99% Recovery of Scaling Cooling Tower Blowdown with a Reverse Osmosis Membrane Demonstration Plant

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ABSTRACT

A reverse osmosis demonstration plant achieved 99% water recovery of highly scaling cooling tower blowdown at an Alberta agrichemical facility. A previous pilot by others achieved 80% recovery. The authors share details of the ultra-high recovery system's design and performance, including system process flow, mass-energy balance, and pilot data. A full scale 1100 m³/day system is in design-construction. The 1% brine achieved eliminates the need for a thermal evaporator or trucking to a discharge facility.

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INTRODUCTION

REVERSE OSMOSIS MEMBRANE DEMONSTRATION – In the latter half of 2020, an onsite pilot project was completed by Saltworks Technologies at an ammonia fertilizer production facility in Carseland, Alberta, Canada. The site had been seeking an economical, high recovery solution to treat cooling tower blowdown (CTB) for reuse. A goal of 98% recovery was set because it would achieve two key benefits: reduced river water extraction in a water security risk area and elimination of off-site transport of residual brine, using a legacy evaporation pond.

A previous trial of leading RO technology by others achieved 80% recovery, but due to frequent pre-treatment issues did not maintain sufficient up-time. The CTB feedwater contained membrane-fouling surfactants, calcium sulfate, and silica scaling ions. To overcome the water chemistry challenges, this demonstration plant included organics reduction and scaling ion removal via precision controlled chemical softening, without coagulants. Chemical softening solid by-products were filtered using an automated, self-cleaning ceramic ultrafiltration (UF) sub-system. Desalination was then accomplished using two stages of reverse osmosis (RO): seawater reverse osmosis (SWRO) rated for 80 bar (1,160 psi) and ultra-high pressure reverse osmosis (UHPRO) rated for 120 bar (1,740 psi).

The plant reached 99% recovery and achieved reliable, automated operation, including unattended overnight operations and automated membrane health protective actions. Extending membrane system recovery from a conventional 80% to 99% results in a twenty-fold decrease in brine volume. The resulting prevention of wastewater trucking or an energy intensive thermal process also saves considerable costs and greenhouse gas (GHG) emissions.

While this paper focuses on technical results, ultra-high recovery treatment economics, including step changes in cost/recovery, were reported in a previous companion IWC paper (Man et al., 2019). Generally, readers should expect a cost range of USD\$2–5/m³ (\$0.50–1.32/USGal) to practice the scaling ion removal and ultra-high recovery RO technologies herein, inclusive of levelized capital and operating costs. These costs are 10X lower than evaporation technologies previously required to boost recovery from conventional RO's 80% to the achieved 99%.

BACKGROUND – Twin challenges of freshwater scarcity and industrial wastewater disposal are of global concern and pose risks to many businesses (Mauter et al., 2018). Thermoelectric power generation in the US accounts for 41% of the country's water withdrawals, or 500 billion liters/day (132B GPD), most of which is for cooling (Childress et al., 2021). Treating CTB is becoming increasingly important as operators seek to reduce water footprints and water volumes become harder to manage; legacy evaporation ponds are filling, limits on CTB land application and sewer discharge are tightening, and regulations are evolving to require increased water reuse.

Advanced, decentralized treatment solutions are highly desired for protection of local water security, reducing wastewater transport costs, and mitigating wider environmental risks (Mauter & Fiske, 2020). However, many remain costly in terms of chemical and energy consumption and

footprint requirements. Others do not recover enough freshwater and/or produce excessive brine volumes. At present, many wastewaters, including CTB, are managed by a combination of conventional membrane treatment (largely RO), greenhouse gas (GHG) intensive trucking to off-site disposal wells, energy-intensive thermal volume reduction, or evaporation/tailings ponds that occupy arable land and require maintenance and remediation.

Recent developments in ultra-high pressure reverse osmosis (UHPRO) membranes have facilitated the development and commercialization of a new class of RO treatment systems. Operating at up to 120 bar (1,740 psi), UHPRO achieves 50% higher brine concentration over conventional 80 bar (1,160 psi) SWRO, generating lower brine volumes and improving economics for operations with disposal constraints. In some applications, UHPRO encroaches on applications previously dominated by more expensive thermal methods. However, the greater risk posed by scaling ions remains a challenge to widespread adoption of the technology (Liu et al., 2019). In this work, the efficient management of both scaling ions and varying inlet chemistry in an integrated and automated plant has been demonstrated to unlock the full potential of UHPRO. This work builds on past IWC papers that previously exhibited the high recovery technology (Albadvi et al., 2018; Man et al., 2017, 2019).

