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Integrated Coagulation-Membrane Processes with Zero Liquid Discharge (ZLD) Configuration for the Treatment of Oil Sands Produced Water

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Abstract: This study explores the feasibility of implementing five hybrid coagulation-membrane processes for the treatment of the boiler blow-down (BBD) water from an oil sands steam assisted gravity drainage (SAGD) operation. The processes involved (1) direct nanofiltration (NF) of the BBD water, (2) pre-treatment of the NF retentate using ion exchanger regeneration wastewater (IERW) as a chemical coagulant followed by NF, (3) pre-treatment of BBD water using IERW followed by NF, (4) dual pre-treatment of BBD water using IERW and soda ash (sodium carbonate, Na_2CO_3) followed by NF, and (5) forward osmosis (FO) treatment of the BBD water using IERW as a draw solution followed by NF treatment of diluted draw solution. These scenarios were compared based on total flux decline ratio (DR_t), flux recovery ratio (*FRR*), and total dissolved solids (TDS) removal over the final NF treatment to suggest an efficient treatment technique to avoid an undesired increase in the capital and operating expenses. It was found that process-1 provided the highest selectivity toward dissolved solids (80%) with a flux decline and recovery ration of 89.6% and 97.4%, respectively. Considering the permeation flux, process-4 exhibited the lowest flux decline (86.1%) and highest recovery ratio (97.5%) compared to other processes, proving the successful role of soda ash softening, as a chemical pretreatment method, in improving the performance of membrane filtration. Process-2 presented a mediocre performance with DRt, *FRR*, and TDS rejection of 93.3%, 97.3%, and 74%, respectively. Finally, process-3 and process-5 showed the lowest performance among all the scenarios with low flux recovery and low permeability, respectively. In addition, process-3 was expected to be cost-efficient since it only uses an on-site generated waste as a coagulant for the chemical pretreatment of the membrane filtration unit. The optimum scenario was proposed to be the two-stage membrane process, with direct NF of BBD followed by the post-treatment of the retentate via a hybrid chemical conditioning using IERW and soda ash softening, followed by a second NF. Overall, this integrated process offered a highly efficient mean with a zero liquid discharge (ZLD) system for the treatment of high pH wastewaters into an uncontaminated stream for the boilers.

Keywords: coagulation-membrane process; produced water treatment; boiler blowdown; SAGD; process integration; ZLD

1. Introduction

With the high demand for water management in industrial operations, a novel hybrid treatment process has attracted considerable attention due to their high efficiency in reusing contaminated wastewaters. During the last decade, numerous studies have focused on improving the water treatment processes to solve the water consumption issue in the oil sands steam assisted gravity drainage (SAGD) operation. SAGD is a commonly used method that is applied for the extraction of bitumen from oil sands reservoir in Alberta, Canada. In this method, steam is produced by once-through steam generators (OTSGs) and injected into the production wells to extract the bitumen by reducing its viscosity. The mixture of water and bitumen is pumped out and moved to the nearby plant for bitumen separation. After the deoiling step, the produced water is treated to be reused as the boiler feed water (BFW) [1–7]. The BFW of OTSGs should meet the minimum specifications to avoid scaling and fouling of boiler tubes, which can lead to lower efficiency and higher maintenance costs. Thus, reusing and recycling of SAGD produced water is necessary for the sustainable production of bitumen. The guideline for the OTSGs operational conditions requires the BFW to have a hardness, silica, and total dissolved solids (TDS) content lower than 1 ppm, 50 ppm, and 7000 ppm, respectively [4,8,9]. Hence, in the water treatment facilities of the SAGD plant, different water treatment processes are being applied to remove contaminants, especially silica, calcium, and magnesium as the main cause of scaling in the boiler tubes [8–10]. The current treatment process in SAGD operations relies on the removal of silica by a warm lime softener (WLS), followed by filtration to remove suspended solids, and hardness removal by strong or weak acid cation exchange (SAC/WAC) process. However, these serial treatments increase the TDS concentration of the BFW. Moreover, although the application of ion exchange is highly effective to remove magnesium and calcium ions, it produces a highly saline brine, known as ion exchange regeneration waste (IERW). The disposal of IERW is challenging as it contains a high concentration of sodium, chloride, magnesium, and calcium, and is limited by provincial environmental and energy regulator sectors. Therefore, extensive efforts have been made to reuse IERW for processes such as sodium chloride extraction or biological denitrifications through connecting an upflow sludge blanket denitrification reactor with ion exchanger columns [11–14].

