 d. Most importantly, the DO-HS technology is very effective in keeping the membrane continuously clean and ensuring stable permeate production.

Keywords: Direct osmosis; Draw solution; Membrane; Lactate salt

1. Introduction

Reverse osmosis (RO) is a pressure-driven membrane process and has been widely applied in water treatment processes as a promising technology. However, RO membrane fouling is a global issue, which limits its operating flux, decreases water production and increases power consumption [1]. Cleaning methods include physical and chemical cleaning. Physical methods are based on mechanical forces to dislodge and remove foulants from the membrane surface such as forward/reverse flushing and air sparging [2–4]. Foulants remaining after physical cleaning need to be removed by a chemical cleaning, depending on chemical reactions to weaken the cohesion forces between the

membranes and foulants [5]. However, these approaches will cause low effectiveness of production due to the downtime of frequent RO operation stoppage and create environmental issues related to the waste chemical disposal [6]. Direct osmosis (DO) technology has been increasingly attractive for backwash cleaning of RO because it is highly efficient and environmentally friendly technique [7] which has been extensively reviewed [8–10]. Recently the new direct osmosis at high salinities (DO-HS) technology offered a novel backwash approach for on-line membrane cleaning in RO operation without stopping the RO pump [11,12]. There was no interruption of RO operation in a new DO cleaning technology where a high salinity (HS) solution was injected

* Corresponding author.

into the feed water over a few seconds that could induce multiple cleaning mechanisms composed of fouling lifting and sweeping as well as bio-osmotic shock and salt dissolve shock, thus could provide high cleaning efficiency [13].

In the past few years, various draw solutes were investigated for DO applications [14–16]. Inorganic salts (e.g., NaCl, MgCl₂) are mostly studied as draw solutions, which can create reasonable water flux but their large-scale applications are severely constricted due to the high energy cost involved in the draw solution recovery with current technologies [17]. Recently, synthetic draw solutes with the controllable molecular size have been investigated to solve the recovery problems and salt leakage, including poly(amidoamine) (PAMAM) functionalized with –COONa (PAMAM-COONa) [18], poly(sodium styrene-4-sulfonate-co-n-isopropylacrylamide) (PSSS-PNIPAM) [19], poly(acrylic acid) sodium salts (PAA-Na) [20], etc. These synthetic draw solutions exhibit a high water flux and a low salt leakage in FO process, but their practical applications in FO remain an uncertainty because of their non-renewable property. So draw solution of natural compounds without regeneration needed could be a desirable choice. Therefore, it is preferable to use the salt that produces a superior osmotic pressure and solve the salt leakage and recovery problems.

In this study, sodium lactate salt was used as draw solute for DO backwash of RO system. Compared with other salts, the lactate salts can release ions in the aqueous solution, resulting in a superior osmotic pressure, whereas its larger molecular size may also render a lower solute leakage in DO process. The objective of this study aimed at further developing the DO-HS backwash cleaning technique for RO in reclamation of the synthetic groundwater. Effects of major parameters and operating conditions were systematically investigated and optimized via a pilot plant operation for future practical implementation.

2. Experimental setup

2.1. Materials

Lactate salts (sodium lactate and calcium lactate) purchased from Sigma-Aldrich (China). Sodium chloride (NaCl, ≥99.5%) was acquired from Sinopharm Chemical Reagent Co., Ltd. Deionized (DI) water was produced in laboratory by a Wuhan Pin Guan Ultrapure LAB purification system and utilized in this work. Typically cellulose acetate coated with polyamide flat-sheet RO membranes were employed (Zhejiang Mei Technology Co., Ltd., China). Some specifications of RO membrane are given in Table 1.

In this paper, synthetic groundwater was prepared according to the ratio of each ion in groundwater in south of Iran, and the main ions in the synthetic solution and their corresponding concentrations are shown in Table 2.

2.2. Pilot system description

Schematic diagram of the RO system assisted by DO-cleaning in a laboratory-scale cross-flow test unit is shown in Fig. 1. The membrane test unit was equipped with pumps, tank (super duplex stainless steel) for feed, saline solution container, air regulator (R07-200-RNKA), feed

Table 1
Membrane specifications

Type	Flat-sheet membrane
Material	Cellulose acetate coated with polyamide
Active area (m ²)	10
Feed spacer thickness (mm)	20
Permeate flow rate (L m ⁻² h ⁻¹)	60.2
Operation pressure (bar)	15–50
Operation pH	2–10
Stabilized salt rejection (%)	99.5