PROCESS DESCRIPTION

Figure 1:

Process flow diagram (PFD) of the pilot plant, employing scaling ion precipitation (SIP), realtime scaling ion monitoring (RTSm), precipitated solids filtration (PSF), 80 bar seawater reverse osmosis (SWRO), brine recycle, and 120 bar ultra-high pressure reverse osmosis (UHPRO), as well as solids removal by filter press.



The process flow diagram (PFD) is shown in Figure 1. The authors developed the process flow, drawing from their RO membrane technologies and industrial desalination unit operations. Nitto-Hydranautics furnished the SWRO and UHPRO membranes and supported pilot plant

development. The pilot plant was designed and built with the following unit operations embedded:

<u>Scaling ion precipitation (SIP)</u> <u>Real-time scaling ion monitoring (RTSm)</u> <u>Precipitated solids filtration (PSF)</u> RO, including SWRO & UHPRO

BRINE RECYCLING – The authors' design approach begins from the plant's back end, namely, determining acceptable and safe brine reject composition that will not foul or scale the UHPRO membrane. To reach ultra-high recovery, sufficient scaling ion mass must be removed to ensure that the final element does not scale. Though antiscalants, cycling concentrations, and good RO hydraulic/control design practices increase recovery, they are insufficient to reach 98% recovery, and they will not prevent membrane fouling due to organics. If the required mass of scaling ions and organics cannot be removed through a simple precipitation step, two options are available:

<u>Option 1</u> Recycle SWRO brine back to the front end of the plant, increasing the scaling ion load and mass removed in the SIP and PSF unit operations.

<u>Option 2</u> Insert a secondary SIP and PSF step midway in the process to remove additional scaling ion load once the raw water is preconcentrated by a primary RO system.

For each unique project, both options should be considered, modelled, and costed because the optimal solution will depend on the water chemistry, capacity, and final brine management costs. Although either option 1 or 2 could be used to reach the ultra-high recovery required, option 1 was found to be more cost-effective and was chosen for the site described in this work because brine recycling offered a reduced number of unit operations (lower CapEx) relative to option 2. Although option 1 increases the total dissolved solids (TDS) fed to the SWRO, thus increasing the SWRO membrane area, option 2 would have required an added lower pressure primary RO (600 psi) and two steps of SIP and PSF. In option 1, the scaling ion mass difference between the inlet and outlet determines the brine recycling flow rate (in other words the flow rate required so that a sufficient mass of scaling ions is recycled to the front end of the plant for removal). Alternatively, were option 2 to have been chosen, the scaling ion mass difference would determine where the secondary SIP and PSF should be inserted.

BRINE RECYCLING OPTIMAL FLOW RATE – Chemical precipitation systems have limits in terms of practical outlet ion concentration. Generally, the authors find that the economic and operational design sweet spot for the outlet of a precipitation system is near 5 mg/L for reactive silica (SiO₂) and 50 mg/L for calcium (as Ca^{2+}). Although longer reaction times and over-dosing chemicals can achieve lower silica and calcium levels, this translates into larger unit operations (higher capital cost) and increased chemical consumption (higher operating cost). Increased

chemical consumption has a further disadvantage in that over-dosed chemicals add to the TDS of the water, reducing downstream RO recovery. When concentrating from a low starting TDS to ultra-high brine concentrations, the concentration factor across the process (e.g. 70–80x the influent TDS) is challenging due to the similarly amplified concentrations of scaling ions and organics. For this project, the process of recycling brine both sufficiently recycled mass of scaling ions for removal while also increasing the TDS at the inlet of the desalination process. This reduced the concentration factor, thereby reducing scaling and fouling risk in the final brine. The brine recycle optimal flow rate must consider the blended chemistry of the influent and brine with respect to both the blended TDS and increasing the blended scaling ions to high enough concentrations above their practical removal limits, while targeting an ideal concentrations post-precipitation, and flow rates, one can then complete the mass balance calculations to solve for the brine recycling flow rate.

A question may be raised as to why a primary RO system was not included upstream of node 1, which would reduce the hydraulic capacity and cost of the PFD in Figure 1. This is an applicable option when the wastewater will not foul the primary RO. In this case, biodispersants and other chemical agents are continuously fed to the cooling water as part of the cooling tower chemical treatment program. These biodispersants were found to be a foulant to the UF and RO membranes if not addressed. This restricts use of a primary RO unless the cooling tower chemical dosing regime was changed. Changing the chemical dosing scheme in an industrial cooling plant that has been operating successfully for decades is a serious undertaking and was not desired unless absolutely necessary. Coupled with the previous experience of executing reliable pre-treatment in previous trials, the project team elected to keep SIP/PSF at the plant inlet to generate 'safe' water for downstream processing.

