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Carbonate scale reduction in reverse osmosis membrane by CO_2 in wastewater reclamation

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Abstract. Reverse osmosis technology is being used on large scale for treatment of ground water, brackish water, wastewater and sea water. The most challenging issue in RO process is carbonate scaling which is directly linked with the efficiency and economy. Considering the natural phenomena of carbonate scaling different adaptations have been made to control scaling on the surface of RO membrane including acid dosage and antiscalant addition. As carbonate scaling is directly related with pH level of feed water, present study describes an experimental approach to reduce scaling on RO membrane by lowering the feed water pH by purging CO_2. In this comparative study four different conditions including control process (without any scale inhibitor), with dosage of antiscalant, with purging of CO_2 and with co addition of antiscalant and CO_2 in a feed stream line; it was established that CO_2 is a better appliance to reduce carbonate scaling on the membrane surface by reduce pH of feed stream. It was also observed that CO_2 and antiscalant mutually function better for scale control.

Keywords: $CaCO_3$; transmembrane pressure (TMP); RO; membrane fouling; CO_2 purging; antiscalant

1. Introduction

Water is a primary and fundamental necessity for life which cannot be replaced by anything else. It always remained most challenging and addressable subject. Around the world one of the major issues is scarcity of water as compared to necessity. To cover the huge gap between necessity and availability, different techniques are being used to ensure the maximum availability of portable water. One of these techniques is to reuse wastewater after treatment (Pype *et al*. 2016, Holloway *et al*. 2016). Wastewater treatment involves physical, chemical and biological processes for treatment of contaminated water. Operational conditions and processing steps directly depend upon the source and the nature of the wastewater.

Reverse osmosis (RO) is a prominent technology which is being used for desalination of water for long time. Use of RO technology is also found successful for wastewater treatment and its demand is constantly increasing as discussed with quantitative data in other studies (Bartels *et al.* 2005, Greenlee *et al.* 2009, Razavi *et al.* 2015). It is a good sign toward reuse of water and

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decreasing discharge of wastewater directly into the environment. As the numbers of RO plant are increasing globally; operational, economic, environmental and other challenges are also growing. Fouling is a major operational challenge which cause loss in performance of membrane resulted in decrease in permeates flux (Greenlee *et al.* 2009). Fouling can be organic, inorganic or biofouling depending on the nature of species, particles or ions (calcium, sulphate, carbonates, bicarbonates, etc.) present in water. In membrane treatment systems, transportation of selective ions or species across the membrane is a key phenomenon. It is directly associated with concentration polarization having principal concern with membrane pressure and flux gradient (Lee *et al.* 1999, Antony *et al.* 2011). Inorganic ion deposition on the surface of membrane is an acute concern.Mechanism of scaling can be heterogonous or homogenous (Okazaki *et al.* 1984, Zeiher *et al.* 2003).

Carbonate scaling is a prime challenge for RO plants which is an alkaline scale and formed due to the breakdown of bicarbonate ions. Equilibrium between bicarbonate ion and $CaCO_3$ can be expressed as follows

$$Ca^{2+} + HCO_3^- \leftrightarrow H^+ + CaCO_3 \tag{1}$$

As reaction directly depends on pH of water, any change in pH of feed water will directly affects the state of equilibrium. For evading carbonate scaling above reaction can be shifted toward left side by keeping pH at acidic side. $CaCO_3$ deposition on the surface of membrane leads to decrease in permeates flux and increase transmembrane pressure (TMP). There are different resistances including concentration polarization resistance, membrane resistance, pore blocking resistance and cake resistance produced by fouling, which mutually reduce permeate flux. Scale can cause physical damage to membrane due to pore plugging. To reduce $CaCO_3$ deposits, controlled acid dosing in feed water streams is a common practice (Greenlee *et al.* 2010).

Langelier saturation index (LSI) is an effective tool to know the degree of saturation of water concerning calcium carbonate. LSI mainly depends upon the calcium hardness and bicarbonate alkalinity of the feed water stream. By knowing the degree of LSI and ionic mass balance within the system, the influent stream line can be regulated to get maximum level of performance. In addition, pre-treatment process and in time clean-in-place (CIP) can also improve membrane performance to avoid scale formation (Fritzmann *et al.* 2007, Lau *et al.* 2014, Wei *et al.* 2007). Generally three different types of scale inhibitors are used including sodium hexametaphosphate (SHMP), organophosphonates and polyacrylates (Prihasto *et al.* 2009). Different studies indicated the by-products generation in concentrate stream due to the chemical dosage in feed stream (Greenlee *et al.* 2010, Lee *et al.* 1999).

In a study by Adriano *et al*. (2011) inorganic ion deposition on RO membrane was successfully avoided by keeping the pH of feed stream at 6.3±2. It was proposed that by modification in MBR with stripping unit for CO_2 recycling could be an effective approach to control cost.