Table 2
Main composition of the synthetic groundwater

Parameter	Composition (ppm)
Mg ²⁺	72.2
Ca ²⁺	62
Fe ⁺³	0.371
Na ⁺¹	663
Cl ⁻	567
SO ₄ ²⁻	1,241
NO ₃ ⁻	24
Al ³⁺	0.15
Ba ²⁺	0.09
Cu ²⁺	0.18
Ni	123

temperature gauge, membrane chamber and membrane inlet and outlet gauge pressure. A HS tank (including sodium lactate) was added for the process of DO-HS. The feed solutions were recirculated by high pressure pump (Wanner Engineering, Inc., USA, F20-111-2400/B) and a pulse of high concentration solution injected into feed water after the membrane was contaminated by foulants. Feed tank is capable of holding about 20 L of the solution. In order to discharge the solution after each test, the bottom of the tank is made bowl-shaped and located on center. Tank outlet is equipped with a drain valve to fully drain the solution from tank. Since by pumping the fluid, tank's temperature rises, in order to control the feed temperature, a cooling coil is placed vertically in the tank. This tank to provide pump's net positive suction head, have been installed at the proper height. Both permeate and retentate were recirculated. The cross-flow velocity and the operating pressure were adjusted by using a bypass valve in conjunction with a back-pressure regulator.

2.3. Membrane cleaning theory

DO-HS cleaning technology can be initiated by a high osmotic pressure solution injected for few seconds into the suction site of feed pump. This slug of draw solution moves along the membrane elements in the pressure vessel and changes the process from RO to forward osmosis. The DO-HS method is applicable in brackish, wastewater or seawater RO plants, because it is not dependent on raw water osmotic pressure.

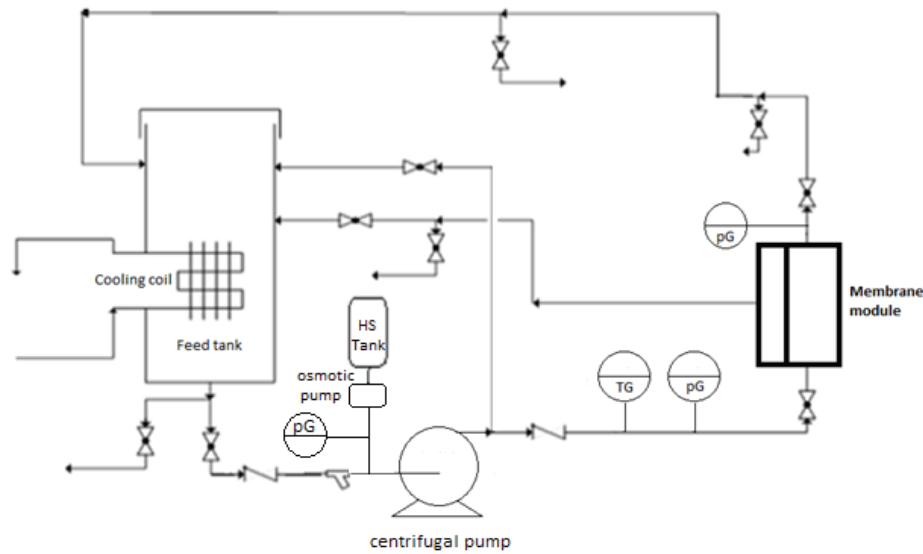


Fig. 1. Schematic diagram of RO semi-industrial pilot plant.

For the DO-HS process to be most effective, the permeate pressure has to be kept high enough, and the reject flow valve has to be open to increase the shearing velocity. Practically, two pumps work simultaneously during the DO-HS process – the high-pressure feed flow centrifugal pump and the osmotic pump. Power for the operation of the osmotic pump is obtained from the draw solution. When high osmotic pressure draw solution moves along the membrane, fouling on the membrane become lifting [21].

In practice, the osmotic pressure difference ($\Delta\Pi$) between both sides of the semi-permeable membrane is used to describe the driving force for water transport although it does not need to apply a physical/hydraulic pressure on the membrane during DO process. The theoretical osmotic pressure Π of a solution can be calculated using the Van't Hoff equation [22] as follows:

$$\Pi = nC\varnothing RT \quad (1)$$

where n is the number of ions; C is the salt concentration (M), \varnothing is osmotic coefficient; R is the universal gas constant and T is the temperature (K).

The water passage through the membrane is generally described and theoretically calculated by following equation:

$$j_w = A \cdot \sigma \cdot \Delta\Pi \quad (2)$$

where j_w is the water flux; A is the water permeability constant of the membrane; σ is the reflection coefficient and $\Delta\Pi$ is the osmotic pressure differential, respectively [23].