In addition to IERW, another wastewater, known as boiler blowdown (BBD) water, is produced from OTSG, which contains a high concentration of organic matter, silica, and TDS. Currently, a portion of the BBD water is treated and reused as BFW, and the remaining volume of BBD is routed to the disposal wells. The disposal of BBD stream, as a highly alkaline wastewater, not only causes serious environmental issues but also leads to several operational problems such as clogging the disposal well due to co-precipitation of silica and organic matter [3,4,6]. Therefore, several studies investigated various water treatment methods such as acidification, Fenton oxidation, and membrane filtration to purify the BBD water [1,3,6,15]. Recently, membrane technology has proven to be a promising technique for the treatment of oil sands produced water owing to its compact design, low operating costs, and straightforward integration into the existing processes. In particular, nanofiltration (NF), reverse osmosis (RO), and forward osmosis (FO) processes have been studied to provide high-quality feed water for the steam generators [6,15–27]. Sadrzadeh et al. [6] showed that the NF is more practical for the treatment of SAGD produced water as it is more energy-efficient than RO by achieving silica and divalent cation rejection of 98% and 99%, respectively. They also reported that increasing the pH of the feed water mitigates the effect of organic fouling on the surface of the membrane. Furthermore, Khorshidi et al. [15,17] showed that the FO process is less prone to fouling compared to the pressure-driven NF and RO processes for the treatment of oil sands produced water. However, in all these studies, the high concentration of organic matter and silica in the oil sands produced water led to severe organic fouling and scaling of the membranes, which increase the water treatment energy consumption and maintenance costs. This issue can be potentially addressed by placing a pre-treatment unit before the membrane separation process. The practice of pre-treatment for membrane desalination has been promising in lowering fouling propensity, enhancing separation performance, and improving the lifetime of the membranes [28–35]. Li et al. [35] demonstrated that applying chemical pre-treatment with polyaluminium chloride (PAC) as the coagulant could enhance the permeation flux of the membrane process. Park et al. conducted a study to prove the significant effect of coagulation as a pretreatment method for improving the membrane separation process [36]. Furthermore, Giagnorio et al. showed that implementing FO as a pretreatment method in water reuse applications can potentially improve the overall performance due to the low vulnerability of the FO process to fouling as compared to pressure-driven filtration processes [37]. In the previous study, it was demonstrated that using IERW as a chemical coagulant could remove a significant amount of organic matter and silica were removed from the BBD water at the expense of increasing the TDS concentration [38]. Such a high TDS concentration may potentially increase the volume of generated BBD in the OTSGs.

In this study, different membrane-based separation scenarios were examined to reduce the TDS concentration of the IERW-treated BBD water. The primary goal was to convert SAGD BBD water, composing high concentration of TOC, TDS, and silica into high-quality feed water for reuse in boilers. The efficiency of the proposed separation scenarios was assessed based on permeation performance, and fouling properties. Accordingly, different membrane processes were compared to nominate an economically feasible module for the treatment of the BBD water.

2. Materials and Method

2.1. Materials

The BBD water and IERW samples were provided by EXEN Pro Ltd. through collection and delivery by Black Pearl Resources Inc., located in Calgary, Alberta, Canada. The BBD water had a high concentration of silica and organic matter. The IERW contained a high concentration of calcium, magnesium, chloride, and sodium. Table 1 presents the properties of the BBD water and IERW sample.

Table 1. Properties of the boiler-blow down (BBD) waste and ion exchanger regeneration wastewater (IERW) sample.

Parameter	Unit	IERW	BBD
TDS	ppm	66,625	6525
pH	-	6.08	11.66
Turbidity	NTU	0.25	0.86
UV absorbance at 254 nm	-	0.07	0.72
$SUVA_{254}$	-	1.04	0.77
TOC	ppm	6.71	229.80
Silica as dissolved	ppm	5.22	77.6
Mg^{2+}	ppm	2201	0.24
Ca^{2+}	ppm	9455	2.97
Na^{+}	ppm	22,165	1806

2.2. Coagulation-Membrane Hybrid Processes

Five coagulation-membrane hybrid processes were evaluated to treat BBD water. The chemical coagulants were either IERW or soda ash solutions or a combination of both. Figure 1 illustrates the treatment processes in a schematic flow diagram. The details of each process are as follows:

Process-1: In the first process, the BBD water (Feed-1) was treated by NF in order to evaluate the capability of a single filtration step with a 50% water recovery. The permeate solution was named BFW-1.

Process-2: In the second process, the concentrate solution (Retentate-1) of process-1 was treated by IERW (IERW conditioning). The details about this pre-treatment are provided in the previous work [38]. In summary, the chemical pre-treatment was conducted by adding IERW as a coagulant to BBD water at a 2:12 (IERW:BBD) ratio. The mixture solution was stirred for 30 min at 60 rpm followed by 30 min of sedimentation. Afterward, the supernatant was decanted and sent to NF unit as Feed-2. The permeate solution after NF was labeled as BFW-2.

Process-3: In the third process, the BBD water was first pre-treated by IERW. The supernatant (Feed-3) was sent to the NF unit. The resulting permeate solution was called BFW-3.

