Evaluation of cellulose triacetate hollow fiber membrane for volume reduction of real industrial effluents through an osmotic concentration process: A pilot-scale study

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ABSTRACT

The current article tackles the challenge of reducing wastewater volumes generated from the gas industry. A forward osmosis (FO) pilot unit, deployed as osmotic concentration (OC) process without the draw solution (DS) recovery step, was applied as an option for volume reduction of real industrial effluents. A commercial hollow fiber (HF) FO membrane fabricated from Cellulose Triacetate (CTA) was firstly tested with synthetic feed solution (FS) to investigate the separation properties of the membrane and to identify the optimum operating conditions of the pilot unit. The pilot plant was then challenged with real industrial wastewater for an extended period of operation, primarily to assess membrane-fouling propensities and other performance parameters. Results revealed that according to the operating conditions, the CTA membrane can achieve feed recoveries between 60%–90%, at water fluxes between 2.24-1.65 L.m^{-2} h^{-1} (LMH)). The operation at 75% feed recovery was identified as the optimum condition since it showed the lowest specific solute flux (20.93 mmol L^{-1}) at a water flux of 1.94 LMH. Outcomes of pilot testing with the real wastewater demonstrated operational stability for over 50 h of continuous operation. The pilot system recovered 75% of the wastewater feed at a stable flux trend with minimal flux decline. Water flux of 1.76 LMH was recorded along with reverse solute flux of 292 mmol h^{-1}. The water flux was observed to decline slightly by only 5.6%, which was attributed to inorganic scaling on the membrane surface where cleaning with citric acid solution demonstrated efficacy in restoring the initial flux.

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1. Introduction

Significant volumes of wastewater are generated during oil and gas (O&G) operations. Managing this wastewater has been a global environmental concern that represents a key challenge to the O&G industry (Zhao et al., 2017). Effluent disposal by deep-well injection is a common practice for O&G wastewater management. Yet, this route is hindered by the limited capacity of underground formations, stringent regulations to minimize potential hazards of contaminating underground water sources due to well leakages, and high costs associated with well-drilling operations (Simpson and
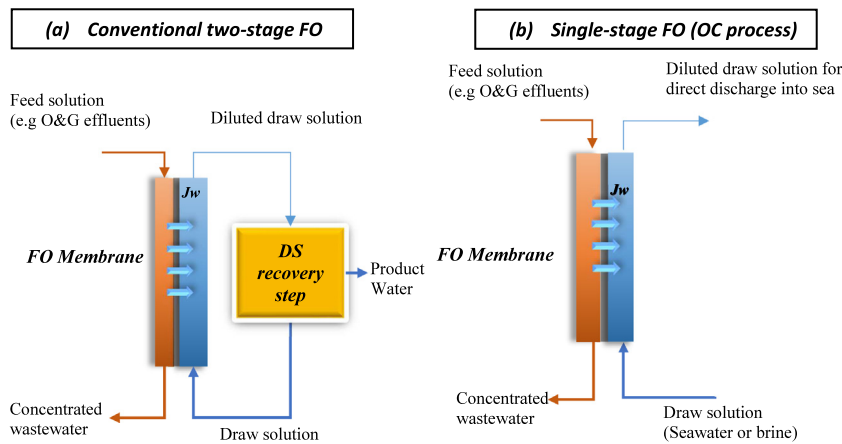
Fig. 1. Process schemes of (a) conventional two-stage FO, (b) OC process.

Lester, 2009). The aforementioned challenges highlight the need for proper treatment of O&G effluents before end-disposal or, in other cases, reuse for well fracturing and other applications.

Forward osmosis (FO) has emerged as a low-cost alternative technology to classical membrane filtration owing to its potential to treat challenging water sources with high solute rejections at low fouling propensity (Bell et al., 2017; She et al., 2016; Jalab et al., 2019). The FO process is based on the osmosis phenomena, where pure water is transferred from a less to more concentrated solution across a semi-permeable membrane. The osmotic pressure gradient across the surface of the membrane allows the transfer of water molecules from the feed solution (FS) to the draw solution (DS) "high concentration stream". In the conventional FO technology, water permeate is extracted from the DS via a further downstream process, which has been historically based on either a pressure-driven membrane or a thermally-based technique (e.g membrane distillation) (Subramani and Jacangelo, 2015). It has been widely acknowledged that the downstream DS recovery step remains a high-energy process (She et al., 2016; Awad et al., 2019; Shaffer et al., 2015; Valladares Linares et al., 2014), rending the overall energy costs of the FO process infeasible (Shaffer et al., 2015; Valladares Linares et al., 2014). FO applications for the treatment of O&G wastewater have been accentuated in recent research studies (Coday et al., 2014; Coday and Cath, 2014; Li et al., 2014; Maltos et al., 2018). However, in all cases, the energy efficiency is constrained by the requirement to recover the DS, the current bottleneck of the process (Awad et al., 2019; Shaffer et al., 2015). If this step is obviated, the stand-alone FO membrane separation stage is close to zero energy.

In this regard, Osmotic Concentration (OC), a relatively new application of FO, obviating the energy-intensive step (DS recovery), has shown the potential to treat O&G effluents. In the OC process, the high concentration solution (DS) is used to draw pure water from the low concentration feedwater (i.e wastewater), thereby the volume of the wastewater is reduced while diluting the DS. Therefore, OC is a favorable application when the main target is to minimize wastewater volumes before end-disposal. Compared to conventional FO (Fig. 1), the OC approach can use readily available seawater or brine as DS, then, instead of recovering the water permeate from the draw solution, the diluted DS stream can be directly discharged into the ocean (Minier-Matar et al., 2015). Consequently, dual benefits arise from reducing the volume of disposal quantity while providing a degree of dilution for a highly saline brine. In addition, compared to conventional seawater RO membrane, the OC process has significantly lower energy consumption, since the FO membrane requires lower applied pressure when the DS recovery step is obviated. It has been reported that the OC has around 81% lower energy demand (0.2 kWh/m^3) as compared to RO (1.1 kWh/m^3) for 50% volume reduction of industrial wastewater, assuming that both processes are fed with similar water quality. Thus, the successful demonstration of the technology can provide suitable management schemes for O&G effluents as well as brine waste streams generated from desalination plants.