On contrary, water driven under hydraulic pressure (ΔP) transfers through a RO membrane from the higher salt concentration side to the lower one and water flux can be calculated using Eq. (3):

$$j_w = A(\Delta P - \sigma \cdot \Delta\Pi) \quad (3)$$

The driving force of RO process is the pressure differential denoted in the following expression:

$$\Delta P_{\text{driving}} = \Delta P - \Delta\Pi = (P_f - P_p) - (\Pi_f - \Pi_p) \quad (4)$$

where P_f , P_p , Π_f and Π_p are the feed pressure, permeate pressure, feed osmotic pressures and permeate osmotic pressure, respectively.

The positive driving force drives water from the feed side to the permeate side in a RO process ($\Delta P_{\text{driving}} > 0$) while in the backwash cleaning (DO-HS) a negative driving force is required to drive water from the permeate side to the feed side ($\Delta P_{\text{driving}} < 0$).

By introduction of a high saline solution to the feed side, high Π_f can be easily gained. So higher driving force for backwash cleaning will be achieved. Therefore, this method is called as DO backwash cleaning will continue since Π_f dominates the backwash process [1].

2.4. Water permeability analysis

Permeate flux was obtained volume of the permeate collected (Q) per unit membrane area (A) per unit time (t) and calculated as follows [24]:

$$J = \frac{Q}{A \times t} \quad (5)$$

Permeate samples were collected for analysis and the membrane permeate flux was measured at specified time intervals. In order to obtain the cleaning efficiency of DO-HS method, flux recovery ratio was calculated by the following equation [25]:

$$\text{Flux recovery ratio (\%)} = \frac{j_w - j_{fw}}{j_{iw} - j_{fw}} \times 100 \quad (6)$$

where J_{iw} is the pure water flux before fouling; J_{fw} is the pure water flux after fouling; and J_w is the pure water flux after cleaning.

After sealing the system, the DI water flux at pressure of 10 bar was measured. When the pressure was adjusted, at least certain time passed, the flux measurements were taken. This allowed the membrane to reach a steady state flux. So, at least certain time passed, the volume of water that collected through membrane was recorded. From this data, the pure water flux before fouling was calculated at certain pressure. As the same way, pure water flux of membrane before and after fouling and again after chemical cleaning was measured. All the pure water fluxes were measured under applied pressure of 10 bar and temperature of $25.0^\circ\text{C} \pm 0.1^\circ\text{C}$.

2.5. Characterization

Scanning electron microscopy (SEM) was used to study the surface morphology of membrane before and after of cleaning. SEM images were made with a KYKY-EM3200 instrument with an accelerating voltage of 25 kV. The membrane was then coated with a thin film of gold to minimize sample charging problems.

To analyze roughness of the membrane atomic force microscopy (AFM) was used. The AFM device was DualScope™ scanning probe–optical microscope (DME model C-26, Switzerland). Small squares of the membrane (approximately 1 cm^2) were cut and glued on glass substrate. The membrane surfaces were imaged in a scan size of $10 \times 10\ \mu\text{m}$.

All samples that collected were diluted to suitable levels for analysis. The content of nickel in permeate samples was analyzed by using the AFS-230 dual-channel atomic fluorescence spectrophotometer. Each sample was measured five times, so as to get more accurate results. The salinity was analyzed by using a conductivity meter (1214000, Thermo Scientific Orion, Beverly, MA).

2.6. Osmotic pressure and relative viscosity of draw solution

The osmotic pressures of lactate salts draw solutions with various concentrations (0.5–2.0 M) were determined according to the freezing point depression method by Eq. (7). Osmotic pressures of NaCl solution was also tested as control [25].

$$\pi = \frac{\Delta T}{1.86} \times 22.66(\text{bar}) \quad (7)$$

where ΔT is the temperature difference between freezing points of pure water and the draw solution.

Relative viscosities (η_R) of sodium lactate and its draw solutions with various concentrations (0.5–2.0 M) and temperatures (25°C , 35°C , 45°C and 55°C) against that of DI water (at the same temperature) were determined using Eq. (8) [26]:

$$\eta_R = \frac{\eta_{\text{lactate}}}{\eta_{\text{water}}} = \frac{t_{\text{lactate}} \rho_{\text{lactate}}}{t_{\text{water}} \rho_{\text{water}}} \quad (8)$$

where t_{gluc} and t_{water} (s) are the respective outflow time of the draw solution and DI water, determined using a commercial Ubbelohde viscometer with temperature maintained by a water bath; their densities were measured by a portable density meter (DMA 35, India).

