



Experimental study of pretreatment of brackish reverse osmosis concentrate streams using seeded precipitative softening process for improvement of water recovery

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ABSTRACT

This study investigates the pretreatment of brackish water reverse osmosis concentrate streams as a feed to secondary reverse osmosis (RO) process aiming at reuse and enhancement of the potable water recovery from the concentrate streams and reduction in its volume. Seeded crystallization process was tested as a pretreatment method. The results obtained indicated that the seeded crystallization process has the potential to accomplish a 96.76% reduction of calcium ion concentration and a reduction of Langelier saturation index from 1.6 to 0.05. The optimum conditions for treating concentrates continuously at flow rate 1 L/min are: pH 10.6; seeds concentration = 3 g/L; seeds size 100 μm , residence time = 1 min. The treated concentrate can be fed to a secondary RO process with low risk of membrane fouling.

Keywords: BWRO; RO; Concentrate; Stream; Recovery; Seeded; Pretreatment; Crystallization; LSI

1. Introduction

Brackish water reverse osmosis (BWRO) is a well-known process for the recovery of potable water with total dissolved solids (TDS) less than 45 mg/L from surface and underground brackish water resources of TDS between 1,000 and 10,000 mg/L. This process requires energy to allow water molecules to transfer through semipermeable reverse osmosis (RO) membranes. The BWRO process produces two streams: the permeate which is a potable water and the concentrate which is a high salt content wastewater. Depending on the feed water and the performance of the process, 10–25% of the feed water is drained as a concentrated saline water (TDS > 10,000 mg/L) that require disposal to meet the strict regulations [1]. The disposal options

are: evaporation ponds, ejection to surface water sewer or sea water [2], deep well injection, and land applications [3].

The high salt content in the BWRO concentrate streams (TDS > 10,000 mg/L) is currently considered as a major environmental challenge due to the discharge of huge amounts of saline wastewater into neighboring water streams, sewer, and soil surface. Another challenge is developing a reuse strategy of the concentrate streams to increase the overall recovery of pure water and consequently improves the economy of the process [4–6]. Moreover, the reuse of the concentrated streams will contribute in minimizing its volume and hence making its disposal in environmentally harmless processes possible [7].

A potential approach to further concentrating BWRO concentrates and increase water recovery is using secondary RO systems [8]. However, a concern hinders direct feeding of the concentrate to secondary RO unit is the fouling of sparingly soluble inorganic salts on the surface of the membrane [9]. This type of membrane fouling is known as scaling and composed mainly from calcium carbonate (CaCO_3) and other scaling materials such as calcium sulfate, barium sulfate, and silica. They precipitate on the membrane surface when their supersaturation concentration is exceeded [10].

The treatment of brackish waters concentrates in RO systems is limited due to the presence of high concentrations of calcium ions which can combine to sulfate and carbonate leading to fouling at the membrane surface. On the other hand, dissolved silica which is present in the natural brackish waters in certain levels that cause silica fouling is one of the major limitations of concentrate streams reused in the RO systems. The silica fouling usually controlled in brackish water desalination industry using chemical inhibitors. The brackish water RO membranes suffering from sulfate scales and carbonate scales are controlled by the acid pretreatment. However, the concentrate streams from the RO units contain large concentrations of calcium ions and will require huge consumption of chemicals (scale inhibitors and acid treatment) to control calcium-related scaling in the secondary RO units. Therefore, calcium removal from concentrates is a crucial issue in the reuse of concentrate streams of BWRO desalination plants.

It has proven in the literature that the reduction of calcium ions concentration in the concentrates using precipitative softening will allow flexible integration of RO-precipitative softening process. Qu et al. [11] reported that the removal of calcium in accelerated precipitation softening was integrated with direct contact membrane distillation to establish a desalination process for high recovery desalting of primary reverse osmosis concentrate. RO concentrate was treated by pH adjustment with sodium hydroxide along with calcite seeding. The 92% removal of calcium in RO concentrate resulted in the whole recovery to be enhanced to 98.8%. Rahardianto et al. [12] demonstrated a two-step chemically enhanced seeded precipitation process for accelerated desupersaturation of reverse osmosis (RO) concentrate water of high mineral scaling propensity. In the investigated process, CaCO_3 precipitation is first induced via lime dosing, followed by subsequent CaSO_4 precipitation via gypsum seeding for concentrate desupersaturation. Enhancement of the overall water recovery from 63% up to 87% or higher appeared to be feasible.