In this study, RO membrane performance was examined with CO_2 purging in influent stream in comparison with antiscalant dosage. Four parallel RO modules were operated with control module, CO_2 purging, antiscalant injection, CO_2 with antiscalant injection respectively. By lowering the pH of feed stream by use of CO_2, this study suggests a way to avoid the scale deposition in reverse osmosis desalination and to reduce the chemical consumption in the system. As there is no specific by-product generation in case of CO_2 usage so there is no probability of damage to the system. By using CO_2 in RO process, the market need for antiscalant can be reduced. Cost effective and environment friendly CO_2 recycling system can be considered to meet the requirement of RO plants operating with CO_2.

Table 1 Quality parameters of UF effluent

Parameters	Quantity
pH	7-7.1
BOD	6-8 mg/L
COD	7-9 mg/L
SS	0.2-0.4 mg/L
TOC	4-7 mg/L
Al	0.02 mg/L
Fe	0.04 mg/L

2. Experimental section

2.1 Material and chemicals

Domestic wastewater of “P” city in Korea treated with ultra-filtration was used as a feed stream for RO modules, quality parameters of UF effluent are described in Table 1. All the chemicals and reagents used were of analytical grade and purchased from Fisher Scientific. CO_2 with over 99% purity was purchased from domestic market. Commercially available antiscalant (MDC-220) was obtained from “P” wastewater reclamation plant, South Korea.

2.2 Operation of the RO module

RO membranes used in this study were manufactured by Toray chemicals Korea. Polyamide Thin-Film composite based spiral wounded RO membrane having 2.5 m^2 area and 800 GPD permeate flow rate was used. Membrane was designed to tolerate up to 45℃ temperature and works perfectly under 600 psi pressures within pH range 2.0-11. Maximum feed flow rate was 6gpm, maximum feed silt density (15 min) is 5.0, maximum turbidity is 1.0 NTU and free chlorine tolerance was <0.1 ppm. Membrane was designed at the 99.4% minimum salt rejection. Permeate flow rate and salt rejection percentage was established on the basis of operation at 1.5 MPa applied pressure for influent having pH 6.75±0.25 at 25℃ containing 2,000 ppm concentration of NaCl resulted in 15% recovery. (Product Specification Sheet / Model RE 2540-BE, Toray Chemical Korea Inc.).

RO system was operated with domestic wastewater of “P” city primarily treated with conventional ultra-filtration at “P” wastewater reclamation plant. Four parallel RO modules were operated with control module, CO_2 purging, antiscalant injection, CO_2 plus antiscalant injection respectively (Fig. 1). All the RO modules were operated on 5.5 bar initial TMP and 4 m^3/day constant influent flow rate leading to get 58% recovery.

CO_2 and antiscalant (MDC-220) were fed through the inline mixer. Pumps were used for controlled dosage (8.4 μL/min) of antiscalant in RO module 3 and 4. CO_2 injection rate was controlled at 300±2 mL/min for RO module 2 and 4 respectively to keep pH of feed stream at level of 6 (Table 2). The value of pH for RO module 1 and 3 was kept at 7. For dechlorinating, 3 ppm sodium bisulfite (SBS, 99% purity) was injected to the influent at a flow rate of 8.4 μL/min. Chemical cleaning was conducted with 1% EDTA, 0.1% caustic and 0.2% HCl with 35 ml/min input flow rate and 30 minute stay time for each chemical. CIP for RO modules 2 and 4 was