2.7. Membrane fouling and cleaning strategy

Fouling studies with synthetic groundwater as feed at 25°C and pressures of 10 bar were conducted to assure steady state RO operation. Other than measuring the water fluxes produced in RO tests, the conductivities were measured per day in order to determine either the solute rejection or the reverse solute transport across the membrane. The conductivities of the feed solution, draw solution and permeate were measured using a conductivity meter (1214000, Thermo Scientific Orion, Beverly, MA), and the solute rejection were then calculated. By calculating the solute concentration before and after each experiment, the solute rejection can be determined using the equation as shown in Eq. (9) for RO experiments [27].

$$\text{Rejection}(\%) = \frac{C_{\text{feed,initial}} - C_{\text{permeate,final}}}{C_{\text{feed,initial}}} \times 100 \quad (9)$$

The experiments for fouling were conducted at time three interval of 10 d. Membrane cleaning protocol includes the following: (1) physical cleaning, which involved recirculating DI water for 20 min with no permeation. (2) Osmotic cleaning, which consisted of 12 pulse of high concentration solution of salt during 2 min followed by physical cleaning. In these experiments, the RO system was adjusted on pressure of 10 bar. Pulse duration should be longer than the residence time for a maximum achievable cycle-averaged permeation rate.

It should be pointed out that several operation conditions such as DO-HS feed flow rate of both draw solution and feed solution, valence state of ions, concentration of HS, the moment to start cleaning and the duration were affected on DO-HS trials. For optimization of operating conditions and the efficiency of DO-HS method on RO fouling control some of them will be considered in the following section.

3. Results and discussion

3.1. Relative viscosity and osmotic pressure of draw solution

To consider the potential of lactate salts as draw solutions comprehensively, the osmotic pressure and the viscosity of their solutions should be investigated, which both have significant impacts on DO cleaning. Generally, a high osmotic pressure of the draw solution can generate a high water flux in DO process, whereas the high viscosity not only leads to high energy consumption for fluid pumping but also causes severe internal concentration polarization (in DO mode).

The physicochemical properties of draw solutes in this study are listed in Table 3.

They are all important properties of the draw solute to determine its final DO performance. Basically, higher molecular weight and higher water solubility of the draw

Table 3
Physicochemical properties of draw solutes

Compound	Molecular weight (g/mol)	Solubility (g/100 g water)	Solution pH (0.4 M, 25°C)
Sodium lactate	112.06	150	8.07
Calcium lactate	218.22	8	7.1
Sodium chloride	58.5	35	7
Glucose	180.06	69	6.67

solute are more desirable, because the former generally corresponds to a lower solute leakage, whereas the latter brings out a higher water flux in the DO process. With regards to the pH value of the draw solution, it needs to be in the range of the membrane tolerance.

Fig. 2 illustrates relative viscosities of sodium lactate, calcium lactate and NaCl solutions as a function of the solution concentration and temperature. As shown in Fig. 2, relative viscosities of three solutions all increase remarkably with the concentration increase, whereas decrease with the temperature increase. But the difference will be greater at higher concentration or temperature. It should be noticed that all viscosities of sodium lactate and calcium lactate solutions with concentration less than 2 M still remain low. Draw solution with a lower viscosity is favorable to enhance the DO performance, this result indicates that sodium lactate draw solution has better DO performance than that of calcium lactate draw solution with the same concentration. Osmotic pressures of glucose, NaCl, and two lactate salt solutions with different concentrations are shown in Table 4. It can be observed that, for all solutions of 0.5 M, osmotic pressures of sodium lactate salt solution is more than that of NaCl and about 2.5 times that of glucose. Generally, lactate salts and NaCl are both electrolytes and can partially or fully dissociate into free ions in the aqueous solution, whereas the glucose (non-electrolyte) cannot.

In general, the draw solution with desirable properties are preferred for DO applications, including high water solubility, appropriate molecular size, high osmotic pressure, low viscosity, etc. However, to consider the potential of any novel draw solutes developed, comprehensive consideration should be taken. A classic example is NaCl, which owns a considerable osmotic pressure and good water solubility, but its small molecular size leads to a severe salt leakage in DO process [26]. Therefore, sodium lactate is suggested as salt for DO-HS cleaning.

3.2. Effect of salt concentration on permeate rate

According to Van't Hoff equation (Eq. (1)) higher feed concentration will induce a larger driving force and a bigger backwash flow rate. In the case of RO cleaning, it was found that a HS with osmotic pressure of 35–100 bar was needed [28] and the corresponding sodium lactate concentration should be 1–2 M.