Process-4: After IERW-conditioning, most of the organic matter and silica particles were removed from the BBD water, but the concentration of the dissolved calcium ions increased significantly. In order to remove the calcium ions, the IERW-treated BBD water was further pre-treated using soda ash in the fourth process [39,40]. 5000 ppm of soda ash was added to IERW-treated BBD water. After 30 min mixing at stirring speed of 60 rpm, the supernatant was sent to the NF unit as Feed-4. The permeate solution after NF was labeled as BFW-4.

Process-5: In the final process, IERW was used as a draw solution to recover water from BBD solution in an FO process. The IERW contained a high concentration of sodium chloride (see Table 1) that can potentially provide high osmotic pressure for the FO application [15]. Afterward, the diluted IERW was sent to NF unit as Feed-5. The resulting permeate solution was called BFW-5.

Table 2 presents the properties of the Retentate-1 and Feed-1 to Feed-5 solutions, which were labeled based on the schematic flow diagram in Figure 1.

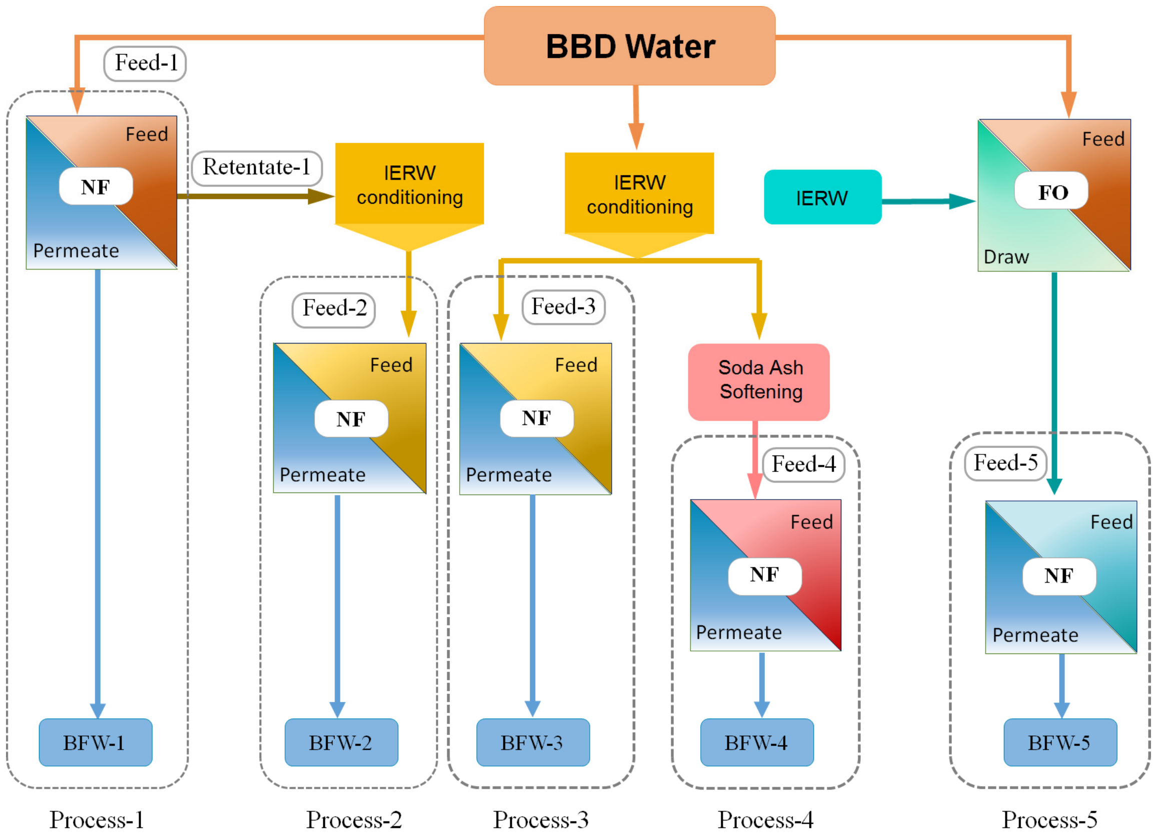

Figure 1. Schematic flow diagram of coagulation-membrane processes for the treatment of steam assisted gravity drainage (SAGD) BBD water.

Table 2. Properties of different feed solutions in coagulation-membrane hybrid processes.

Parameter	Unit	Retentate-1	Feed-1	Feed-2	Feed-3	Feed-4	Feed-5
TDS	ppm	8500	6525	16,750	11,350	16,665	34,535
pH	-	10.90	11.66	11.60	10.75	10.96	6.20
Turbidity	NTU	0.90	0.86	1.20	1.40	1.80	0.80
TOC	ppm	443.30	229.80	107.70	17.00	16.60	3.00
Silica as dissolved	ppm	111	77.60	3.17	1.43	0.93	3.00
Mg^{2+}	ppm	0.16	0.24	0.07	1.29	0.01	1131
Ca^{2+}	ppm	2.78	2.97	3.82	1084	0.00	5325
Na^{+}	ppm	3975	1806	8069	4311	6973	11,436

2.3. Membrane Element

The NF was performed using NF90 (DuPont Water Solutions (FILMTEC^{TM})) membrane. The FO experiments were conducted by a commercial thin film composite (TFC) polyamide membrane, which was purchased from Hydration Technology Innovation (HTI, Albany, NY, USA).

