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Cleaning strategy of fouled reverse osmosis membrane: Direct osmosis at high salinities (DO-HS) as on-line technique without interruption of RO operation

S.M. Tabatabaee Ghomshe*

Department of Chemical Engineering, Mahshahr Branch, Islamic Azad University, Mahshahr, Iran.

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This paper summarize the critical examination of the cleaning performance of the polyamide RO membrane with DO-HS method as novel backwash approach for on-line membrane cleaning in RO operation. In this work, the effect of pulse injection of salt demonstrate on RO membrane cleaning. A short injection of feed water with increased salt concentrations (8 and 10% NaCl solution) with an associated osmotic pressure of 35 and43 bar overcomes feed pump gauge pressure and reverse osmosis shifts to direct osmosis, leading to a permeate backwash stream through the reverse osmosis membrane. The results illustrate that there is more increasing in water permeate flux and permeability coefficient of RO membrane by pulse injection of 10% NaCl than the other one(8% NaCl solution) .Results observed a rise of the permeability coefficient of membrane, indicating the foulant removal from the membrane. Permeability coefficient of membrane reached to approximately 90 percent after pulse injection in 10% of NaCl. As result salt concentration of 10% of NaCl s that injected to RO system was chosen as the optimal concentration.

Keywords: Membrane cleaning, Direct osmosis, Membrane fouling, Reverse osmosis, Osmotic backwashing

INTRODUCTION

The use of reverse osmosis (RO) technology has grown rapidly through the 1990's and early 2000's. The ability of RO to replace or augment conventional ion exchange saves end users the need to store, handle, and dispose of large amounts of acid and caustic, making RO a "greener" technology. Reverse Osmosis (RO) is a membrane-based demineralization technique used to separate dissolved solids and it has become one of the major technologies for producing potable water throughout the world [1]. Reverse osmosis has become one of the high potential technologies for producing either purify water or to concentrate and recover dissolved solids in the feed water[2]. In addition to providing drinking water with this method, this method is much used in industry. For example the most common application of RO in industry is to replace ion exchange, including sodium softening, to purify water for use as boiler makeup to low- to medium-pressure boilers. Other common applications of RO include: desalination of seawater, generation of high-purity water for pharmaceuticals, generation of ultrapure water for the microelectronics industry and pharmaceuticals, processing of dairy products, waste treatment for the recovery of process materials and so on. In this method, membranes in general act as perm-selective barriers, barriers that allow some species (such as water) to selectively permeate through them while selectively retaining other dissolved species [3]. Specifically, fouling of membranes is the most important problem in reverse osmosis since economics of the process is still highly influenced by membrane fouling rate and effectiveness of fouling control [4]. Membrane lifetime and permeate productivity are primarily affected by concentration polarization and fouling at the membrane surface [5]. In this regard, the performance of a membrane due to the deposition of suspended or dissolved substances on its external surfaces, at its pore openings or within pores decreases. The mechanisms of membrane fouling are adsorption, Pore blockage, gel formation and biofouling [6]. As a result of membrane fouling, membrane resistance increases with time due to accumulation of foulants on membrane surface and inside the membrane [7]. The main results of fouling are: flux decline, permeate quality deterioration and energy consumption increase. Since operating costs of reverse osmosis highly depend on membrane useful life, fouling control is essential for increasing membrane operational life and thus reducing economics of the process [8].the ways for fouling control are feed pre-treatment, operation conditions and membrane cleaning. Good pre-treatment avoid or minimize fouling, but in spite of good pre-treatment, membranes have to be periodically cleaned to remove reversible fouling. Operation conditions i.e. temperature, pressure and crossflow velocity can be considered to reduce fouling [9].Despite the methods that can help to

* To whom all correspondence should be sent:
E-mail: mostafa.tabatabaee@yahoo.com