Sodium lactate solutions with concentration in the range of 1–2 mol/L were studied, so as to obtain a required osmotic

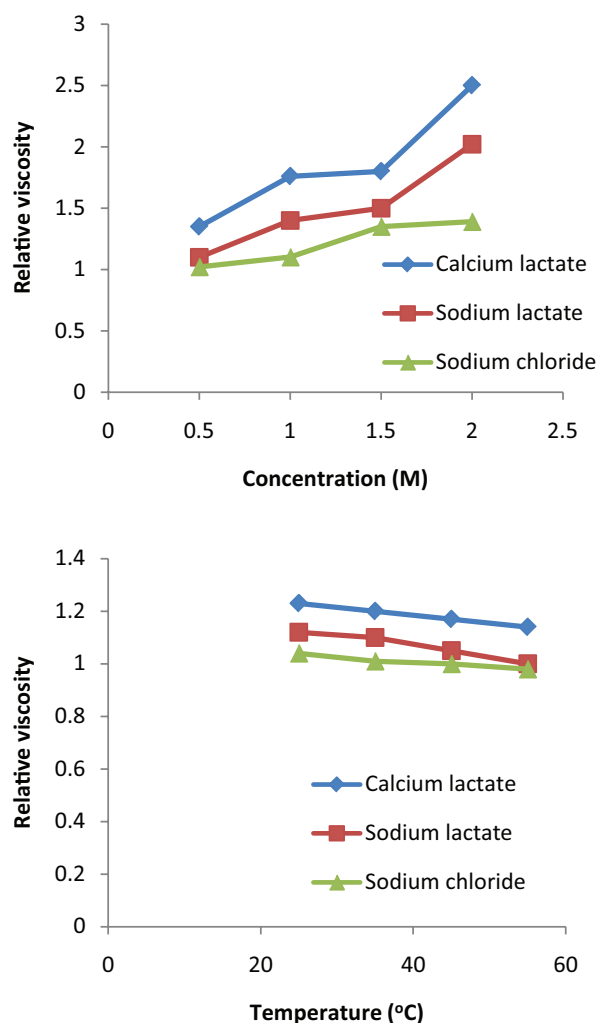


Fig. 2. Effects of concentration (at 25°C) and temperature (0.4 M) on the relative viscosities of draw solutions.

Table 4
Osmotic pressure of two lactate salts, glucose and sodium chloride solutions at different concentrations

Osmotic pressure (bar)				
Concentration (M)	Sodium lactate	Calcium lactate	Glucose	Sodium chloride
0.5	31.23	15.46	10.01	17.43
1	78.3	36.4	33.4	41.85
1.5	105.32	61.2	49.7	65.1
2	114.5	94.3	58.5	100.02

pressure for RO membrane backwash. Osmotic pressure of sodium lactate salt solutions at different concentrations is investigated in Table 4. The effect of HS concentration on permeate rate is shown in Fig. 3. In the case presented, the permeate flux of the DO-HS with different sodium lactate concentrations was calculated based on the backwash flow rate. As shown, the flux increased with osmotic pressure in the range of 1–2 mol/L of this salt solution. While the permeate

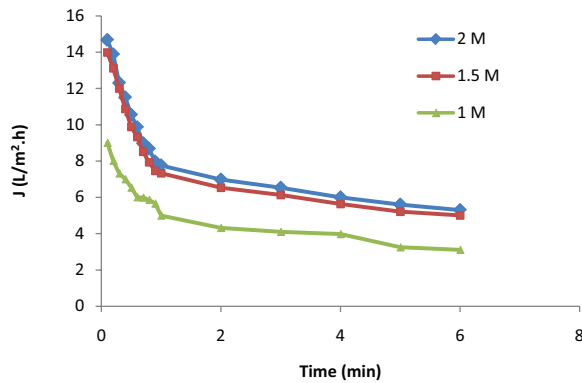


Fig. 3. Effect of HS concentration on permeate rate in DO-HS cleaning.

flux was almost the same with salt concentration increased from 1.5 to 2 mol/L. So in order to economize the consumption of chemical salts, the sodium lactate salt concentration of 1.5 mol/L was selected for DO-HS cleaning processes.

3.3. Effect of DO-HS treatment on RO membrane fouling tendency

Fig. 4 shows the relative permeability (which is the ratio of permeability at the time over that on Day 1) of RO membranes as a function of passing time of the pilot plant operation. It can be seen that the RO fouling rate with the DO-HS treatment was 19%, 30% and 39% of that without DO-HS treatment in 15, 30 and 45 d, respectively. The preliminary results indicated that DO-HS treatment indeed demonstrated a benefit to low RO fouling tendency. In addition, the fresh membranes in the baseline study without DO-HS treatment performed faster fouling rate at the beginning of the plant operation and the fouling rate reduced with the operating time.