The reduction of saturation index (SI) with respect to calcium carbonate to certain levels that ensure inhibition of membrane scaling with calcium carbonate is the key success for the reuse of the RO concentrate systems for enhancing the water recovery. On the other hand, reduction of calcium ion concentration to the lowest level will prevent scaling with other calcium salts like calcium sulfate scaling. The precipitation of these scaling elements on the membrane surface causes damage of membrane and decreases its performance [13]. The feasibility of application of the RO systems for the reuse of BWRO concentrate and to increase the water recovery requires developing effective technologies that can be practically implemented for removing scale forming elements from the concentrates [14,15].

Treating the BWRO concentrate streams before feeding to secondary RO process by precipitation of scaling salts has been researched previously. The studies reported that precipitation and seeded crystallization are the effective methods for the separation of scaling materials from BWRO concentrates. Van Houwelingen et al. [16] investigated the application of pellet softening as an important step of a treatment train for the reuse of RO concentrates aiming at achieving zero liquid discharge. An interesting finding is that the pellet softening is an attractive method not only to reduce the concentration of calcium ions and bicarbonate ions but also a significant removal of silica was achieved. The feasibility of using pellet reactor and electrodialysis to treat RO concentrates for high recovery and zero liquid discharge of RO system was investigated by Tran et al. [17]. It was reported that 70–80% removal of calcium was achieved using the pellet reactor and the calcium carbonate scaling potential of the treated brine was reduced significantly, suitable to be treated by electrodialysis process before feeding to secondary RO process. Rahardianto et al. [18] investigated a treatment process for concentrate desupersaturation by CaCO_3 precipitation followed by a subsequent CaSO_4 precipitation via gypsum seeding. Van der Bruggen et al. [19] reported simulated results about the application of crystallization processes in line with membrane systems applied for water reuse in textile industry. Van der Bruggen et al. [20] presented a case study for softening of drinking water before distribution using the pellet reactor technique based on the precipitation of CaCO_3 by NaOH addition. Omar et al. [21,22] and Al-Ghamdi and Omar [23] reported about the application of seeded crystallization for the pretreatment of seawater aiming at reducing its supersaturation. The status of seawater was changed from high-scaling potential to low-scaling potential after being subjected to seeded crystallization process.

The seeded crystallization process referred as concentrate softening. A disadvantage of this process is the large residence times which represent a difficulty in adapting this process to RO system in a continuous operation. These restrictions represent a major challenge for the implementation of precipitation in line and continuously as a pretreatment approach for RO concentrates. Moreover, the process parameters and their role in accelerating the scale reduction still needs more investigations to develop this process.

Desupersaturation of scaling ions in brines by seeded crystallization is a mass transfer process where the ions migrate toward the seeds surface and integrate in the crystal lattice. One important process parameter is the pH of water which plays an important role in carbonate system in water. High pH will support the formation of carbonate ions and therefore will enhance the separation of calcium carbonate from the brine. Another important parameter is the size and amount of seeds which affects directly the available active sites for crystal growth. The contact time between the seeds and the brine is a critical parameter, which plays an important role in the separation of scaling ions from the brine.

Other scaling compounds can precipitate like silica and barium sulfate... etc. in the real concentrate streams, however it is not considered in this study because of the well-known application of chemical inhibitors for non-calcium related scaling materials.

This study presents an experimental evaluation of the parameters involved in the reduction of calcium ions by seeded crystallization. The factors investigated are: pH, seeds size, flow rate, and seeds concentration.