reduce fouling, membrane cleaning is necessary. Membrane fouling can be mitigated with chemical, physical and physio-chemical. In practice, physical cleaning methods includes sponge ball cleaning, forward and reverse flushing, backwashing, air flushing, Ultrasonic, electrical fields and magnetic fields followed by chemical cleaning methods and are widely used in membrane applications. However, only the chemical cleaning methods are widely applied for RO processes. In chemical cleaning the choice of the cleaning agent is critical. The optimal selection of the cleaning agent depends mainly on membrane material and type of foulant. Chemical method is an effective method to remove foulants, but this method, resulting in process downtime, membrane degradation and increased operation costs is costly and requires to stop ping the RO plant operation. Physico-chemical cleaning methods are not implemented widely in the RO industry. The applications usually consist in forward flushing with permeate between cleanings when more than one chemical cleaning is used [10], but not in a simultaneous use of physical and chemical cleaning actions. Recently innovative and non-conventional method for membrane cleaning is used. There have been significant developments in new devices for energy recovery, new membrane materials, and new sizes and orientations of RO plants, all designed to reduce costs and improving efficiency. Alternative methods of recovering RO membrane performance include Electro Magnetic Fields (EMF), [11] Direct Osmosis at High Salinities (DO-HS) [12] air scouring using compressed air [13] and combined hydrodynamic and chemical cleaning [14]. Some of these techniques have been applied to RO membranes but most work appears to have been conducted on UF membranes. There has not been a wholesale adoption of any new cleaning techniques for RO membranes [10].

Advanced processes are sought that can enhance water recovery without the limitations associated with the current processes [15]. Forward osmosis (FO) or Direct Osmosis (DO) as a novel process has attracted much interest because of its potential applications in seawater desalination [16], wastewater reclamation [17], energy production [18-19-20-21-22-23-24], and so on. The water permeation in the DO process is brought about by an osmotic pressure difference across semi-permeable membranes. The water molecules can be transferred from a low concentration solution to a high concentration solution without applied hydraulic pressure. The DO process has great potential to enable versatile novel applications such as pure water drawing for product enrichment, hybrid desalination [25], energy production, and so on. Although a few DO processes have already been commercialized, further efforts are required to achieve their industrial use worldwide [26].Pressure driven backwashing is common practice in filtration processes, including microfiltration and ultrafiltration, offering an effective means of fouling control. However, it was not extensively employed for membranes used in RO, due to high back-pressure required for a hydraulically driven backwash since it may rupture the composite membrane used[27].In this study, Osmosis-assisted cleaning of polyamide RO membrane carried out using direct osmosis (DO) in RO plant operation in continues mode without stopping of RO plant. In these experiments the membrane fouling trend is shown and then high concentration solution of salt injected in order to create osmotic pressure. This means that the permeate flow is improved .The aim of this project is to investigate the optimum concentration of salt for the implementation of this method and identify practical feasibility of this approach.

MATERIALS AND METHODS

Membrane characteristics

A polyamide Dow Filmtec membrane was used, which was flat sheet, hydrophilic, with a nominal pore size of 0.01μm. Some other specifications of membrane given in Table 1.

Test equipment

In this project for cleaning the polyamide RO membrane assisted by directly osmosis with saline solution, the semi-industrial pilot plant is designed and built.

Table 1. Membrane Specifications

Membrane	Part number	Active area, ft^2 (m^2)	Feed spacer thickness (mm)	Permeate flow rate, gpd (m^3/d)	Stabilized salt rejection (%)	Minimumsaltrejection (%)
polyamide Dow Filmtec	Lc-HR-404	94(8.7)	28	2900(11)	99.7	99.5

The pilot includes pumps, tank(Super Duplex stainless steel) for feed, saline solution container, air regulator (R07-200-RNKA),feed temperature gauge, membrane chamber and membrane inlet and outlet gauge pressure. A NaCl solution as the high salinity solution and cooling water as feed solution were connected to Membrane chamber. The feed solutions were recirculated by high pressure pump (Wanner Engineering, Inc., USA, F20-111-2400/B) and a pulse of high concentration solution called hypersaline solution (HS)injected in to feed water after the membrane contaminated by organic and mineral foulants. Feed tank is capable of holding about 20liters of the solution. In order to discharge of 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 full drain the solution from tank. Since the tests carry out at intervals of 2 to 4 hours, and during this time, 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 NPSH, have been installed at the proper height. Fig. 1 shows a schematic diagram of a pressure assisted osmosis evaluation system.