3.4. Membrane surface analysis

Analysis by SEM provides a visual and quantitative characterization of the surface of RO membrane after and before DO-HS cleaning. Fig. 5 shows the SEM images of the surface structure of the membrane. Fouled membranes being cleaned for different time were characterized by SEM (Fig. 5). As shown in SEM images, the clean degrees of membrane surface after cleaning for different time increased in the sequence of 1 min < 2 min. A large amount of white crystal substances disappeared in the fouling layer after 1 min cleaning, indicating that the membrane was cleaned to some extent. And the SEM images of the membrane cleaned for 2 min were quite similar to that of the membrane before fouling. The foulants were cleaned with the backwash force by two steps: foulant lifting and sweeping. The thickness of fouling layer on membrane surface was different. Inevitably, it would take different time to loosen up the foulants. So the membrane surface became increasingly cleaner during the 2 min and then leveled off. This confirmed that the best cleaning time for the membrane was 2 min.

AFM images of the virgin and fouled membrane are shown in Fig. 6. These images confirm that the

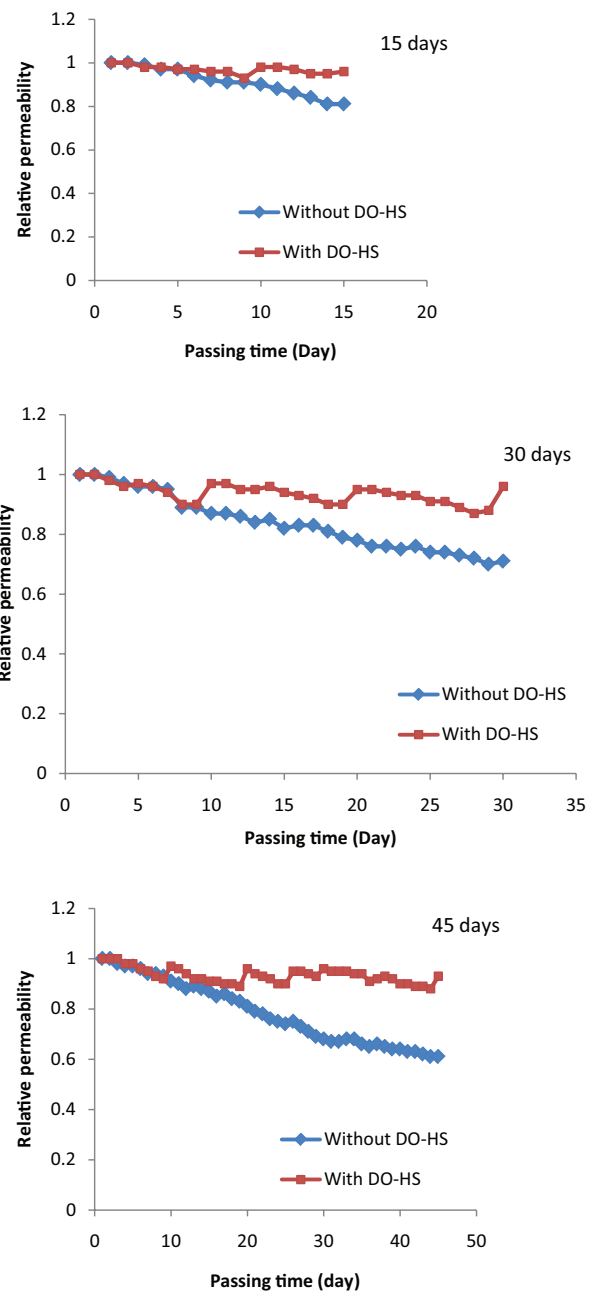


Fig. 4. Effect of DO-HS treatment on RO membrane fouling tendency.

virgin RO membrane has smoother structure than the fouled membrane. The surface roughness parameters of membranes in scan areas of $10 \times 10 \mu\text{m}$ were calculated by DME SPM software and are presented in Table 5. The R_a for the membrane without cleaning was about 25.32 nm and for others with 1 and 2 min cleaning time were 7.5 and 3.9 nm, respectively. The RMS is the root mean square average of height deviations taken from the mean data plane. The high RMS means high surface roughness. As shown in Table 5, the surface roughness of RO membrane with 1 min cleaning time is higher than 2 min.