Notwithstanding the recent surge of interest in FO research and development (Awad et al., 2019; Suwaileh et al., 2020; Corzo et al., 2017, 2018; Akther et al., 2015), limited studies have exploited the FO as a low-energy process (i.e single-stage process), when recovering the DS is not required (Chekli et al., 2017; Phuntsho et al., 2016; Kim et al., 2018; Hancock et al., 2013a; Choi et al., 2017). In the O&G field, Hutchings et al. (2010) deployed a single-stage FO to reclaim 473.17 m^3 drilling waste, while utilizing the diluted DS as completion fluid for subsequent well-drilling operations. They reported a 50%–80% volume reduction of drilling wastewater generated from shale gas field operations (Hickenbottom et al., 2013). In another bench-scale study, Joel and his coworkers (Minier-Matar et al., 2015, 2016) demonstrated the potential of the OC process for volume reduction of combined produced and process water (PPW) from Qatari's gas processing facilities. Their results revealed the feasibility of the process at a bench-scale for 50% volume reduction.

The separation properties of FO membranes are significantly influenced by the operating conditions encompassing FS, DS cross flowrates, as well as the osmotic pressure gradient. These parameters determine the attainable, water flux (J_w),

feed recovery, and reverse solute flux (J_s). It has been demonstrated that high flowrates of the feed and draw solution streams are favorable for higher water flux, and lower J_s, mainly due to the reduced effect of concentration polarization on the surface of the membrane (Hawari et al., 2016). The technical viability of the osmotic unit is also robustly linked to the attainable water flux, membrane fouling, and cleaning efficacy for fouling mitigation. The severity of fouling as well as the membrane cleaning requirements are generally a function of several factors such as contaminants' type and concentration, hydrodynamic conditions of the membrane module, and the imposed initial flux (Im et al., 2020). During the bench-scale study of the OC process for PPW volume reduction, around 15% flux decline was observed due to organic fouling. This flux deterioration was, nonetheless, 100% recoverable after membrane cleaning with sodium dodecyl sulfate (SD). Furthermore, with the use of a proper pretreatment, no membrane fouling was observed for a TFC FO membrane (Minier-Matar et al., 2015). In another work, Hickenbottom et al. (2013) reported minimal irreversible fouling when CTA FO membranes were applied for the osmotic concentration of drilling wastewater.

Despite their promising outcomes, prior investigations of the OC process are predominantly based on the laboratory scale (Zhao et al., 2017; Minier-Matar et al., 2015), and the reliability of the process at a larger scale, concerning the real operation challenges during an extended period of operation, is not yet investigated. This includes performance deterioration due to fouling in long-term operation and the subsequent cleaning requirements for fouling mitigation. Additionally, while HF-FO membranes are commercially available, their viability for the full-scale field implementation of the OC of the O&G effluents stream before end disposal has not previously been well assessed.

In light of the above, the current article provides a rigorous demonstration of the OC technology at a pilot-scale challenged with real wastewater and representative conditions. A pilot unit was constructed for volume reduction of process water generated from the gas field operations. The OC unit was firstly challenged with synthetic water, mimicking the salinity of Qatari's real PW stream and salt-rich solution of 40,000 mg L^{-1}; representing the salinity of seawater. These tests are intended to investigate the impact of the operating conditions (i.e FS, DS flowrates) on the water flux, recovery rate, and reverse solute flux. The pilot unit was operated at feed recovery rates of 60% to 90%, which correspond to a feed flowrate of 1–2 L min^{-1} and a DS flowrate of 0.33–0.4 L min^{-1}. After identifying the best conditions for the unit operation, the pilot unit was tested under the real process water stream at a 75% feed recovery rate. For the first time, the robustness of the OC process is examined by testing the pilot unit for 50 h of continuous operation; using a CTA HF FO module. This study also explores membrane fouling propensity and cleaning efficacy for fouling amelioration of the OC process.

2. Materials and methods

2.1. OC pilot-scale system

A pilot-scale plant was used to evaluate the OC process for reducing wastewater volumes from the gas field's operations. The pilot unit was constructed in a 4.81 m × 2.26 m platform skid at the Pilot Hall at the Gas Processing Center (GPC), Qatar University. The unit system was designed based on a batch operation of 5000 L wastewater feed. Fig. 2 shows the schematic diagram of the pilot unit, which consists of four 5000 L storage tanks, two diaphragm pumps (*Sterlitech, Switzerland*), one transfer pump, a clean-in-place tank, $5\mu m$ cartridge filters, and HF FO membrane (Toyobo, Japan). The diaphragm pumps were used to circulate FS and DS within the membrane element, while the cartridge filters were employed to ensure the removal of any suspended solids from the solutions before entering the membrane module. The pilot plant also encompassed sensors (*Omega, UK*) for on-line monitoring of flows, pressures, temperatures, and water salinity, the latter primarily based on conductivity meters (*Thermo Fisher Scientific, US*). More details about the pilot system design can be found in our previous publication (Jalab et al., 2020).

The pilot system was operated with a real-time control system implemented by LabVIEW (*cRIO 9035, National Instruments, USA*). The LabVIEW user interface allows monitoring and recording different performance parameters, while a PID controller was used to maintain constant cross-flow rates of the FS and DS. The water flux of Toyobo membrane was measured from the difference between the inlet and outlet flowrates. All wiring and control system components were installed in a control panel with an emergency shut down button and alarm indicators to ensure the safe operation of the pilot unit.

2.2. FO membrane

A commercial hollow fiber FO membrane developed by Toyobo Co., Ltd, (Japan) was utilized in the pilot unit. The membrane is fabricated from cellulose triacetate (CTA) with a large number of fibers, packed in a cylindrical pressure vessel, which provides high packing densities (large effective surface area to volume ratios). Table 1 shows detailed specifications of Toyobo membrane.