2. Experimental

2.1. Materials

For each experiment, 20 L of synthetic BWRO concentrate was prepared in a well-mixed and closed

Table 1

The composition of the artificial BWRO concentrate prepared from analytical grade salts and deionized water

Concentration	mg/L	mmol/L
Calcium (Ca^{2+})	361	9
Bicarbonate (HCO_3^-)	1,199	19.7
Sodium (Na^+)	3,407	148
Chloride (Cl^-)	5,200	146
TDS	10,168	–
Ionic strength	0.132 mol/L	
Temperature	24°C	
pH	8.1	

tank by dissolving analytical grade pure salts of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ (SIGMA-ALDRICH, $\geq 99\%$), NaHCO_3 (MERCK, 99.9), and NaCl (ACROS, $>99.5\%$) in double deionized water. A predetermined amount of NaCl was added to substitute the other positive and negative ions and to achieve the same ionic strength as in BWRO concentrates. The prepared concentrate simulates BWRO concentrate from plants with the recovery range from 75 to 90% and average TDS of 10,000 mg/L and calcium ion concentration of 360 mg/L. The prepared concentrate has the composition listed in Table 1.

Limestone seeds were prepared from natural raw limestone deposits with a mineralogical composition ($\text{CaCO}_3 > 93\%$, $\text{SiO}_2 < 3\%$, $\text{MgO} < 1.2\%$, and $\text{Fe}_2\text{O}_3 < 1.5\%$). The particles were sieved using OCTAGON-Digital test sieve shaker. After 2 h shaking, the required seeds size fractions were collected.

2.2. Methods

The experiments were conducted in a 1 L continuous well-mixed crystallizer (Fig. 1). The prepared concentrate was kept in a well-mixed tank. The pH of the concentrate was adjusted by injecting concentrated NaOH solution (16.1 M) to the concentrate tank. A concentrated seeds slurry was prepared by mixing 5 L of the concentrate with limestone seeds in a well-mixed tank. The concentration of the seeds was adjusted to be 35 g/L. All the experiments were conducted at a constant room temperature of 24°C and a constant agitation speed of 200 revolutions per minute (RPM). The concentrate was supersaturated with respect to calcium carbonate by changing the pH of the concentrate. The pH, concentrate flow rate, and seeds slurry flow to the batch crystallizer were adjusted to achieve certain predetermined residence

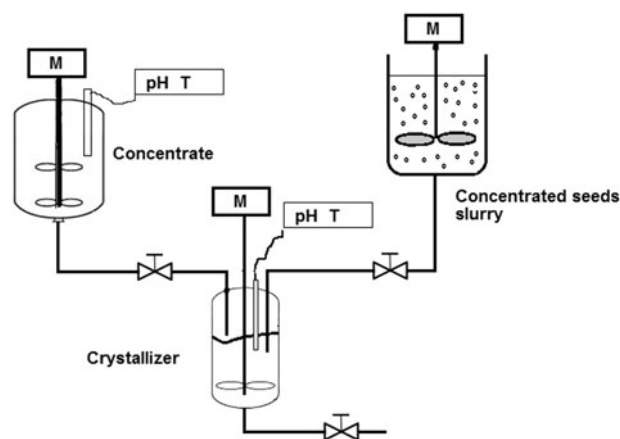


Fig. 1. Schematic diagram for the experimental setup.

time of the concentrate and seeds concentration in the crystallizer. The contact between the seeds and the concentrate will allow the removal of calcium ions by crystallization on seeds. The removal of calcium ions was measured by taking samples at different time intervals from the effluent of the crystallizer. The samples were filtered using 0.3- μm filter paper and then subjected to calcium ions analysis using inductively coupled plasma method (ICP-OES, Thermo Scientific iCAP 6000 Series Duo). The calcium ion concentration was measured using this method due its high accuracy and very low calcium detection limit (2 $\mu\text{g/L}$). The pH was measured using the pH meter (WTW-pH 340i) within the accuracy of ± 0.01 . The average steady state value of the calcium ion concentration and pH in the effluent was recorded.

The experiments were conducted to investigate the possible treatment of continuous flow of concentrates to be suitable feed for further RO system. The main governing parameter studied are the rate of removal of calcium ions which contributes directly to membrane fouling with different anions. The removal of calcium ions as carbonate was studied experimentally at different operating condition such as pH, residence time, seeds concentration, and seeds size. The experimental conditions studied are summarized in Table 2.