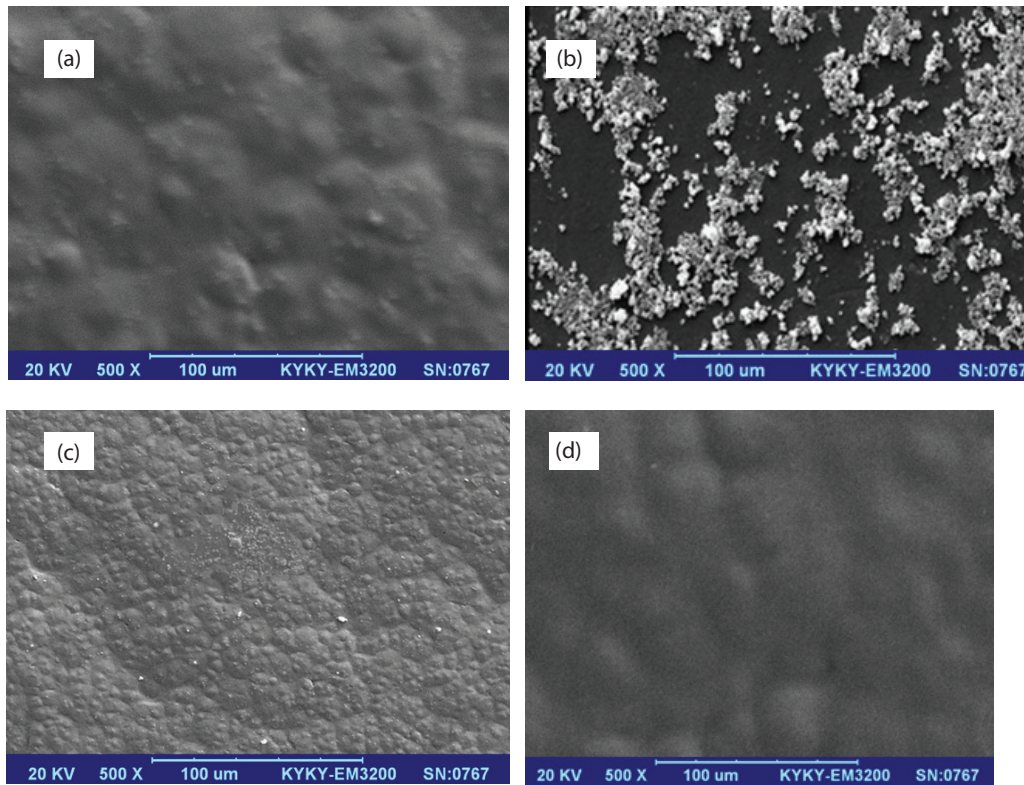


Fig. 5. SEM images of RO membrane samples with different cleaning stages: (a) virgin membrane, (b) membrane without cleaning, (c) membrane cleaning for 1 min and (d) membrane cleaning for 2 min.

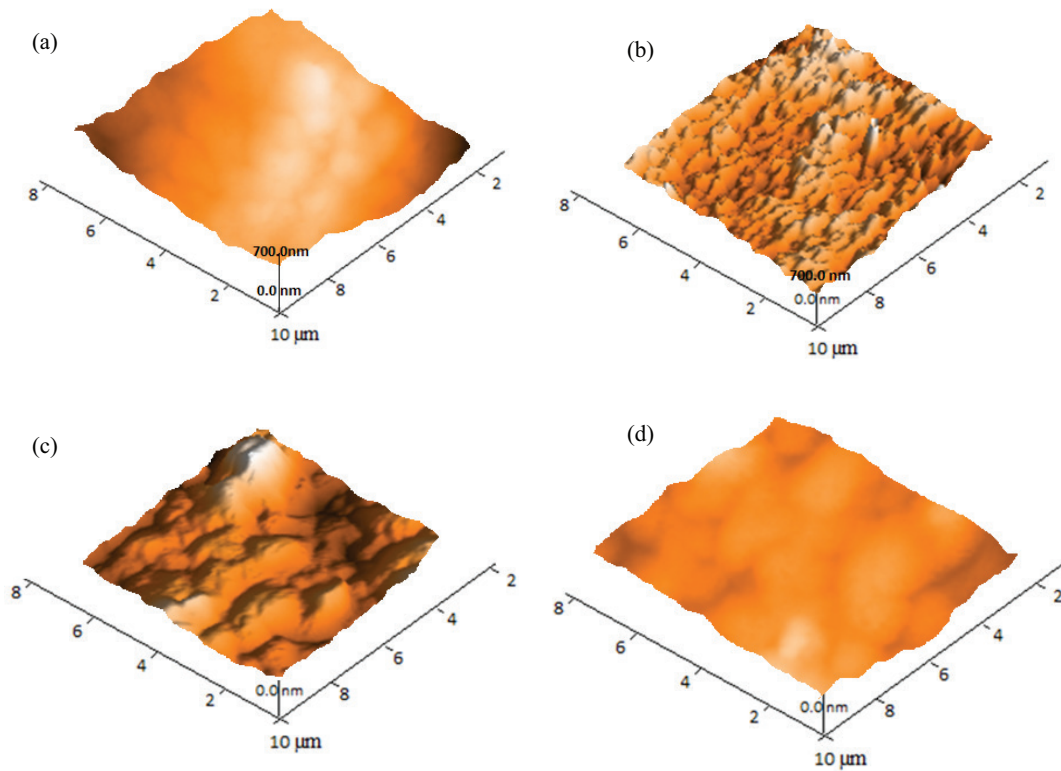


Fig. 6. AFM images of (a) virgin membrane, (b) fouled membrane, and membranes cleaned for (c) 1 min and (d) 2 min.