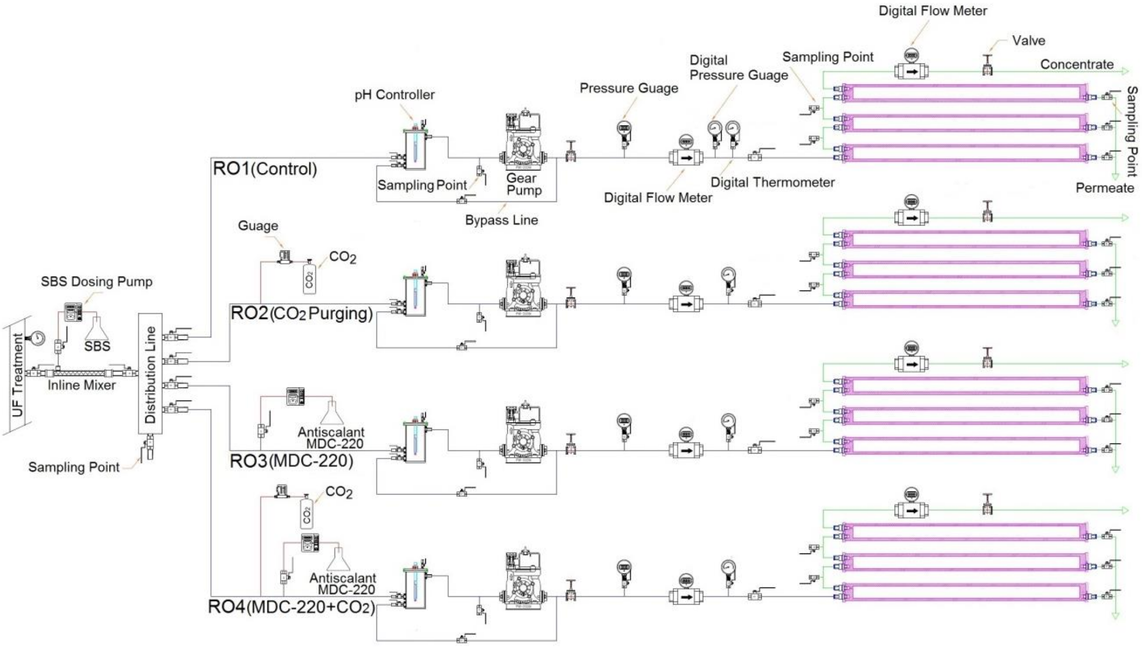


Fig. 1 Schematic diagram of the RO modules

Table 2 Characterization of the RO modules on the basis of scale inhibition methods

RO System	Scale inhibition method	Remark
RO 1	Control (No scale inhibition)	-
RO 2	CO_2 purging	300±2 mL/min
RO 3	Antiscalant (MDC-220)	8.4 μL/min
RO 4	CO_2 +Antiscalant	300±2 mL/min + 8.4 μL/min

conducted without CO_2 purging.

2.3 Analysis of water quality constituents

Conductivity and pH were analysed with EC96 (M.Cubic) and 96pH-L2 (samsan) respectively. The other water quality constituents were analysed according to *Standard methods* (APHA).TMP was monitored by real-time monitoring system.Ionic concentrations of monovalent and divalent ions (Na^+, Mg^+, K^+, Ca^+, Cl^-, NO_3^{2-},and SO_4^{2-}) was analysed by Ion Chromatograph(ICS-1000 and ICS-5000 for cations and anions respectively). Scanning Electron Microscopy and energy Dispersive Spectroscopy (SEM-EDS, S-4300) was used to analyse the elemental analysis and morphological changes on the surface of each RO membrane. LSI was calculated as follows:

$$LSI = pH - pH_s$$

where,

pH is the measured feed water pH

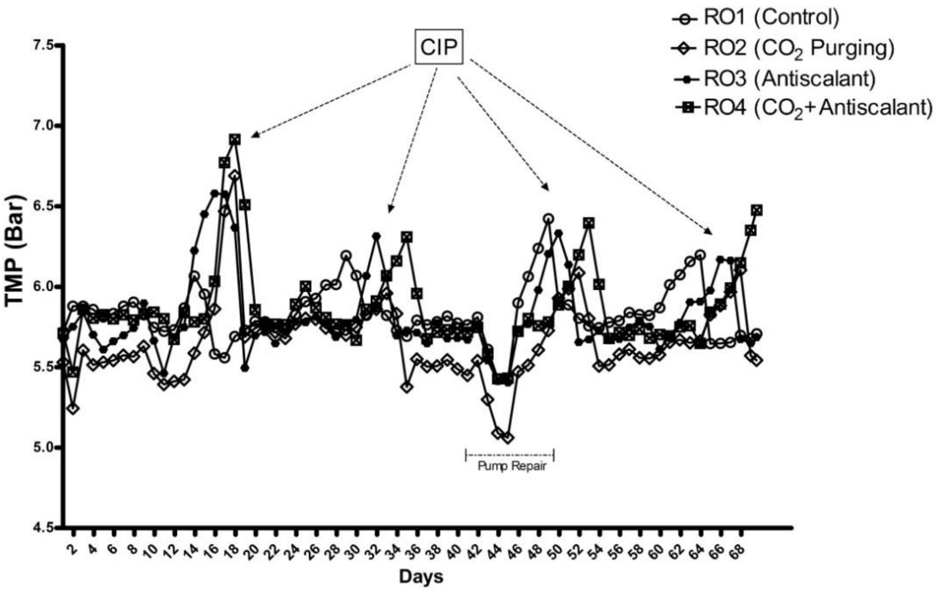


Fig. 2 Comparison of RO modules, change in TMP

pH_s is the pH at saturation and it is defined as:

$$pH_s = (9.3 + A + B) - (C + D)$$

where,

A = (Log_{10} [TDS] - 1) / 10
B = -13.12 x Log_{10} (C + 273) + 34.55
C = Log_{10} [Ca^{2+} as $CaCO_3$] - 0.4
D = Log_{10} [alkalinity as $CaCO_3$