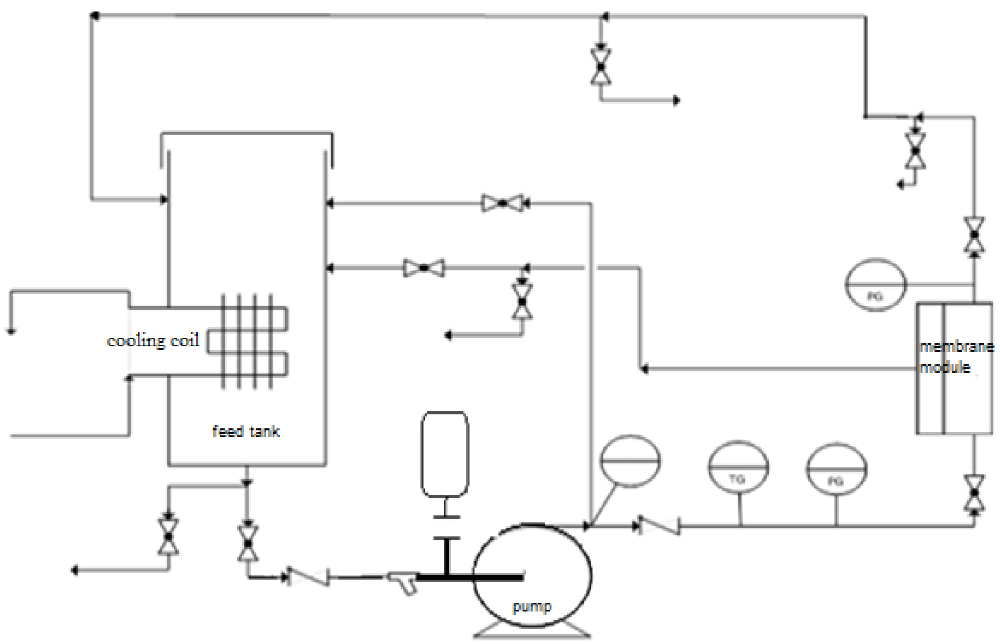


Fig.1. Schematic diagram of RO semi-industrial pilot plant

Determination of pure water permeability of clean membrane

After sealing the system, the Demineralized Water (DM) flux at pressures of 2, 4, 6, 8, 10 and 12 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 was calculated at each different pressure. The general equation describing the water flux in direct osmosis (DO), reverse osmosis (RO), or pressure-retarded osmosis (PRO) is as follows [28]:

$$j_w = A(\Delta\pi - \Delta P), \quad (1)$$

Where, Jw is the water flux, A is the water permeability constant of the membrane, Δπ is the osmotic pressure difference across the membrane, and ΔP is the applied hydraulic pressure difference.

In equation 1 the calculation of osmotic pressure is an important consideration. The van't Hoff equation provides the basis for that through the following simple relation for the estimation of osmotic pressure (π) for any dilute solution [29]:

$$\pi = MRT \ , \quad (2)$$

Where, M is the molar concentration of the solution, R is the universal gas constant, and T is the absolute temperature. If the solute is a strong electrolyte that completely dissociates in water and contains m ions, then the Van't Hoff equation becomes the following [30]:

$$\pi = \frac{m x_1 RT}{V_2}, \quad (3)$$

Where, x_1 is the mole fraction of species 1 (electrolyte) and V_2 is the molar volume of water. Pure water permeability coefficient calculation were done at 2, 4, 6, 8, 10 and 12 bar. Osmotic pressure calculated by equation 2.Based on experimental data by calculating the slope of J_w versus (P-π) membrane permeability coefficient obtained.

Fouling experiments

Fouling studies with cooling water(collected from cooling tower of Marun petrochemical company, Mahshahr, Iran) as feed at 25^0C and pressures of 2,4,6,8,10 and 12 bar were conducted until a steady-state flux was observed. 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. Prior to using the conductivity meter, calibration curves associating individual solute concentration in water with conductivity were conducted to facilitate the calculation of solute concentration following the measurement of conductivity during the tests. By calculating the solute concentration before and after each experiment, the solute rejection can be determined using the equation as shown in Eq. (4) for RO experiments.

$$Rejection, \% = \frac{C_{feed,initial} - C_{permeate,final}}{C_{feed,initial}} 100\%, \qquad (4)$$

In order to investigate the membrane fouling, the tests were carried out at different pressuresin11 days. Permeability coefficients at different pressures calculated in the11daysandits results will be reported.