The pure water and salt (NaCl) permeability coefficients were measured for the CTA membrane. The FS was either deionized (DI) water for the permeability coefficient test or a 500 mg L^{-1} NaCl solution for the salt rejection experiment. The system was pressurized to 2 bar. When the operation reached a steady state (equilibrium), the permeate water was collected for 60 s, and the values of A and B were calculated. Table 2 shows the values of water and salt permeability coefficients, as well as rejection values at different temperatures.
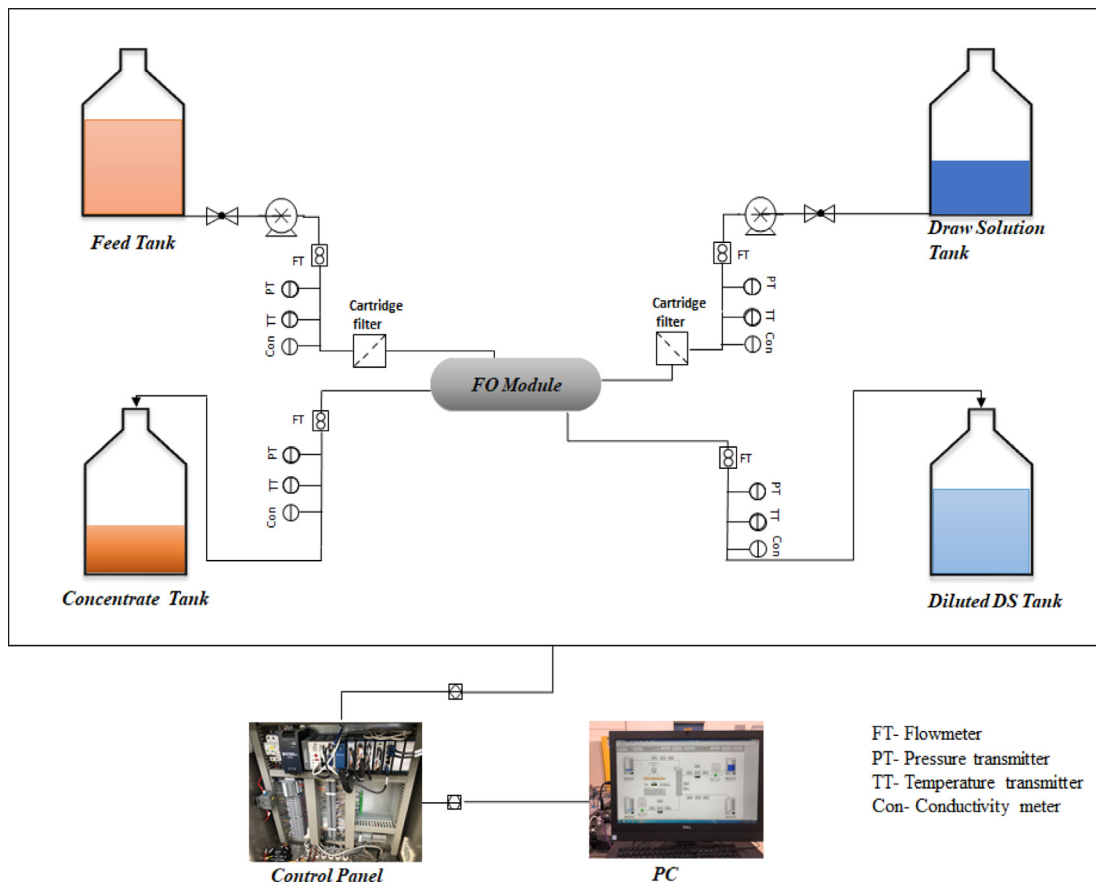
Fig. 2. Schematic of the OC pilot unit.

Table 1
Detailed membrane specifications for Toyobo module.

	Membrane configuration	HF
Membrane	Outer diameter, μm	175
	Inner diameter, μm	85
	Membrane Surface Area, m^2	31.5
Module	Vessel material	polyvinyl chloride (PVC)
	Dimension, mm	Inside diameter $\sim$ 103, Length $\sim$ 830
Operating Conditions	Pressure, bar[a]	Shell $<$ 4.9, Bore $<$ 2
	Temperature, °C	5–40
	pH	3–8
	Residual Chlorine, mg L^{-1}	$\leq$1

[a]These values are the maximum operating conditions, provided by the manufacturer. The accrual operating pressure of the conducted experiments ranged from 0.05 to 0.365 bar.

Table 2
Water and salt permeability coefficients at different temperatures for the CTA-Toyobo membrane used in this study.

Temperature, °C	Water permeability coefficient (A), LMH/bar	Salt permeability coefficients (B), LMH	NaCl Rejection, %
20	0.195	0.049	94.2%
24.7	0.225	0.053	94.5%
29.3	0.255	0.058	94.4%

Table 3
Water quality of real wastewater, synthetic feed, and draw solution.

Parameter	Real wastewater (PW)	Synthetic FS*	DS
TDS, mg L^{-1}	1120	2000	40000
TSS, mg L^{-1}	–	1.30	15.20
Inorganic carbon (IC), mg L^{-1}	76	18.75	15.40
Total Organic Carbon (TOC), mg L^{-1}	10.	0.32	0.70
pH	7.10	7.90	7.74
Conductivity, μS cm^{-1}	2155	4000	64400
Turbidity, NTU	0.34	0.14	0.72
Residual Cl_2, mg L^{-1}	<0.10	<0.1	<0.1
Chloride, mg L^{-1}	253	<0.1	<5
Sodium, mg L^{-1}	481	<0.1	<5
Sulfate, mg L^{-1}	245	<0.1	<5
Phosphate, mg L^{-1}	17.80	<0.1	<5
Calcium, mg L^{-1}	3.30	<0.1	<5
Magnesium, mg L^{-1}	3.20	<0.1	<5

• Feed solution used for the membrane integrity test.

2.3. Feed and draw solutions

2.3.1. Synthetic waters

The pilot unit was firstly tested under synthetic feed and draw solutions; primarily to evaluate the basic separation properties (i.e water flux, recovery rate) of the CTA FO membrane at various recovery rates, and to optimize the operating conditions of the pilot unit. For these experimental trials, an industrial-grade sodium chloride (NaCl) salt (*99% purity, ESS-Middle East*) was used to prepare the synthetic FS and DS. The FS was prepared by dissolving NaCl in tap water that is filtered by three stages cartridge filter (5 μm, Altlas Filtri, Italy).

A synthetically prepared NaCl solution was used as DS for this pilot study. The DS was prepared with a salinity of 40,000 mg L^{-1}, mimicking the salinity of the Arabian Gulf seawater. Detailed water quality of the synthetic solutions is shown in Table 3.

2.3.2. Real wastewater

The wastewater feed used in the demonstration plant was gas field onshore process water (PW) used during natural gas conditioning and treatment. 5000 L of PW was collected from Qatargas industrial site at Ras Laffan. This wastewater is a mixture of different process water streams; previously pretreated with membrane bioreactor (MBR). The effluent was characterized to identify organic and inorganic constituents and to ensure that the pH and chlorine residual amounts are within the range specified by the membrane manufacturer. The pH of the feed water was adjusted to 7–8, by adding hydrochloric acid (HCL) solution. Table 3 shows the chemical composition and characteristics of the wastewater after pH adjustment.

2.4. Experimental procedures

The course of the experimental program entailed testing the CTA membrane with both synthetic and real wastewater. Short-term experiments (i.e., up to 4 h) were conducted using synthetic FS and DS, to examine the role of operating conditions in the performance of the pilot unit. The experiments were conducted, targeting feed recovery rates between 60% to 90%, at a constant dilution of the DS, and an operating temperature of 27 °C. A long-term experiment using the real wastewater was conducted at a 75% volume reduction of the PW feed at a constant DS dilution of 4X. This percent recovery was identified as the optimum condition after testing the membrane at different feed recoveries (60%, 68%, 75%, and 90%.) using synthetic solutions as revealed by the short-term experiments.