3. Results and discussion

3.1 Operational behaviour

All modules were operated for continuous 69 days with chemical cleaning at certain interval of time. All the RO modules showed effective functioning on 5.5 bar TMP for continuous 12 days operation. An increase in TMP was observed on 12th day for RO module 1 followed by the next five days up to 6.8 bar. In case of RO module 2 and 4, TMP remained constant for first 14 days then it gradually increased for following three days up to 6.7 bar. TMP for RO module 3 remained constant for first 12 days and later an increase in TMP was observed for following four days which finally attained the value of 6.9 bar (Fig. 2). Increase in TMP after a certain time is a symbolic representation of scale deposition on the surface of membrane. In following operational days, gradual increase in ion deposition caused blockage in feed channels and increase in conductivity of the system. This led to increase in TMP and decrease in salt rejection due to restraint back diffusion of ions. By reducing the pH of influent from 7 to 6 with CO_2 purging for RO module 2, it was observed that TMP remained constant for two additional days passing about additional 8 $m^3$of influent as compared with control RO module 1 which was operated without any scale inhibitor. Similarly in case of RO module 4, TMP remained constant for longer time as compared to RO module 1. TMP slightly increased in case of RO module 4 operated with CO_2 and antiscalant jointly. It was observed that by lowering the pH (pH 6) with the purging of CO_2 RO modules indicated the better stability for TMP as compare to RO modules operated with feed stream having pH 7. CO_2 was found better in maintaining equilibrium between ionic concentrations resulting in decrease in rate of deposition on membrane surface and preventing blockage of feed channels. In a comparative study on membrane fouling at constant flux and constant TMP, Daniel *et al.* (2014) found an increase in TMP as a result of scale growth on the surface of the membrane.

3.2 Scale production behaviour

A significant change was observed for LSI between RO module 1 and the other modules. LSI value remained at positive value for RO module 1 and 3 i.e., 0.4±0.15 and 0.2±0.05 respectively, while a shift in LSI value toward negative side was observed for RO module 2 & 4 i.e., -0.40±0.1 for both modules. Trend towards the negative side shows the system is away from the point of saturation. Although chemical cleaning aided to decrease the LSI value but after each CIP phase there was a little switch toward higher side in LSI value as compare to previous CIP phase. It indicates the supersaturation state of system. Slight variation was also observed for module 3 but chemical cleaning was found effective to regain the LSI value similar to the value at the beginning of operation. Comparative behaviour of modules regarding LSI values showed that lowering the pH for one degree by CO_2 purging is an effective way toevade saturation of $CaCO_3$ as compare to normal operation (Fig. 3).

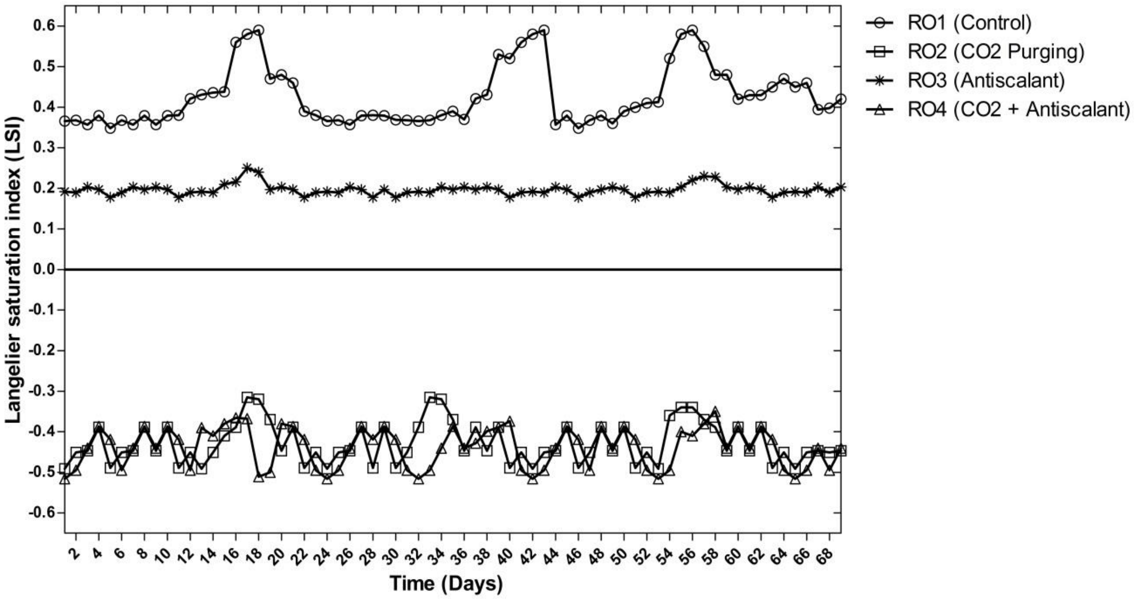


Fig. 3 Comparison between LSI data of all RO modules

Table 3 Principal ion concentration for RO systems (Average ± standard deviation)