Membrane cleaning experiments

The cleaning procedures applied were either: (1) physical cleaning, which involved recirculating DM water for 20 min with no permeation. (2) osmotic cleaning, which consisted of recirculating a high salinity feed solution for 21 second followed by physical cleaning as described above in procedure1. 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. A shorter pulse is ineffective in inducing osmotic permeation. In fact, short pulse is significantly diluted on the membrane surface to the point where its concentration may drop below that required for inducing osmotic flow. The pulse concentration and duration must be optimized if efficient osmotic cleaning is to be achieved throughout the full length of a membrane train. So osmotic backwash cycle induced by seven pulse of high concentration solution of NaCl during 21 second that create osmotic pressure more than hydraulic pressure. Duration of each pulse was 3 second. Two draw concentration 8% and 10% NaCl solution are injected to RO system. Simultaneously with per pulse permeate flow is almost zero and DO were made. After the injection of salt solution and stability of RO system; the tests take place to measure the permeate flux again. Salt injection into the system at both levels will continue to 6 days. Permeate flow is measured per day in each of concentration separately.

RESULTS AND DISCUSSION

Permeability coefficient calculation of clean membrane

The result of permeate flux of clean membrane at different pressures is given in Table 1.

Permeability coefficient calculated by equation 1.

As can be seen in table 1, the permeate flux increased with increasing ΔP because the additional applied hydraulic pressure could work as a high driving force in all experiments. By calculating theslope of J_w versus (P-π) membrane permeability coefficient obtained. Result given in figure 2.

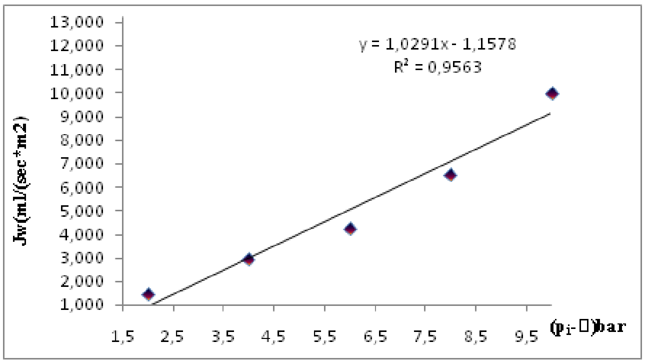


Fig. 2. Permeate flux of clean membrane at different pressures

Membrane fouling

Permeate flux of fouled membrane at different pressures in 11 days measured and subsequently the permeability coefficients of membrane calculated by equation 1.The results are given in Table 2.

As can be seen, permeability coefficients of membrane are reduced per day which this indicates fouling of membrane during these days. Accordingtomeasurementstakenwithin11daysandtaking into account the operating pressure is 10 bar, the reduction in permeate flow and the TDS concentration of feed, permeate and rejection flow are given in Table 3 & 4.

Table 2. Water permeate flux of clean membrane

i	P_{in}(bar)	P_{out}(bar)	t(sec)	V(ml)	Q_P(ml /s)	Temp(C)	J_W(ml /m^2.sec)
1	2	0.6	20.29	258	12.71	37	1.462
2	4	1.4	20.58	523	25.41	37	2.921
3	6	2.1	20.35	750	36.85	37	4.236
4	8	4	20.46	1158	56.59	37	6.506
5	10	7.4	20.31	1760	86.66	37	9.961
6	12	10.5	20.23	2329	115.12	37	13.233

Table 3. Permeability coefficients of fouled membrane in 11 days

	First day	Second day	Third day	Fourth day	Fifth day	Sixth day	Seventh day	Eighth day	Ninth day	Tenth day	Eleventh day
Permeability Coefficient (ml/m^{2}s.bar.)	1.01	1.0	0.98	0.93	0.83	0.8	0.78	0.74	0.69	0.65	0.6

Table 4. Results of membrane fouling at pressure of 10 bar

Test number (day)	TDS_F (mg/lit)	$Cond_F$ (µs/cm)	TDS_R (mg/lit)	$Cond_R$ (µs/cm)	TDS_P (mg/lit)	$Cond_P$ (µs/cm)	Q_P (Lit/s)	Reduc-tion of permeate flow(%)
First	285	570	340	680	2.4	4.6	0.086	0
Second	303	604	342	687	2.35	4.6	0.083	3
Third	287	574	353	705	2.4	4.75	0.08	6.82
fourth	391	782	469	936	3.7	7.3	0.083	3.09
fifth	438	875	627	1255	3.7	7.4	0.058	32.29
sixth	1370	2750	1528	3100	5.1	10.3	0.057	33.93
seventh	1370	2750	1528	3100	5.1	10.3	0.051	40.54
Eighth	1650	3300	1817	3640	6.2	12.3	0.062	28.26
Ninth	1970	3940	2350	4700	6.5	13	0.051	40.54
Tenth	2270	4530	2430	4870	8	16.1	0.049	43.02
Eleventh	2530	5110	3310	6600	17	34	0.047	45.35