3. Results and discussion

Removal of calcium ions as calcium carbonate was studied experimentally in a continuous manner. The

reduction in calcium ion concentration will reduce the scaling tendency of calcium carbonate and calcium sulfate which are the main scaling compounds hindering the reuse of BWRO concentrates. The fast removal of calcium ions can be achieved by seeded crystallization if certain level of supersaturation with respect to calcium carbonate is created in the concentrate. The studied factors that affect the precipitation of calcium carbonate from concentrates on the seeds are pH, flow rate, seeds concentration, and seeds size.

3.1. Effect of pH

The measured percent removal of calcium ions at different pH levels of the concentrate is presented in Fig. 2. The experimental conditions are given in Table 3 for the experiments from 1 to 4. The experiments were conducted with a concentrate flow rate of 1 L/min and seeds concentration of 1 g/L and seeds size of 250 μm . As Fig. 2 shows, the percent removal of calcium ions are 90.11, 34.26, 23.6, and 9.8% at pH levels of 10.6, 9.8, 9, and 8.4, respectively.

The highest percent removal of calcium ions was achieved at higher pH levels (pH 10.6). Higher removal of calcium ions by crystallization as calcium carbonate at the highest pH value is attributed to the direct influence of pH on the level of supersaturation and the conversion of bicarbonate to carbonate. Therefore, increasing the pH leads to a decrease in H^+ concentration and consequently intensifications in the generation of carbonate ions which reacts with

Table 2

Summary of the experimental conditions for seeded continuous crystallization of CaCO_3 from supersaturated synthetic BWRO concentrates

Experiment #	Concentrate pH	Seeds size μm	Seeds concentration g/L	Concentrate flow rate L/min	Concentrate residence time min	Slurry flow rate mL/min
1	10.6	250	1	1	1	29.5
2	9.8					
3	9					
4	8.4					
5	10.6	250	1	0.1	10	3
6				0.3	3.33	9
7				0.5	2	15
8	10.6	250	1.5	1	1	45
9			3			95
10			3.5			110
11	10.6	200	3	1	1	95
12		150				
13		100				

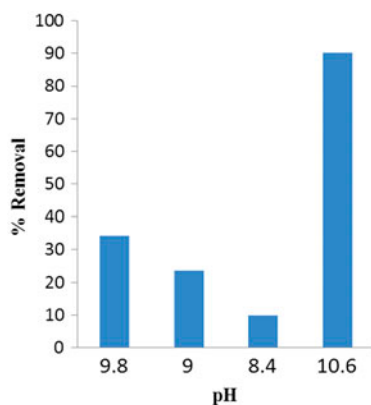


Fig. 2. Percent removal of calcium ions at different pH levels of the concentrate. Concentrate flow rate = 1 L/min, seeds concentration = 1 g/L, and seeds size = 250 μ m.

calcium ions to form the solid calcium carbonate that reflects growth of the seeds.

The measured calcium ions concentrations in the feed and effluent are 361 mg/L and 35.7 mg/L, respectively. However, this reduction in calcium ion concentration is not adequate to ensure feeding the water to RO systems without the risk of fouling of the membranes.

3.2. Effect of concentrate flow rate

The influence of concentrate flow rate was examined experimentally (experiments 5, 6, and 7) by setting the pH of the concentrate at a level of 10.6 and seeds density of 1 g/L and seeds size of 250 μ m. Fig. 3 displays the influence of flow rate on the percent removal of calcium ions. By examining Fig. 2, it can be concluded that reducing the flow rate leads to intensification in the reduction of calcium ions. The measured percentages of calcium ions removal are 92.88, 92.05, 90.91, and 90.11% at the flow rates 0.1, 0.3, 0.5, and 1 L/min, respectively.