		RO 1 (Control)		RO 2 (CO_2 purging)		RO 3 (Antiscalant)		RO 4 (CO_2 + Antiscalant)	
	Influent (mg/L)	Permeate (mg/L)	Concentrate (mg/L)	Permeate (mg/L)	Concentrate (mg/L)	Permeate (mg/L)	Concentrate (mg/L)	Permeate (mg/L)	Concentrate (mg/L)
Na^+	500±20	20±2	760±10	20±3	900±10	20±3	920±10	17±2	960±10
K^+	50±5	2.2±0.2	100±5	2±0.2	100±5	2±0.2	110±5	2±0.2	120±5
Mg^{2+}	80±10	0.1±0.02	165±5	0.05±0.01	175±5	0.05±0.02	170±5	0.05±0.01	175±5
Ca^{2+}	160±15	0.6±0.5	335±5	0.45±0.02	375±5	0.43±0.02	365±5	0.48±0.05	378±5
Cl^-	820±20	17±1	1700±15	12±2	1840±15	17.5±2	1810±15	15±2	1945±15
NO_3^{2-}	32±5	4±0.1	60±5	3.3±0.2	60±3	4±0.2	58±5	4±0.2	62±5
SO_4^{2-}	364±10	0.2±0.02	760±10	0.25±0.02	830±10	0.26±0.02	795±10	0.25±0.02	865±10

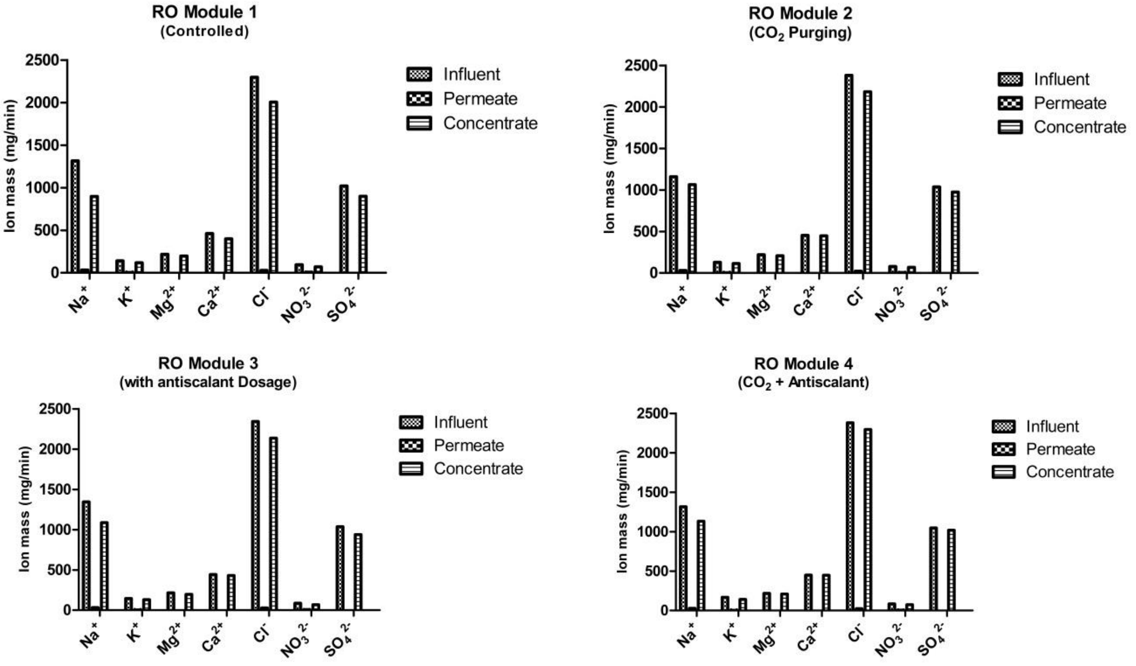


Fig. 4 Ion mass balance of RO modules

3.3 Mass balance of the principal ions

Ionic mass balance was calculated on the basis of concentration of specific ions including Na^+, Mg^+, K^+, Ca^+, Cl^-, NO_3^{2-}&SO_4^{2-} in influent, permeate and concentrate stream (Table 3, Fig. 4). Comparative data for ionic concentration in permeate and concentrate stream showed the higher discharge for monovalent and divalent ions in RO modules 2, 3 & 4. Relatively low ionic discharge was observed for RO module 1 which indicates the accumulation of ionswithin the membrane in a system. Successful discharge of rejected ions in the RO modules withCO_2 injection describes that CO_2 can effectively control ion deposition on membrane surface.