2.4. Membrane Filtration Setups

NF tests were carried out using a dead-end filtration setup that is shown schematically in Figure 2. A nitrogen tank was used to provide the required pressure. The feed solution was stirred using a magnetic stirrer at 500 rpm during the experiments to mitigate the adverse effects of concentration polarization and fouling due to deposition of the contaminants on the surface of the membranes.

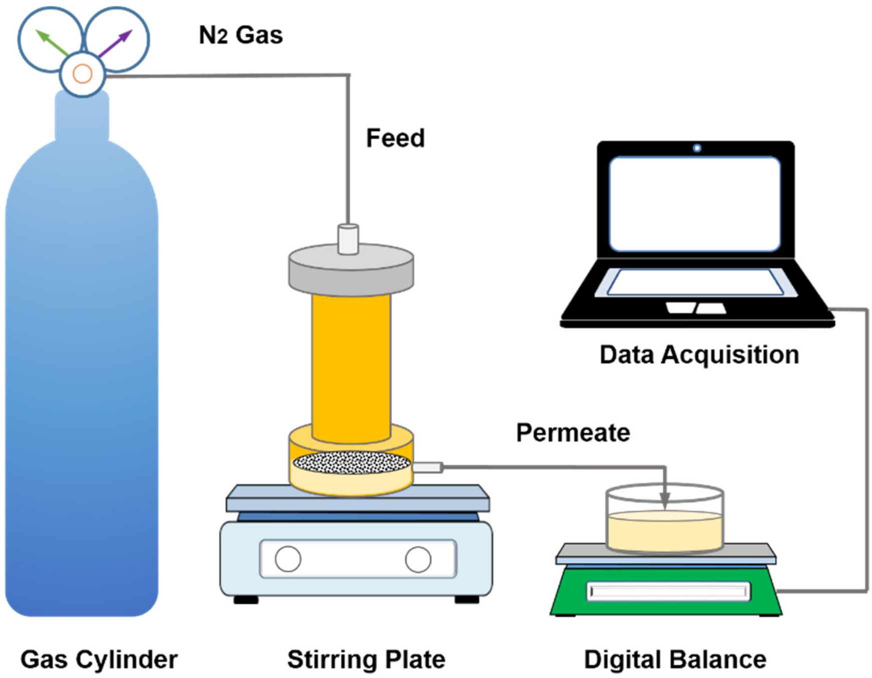

Figure 2. Schematic view of the dead-end nanofiltration (NF) setup. All of the filtration tests were conducted at 25 °C. The operating pressure and stirring rate were between 250 psi to 350 psi and 500 rpm to 1500 rpm, respectively.

The permeate water flux (J_w) was calculated by:

$$J_w = \frac{\Delta m}{\rho A \Delta t}, \quad (1)$$

where A is the effective surface area of the membrane (14.6 cm^2), ρ is the water density, and Δm is the mass of the collected permeate over a certain time Δt. The permeate flux was directly calculated using LabVIEW data acquisition software. The apparent rejection (R) of the contaminants was measured as:

$$R\ (\%) = \left(1 - \frac{C_p}{C_f}\right) \times 100, \quad (2)$$

where C_p and C_f are the concentration of solutes in permeate and feed solutions, respectively. The concentration of solutes was evaluated by measuring the conductivity of permeate and feed solutions.

The fouling behavior of the membrane elements was evaluated by conducting the following consecutive tests: (i) Pure water filtration for 30 min, (ii) wastewater filtration for 3 h, and (iii) hydraulic washing of the membrane followed by filtration of pure water for 30 min. For hydraulic washing, 300 mL pure water was used as the feed solution and the operating pressure and temperature were set to 250 psi and 25 °C, respectively. The total flux decline ratio (DR_t), reversible flux decline ratio (DR_r), irreversible flux decline ratio (DR_{ir}), and flux recovery ratio (FRR) were measured by the following equations [41]:

$$DR_t\ (\%) = \left(1 - \frac{J_p}{J_{w1}}\right) \times 100, \tag{3}$$

$$DR_r\ (\%) = \left(\frac{J_{w2} - J_p}{J_{w1}}\right) \times 100, \tag{4}$$

$$DR_{ir}\ (\%) = \left(\frac{J_{w1} - J_{w2}}{J_{w1}}\right) \times 100, \tag{5}$$

$$FRR\ (\%) = \left(\frac{J_{w2}}{J_{w1}}\right) \times 100, \tag{6}$$

where J_{w1}, J_{w2}, and J_p are the initial pure water flux, pure water flux after hydraulic cleaning, and permeate water flux of the wastewater, respectively.