Table 5
Roughness parameters of membrane surface calculated with DME SPM software

Membrane	R_a (nm)	RMS (nm)
Virgin membrane	3.21	8.2
Fouled RO membrane	25.32	65.02
RO membrane with 1 min time cleaning	7.5	22.82
RO membrane with 2 min time cleaning	3.9	10.2

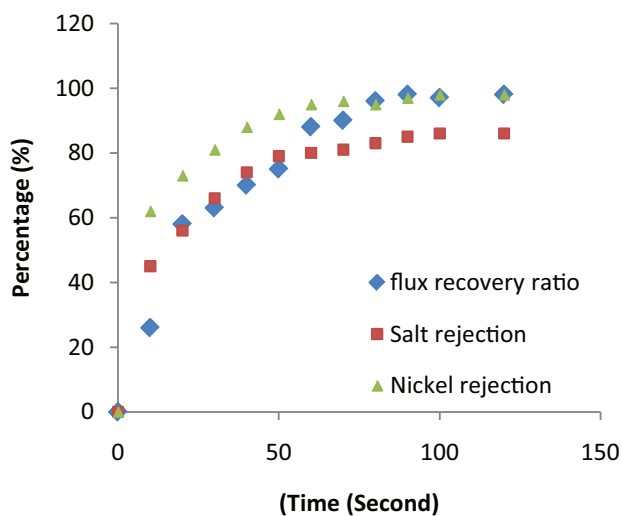


Fig. 7. Membrane flux recovery ratios and rejection with respect to cleaning time.

Table 6
Typical analysis of RO feed and permeate

Parameter	RO feed	RO permeate	NEWater
Mg ²⁺ (ppm)	72.2	12.2	<30
Ca ²⁺ (ppm)	62	5.33	4–20
Fe ³⁺ (ppm)	0.371	0.12	<0.3
Na ¹⁺ (ppm)	663	24.1	<50
Cl ⁻ (ppm)	567	13.43	<20
SO ₄ ²⁻ (ppm)	1,241	1.23	<5
NO ₃ ⁻ (ppm)	24	2.3	<15
Al ³⁺ (ppm)	0.15	0.05	<0.1
Ba ²⁺ (ppm)	0.09	0.03	<0.1
Cu ²⁺ (ppm)	0.18	0.01	<0.05
Ni	123	0.009	<0.02
pH value	7.2	6.7	7–8.5
Conductivity (Us/cm)	553	92	<250
Total dissolved solid (TDS)	432	23	<150
Turbidity (NTU)	0.5	0.1	<5

3.5. Membrane flux recovery ratios and rejection

Membrane flux recovery ratios (FRR) and retention performances were shown in Fig. 7. The FRR increased obviously with cleaning time at the first 1 min and it reached to 60% at the time of 20 s. For the fouled membrane, the salt and nickel rejections were 45.43% and 62.51%, respectively, while for virgin membrane the corresponding values were 86.5% and 98.7%. The slope of the flux recovery ratios diagram in the first 20 s is very high. This is due to fast cleaning of non-organic foulants from the membrane surface during DO-HS process.

The salt and nickel rejections were both increased rapidly in the 20 seconds and attained to a steady status at the time between 1 and 2 min. So after 10 d of fouling operation, it was found that the best cleaning time for DO-HS was 2 min. It could regenerate the membrane in a huge way. Meanwhile, this cleaning method was a membrane eco-friendly cleaning method with the dose of chemical salt reducing to the minimum.

3.6. Analysis of RO feed and permeate

Table 6 shows the typical analysis of RO feed and permeate during the study. RO feed quality was well within the standard limits. RO permeate quality met the requirement of NEWater.

As it is seen, in an interval of 45 d, after three DO-HS cleaning process (per 10 d), the concentration of ions and indicators decreased to the optimal level.

4. Conclusion

The DO-HS method is easy to RO membrane cleaning without interruption of RO operation. In this study, two gluconate salts (sodium lactate and calcium lactate) are employed as novel draw solutes for the DO cleaning. Among the two draw solutions, sodium lactate draw solution (concentration of 1.5 M) with desirable big molecule size, highest water solubility, low relative viscosity and highest osmotic pressure exhibits a comparable water flux to that of NaCl draw solution. Results showed that there is an increase in permeate flux by 12 pulse injection of 1.5 M sodium lactate solution for 2 min in DO process. The removal efficiency of Ni increased with the cleaning time and reached 98% at the time of 2 min. According to this, the fouling could be almost fully reversible (more than 99% permeate water flux recovery) and this method demonstrated a benefit to low RO fouling tendency.

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