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Permeate quality, advanced oxidation process treatability, and cost for two concentrate treatment technologies to enhance recovery for potable reuse

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ABSTRACT

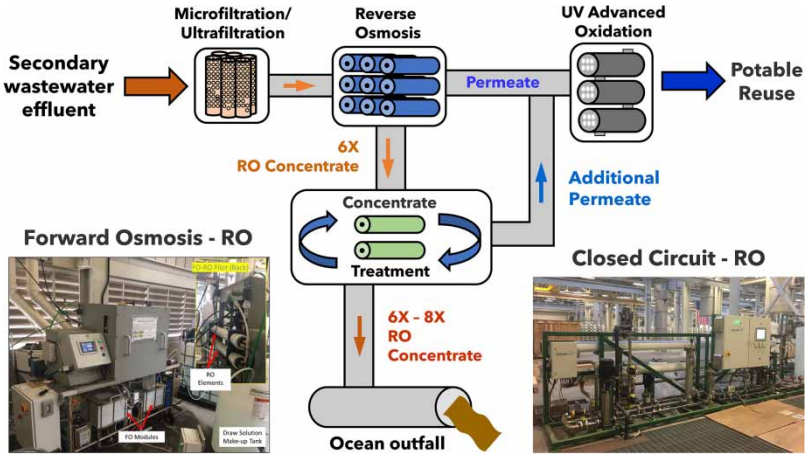
Closed circuit reverse osmosis (CCRO) and forward osmosis-RO (FO-RO) were evaluated at a pilot scale to generate additional permeate from RO concentrate – achieving a recovery of 61% for CCRO and 35% for FO-RO – at a full-scale advanced water purification facility. This study assessed permeate water quality, suitability of the permeate for treatment by an ultraviolet-advanced oxidation process (UV-AOP), and cost/footprint for a conceptual 10- or 20-mgd system. Both technologies demonstrated inorganic, organic, and microbiological constituent removal suitable for blending with primary RO permeate. Virus challenge testing with MS coliphage demonstrated ≥3.7-log removal by both technologies. Pilot-scale UV/hydrogen peroxide AOP treatment of CCRO or FO-RO permeate yielded similar performance (~1.4-log *N*-nitrosodimethylamine removal and ~0.5-log 1,4-dioxane removal) as the full-scale UV-AOP that treats the RO permeate from the purification facility. The estimated full-scale total unit cost (capital plus operation and maintenance costs) of product water produced by the two technologies was estimated to range from $0.91 to $1.12 per cubic meter, depending on the design flow rate of RO concentrate treated, and is estimated to be similar between the two technologies given the +50%/–30% expected accuracy of the Class 5 cost estimate.

Key words: closed circuit RO, concentrate treatment, cost analysis, forward osmosis, permeate quality, water reuse

HIGHLIGHTS

- Reverse osmosis (RO) concentrate treatment from municipal potable reuse to generate purified water is feasible and economical.
- Closed circuit reverse osmosis (CCRO) or forward osmosis-RO (FO-RO) permeate was suitable for blending with primary RO permeate.
- Spike tests with MS coliphage showed ≥3.7 and ≥6 log removal for CCRO and FO-RO.
- UV-AOP treatment of CCRO or FO-RO permeate was effective and consistent.

GRAPHICAL ABSTRACT



INTRODUCTION

Reverse osmosis (RO) accounts for more than half of the share of the desalination market and is the leading form of treatment technology for the modern water desalination industry. RO systems can produce high-quality permeate by rejection of dissolved mineral salts in feed water. During treatment, RO can effectively concentrate dissolved mineral salts by 5–7 times, generating a purified permeate product and a concentrate (brine) waste (Ramasamy 2019; Heo *et al.* 2020). Conventional RO systems for potable reuse typically recover 75–85% of the feed water, leaving a large volume of concentrate that must be discharged. This is especially challenging for inland regions where ocean discharge is not available.

The Orange County Water District (OCWD) manages a large potable reuse plant – the Groundwater Replenishment System (GWRS) Advanced Water Purification Facility (AWPF) – which features a three-step treatment process consisting of microfiltration (MF) or ultrafiltration (UF), RO, and UV-AOP. The final expansion of the AWPF (hereafter referred to as the 'plant') has increased the treatment capacity from 100 mgd (378,541 m^3/d) to 130 mgd (492,104 m^3/d), which increased the RO concentrate generated to 23 mgd (87,064 m^3/d). Increasing the recovery rate from 85 to 90% (hypothetical example) via a 'fourth-stage' concentrate treatment facility could generate ~8 mgd (30,283 m^3/d) of additional RO permeate and reduce concentrate disposal to 15 mgd (56,781 m^3/d). OCWD serves as a representative model for potable reuse facilities that utilize membrane-based treatment and aim to identify cost-effective treatment processes to improve water recycling efficiency and increase water supply.