The pilot unit was operated in one pass-through (FS and DS are both in once-through mode), while the concentrated FS outlet and the diluted DS outlet are sent into separate storage tanks. The CTA HF module was positioned horizontally and operated in counter-current mode (Fig. 3), with the FS flowing in the shell side, while the DS flowing into the bore-side (outside-in configuration). Manipulation of both FS and DS flowrates permits operating Toyobo module for a feed recovery up to 90%. The percentage volume reduction (recovery rate) of the wastewater and DS dilution rate can be calculated as follows:

$$Volume\ reduction, \% = \frac{Q_{F_{in}} - Q_{F_{out}}}{Q_{F_{in}}} \times 100 \tag{1}$$

$$DS\ Dilution, \% = \frac{Q_{DS_{in}}}{Q_{DS_{out}}} \times 100 \tag{2}$$

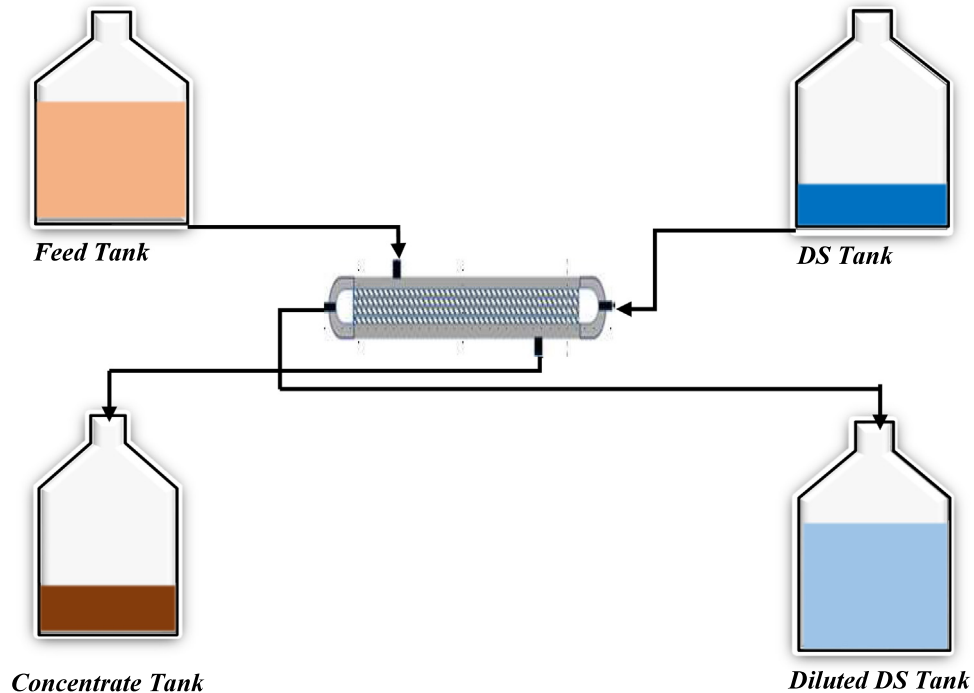**Fig. 3.** One-pass through operation mode of the pilot unit.

where $Q_{F_{in}}$ and $Q_{F_{out}}$ are the flowrates in L min^{-1} of inlet and outlet feed streams. $Q_{DS_{in}}$ is the inlet DS flowrate, and $Q_{DS_{out}}$ is the outlet DS flowrate in L min^{-1}.

The water flux was estimated based on the flowrates of the inlet and outlet of each stream according to the following equations (Eqs. (3) and (4)):

$$J_{W,FS} = \frac{Q_{F_{in}} - Q_{F_{out}}}{A} \tag{3}$$

$$J_{w,DS} = \frac{Q_{DS_{out}} - Q_{DS_{in}}}{A} \tag{4}$$

where $J_{W,FS}$ and $J_{w,DS}$ are the water fluxes (in L m^{-2} h^{-1} (LMH)) based on the FS and DS stream; respectively. $Q_{F_{in}} - Q_{F_{out}}$ represents the amount of water (in L m^{-1}) transferred from the feed to draw solution side across the membrane area (A in m^2).

The reverse solute flux was determined from the overall mass balance of NaCl in either the feed or draw solution stream. The online conductivity measurements were used to obtain the NaCl mass flowrate and the J_s in mmol h^{-1} m^{-2} was calculated as follows (Eq. (5)):

$$J_s = \frac{(C_{in}Q_{Fin} - C_{out}Q_{F\_out}) \times 60}{AM_w} \tag{5}$$

where C_{in} and C_{out} are NaCl concentrations in the inlet and outlet feed streams, respectively. M_w is the molecular weight of NaCl.

The experiments on synthetic wastewater were duplicated to assure reproducibility of the data generated by the system. Experimental outcomes were reproducible with a maximum absolute error of less than 3% (i.e. the difference between the duplicated experiments).

2.5. Membrane integrity test and cleaning protocol

Membrane integrity tests (using synthetic solutions) were conducted before and after the trial on the PW; primarily to assess the water flux and Js of the virgin membrane and to assess the fouling tendency of the membrane after trials on the real wastewater. After each experiment with the synthetic water, the surface of the membrane was flushed with tap water for 30 min. However, after trials on the real wastewater, the membrane was subjected to two chemical cleaning cycles, targeting the removal of organic and inorganic foulants from the surface of the membrane. In the first cleaning cycle, Toyobo membrane was cleaned by running a 10 mmol L^{-1} SDS solution on the feed side of the membrane at a pH
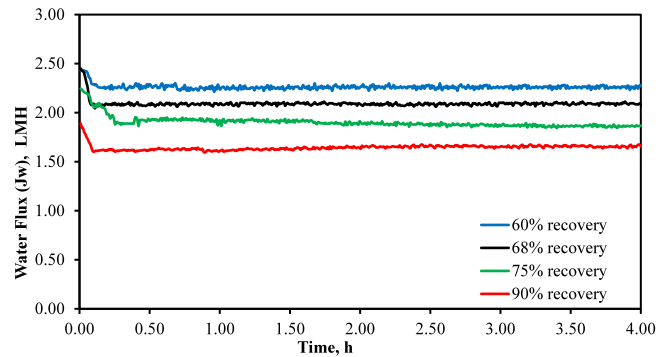
Fig. 4. Water flux of CTA HF Toyobo membrane at different feed recoveries at 27 °C.

of 4 and a flowrate of 1.5 L min^{-1} for 15 min. Then the module was flushed with tap water at a flow rate of 1.5 L min^{-1} for 30 min. The second cleaning cycle was mainly aimed to remove inorganic scaling (calcium carbonate, and calcium and iron phosphates). For this duty, citric acid solution (2%) was employed as a cleaning reagent. Toyobo was cleaned with citric acid at a pH 5 and a flowrate of 1.5 L min^{-1} for 15 min in recirculation mode. After the second cleaning cycle, the membrane was flushed with tap water for 30 min.