As it is seen, conductivity and subsequent TDS increase during 11 days, it means that the membrane fouling is increasing. With putting the RO system in service and daily calculation of important parameters such as TDS, conductivity for feed, permeate and rejection flow, Input and output pressure of the membrane and calculation of permeate flux, the percentage of membrane clogging is investigated. The results show that the percentage of fouling in the membrane of the 11 daysis increased approximately45percent.

Osmosis-assisted cleaning of fouled membrane

The osmotic pressure assisted operation has a potential use in membrane cleaning by high salinity solutions. Therefore, to estimate the potential use for salinity solutions, we investigated the effect of salt concentration of NaCl on permeate flux in RO membrane and membrane cleaning during DO process. Draw solutions used in the osmotic cleaning experiments are NaCl solutions with a concentration of 80 and100 gr/l. These concentrations of salt create osmotic pressure of 35 and 43 bar respectively.

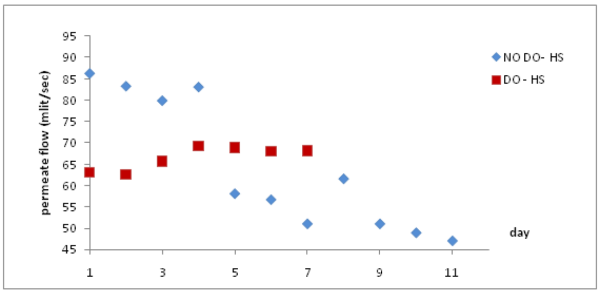


Fig. 3. Influence of performing DO process on permeate flow(pulse injection salt's concentration of 8%)

The change of permeate flux after salt injection with concentration of 8% at pressure of 10 bar is given by Table 5.

In the pulse injection, desorption process take place. Since desorption is endothermic process, so the temperature of system reduce. As shown in the table above, after a week of injection high salinities (8% NaCl solution), permeate flux increased about 45percent than the last day before injection. Influence of performing DO process on permeate flow illustrate in figure 3.

In order to create a strong net driving force of 43 bar for the DO backwash process high-saline solution made of 10% NaCl could be injected into the feed stream over a few seconds before the injection of high-solution, the RO system put in service to membrane fouling reached up to 35 %.

Table 5. Result's data after pulse injection in 8% of salt concentration

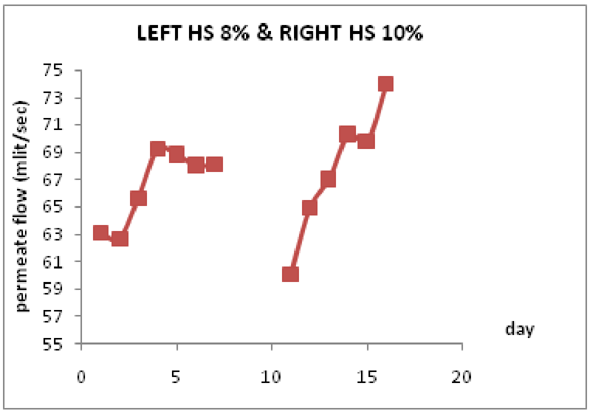
P_{in} (bar)	Test number (day)	P_{out} (bar)	$Cond_p$ (µs/cm)	Q_p (ml/s)	J_W (ml/m^2.s)	T (ºC)
10	The day before salt injection	8.7	16.7	47	5.387	33
10	1	8.9	3.54	63.12	7.205	30
10	2	9	3.07	64.69	7.21	29
10	3	9	2.78	65.65	7.55	29
10	4	9	2.5	67.85	7.63	29
10	5	8.95	2.41	68.04	7.71	28
10	6	8.95	2.23	68.04	7.82	28
10	7	8.95	2.1	68.12	7.83	28