Figure 3 illustrates the schematic view of the FO setup. The effective membrane area in the FO cell was 140 cm^2. Two gear pumps were used to circulate the feed and draw solution. In all experiments, a flow rate of 2.5 liters per minute (LPM) was maintained for feed and draw solutions. The temperature of the feed and draw solution was adjusted at 25 °C using a circulating water bath (Isotemp 3013, Fisher Scientific). At the beginning of the experiment. The volume of the feed and the draw solutions for all of the tests was 1 L. The conductivity was monitored using inline conductivity meters. The water flux was calculated with the same procedure as the dead-end NF setup using Equation (1). Furthermore, the reverse solute passage (J_s) was calculated using [42]:

$$J_s = \frac{C_t V_t - C_0 V_0}{A \Delta t}, \tag{7}$$

where the C_t and V_t are the concentration of solute and the volume of the feed water measured at the time t, respectively, and the C_0 and V_0 are the concentration and volume of the feed water at the beginning, respectively.

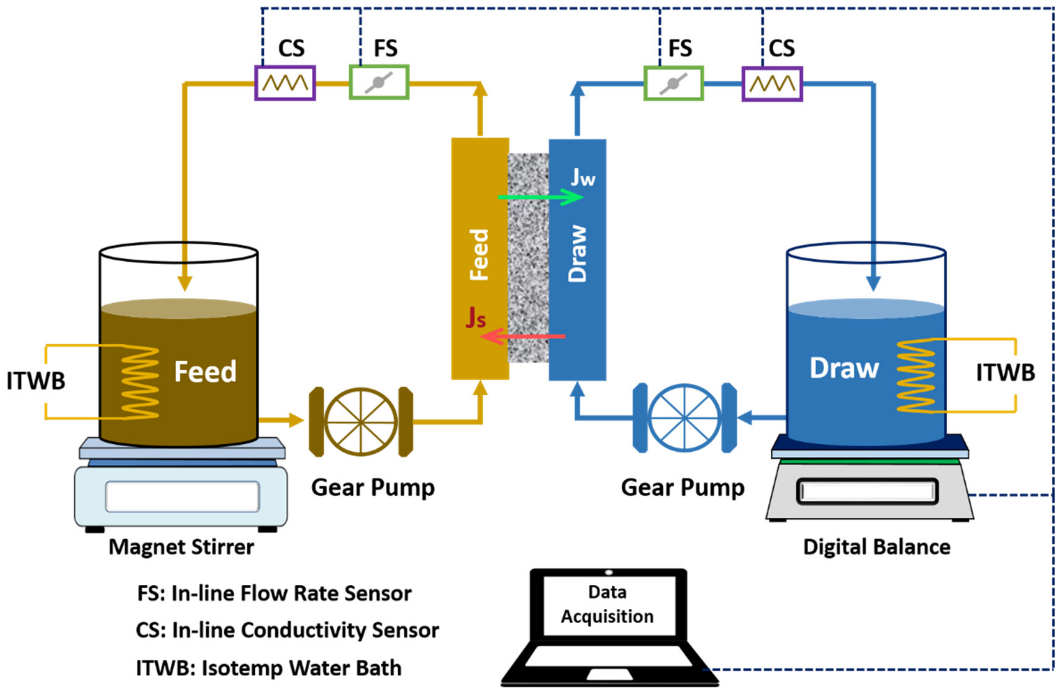

Figure 3. Schematic view of the forward osmosis (FO) setup. The flow rate and operating temperature of feed and draw solutions were set at 2.5 LPM and 25 °C.

2.5. Characterization of Water Samples

The TOC was analyzed using a Shimadzu TOC-VCSH analyzer. The concentration of calcium, magnesium, sodium, and silica in water was obtained using inductively coupled plasma optical emission spectroscopy (ICP-OES). A Hach DR 5000 spectrophotometer was employed to measure the UV absorbance. The turbidity was analyzed with a unit of the nephelometric turbidity unit (NTU) using a turbidity meter.

3. Results and Discussion

3.1. Direct NF Treatment of BBD Water with IERW-Conditioning (Processes 1 and 2)

Figure 4 presents the separation performance and fouling behavior of the NF operation in process-1 and process-2. The filtration of BBD water with NF90 membrane (process-1) caused a gradual flux decline from 77 L/m^{2}h (LMH) to 8 LMH (DR_t of 89.6%) over 3 h of the filtration test. The flux decline can be attributed to progressive fouling due to co-precipitation of organic matters and silica on the surface of the membrane. The irreversible fouling, presented by DR_r, was as low as 4%, implying that the flux decline was primarily due to the concentration polarization and loose attachment of contaminants, which could easily be washed out by hydraulic cleaning. Moreover, the direct nanofiltration of BBD water provided a TDS, silica, and TOC rejection of 80%, 88%, and 90%, respectively. In contrast to process-1, the filtration of IERW-conditioned BBD concentrate (Feed-2) in process-2 resulted in a severe flux loss from 75 LMH to 14 LMH at the onset of the filtration test, followed by a slight decline in permeation flux to 5 LMH. The severe initial flux loss of process-2 is caused by the higher osmotic pressure of Feed-2 due to its high TDS concentration. Similar to process-1, a high flux recover of 97.3% was observed for process-2. The TDS, silica, and TOC rejection of this process were 74%, 85%, and 92%, respectively. In conclusion, both processes demonstrated a severe flux decline with a high flux recovery ratio and low irreversible fouling.