2.6. Analytical methods

During the long-term experiment with the PW feed, two sets of samples were collected daily and analyzed. A set of a sample includes four streams: PW feed, concentrated wastewater, DS, and diluted DS. A total of 18 samples for each membrane were collected characterized for turbidity (*2100N Turbidmeter, Hach, USA*), pH (*907 Titrando, Metrohm, Switzerland*), and residual chlorine (*CN-66, Hach, USA*). The total organic and inorganic contents were measured by *TOC-L, Shimadzu, Japan*. Moreover, at the end of each long-term test, the four water streams were analyzed for cation and anion concentrations using ion chromatography (*ICS 6000, Thermo Scientific, USA*). Inductively coupled plasma (*ICAP 6500, Thermo Scientific, USA*) was used to analyze metals.

3. Results and discussion

3.1. OC process performance with synthetic wastewater

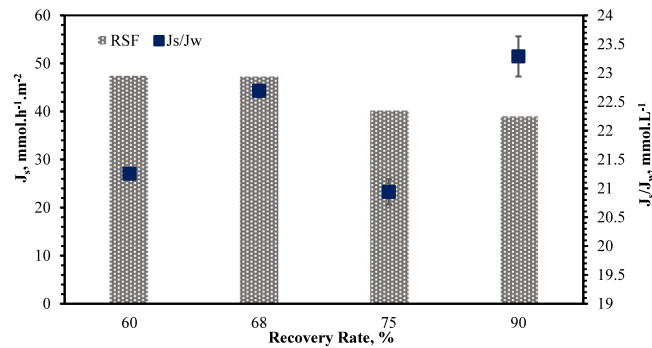#### 3.1.1. Water flux and recovery rate

Water fluxes generated from the experimental trials on synthetic solutions are illustrated in Fig. 4. It can be seen that the attainable water flux varies with the target feed recovery. Based on the experimental procedure of the Toyobo module, manipulation of FS and DS flowrates permits operating the pilot unit at different feed recoveries. For all experiments, the target DS dilution rate was 75% which was achieved by changing the DS flowrate. Table 4 shows that as the flowrate of feed increases, a higher draw solution flowrate is required to obtain a 75% DS dilution rate.

As shown in Fig. 4, the water flux ranged between 2.24 LMH (for 60% feed recovery) to 1.65 LMH (for 90% recovery rate) at an operating temperature of 27 °C. Fig. 4 shows stable flux trends, which indicate that the osmotic pressure gradient was maintained during the entire period of operation. Nevertheless, the water flux declined slightly at the beginning of each run which can be ascribed to the instability of water permeate through the membrane while adjusting the flowrates of feed and draw solutions. The flux values (2.24–1.65 LMH) observed in the current study are comparable to those reported in the literature under approximately the same osmotic pressure difference (Corzo et al., 2018; Shibuya et al., 2015; Minier-Matar et al., 2021). Shibuya and his coworkers (Shibuya et al., 2015) demonstrated that CTA Toyobo membranes can attain a water flux of 3 LMH when a 0.69M NaCl was used as a DS to remove pure water from 1.5 g L^{-1} NaCl feed water. In a bench-scale study (Minier-Matar et al., 2021), CTA membranes showed water fluxes of 1.7–2.1 LMH for synthetic feed solution with a salinity of 1.7 g L^{-1} and a DS concentration of 40 g L^{-1}. In another study, Corzo et al. (2018) reported a flux value of 2.2 LMH for thin-film composite membrane, treating feed water with a salinity of 1.7–2.8 g L^{-1} using 36 g L^{-1} MgSO$_4$ solution as a DS.

Fig. 4. Indicates the inverse relation between membrane water flux and feed recovery rate. It was observed that the operation at higher feed recoveries requires adapting lower flowrates of both FS and DS (Table 4). It is known that better turbulence and fluid mixing conditions can be obtained at higher flowrates which has a favorable role in minimizing the impact of the concentration polarization (CP) phenomenon. Therefore, the reduced water fluxes at lower flowrates (or higher recoveries) were likely resulted from the reduced impact of CP on the surface of the membrane. Similar observations were reported in the literature for the favorable impact of the high cross-flow velocity of FS and DS streams (Chowdhury and McCutcheon, 2018; Holloway et al., 2015).

Table 4
Flowrates of feed and draw solutions at different feed recoveries and 75% DS dilution..

FS Flowrate, L min^{-1}	DS flowrate required to maintain 75% DS dilution, L min^{-1}	Feed Recovery, %
2	0.4	60%
1.6	0.37	68%
1.35	0.35	75%
1	0.33	90%

Fig. 5. Reverse solute flux and specific reverse solute flux for Toyobo testing at different feed recoveries at 27 °C.

Table 4 demonstrates that the achievable recovery rates are inversely proportional to the FS and DS flowrates. Adopting higher flowrates results in lower feed recoveries. As shown in Table 4 a maximum recovery rate of 90% was recorded at flowrates of 1 and 0.33 L min^{-1} for the feed and draw solutions respectively. Increasing the FS flowrate to 1.6 L min^{-1} and the DS flowrate to 0.37 L min^{-1} led to a 9.3% reduction in the feed recovery. The lowest feed recovery of 60% was obtained at flowrates of 2 L min^{-1} and 0.4 L min^{-1} for FS and DS respectively. Similar observations of this relationship between FS flowrates and feed recoveries were reported for using a spiral wound FO membrane (Im et al., 2018).

3.1.2. Reverse solute flux

Fig. 5 illustrates the J_s obtained from the pilot testing of Toyobo module at different feed recoveries (60%, 68%, 75%, and 90%) at a temperature of 27 °C. The J_s was measured based on the mass balance determined by the conductivity measurements of the feed and draw solution streams. The J_s is an indication of the reverse flow of salts – NaCl in this case – from DS to FS, in a direction opposes to the conventional water flow. When the J_s increases, more amount of solutes is lost from the DS to the FS stream, which has adverse effects on the net osmotic driving force across the membrane surface. This eventually increases the frequency of periodical DS replenishment to restore the concentration of the DS and to maintain an adequate osmotic separation force. Fig. 5. demonstrates that as the target feed recovery increases, fewer amounts of solutes are transported from the DS into the FS side.