PILOT PLANT DESCRIPTION

The pilot plant is packaged in two 40' ISO shipping containers for portability and ease of site setup, incorporating the process flow shown above in Figure 1. The plant is sized for a nominal inlet flow rate of $2-10 \text{ m}^3$ /day (0.4–1.8 GPM), with capacity dependent on inlet water chemistry; higher TDS and increased scaling ion concentrations result in lower capacity because of reduced flux through the membrane operations. The pilot is fully automated with Siemens Simatic, inclusive of auto start-stop-hibernate and self-clean processes, and readily configurable to trial alternative process configurations. The process and automation design are flexible to accommodate a wide variety of wastewaters and their respective chemistries.

The plant can be operated and adjusted remotely from Vancouver, Canada in cooperation with on-site support. The plant carries extensive instrumentation for process controls to secure fulsome suites of data for full-scale plant design. All data is logged with easy-to-use trending

features. The trending features allow for rapid plots of membrane flux and productivity, which enable appropriate tuning of the built-in self-cleaning features.

SUB-SYSTEMS – The SIP sub-system is configurable with a variety of chemicals so it can perform dosing to precipitate a variety of targeted ions-of-concern. For this trial, surfactants, silica, and calcium were the primary concern. Therefore, the SIP utilized sodium hydroxide (NaOH), which formed magnesium hydroxide with the native Mg²⁺ and co-precipitated silica. The high pH step also precipitated calcium associated with native alkalinity in the water. Sodium carbonate (soda ash) dosing then further lowered the calcium levels, enabling safe RO operation at ultra-high recovery. High pH chemical softening followed by soda ash addition to reduce calcium levels is well known chemistry not covered in detail herein.

Revealed through comparative run data, the authors also found that bio-dispersants (surfactants) were beneficially removed by the above chemistry, preventing UF fouling risk. A first run was completed without SIP (chemical softening) and a rapid decline of PSF ceramic UF membrane flux was observed, with 100 liters per m² per hour (LMH) (59 gallons/ft²/day (GFD)) achieved through frequent chemical cleans. After initiating SIP, PSF UF membrane flux over 300 LMH (176 GFD) could be sustained without chemical cleans. The authors theorize that the mechanism of surfactant removal was entrapment of surfactants into calcium carbonate solid matrices during growth and aggregation of precipitating calcium carbonate particles.

Although chemical softening offers many benefits for downstream processing, chemical costs must be managed, particularly soda ash. Soda ash can be used to reduce calcium levels, but it is an expensive chemical when employed on an annual basis. Optimizing soda ash consumption on waters with variable chemistry can lead to frequent changes and adjustments in dosing rates based on measured calcium levels. However, until recently, the authors were not able to locate a device for reliable real-time measurement of calcium on water with TDS > 500 mg/L.

The authors developed the RTSm sub-system to measure silica, calcium, or sulfates in high TDS solutions after existing off-the-shelf sensors failed to provide reliable measurement. Existing sensors are largely intended for low TDS operation, for example in the ultra-pure water market. High TDS solutions in brine management applications resulted in erroneous readings with these conventional sensors. Therefore, the authors developed new real-time sensors to enable reliable, repeatable measurement of scaling ions and therefore automatic adjustment of chemical feeds and/or dynamic RO recovery control. The RTSm simultaneously protects RO membrane health while optimizing chemical dosing. In this project, the calcium-configured RTSm variants enabled both. Because of soda ash cost savings, the cost of the integrated RTSm offers a payback period of approximately one month if implemented at full-scale.