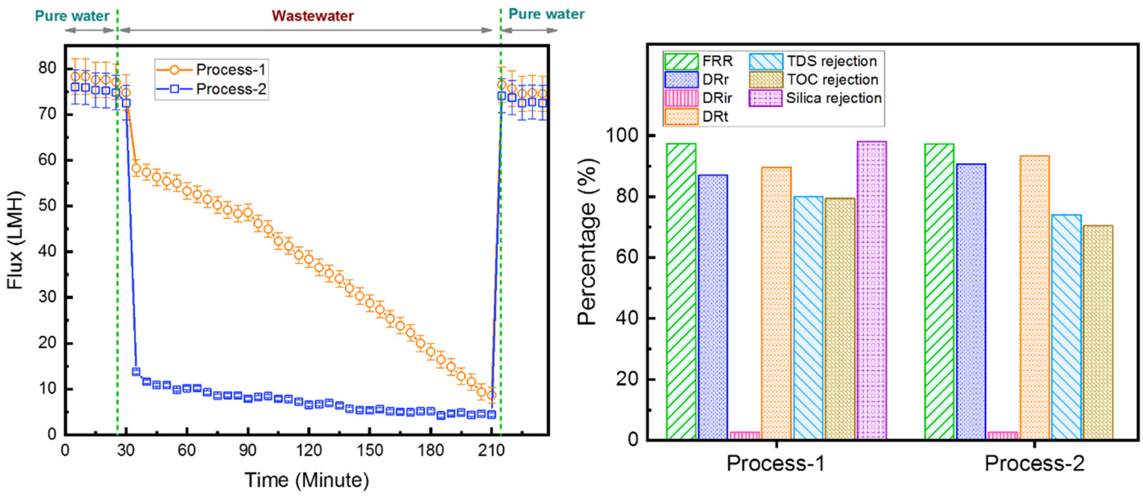

Figure 4. The permeation performance and fouling characteristics of NF tests in processes 1 and 2. Transmembrane pressure is set at 250 psi and operating temperature of 25 °C.

3.2. Nanofiltration with Chemical Pre-treatment of BBD Water

Figure 5 illustrates the permeation flux and fouling characteristics of process-3 and process-4. In these processes, BBD water was pre-treated by IERW solution. Process-4 had additional conditioning by using soda ash. The resulting feed solutions (Feed-3 and 4) were sent to the NF unit for final separation. Both processes resulted in a considerable flux drop at the onset of filtration because of their TDS concentration and thus large osmotic pressure. During the filtration period, the process-3 showed a slight flux decrease of about 6 LMH while a negligible flux loss was measured for process-4. This observation can be attributed to scaling of the membrane surface by a high concentration of Ca^{2+} in

Feed-3 (1084 ppm), which were rejected about 98% by the NF membrane. The total flux decline (DR_t) of the two processes was almost identical (~88%). However, their flux recovery ratio was quite noticeable. While the *FRR* of the process-4 was about 98.7%, only 70% of the flux was recovered in process-3 after hydraulic washing, which implies the strong attachment of calcium ions on the surface of the membrane in this process. Comparing the TDS removal, a slightly lower TDS rejection about 5% was obtained for process-3 than process-4. This observation can be attributed to the higher concentration of dissolved solids in Feed-4 than Feed-3. In a pressure-driven membrane filtration process, the effect of concentration polarization becomes more severe when the feed solution has high TDS concentration. The enhanced solute concentration difference between the feed side and the permeate side results in a large driving force that causes the solutes to move to the permeate side to maintain the osmotic balance.