Table 6.Result's data after pulse injection in 10% of salt's concentration

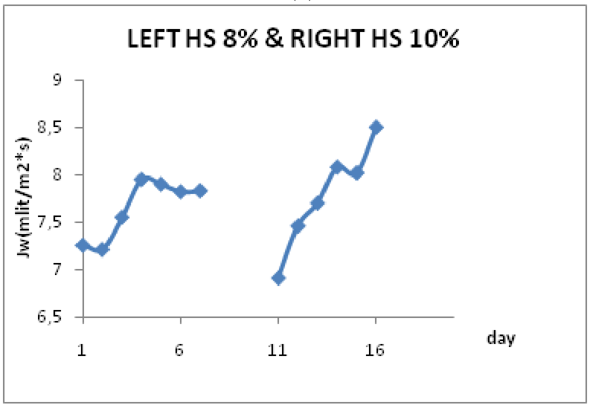
Test number (day)	P_{in} (bar)	P_{out} (bar)	$Cond_p$ (µs/cm)	Q_p (ml/s)	J_W (ml/m^2.s)	T (ºC)
The day before salt injection	10	9.2	7.2	55.26	6.35	31
1	10	9.25	6.94	60.10	6.91	30
2	10	9.3	2.49	64.94	7.46	29
3	10	9.25	2.57	67.03	7.70	30
4	10	9.2	2.89	70.29	8.08	30
5	10	9.2	3.18	69.81	8.02	30
6	10	9.2	3.7	73.96	8.5	30

Table 7. Result's data after and before pulse injection in 8,10% of salt's concentration

	P_i (bar)	P_{out} (bar)	Q_p (ml/s)	J_w (ml/s.m^2)	T (ºC)	Π(bar)	Permeability Coefficient (ml/s.m^2.bar)
Before salt injection	10	7.4	86.66	9.961	37	0	1.0291
After salt injection with concentration of 8 %	10	8.9	68.12	7.83	28	0.00578	0.88
After saltinjection with concentration of 10%	10	9.2	73.96	8.5	30	0.023	0.924



(a)



(b)

Fig 4. Significant increasing in the a(permeate flux b)permeate flow after solution injection.

As shows Table 5, after 6 days of injection high-saline permeate flow increased about 34% Than the last day before injection. Increase permeate flow after solution injection has been demonstrated in figure 4.

Figure 4 indicate that the slope of permeate flux and permeate flow with a short injection of feed with increased salt concentration (10% NaCl solution) with an associated osmotic pressure of 43 bar is growing faster than the other one(8% NaCl solution).also it is indicate that an increase in salt concentration, caused more stable system and increasing in permeate flow and permeate flux can be observed. The reversible flow helps to dislodge any foulants (mineral and organic foulants) and scaling on the membrane surface and promotes lifting, sweeping and removing of the concentration polarization CP layer. In this case the percent of salt rejection calculated by equation 4 is 99.5% after solution injection. so this method considered as effective and potentially technique that able to membrane cleaning.

Permeability coefficient calculation of membrane after salt injection with concentration of 8 and 10% NaCl

As in the starting up of process the permeability coefficient calculated by DM water, at this stage instead of cooling water (feed) ,DM water is used and permeability coefficient calculated after salt injection. Results showed in Table 6.

As can be seen permeability coefficient of membrane is 1.02 at first day (before salt injection) while it is reached to approximately 90 percent of itafterpulse injection in 10% of salt concentration. Also in the short time we have achieved the desired result by injection in 10% of salt concentration.

CONCLUSION

There is clearly much scope for the development of assisted pressure process like DO for membrane cleaning. In general this method is considered an innovative, effective and potentially chemical–free cleaning technique and was used as a novel backwash approach for on-line polyamide membrane cleaning in RO operation without stopping the RO pump in this research. In the system studied, foulant removal was facilitated by the combined effect of DO process by high saline solution of NaCl with RO process. Draw strength (concentration of salt) optimization may be achieved by considering the amount of permeate flux and toleration of osmotic pressure by polyamide membrane .Results showed that there is more increasing in permeate flux by pulse injection of 10%NaCl than the other one(8% NaCl solution).On the other hand, there was not polyamide membrane rupture under osmotic pressure. Permeability coefficient of membrane reached to approximately 90 percent after pulse injection in 10% of NaCl as well as. So 10% NaCl as salt concentration. To the best of our knowledge the current study is one of the first one to have demonstrated the application of DO process in polyamide RO membrane cleaning. It can be concluded from the work reported here in that the membrane cleaning by hypersaline solutions ensured stable operation with significantly improved membrane performance, consistently reduced low pressure drop and increased membrane salt rejection. Nevertheless it deserves further study.

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