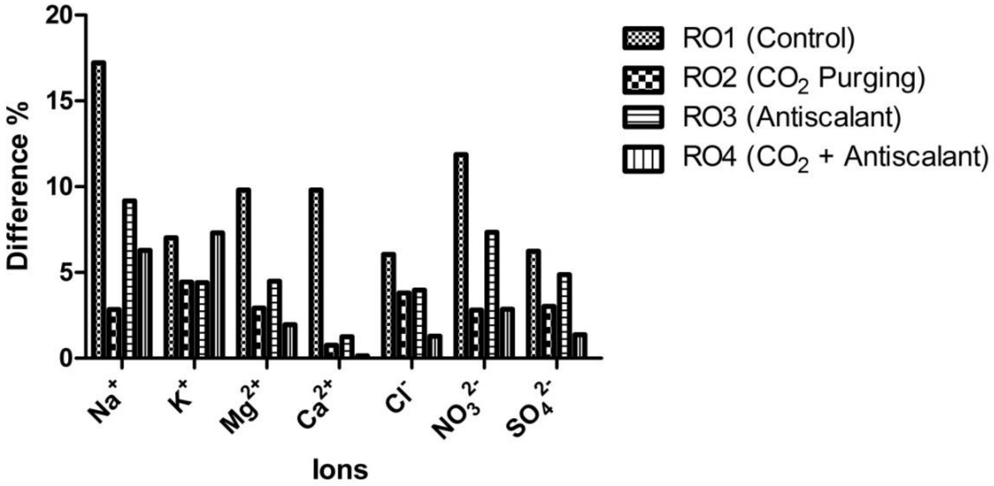


Fig. 5 Difference % obtained from ion mass balance

Difference percentage for ions was calculated on the base of data obtained from mass balance equation which is expressed as; (Feed flow rate×Ion concentration $_{influent}$)=(Permeate flow rate×Ion concentration $_{Permeate}$)+(Concentrate flow rate×Ion concentration $_{Concentrate}$).

Difference percentage in mass balance for all RO modules signified the comparison among the modules regarding ion deposition within the system (Fig. 5).

RO module1 was found with high precipitations of monovalent as well as divalent ions within the system. About 18%, 11%, 8%, 12%, 8%, 10% and 6% of the Na^+, Mg^{2+}, K^+, Ca^{2+}, Cl^-, NO_3^{2-} and SO_4^{2-} respectively gathered within the membrane as a precipitation supporting alteration of overall system performance influencing flux, TMP, scale index and other related parameters. RO module2 was observed with controlled ion deposition within the system having difference value less than 4% for all ions.

RO module3 represented a little higher percentage for Na^+ andNO_3^{2-} ions deposition within the membrane i.e., 8±1% but the other ions remained below 4%.RO module4 showed slightly higher ion deposition percentage (6±1%) for monovalent ions like Na^+& K^+. Precipitation of calcium ions remained less than 1% for RO module 2 and 4. For RO module 3 it was observed about 2% but comparatively high and severe precipitation of $CaCO_3$ (12%) was observed for RO module 1.

It was established that carbonate ion deposition can be effectively controlled within the system at pH 6 by use of CO_2. Adriano *et al.* (2011) concluded in an experimental study that by lowering the pH at level of 6.3±0.2 all ion deposition might be evaded with the exception of SiO_2.

3.4 Morphological results

SEM-EDS analyses were carried out for the examination of membrane surface after the completion of operational cycle. Membranes for analysis were obtained from lab scale RO system which was operated on same conditions as discussed in Table 2. All the membranes were examined in comparison with virgin membrane (Fig. 6).

In comparison of EDS spectra of virgin and used membranes; virgin membrane indicated the existence of carbon, oxygen and sulphur, the chief constituents of polyamide membrane. The

Table 4 EDS analytical results for used RO membranes in comparison with virgin membrane