The PSF sub-system consisted of CPVC pipework rated for 10.3 bar (150 psi), titanium ceramic UF membrane vessels, and zirconia ceramic membranes on alumina substrates, rated to remove solid particles greater than 0.1 µm. Commodity ceramic membranes with standard sizes are employed to ensure that end-users can be re-supplied quickly, at competitive prices, and without being confined to a single supplier's membrane form factor. In this pilot, the membranes were 30 mm x 1200 mm with 19 x 4 mm channels (30-19-4). The PSF system operates at maintained flux and cleans the membranes prior to irreversible fouling via continuous online monitoring of membrane performance and proprietary control algorithms. Residual solids are concentrated to a low volume and removed by a filter press, with the liquids returning for further filtration. The solid filter cake can, generally, be inexpensively disposed of; the solids from this project met the requirements of an Alberta Class II (non-hazardous) landfill. Filtrate from the PSF is RO friendly and in this project, had turbidity <0.2 NTU and a silt density index (SDI) of 0.35. The system selects from multiple levels of cleaning cycles to enable the membrane to maintain flux without irreversibly fouling, while minimizing energy and chemical consumption. The automated selfcleaning cycles included rapid high crossflow (forward flush), back flush, and chemically enhanced cleans. The PSF ceramic membranes can tolerate organics up to 1,000 mg/L, temperatures up to 300°C (570 °F), and pH from 0 to 12.

The SWRO sub-system incorporates one 80 bar (1,160 psi) ASME-rated FRP vessel housing three 8"x 40" membrane elements. The UHPRO sub-system incorporates one 120 bar (1,740 psi) ASME designed FRP vessel housing one 8"x 40" membrane element^{*}. Both units are automated for forward and reverse permeate flushes and chemical cleans. However, no chemical cleans were performed during the pilot owing to the effectiveness of the upstream pre-treatment technologies and the high performance and resilience of the membranes used within.

PILOT RESULTS

The pilot plant operated for 60 days in total. Operation was continuous over 24 hours, with an nominal feed flow rate of 10.4 m³/day (1.91 GPM), showing excellent performance and full uptime. A mass balance with labels corresponding to those included in Figure 1 is presented in Table 1, showing nominal flowrate, pH, TDS, and salt mass/day (for solids added or removed) at the listed nodes. Table 2 presents analytical chemistry data for selected nodes.

^{*} At time of construction, ASME BPVC-X did not extend beyond 1,500 psi. The 2021 release is expected to update to align with this new emerging technology.

Table 1:

Statepoint		Nominal flowrate		pН	TDS	Salt Mass/Day	
		(m ³ /day)	(GPM)		(mg/L)	(kg/day)	
1	Raw	10.4	1.9	7.9	1,550	16.1	
2	SIP inlet	12.4	2.3	-	14,374	178	
3	SIP chemicals	-	-	-	-	NaOH: 4.86	
						Na ₂ CO ₃ : 3.68	
4	SIP solids	-	-	-	-	Dry: 5.78	
5	PSF filtrate	12.4	2.3	11.0	14,597	180	
6	SWRO chemicals	-	-	-	-	H ₂ SO ₄ : 3.59	
						Antiscalant: 0.052	
7	SWRO reject	2.2	0.40	5.3	82,429	178	
8	Brine recycle	2.0	0.36	5.3	82,429	162	
9	UHPRO inlet	0.20	0.36	5.3	82,429	16.8	
10	UHPRO brine	0.14	0.024	5.0	119,967	16.7	
11	SWRO permeate	10.2	1.87	6.3	574	5.85	
12	UHPRO permeate	0.06	0.01	6.1	800	0.0512	
13	Blended permeate	10.3	1.88	6.3	575	5.90	

Mass balance with reference to the statepoint nodes labelled in Figure 1.

Table 2:

Analytical chemistry data at statepoint nodes labelled in figure 1.

	1	2	5	13	10
Statepoint	Raw	SIP Inlet	PSF Filtrate	Blended Permeate	UHPRO Brine
Total Dissolved Solids (TDS, mg/L)	1,550	13,392	15,500	575	119,967
Sodium (Na ⁺ , mg/L)	199	4,479	5,220	218	43,000
Calcium (Ca ²⁺ , mg/L)	120	162.5	63.6	1.2	545
Silica (SiO ₂ , mg/L)	17	16.8	3.6	1.5	15
Carbonate (as CaCO ₃ , mg/L)	<5	<5	186	<5	<5
Chloride (Cl ⁻ , mg/L)	333	1,510	1,670	157	12,100
Sulphate (SO4 ²⁻ , mg/L)	723	6,791	1,560	198	61,400

Table 3 summarizes results, including: 99.1% CTB recovery by volume; feed, brine, and permeate TDS; and salt mass recovery. Both total raw feed volume and the total permeate volume were measured by flow sensors on the plant inlet and outlet. The volume recovery percentage is calculated from the ratio:

[Total Permeate Volume Out]/[Total Raw Feed Volume in]

TDS and specific ions were measured for the feed, brine, and permeate at an accredited thirdparty lab. The salt mass recovery percentage is calculated from:

([Feed TDS] – [Brine TDS])/([Permeate TDS] – [Brine TDS])

Table 3:

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SWRO, brine recycle, and UHPRO recovery and TDS summary.
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Volume:	Feed TDS	Brine TDS	Permeate TDS	Salt Mass
Recovery	(mg/L)	(mg/L)	(mg/L)	Recovery
99.10%	1,550	119,967	575	99.18%

Figure 2 shows the cumulative feed and permeate volumes on the left axis and the volume recovery on the right axis. As the plant operates, both volumes increase linearly, and the recovery volume percentage converges around 99.1%.