The results show that the percent reduction of calcium ions increases with increasing concentrate

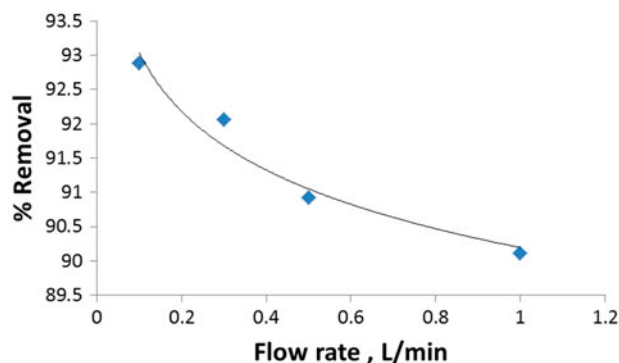


Fig. 3. Percent removal of calcium ions at different concentrate flow rates. pH 10.6, seeds concentration = 1 g/L, and seeds size = 250 μ m.

residence time for the contact between the supersaturated concentrate and the limestone seeds. However, the increase in percentage removal of calcium ions was moderate. As the flow of concentrate is reduced from 1 L/min (residence time 1 min) to 0.1 L/min (residence time 10 min), the percent reduction increased from 90.11 to 92.88%.

At the flow rate of 0.1 L/min, the measured concentration of calcium ions in the feed and effluent was 361 and 24.6 mg/L, respectively. Intensification of the reduction of calcium ion concentration at low flow rates might be operationally not advantageous if this process will be operating in line with RO system for concentrate reuse as such arrangement requires treating higher flow rates of the brine from PWRO plants. It is practically more attractive for this pretreatment process to be operated with higher flow rates and maximum removal of calcium ions.

3.3. Effect of seeds concentration

Increasing the seeds concentration at greater concentrate flow rate (1 L/min) was experimentally tested (experiments 8, 9, and 10). The measurements were conducted at pH value of 10.6 and seeds size of

Table 3
Comparison between the feed and treated concentrate

	Feed concentrate	Treated concentrate
Calcium ion concentration	361 mg/L	11.6 mg/L
HCO ₃ ⁻	1,199 mg/L	647 mg/L
pH	8.2	8.4
TDS	10,168 mg/L	9266.6 mg/L
Langelier saturation index	1.6	0.05
Stiff–Davis saturation index (SDSI)	1.62	0.2

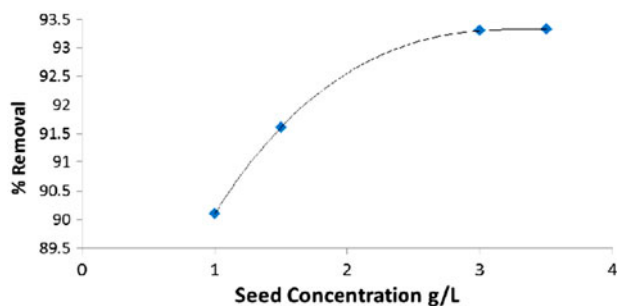


Fig. 4. Influence of seeds concentration on the percent removal of calcium ions at pH 10.6, seeds size = 250 μm , and flow rate 1 L/min.

250 μm . Fig. 4 represents the variation of the percent removal of calcium ions with seeds concentration. Obviously, there is an increase in the calcium ion removal from 90.11 to 93.3% as seeds concentration increases from 1 to 3 g/L. This is explained by the supplementary surface area available for seeds–concentrate interaction.

It is however worthwhile to notice that increasing the seeds concentration from 1 to 3 g/L has the same influence on the intensification of calcium ions removal as decreasing the flow rate from 1 to 0.1 L/min. However, increasing the seeds concentration with treating higher flow rates is more favorable for continuous in line reuse of the concentrate although increasing the seeds concentration always accompanied by additional filtration cost to separate the treated concentrate and recycle the seeds.

3.4. Effect of seeds size

The effect of particle size added to the crystallizers as seeds produced from limestone is studied experimentally (experiments 11, 12, and 13) at higher flow rate (1 L/min), seeds concentration of 3 g/L, and pH level of 10.6.

The crystal growth occurs when the ions migrate to active sites on the surface of the seed material and are incorporated into a crystal lattice. This phenomenon occurs on the surface only and the porosity of the seeds has minor role in this process. However, the available active sites at the surface are the main factors affecting the separation of scaling ions from the brine to the seeds surface.