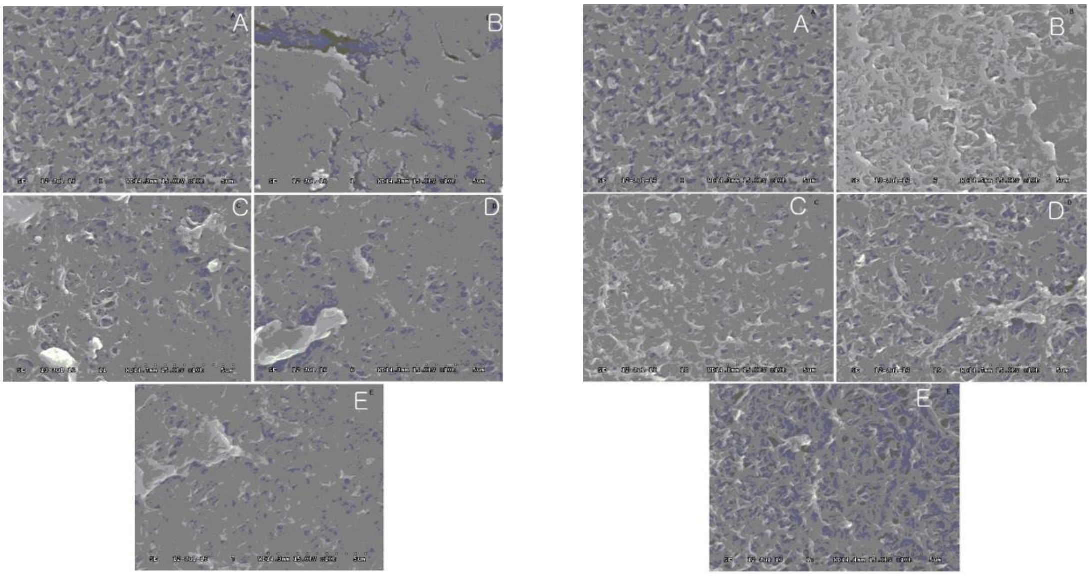
	Virgin		RO1 (Control)		RO2 (CO_2 Purging)		RO3 (Antiscalant)		RO4 (CO_2+ Antiscalant)	
Element	Wt. %	At. %	Wt. %	At. %	Wt. %	At. %	Wt. %	At. %	Wt. %	At. %
C	70.07	79.9	51.37	62.65	58.9	68.47	69.2	78	73.34	81.31
O	20.42	17	33.11	30.31	31.19	27.21	21.2	17.94	18.3	15.23
Na	-	-	0.63	0.4	0.17	0.1	0.19	0.11	0.09	0.05
Mg	-	-	0.63	0.38	0.28	0.16	0.16	0.09	0.01	0
Si	-	-	0.22	0.12	0.29	0.14	0.09	0.04	0.01	0
P	-	-	2.86	1.35	-	-	-	-	-	-
S	7.31	3.04	6.88	3.14	7.9	3.44	8.2	3.46	7.77	3.23
Cl	0.19	0.07	1.37	0.57	0.6	0.24	0.6	0.23	0.38	0.14
Ca	-	-	2.91	1.07	0.66	0.23	0.35	0.12	0.1	0.03

Table 5 EDS analytical results after CIP for used RO membranes in comparison with virgin membrane

	Virgin		RO1 (Control)		RO2 (CO_2 Purging)		RO3 (Antiscalant)		RO4 (CO_2 + Antiscalant)	
Element	Wt. %	At. %	Wt. %	At. %	Wt. %	At. %	Wt. %	At. %	Wt. %	At. %
C	70.07	79.9	70.78	77.68	70.72	77.5	70.72	77.5	72.79	79.34
N	-	-	10.63	10	10.54	9.9	10.54	9.9	9.70	9.07
O	20.42	17	11.28	9.3	11.86	9.77	11.86	9.77	10.8	8.84
Na	-	-	0.01	0.01	0	0	0	0	0.04	0.02
Mg	-	-	0	0	0.02	0.02	0.02	0.02	0	0
Si	-	-	0.11	0.05	0.05	0.02	0.05	0.02	0.06	0.03
P	-	-	0.4	0.17	0.4	0.17	0.4	0.17	0.44	0.19
S	7.31	3.04	6.68	2.75	6.17	2.53	6.17	2.53	6.03	2.46
Cl	0.19	0.07	0.1	0.04	0.24	0.09	0.24	0.09	0.13	0.05
Ca	-	-	0	0	0	0	0	0	0	0