Figure 2:

SWRO, brine recycle, and UHPRO cumulative feed and permeate volumes, and cumulative recovery percentage over 10 days of operation.



Figure 3 shows conductivity of the RO permeates, both separately and blended. Conductivity was generally kept very low (~1 mS), indicating the high quality of both the SWRO and UHPRO permeate. The permeate quality met the site specifications for reuse as cooling tower makeup, enabling recycle of the treated water at scale. By contrast, Figure 4 shows consistently high UHPRO brine conductivity (~100mS) and the calculated TDS of the UHPRO brine reject.

Figure 3:

Conductivity of the (a) SWRO, (b) UHPRO, and (c) blended permeates, respectively. Outlier points above 5 mS are excluded.



Figure 4:





Membrane flux, an indication of membrane health, is shown in Figure 5. Both RO systems maintained a consistently healthy flux over the course of the project. After an initial break-in period, flux converged in the 16–18 LMH (9.4–11 GFD) and 10–17 LMH (5.9–10 GFD) ranges for the SWRO and UHPRO systems, respectively. Flux was automatically adapted to fluctuations in inlet TDS and chemistry in the pilots' semi-batch process and no evidence of irreversible flux decline presented.

Figure 5:

SWRO and UHPRO system flux with data points in blue and a rolling average in gold.



The project began with the clear goal of advancing the technology to a full-scale permanent installation. Full-scale plant configuration options, process modelling, and economic modelling were established in advance of the pilot. This informed the test plan and meant that the pilot could then be operated most meaningfully, maximizing its representativeness of the full-scale design. The end-user was actively involved throughout the project, enabling the pilot to focus on

the site's overall needs while exploring innovation options. Open communication was frequent, and the end user was able to clearly set ambitious yet realistic technical and economic objectives. It was well understood that treated water needed to meet a permeate specification and that a superior economic result would be achieved with plant recovery $\geq 98\%$, otherwise substantial brine management costs of USD\$25/m³ would be incurred.

Off-site pre-pilot tests on shipped water provided meaningful results, enabling a faster on-site pilot start-up. On-site pilots are more costly and time-limited, so maximizing utility of the pilot plant while at the end user site is critical. The low cost to complete off-site tests on shipped water offers a very high return-on-investment, even if there is the caveat that some chemistry may change during shipment. There is also a considerable return-on-investment on generating a detailed water chemistry matrix, ascertaining variability in flows and chemistry, and piloting during a period that spans this variability.

Placement of the SIP and PSF processes at the front end of the plant are important for protecting and stabilizing downstream processing. Cooling tower blowdown can contain unknown species such as organics from biodispersants and other added chemicals. The SIP and PSF processes effectively insulate all downstream processes from the negative impacts these chemicals can have such as membrane fouling. Furthermore, the brine recycling approach not only removed scaling ion mass, but also served to further remove residual organics after they have been concentrated up in the SWRO.

Health and safety risks can be mitigated through good practices, training, foresight and planning, open communication between all parties, and safety-by-design consideration in the plant. The pilot was successfully completed without any injuries, spills, or accidents and although the pilot took place at the height of the COVID-19 pandemic there was no COVID-19 related incident.

CONCLUSION

The ultra-high recovery reverse osmosis demonstration plant demonstrated reliable 99% recovery of highly scaling cooling tower blowdown. Its success means that the 1% brine-by-volume remaining can be managed by a small legacy evaporation pond, alleviating the need for a thermal evaporator or wastewater trucking to a discharge facility.

OUTLOOK – Detailed design of a full-scale RO-based plant utilizing brine recycling is 90% complete at time of writing and planned for implementation in 2023. The results of this study are directly translatable to other sites and wider applications including mining, oil and gas, factory wastewater treatment, flue gas desulfurization (FGD), lithium extraction, indoor farming, and bio-energy digestate treatment. This study and subsequent pilots by the authors using the pilot plant have demonstrated the brine recycle process as a reliable means of achieving extremely high recoveries, even in the presence of high organics.

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