During the membrane testing, a minimum J_s value of 39 mmol h^{-1} m^{-2} was observed at the highest feed recovery (90%). On the other hand, operating the pilot unit at the lowest feed recovery (60%) showed the maximum J_s value of 47.4 mmol h^{-1} m^{-2} which is around 21% higher than the J_s observed at 90% recovery rate. It is worth mentioning that at modest feed recovery (75%), the J_s was 3% higher and 15% lower than the values recorded at 90%, and 60% recoveries respectively. There is a direct relationship between reverse solute flux and water flux, primarily due to the influence of FS and DS flowrates on the flux and J_s. It was found that J_s was higher at lower feed recovery rates when the average water flux was higher. Heo et al. (2016) also reported on the direct relationship between water flux and J_s for several draw solutions at varying concentrations.

The impact of reverse solute flux on the process performance can be better analyzed by the specific reverse solute flux, which represents the loss of the draw solute per the amount of water pass through the membrane (J_s/J_w). Fig. 5 also illustrates that the specific reverse solute flux exhibited a fluctuating trend with feed recovery rate. This is because the values of J_s/J_w depend on the rate of change of both J_s and water flux when the recovery rate is changed. For instance, increasing the feed recovery rate from 75% to 90% resulted in a 10% lower flux, while the J_s was reduced by only 3%, which led to higher J_s/J_w at a 90% recovery rate. On the contrary, when the feed recovery was increased from 68 to 75%, the rate of reduction in the J_s was higher than the reduction in the water flux which resulted in a 7% lower J_s/J_w at 75% recovery compared to 68% feed recovery. Overall, operating the pilot unit at 75% feed recovery showed the lowest J_s/J_w of 20.93 mmol L^{-1}. It is worth noting that a low value of the J_s/J_w is preferable; since it indicates a lower loss of the draw solution solutes.

It is evident that the operating conditions of the pilot unit can play a vitally important role in determining the performance of the OC process. Results demonstrated that there is a trade-off between achieving sufficiently high water
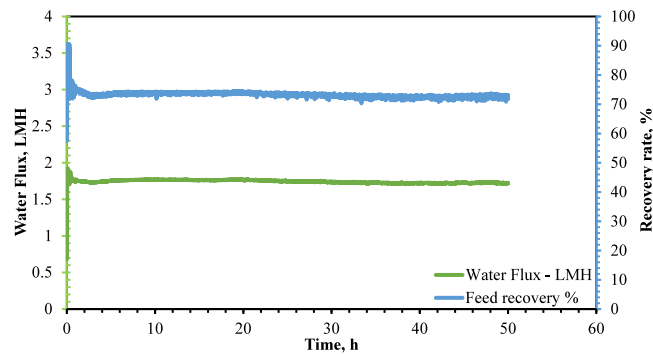
Fig. 6. Water flux and feed recovery rate for Toyobo testing with PW feed at 22 °C.

flux and recovery rate while minimizing the J_s. The operation at the lowest feed recovery of 60% (i.e highest flowrates) resulted in the greatest generated flux (2.2 LMH) as well as the highest J_s. In contrast, the OC system can achieve the highest recovery rate of 90% at the lowest flowrates when the flux and J_s were at their minimum values. Moreover, the operation at the highest flowrates would result in greater stress on the membrane fibers while the operation at very low flowrates can lead to an increased effect of fouling and concentration polarization.

In light of the above discussion, it was evident that operating the pilot unit at intermediate flowrates to achieve 75% feed recovery can provide the best performance of the OC system since a reasonable water flux (1.94 LMH) and the lowest J_s/J_w (20.93 mmol L^{-1}) were recorded at a reduced effect of membrane fouling or feed concentration stress.

3.2. OC process performance with real wastewater

3.2.1. Water recovery, flux, and reverse solute flux

The performance of the OC process for 50 h of continuous operation at 22 °C is shown in Fig. 6. The pilot unit was operated to achieve 75% recovery (volume reduction) of the wastewater feed at 4X dilution of the DS. Based on the experimental procedure of Toyobo module, manipulation of FS and DS flowrates permits operating the pilot unit at the target feed recovery, which was obtained by setting flowrates of the FS and DS at 1.2 and 0.30 L min^{-1}; respectively. It can be seen that (Fig. 6), the feed recovery rate was maintained stable between 75%–74% for around 29 h, before a slight decrease to 72% at the end of the experiment. This lower recovery rate was primarily associated with the decline in the membrane water flux, which resulted in lower permeate across the membrane, and a higher flow rate of the concentrate feed stream. Overall, Toyobo was able to recover around 3262 L of the wastewater during the 50 h of operation at a constant DS dilution of 75%.

The attainable water flux for Toyobo membrane with the real PW is illustrated in Fig. 6, where a slight loss in the water flux can be observed due to the instability of water permeate across the membrane at the beginning of the experiment when the FS and DS flowrates were adjusted. After 5 h of operation, the pilot unit was able to achieve a stable flux of 1.76 LMH for around 22 h, which then slightly descended to reach 1.72 LMH at the end of the experiment. This lower flux was mainly caused by the deposition of foulants on the surface of the membrane after exposure to the organic and inorganic constituents existing in the PW feed (Table 3).

It is worth mentioning that the attainable water fluxes for the osmotic unit operation relate mainly to the concentration driving force (concentration gradient) across the membrane surface. This concentration gradient is mainly controlled by the type and concentration of the DS and the feed concentration. Membrane fluxes increase as this concentration difference increases. During previous bench-scale testing (Minier-Matar et al., 2015), the OC process showed 42% higher water fluxes than those observed in the current pilot testing. The greater water flux of the bench-scale study is ascribed to the improved driving force resulting from the use of brine (instead of seawater) as DS. Highly saline brine can provide high osmotic pressure, consequently, a greater driving force for water permeation across the membrane. Thus, the selection of a suitable DS plays a vitally important role in determining high water fluxes for the OC membranes. It is worth mentioning that, the flux values achieved during both the bench and pilot testing are sensitive to the quality of the feed-water (i.e specifically the salinity). The pretreated wastewater used in our experimental program is of comparatively low conductivity (∼2 g. L^{-1} TDS). However, if the OC system is fed with highly saline wastewater, flux values, as well as the degree of the volume reduction (recovery %), might diminish significantly. Therefore, the most favored application for the OC technology is for the concentration (i.e. volume reduction) of low-salinity waters.