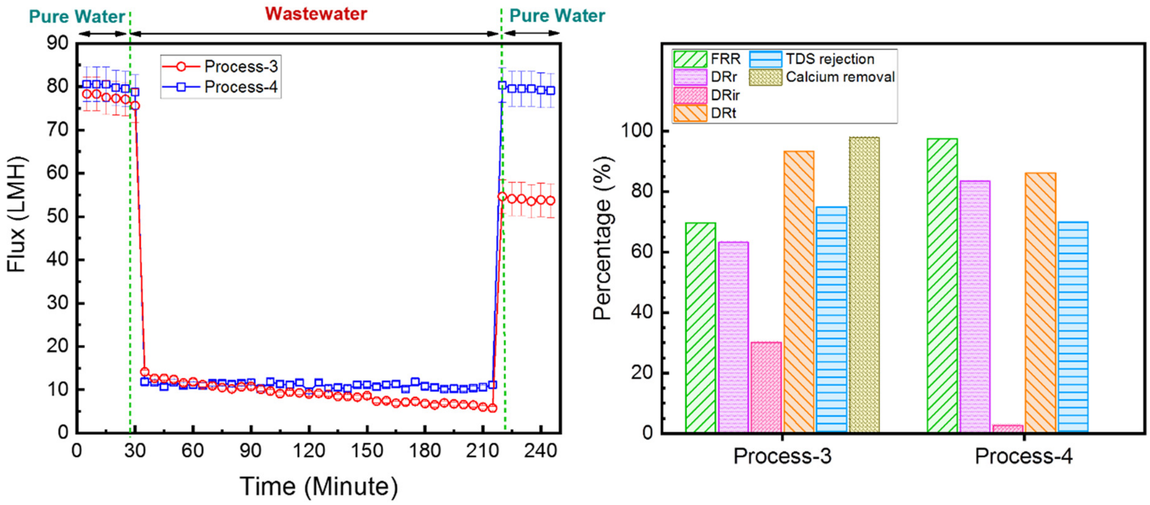

Figure 5. Permeation flux and fouling characteristics of process-3 and 4, which involved chemical pre-treatment followed by nanofiltration. Transmembrane pressure is set to 250 psi and operating temperature of 25 °C.

3.3. Hybrid FO/NF Treatment of BBD Water

The final hybrid process involved water recovery from BBD water by IERW solution in an FO process. The TFC FO membrane was placed in the cell with its active layer toward the feed solution (ALFS configuration). Under approximately 266 psi of initial osmotic pressure gradient, a water flux of 6.7 LMH with 4.9 g/m^{2}h (gMH) of reverse solute flux was obtained, which resulted in about 50% dilution of the IERW solution over five hours of the FO filtration test. The diluted IERW (Feed-5) was then sent to the NF unit for further treatment. Due to the high TDS concentration of Feed-5, the filtration test was conducted at a transmembrane pressure of 350 psi. Figure 6 illustrates the fouling behavior of the NF membrane in process-5. The permeation flux dropped sharply from 74 LMH to 20 LMH at the start of the filtration test due to large osmotic pressure difference, followed by gradual flux decline to 5 LMH as a result of scaling and concentration polarization of Ca^{2+} and Mg^{2+} on the membrane surface. The TDS, calcium, and magnesium rejection for process-5 was 86%, 97%, and 97%, respectively. The total flux decline of this filtration was about 95% with 89% flux recovery ratio. The low value of irreversible fouling, despite a high concentration of Ca^{2+} and Mg^{2+} in the feed solution, can be attributed to the low pH (6.2) of Feed-5. The scaling of the membrane surface by Ca^{2+} and Mg^{2+} becomes more severe at higher pH values due to the generation of solid substances with lower solubilities [43].

3.4. Comparison of Different Hybrid Processes

Figure 7 presents the total flux decline, flux recovery ratio, and TDS rejection of different processes after the nanofiltration unit. These parameters were considered to select the most efficient process for the treatment of the BBD water. The total flux decline ratio demonstrates the efficiency for achieving a higher volume of the treated water under similar transmembrane pressure, and the flux recovery ratio is a measure of the fouling resistance of the membrane. Among all the processes, process-3 showed the lowest performance with high DR_t (93.4%) and low FRR (69.7%). Process-5 can also be eliminated from the candidate pool as the transmembrane pressure for this process was elevated to 350 psi, and the permeate flux declined about 96.2%. The three remaining processes (1, 2, and 4) showed an almost identical flux recovery ratio above 97%. Among these three processes, process-2 showed lower performance with higher flux decline and lower TDS rejection. Process-4 showed a lower flux decline (about 3.5%) than process-1, suggesting that this process can be chosen if a higher permeation rate is the selection criterion. However, if higher TDS removal is required, process-1 was more efficient than process-4 with 10% higher TDS rejection percentage.