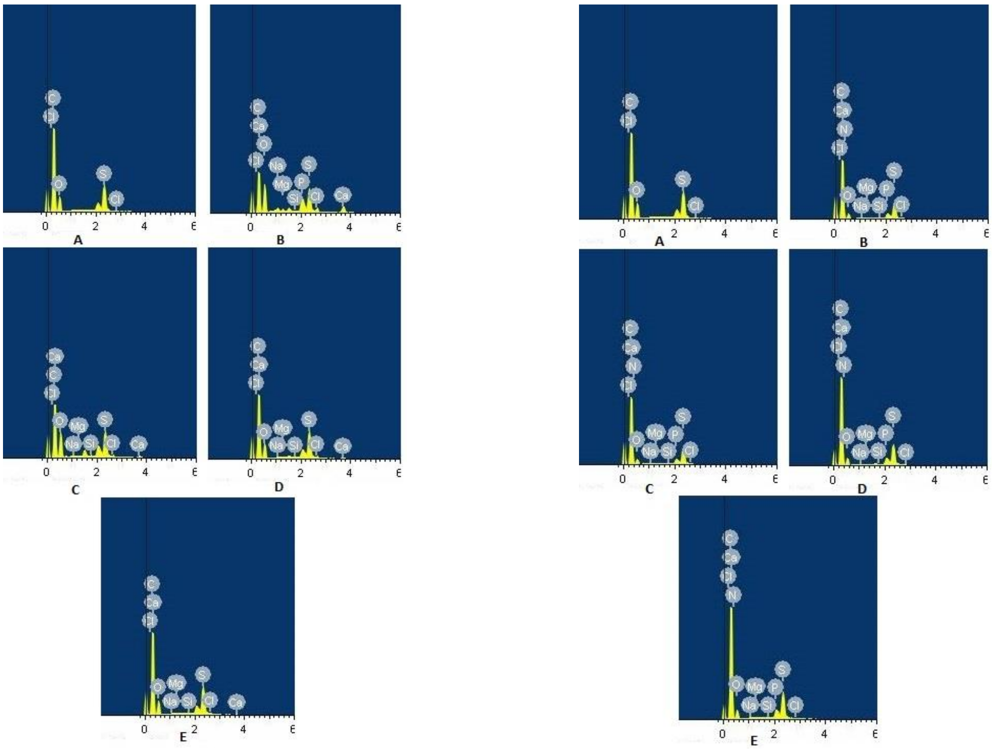
membrane from control module 1 observed with overcrowded elemental deposition including calcium and phosphorus (Table 4). A serious increase in the oxygen weight percentage on the surface of membrane was observed which might be a sign of organic matter deposition, as discussed in a study by Zhao *et al.* (2010). Morphological structure of the surface of membrane was highly spoiled after operation as shown in Fig. 6. Relatively low calcium content was observed on the membrane surface from RO module operated with CO_2 purging but prominent crystallization of inorganic matters was observed.

The atomic content (weight %) of the divalent cations (Ca^{2+} and Mg^{2+}) was the lowest level in RO module 4, which indicates the highest inhibition of scale formation. Although a little higher percentage of the cations was found on the membrane surface of RO module 2; it seems much better scale control as compared with RO module 1 where proportion of calcium and magnesium is relatively high. Moreover, serious accumulation of phosphorus was observed on the membrane surface of RO module 1. In this study CO_2 was used as a tool for lowering pH to control ion deposition on the membrane surface and it was agreed by SEM images as well as operational



After Operation After CIP

Fig. 6 SEM images of RO membranes of virgin membrane (A) and different operational modules i.e., Control (B), CO_2 purging (C), Antiscalant addition (D), Antiscalant + CO_2 (E).



After Operation After CIP

Fig. 7 EDS spectra of virgin (A), control (B), CO_2 injected (C), Antiscalant dosed (D), Antiscalant dosed + CO_2 injected (E) membrane behaviour.

Membranes were also examined for SEM-EDS after the chemical cleaning with 1 % EDTA, 0.1% caustic and 0.2% HCl to understand the behaviour of all modules in comparison. SEM-EDS results for membranes after chemical cleaning indicated the successful removal of most of the elemental traces from the surface of membranes especially the membranes operated with CO_2 purging (Table 5, Fig. 7). Different cleaning agents were used in different experimental studies representing successful removal of inorganic fouling by comparison virgin membrane and used membrane surfaces after and before cleaning (Emmanuelle *et al.* 2015, Pervov 1991). Fig. 7 indicated the successful restoration of membrane surfaces operated with CO_2 while the other membranes were observed with deposits even after CIP. The existence of deposits on membrane surface even after CIP shows the ineffectiveness of other modules for scale inhibition. It was concluded that CO_2 purging with periodic CIP can successfully inhibit scale deposition in RO system.

4. Conclusions

In this study the effectiveness of CO_2 purging for scale inhibition in RO system was examined. The effect of CO_2 purging was compared with antiscalant dosage. On the basis of continuous operation of the four different RO modules for 69 days, it was concluded that carbonate scaling can be reduced with continuous purging of CO_2. RO modules with CO_2 purging indicated more stability for TMP as compared with the other modules. It was established that by dropping the pH of influent at the level of 6, RO membrane works more efficiently to constrain scale production with more permeability as compared to be operated at pH 7. In order to treat domestic wastewater with CO_2 purging at commercial scale to reduce scaling and flux improvement of the RO membrane, this study provides an eye opening door for further developments. CO_2 purging devices may be installed commercially to replace or reduce the quantity of antiscalant addition in the process. Comparative morphological study of membranes showed that trace element deposition on the surface of membrane during operational period can be more effectively removed by CIP in the RO module operated with CO_2 purging.

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