Fig. 7 illustrates the solute flux (of NaCl) and the conductivity measurements of the concentrate feed obtained from testing Toyobo module for the 50 h operation period at 22 °C. Toyobo exhibited a stable J_s between 8–9 mmol m^{-2} h^{-1}, however, after 22 h of operation, the J_s trend started to fall and reached around 6 mmol m^{-2} h^{-1} near the end of the experiment (after 50 h). This can be attributed to the decline in the membrane salt permeability due to the formation of a foulant layer on the membrane surface, which can present a barrier to reverse solute transport. Similar observations
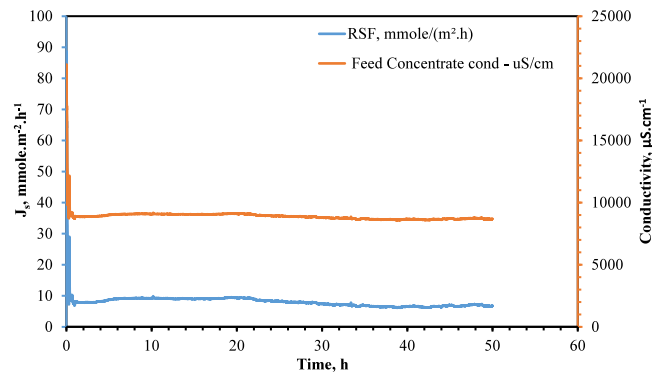
Fig. 7. The reverse solute flux of Toyobo membrane under 75% feed recovery at 22 °C.

for the impact of fouling on the J_s were reported in the literature (Tang et al., 2010; Boo et al., 2012) for other TFC-PA FO membranes. Hancock et al. (2013b) also reported around a 16.8% reduction in the J_s of multiple solutes as a result of membrane fouling. Moreover, Fig. 5 reveals that after 27 h of experimentation, the conductivity measurements of the concentrated FS was reduced slightly from around 8897 to 8658 μS.cm^{-1}. The instantaneous decline (after 27 h) in both the membrane water flux and NaCl concentration (conductivity) of the concentrate feed, confirms that the lower reverse salt transport (i.e J_s) observed in Fig. 6, was mainly resulted from membrane fouling. To determine the solute transport of other organic constituents present in the wastewater feed, mass balance calculations were performed for the feed and draw solutions. Toyobo showed a forward organic solute flux of 1.6 mg. m^{-2} h^{-1}, which represents the amount of the organics transferred from the FS to the DS stream. When the organic solute flux is normalized by the water flux, the normalized organic flux value was 0.9. These results are in agreement with published data for CTA FO membranes (Sauchelli et al., 2018; Zou et al., 2019).

3.2.2. Membrane fouling

To assess the severity of membrane fouling, a baseline test was conducted before (initial) and after (final) trials on the PW stream. Both baseline experiments were conducted with synthetic solutions (2000 mg L^{-1} FS and 40,000 mg L^{-1} DS). As shown in Fig. 8, for Toyobo membrane the water flux observed in the initial baseline test (before introducing the PW to the membrane) was 1.64 LMH, however, when the real PW was used as a feed, the membrane generated slightly higher water flux (1.76 LMH). The lower flux of the initial baseline was primarily attributed to the reduced driving force related to the higher salinity of the synthetic feed water (4000 c.f ~2155 uS/cm) as compared to the real PW stream. Fig. 8 clearly illustrates a noticeable flux decline from 1.64 LMH for the initial baseline to 1.55 LMH for the final baseline (after the experiment on the real PW). Thus, around 5.6% loss in water flux was observed as a consequence of membrane fouling, yet, it was still not clear whether the membrane surface was fouled by the organic or inorganic contaminants. Therefore, to determine if the flux decline was due to inorganic fouling (scaling), OLI simulation software was used to assess the scaling tendency of the compounds present in the industrial wastewater. OLI simulations revealed that under the testing conditions, calcium phosphate (Hydroxyapatite) would precipitate on the surface of the membrane. OLI simulations also revealed that if the wastewater outlet pH is below 6.5, calcium phosphate will not precipitate on the membrane, thus, the observed flux decline in Fig. 8 was likely caused by the accumulation of inorganic foulants on the surface of the membrane.

The conclusion of the insignificance impact of the organic fouling makes sense since the total organic concentration in the feedwater is relatively low (<10 mg/L), therefore, in this case, it is unlikely that some organic would be attached to the surface of the membrane. In the previous OC bench-scale study (Minier-Matar et al., 2015), it has been demonstrated that only a 15% flux decline was due to organic fouling when PPW stream, with TOC of 120 mg/L, was used as feed. However, in the current study, the feed solution was only pretreated PW with a relatively low TOC concentration (~10 mg/L).

3.2.3. Cleaning and membrane integrity test

To further investigate the impact of organic fouling, and to determine if the fouling was reversible, series of cleaning cycles were performed, targeting the removal of organic and inorganic constituents from the surface of the membrane. The CTA membrane was subjected to two chemical cleaning cycles, and the baseline experiment was repeated after each cleaning cycle. Water fluxes obtained from the baseline experiments (for 24 h) before and after membrane cleaning are shown in Table 5. The first cleaning cycle was aimed to remove the organic foulants from the surface of the membrane. For this duty, 10 mmol L^{-1} SDS, was employed as a cleaning reagent. On the other hand, the second cleaning cycle targeted the removal of inorganic compounds by using 2% citric acid solution. The most relevant observation is that the water flux did not improve after cleaning the membrane with SDS, which infers the insignificance impact of the organic fouling. On
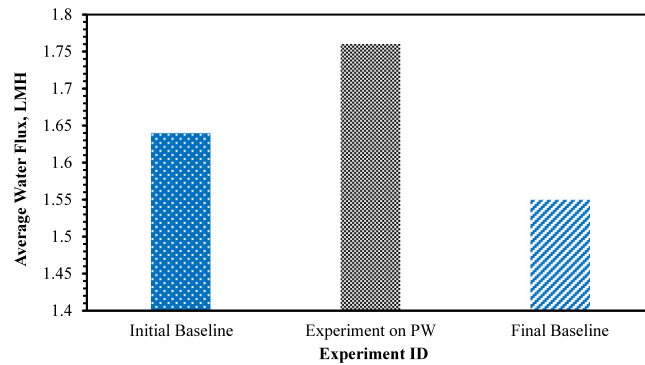
Fig. 8. Comparison between the average water fluxes obtained from the baseline experiments before (initial) and after (final) trails on PW; all baseline experiments were conducted with synthetic solutions (FS: 2000 mg L^{-1} NaCl and DS: 40,000 mg L^{-1}).

Table 5
Water flux of CTA-Toyobo membrane before and after membrane cleaning. All experiments were conducted with synthetic solutions (FS: 2000 mg L^{-1} NaCl and DS: 40,000 mg L^{-1}).

Baseline Experiment	Water Flux, LMH[a]
Before membrane cleaning	1.554
After membrane cleaning with SDS	1.56
After Membrane cleaning with citric acid	1.76

[a]The stable fluxes observed during 24 h of operation. These values were attained in less than 1 h.

the contrary, there was a marked increase in the average flux when the membrane was cleaned by citric acid solution. The water flux of Toyobo improved by 12.9% from 1.55 to 1.75 LMH.