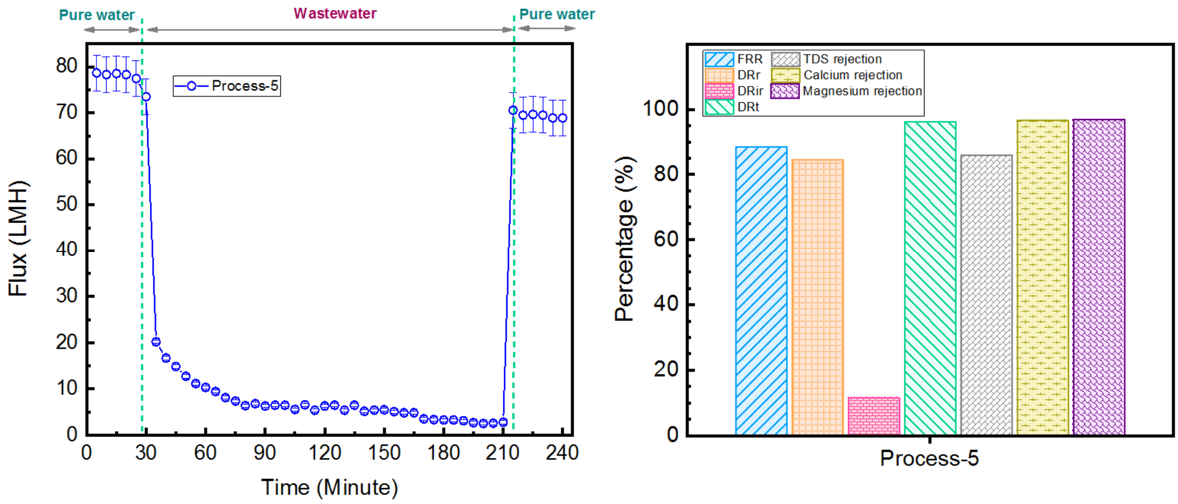

Figure 6. Permeation flux and fouling characteristics of Process-5. Operating conditions were set at a transmembrane pressure of 350 psi, solution temperature of 25 °C and stirring speed of 1500 rpm.

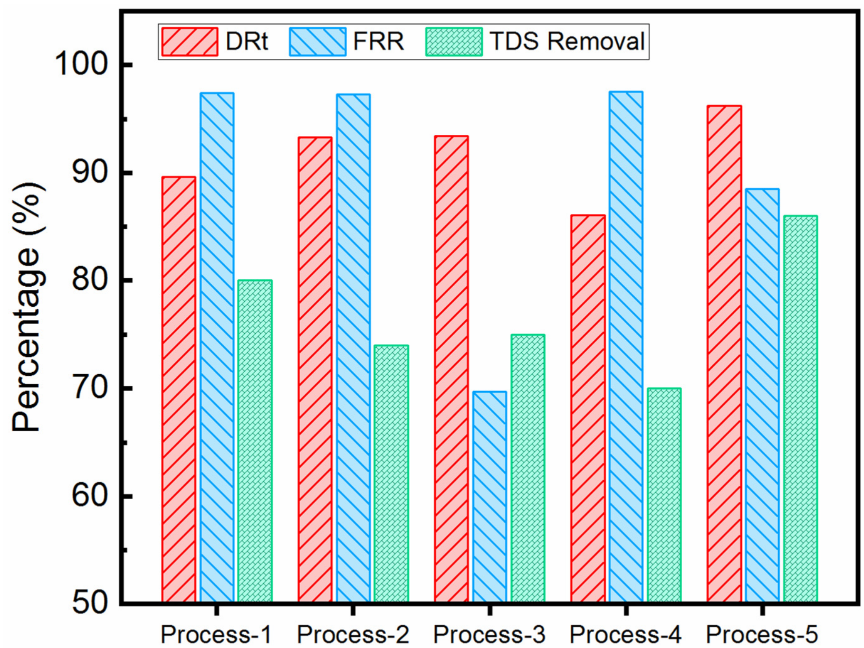

Figure 7. Comparison of total flux decline (DR_t), flux recovery ration (FRR), and total dissolved solids (TDS) removal of different processes.

4. Conclusions

The present study evaluated different chemical-membrane hybrid processes for the treatment of BBD water in order to be reused as BFW. It was found that a direct treatment of BBD water using single-stage nanofiltration could result in the highest TDS removal from the feed solution. Although a flux recovery of 97% was obtained after simple hydraulic washing, the high flux decline (~90%) was the notable adverse side of the direct NF treatment of BBD water. This observation emphasizes the necessity of chemical treatment prior to the membrane filtration unit for such industrial waters. Application of dual chemical pre-treatment using IERW and soda ash solutions resulted in the highest permeation rate with lowest flux decline and highest flux recovery, demonstrating a potential solution to the fouling issue, which was also observed in other works that treated SAGD produced water with one-stage membrane separation processes [6,7,15]. Additionally, the IERW conditioning only uses a waste stream as the coagulant minimizing the operating expanse of chemical coagulant. However, lower TDS rejection compared to direct NF treatment (70% compared to 80%) can be mentioned as the main drawback of this process. In overall, a combination of these two processes could be used as a zero-liquid discharge (ZLD) scheme by reusing the waste products in different applications. For instance, the produced sludge from the IERW conditioning unit can be used for extraction of calcium sulfate, which is used as a direct additive in many applications such as cement, water treatment, and food industries. Moreover, the concentrate solution from the NF of soda ash treated water can be potentially used as a regeneration solution for the ion exchanger as it contains a high concentration of sodium ions. For future work, different types of membrane filtration such as RO or ultrafiltration (UF) can be studied to achieve a higher efficiency or to provide a higher quality BFW for the boilers. Moreover, different levels for the operating conditions and other types of solutions for hydraulic backwashing can be selected in order to recommend an optimized condition for the hybrid process.

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