Fig. 5 displays the influence of seeds size on the percent removal of calcium ions. The highest calcium ion reduction was achieved (96.76%) at smaller size of the seeds (100 μm). Smaller size seeds provides increased number of crystals. The active sites on the seed which is available for calcium carbonate growth

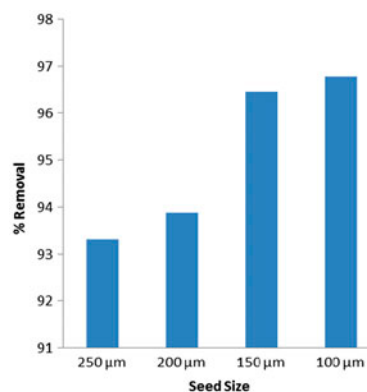


Fig. 5. Influence of seeds size on the percent removal of calcium ions at pH 10.6, seeds concentration = 3 g/L, and flow rate = 1 L/min.

are limited for each particle. Increasing the number of seeds means more active sites available for calcium carbonate growth. The relatively high number of active sites implies enhancement removal of calcium ions.

3.5. Treated concentrate evaluation

The analysis of the feed concentrate and treated concentrate at the above mentioned conditions (pH 10.6; seeds concentration = 3 g/L; flow rate = 1 L/min; seeds size 100 μm ; and residence time = 1 min) is given in Table 3. It can be concluded from Table 3 that the treatment of the concentrate resulted in a reduction of Langelier saturation index (LSI) from 1.6 to 0.05. The LSI represents an indication of the degree of supersaturation with respect to calcium carbonate and thus the tendency to membrane fouling. The LSI value of 0.05 for the effluent stream reveals that the treated concentrate has insignificant supersaturation with respect to calcium carbonate and therefore can be fed to RO system with high degree of confidence that the scaling will not occur at the surface of the membrane.

The Stiff–Davis saturation index (SDSI) is more appropriate to evaluate the tendency toward calcium carbonate scaling for highly concentrate waters (> 5 g/L). Table 3 shows a strong reduction in SDSI from 1.62 to 0.2 of the concentrated treated by the seeded crystallization method. Although the positive value of SDSI indicating small level of calcium carbonate scaling potential, however this small amount means very large induction period and negligible supersaturation for crystal growth. The evaluation using SDSI gives also an indication that the seeded crystallization pretreatment process has the potential to reduce the scaling potential of the concentrate

produced in the RO plants. On the other hand, the low concentration of calcium ions in the treated concentrate diminish the chance for other calcium scaling components such as calcium sulfate to precipitate on the membrane system.

The properties of the treated brine and the process parameters investigated in this study indicated that the industrial application of seeded crystallizer inline between the principal RO and the secondary RO systems seems to be beneficial due to the advantage of fast crystallization with small residence time. The small residence time (1 min) is advantageous in terms of smaller crystallization equipment and higher operational flexibility. For example, in a RO train with a feed of 1,525 gpm, the concentrate is 305 gpm (based on 80% recovery). The crystallizer size is 305 gallon if the residence time is 1 min.

Industrially, the effluent from the seeded crystallizer requires solid–liquid separation to recover the seeds and to provide a feed to secondary RO system with the required specifications in terms of solid content. The solid–liquid separation should be of two stages: cyclone/settling followed by microfiltration membrane.

4. Conclusion

Reuse of BWRO by-product concentrates which produced in large quantities is an important environmental and economic challenge for desalination of underground and surface saline waters using RO systems. These concentrates contains high amounts of salts and unrecovered water. Recycling of these concentrates to secondary RO units for potable water recovery is restricted due to the treat of scaling. This work investigated the treatment of these concentrates in a continuous manner to be operated in line between the primary and secondary RO systems to increase the water recovery from one side and to decrease the volume of the concentrate on the other side. The results obtained revealed that the continuous seeded crystallization is a potential and attractive process for reduction of scaling tendency with respect to calcium carbonates and can be implemented in a continuous manner.

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