It can be concluded that, although the fouling propensity of the OC membranes was low, simple physical cleaning (membrane flushing) was not sufficient to recover the initial flux, and chemical cleaning was required to sustain a stable water flux. The flux decline due to inorganic scaling can be recovered if the proper cleaning protocol is followed. Outcomes of the membrane-cleaning program suggest that citric acid is a suitable cleaning reagent for the OC process when the target is to remove the inorganic foulants from the surface of the membrane. However, other chemicals such as SDS, and Ethylenediaminetetraacetic acid (EDTA) have demonstrated the effectiveness of removing organic foulants from the membrane surface and restoring the initial flux (Zhao et al., 2017; Minier-Matar et al., 2015). Consequently, they might be required if the feed-water source contains significant TOC concentrations.

3.2.4. Water quality

The characteristics and chemical compositions of the water samples obtained during the pilot testing are shown in Table 6. During the experimentation, PW feed, concentrate feed, DS, and diluted DS streams were characterized for organic and inorganic constituents. For each stream, Table 6 represents the average of five independent samples, collected over 2 days period of operation. It can be seen that the pH and residual chlorine of all streams were within the limits specified by the membrane manufacturer (pH<8, and Cl$_2$<0.1 mg L^{-1}). In addition, the turbidity measurements were constantly below 0.5 NTU, as FS and DS were passed through 5 μm polypropylene cartridges before entering the membrane to assure the removal of any suspended solids. TOC concentration in the outlet feed stream was 25.89 mg L^{-1}. A small loss of TOC from the feed stream to the DS side was attributed to the forward organic flux, however, TOC concentrations of both DS and diluted DS streams were less than 1 mg L^{-1} for the two membranes. Table 6 also reveals a loss, of the Na and Cl content, from the draw stream, which can be explained by the NaCl reverse flux discussed earlier. Measurements of sulfate, phosphate, calcium, and magnesium in the diluted DS were below detection limits, indicating the high rejection of these ions. Overall analysis of the diluted DS showed insignificant amounts of TOC and inorganic substances, demonstrating the feasibility of direct discharge of the diluted DS to the ocean. Actually, this stream is basically NaCl solution diluted in water so no significant organic and inorganic compounds were detected (measurements of inorganics were below detection limits).

4. Conclusions

The current study examines the feasibility of deploying the FO technology as an OC process for reducing wastewater volumes generated from the gas field operations. The technical viability of the OC technology was validated at a pilot scale using CTA HF FO membrane under synthetic and real wastewater streams. The obtained results and trends revealed the following conclusions:

Table 6

Water quality of the PW feed, concentrate feed, DS, and diluted DS obtained during the pilot testing.

Parameter	PW feed	Concentrate solution	DS	Diluted DS
pH	7.04	7.73	7.93	5.90
Conductivity, $\mu S\ cm^{-1}$	2155	8855.6	63234	19692.8
Turbidity, NTU	0.34	0.36	0.30	0.09
Chlorine Residual, mg L^{-1}	<0.05	–	<0.07	–
Total organic carbon (TOC), mg L^{-1}	10	25.89	0.05	0.36
Inorganic carbon (IC), mg L^{-1}	80.01	251.96	19.36	30.24
Chloride, mg L^{-1}	253	1403	23056	6823
Sodium, mg L^{-1}	481	1791	18820	4578
Sulfate, mg L^{-1}	245	668	*	*
Phosphate, mg L^{-1}	17.8	74	*	*
Calcium, mg L^{-1}	3.3	16	*	*
Magnesium, mg L^{-1}	3.2	9	*	*

*Below detection limits (<5 mg/l), - Not measured.

i. The operating conditions of the pilot unit were found to play a vital role in determining the performance of the OC process. Manipulation of both FS and DS flowrates permits operating the pilot unit at various feed recovery rates (up to 90%).

ii. The higher the achieved feed recovery, the lower the water flux and J_s. Commercial CTA HF membrane showed water fluxes between 2.24–1.65 LMH for feed recoveries between 60%–90%. The higher water fluxes at lower feed recoveries were resulted from the reduced impact of concentration polarization at higher flowrates of the feed and draw solutions. However, increasing the flowrates also led to higher diffusivity of solutes, consequently, higher J_s values were obtained at lower feed recovery rates.

iii. Operating the pilot unit for 75% volume reduction was identified as the optimum operating conditions since it demonstrated reasonably good water flux (1.94 LMH) at the lowest J_s/J_w (20.93 mmol L^{-1}). This condition at intermediate flowrates is expected to have lower membrane fouling and feed concentration stress Therefore, the trial on real PW was conducted to achieve 75% volume reduction.

iv. Results of experiments on the real PW demonstrated the robustness of the OC process for an extended period of operation (over 50 h of continuous operation). The CTA membrane successfully reduced the volume of PW by 75% at a stable flux trend with insignificant membrane fouling.

v. The CTA Toyobo membrane showed a water flux of 1.76 LMH with reverse solute transport of 9.3 mmol m^{-2} h^{-1} ~292 mmol h^{-1}). A slight decline in the water flux was observed which was attributed to inorganic scaling, as confirmed by the OLI water quality simulations and cleaning outcomes.

vi. The membrane was subjected to a series of physical and chemical cleaning cycles, targeting the removal of organic and inorganic foulants from the membrane surface. Cleaning with a citric acid solution demonstrated the efficacy of removing inorganic scaling and restoring the initial flux.

vii. Results of analytical measurements revealed insignificance amounts of the organic and inorganic compounds in the diluted brine stream, which renders the option of direct brine discharge to the ocean, a technically feasible and environmentally friendly solution.

viii. Compared to conventional RO, the OC process has the advantages of: (1) Lower energy consumption since the process operate a lower pressure, (2) lower waste volume generation since high recovery can be achieved, and (2) beneficial environmental impact due to the dilution of the brine discharged to the sea. Nonetheless, the OC is still confronted by limited full-scale applications and the fact that no water permeate is produced from the process.

CRediT authorship contribution statement

Abdelrahman M. Awad: Conceptualization, Original draft, Methodology, Formal analysis. **Rem Jalab:** Writing, Methodology, Investigation. **Mustafa S. Nasser:** Conceptualization, Supervision, Funding acquisition, Project administration. **Muftah El-Naas:** Review & editing, Funding acquisition. **Ibnelwaleed A. Hussein:** Writing – review & editing. **Joel Minier-Matar:** Methodology, Formal analysis, Conceptualization. **Samer Adham:** Writing – review & editing, Validation, Project administration.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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