Membrane-based technologies have been successful in increasing concentrate recovery for industrial and municipal wastewater reuse. Closed circuit RO (CCRO) is a high-recovery RO system that operates in a single-stage mode, recirculating RO concentrate back into the feed of the membrane array until a set-point trigger is reached based on recovery, pressure, and/or a conductivity threshold (concentrate or permeate) (Efraty 2009, 2012). At this point in time, the system changes to plug-flow mode, flushing the concentrate from the system (Quay *et al.* 2018; Ruiz-García *et al.* 2018). In 2019, OCWD piloted the CCRO technology to treat plant RO feed and later RO concentrate, and investigated the optimal CCRO operating parameters (Gu *et al.* 2021). Treating RO concentrate as the feed water (i.e., representing a fourth-stage RO), the study found that CCRO recovery could be sustained at 61% (i.e., CCRO permeate divided by feed flow), which corresponds to a theoretical overall recovery of 91% if the CCRO production is added to primary RO system production at this plant. This was achievable at a permeate flux of 6.4 gfd (gallons per square foot of membrane per day) (11.0 LMH) (liters per square meter per hour) while meeting the minimum >30-day clean-in-place interval requirement, though successful operation required side-conduit flushing with plant RO feed water (Juby *et al.* 2020; Gu *et al.* 2021).

Another promising concentrate recovery technology for municipal reuse is forward osmosis-RO (FO-RO), an emerging hybrid (two-barrier) membrane process (Suwaileh *et al.* 2020). FO-RO separates water from the contaminants by inducing flow through an FO membrane into a high solute-concentration draw solution (Cath *et al.* 2006; Cai 2016; Korenak *et al.* 2017). The FO permeate is then separated from the draw compound by an RO membrane. FO technology has been used to treat municipal wastewater with varying degrees of success (Chaoui *et al.* 2019; Suwaileh *et al.* 2020). Alongside the

CCRO, OCWD piloted a FO-RO system to treat the concentrate from the full-scale three-stage RO process. The study demonstrated 35% recovery by FO-RO, which corresponds to a theoretical 89.5% overall recovery when combined with the primary RO system production (Desormeaux *et al.* 2019; Gu *et al.* 2020).

The abovementioned CCRO and FO-RO pilot studies were part of a Phase I investigation that focused on operational optimization (Desormeaux *et al.* 2019; Gu *et al.* 2020, 2021; Juby *et al.* 2020). The objectives of the Phase II study reported herein were divided into four areas: comprehensive water quality evaluation of the product water; determination of virus log removal via challenge testing; confirmation of suitability for subsequent ultraviolet-advanced oxidation process (UV-AOP) treatment; and full-scale cost and physical footprint analysis.

Permeate from both pilots was sampled and analyzed for minerals, microorganisms, disinfection byproducts (DBPs), and wastewater-derived organic compounds (i.e., contaminants of emerging concern (CECs) such as pharmaceuticals and personal care products) (Fairbairn *et al.* 2016). Most CECs are removed from the wastewater by the RO and thus are expected to occur in the RO concentrate that served as the feed water to the CCRO and FO-RO pilot systems (Fairbairn *et al.* 2016; Farrokh Shad *et al.* 2019; Heo *et al.* 2020). Some organic compounds, such as *N*-nitrosamines and halogenated DBPs, represent challenge chemicals for membrane-based potable reuse trains because they are inefficiently removed by RO membranes (Xu *et al.* 2018; Szczuka *et al.* 2020).

With respect to microbial removal, implementation of CCRO or FO-RO for potable reuse must protect the public from pathogens exposure. California, for example, requires 10-log reduction of *Giardia*, 10-log reduction of *Cryptosporidium*, and 12-log reduction of viruses for groundwater replenishment facilities (i.e., indirect potable reuse via groundwater augmentation) which may be summed across the unit treatment steps from raw influent wastewater to extracted groundwater (CDPH 2014). This study evaluated the U.S. EPA recommended microbial indicators that are indigenous to wastewater, including fecal indicator bacteria (FIB), male-specific (MS) and somatic (SOM) coliphage, and enteric viruses.

To assess virus removal, bacteriophage is commonly used as a surrogate due to its small size (up to 28 nm) compared to other viruses (Antony *et al.* 2012; Chahal *et al.* 2016). While RO prevents viruses from passing through due to the membranes' dense polymer structure, complete removal is not always observed (Antony *et al.* 2012). In this study, the pilot systems' feed (essentially concentrated MF effluent) was seeded (spiked) with MS coliphage as a challenge test to determine the virus log removal capability of CCRO and FO-RO treatment processes (Rodriguez *et al.* 2009).

This research also aimed to confirm the effective treatment of CCRO and FO-RO product water using UV-AOP. Potable reuse facilities typically employ UV or UV-AOP treatment after RO to remove certain trace contaminants such as *N*-nitrosodimethylamine (NDMA) and 1,4-dioxane (Roback *et al.* 2018, 2020). If full-scale RO concentrate treatment (as a fourth-stage RO) is implemented, the product water would likely be blended with primary RO permeate and treated by UV-AOP. Therefore, separate spike studies were conducted to confirm removal of NDMA and 1,4-dioxane by UV/hydrogen peroxide AOP (UV/H_2O_2 AOP) from permeates produced by CCRO and FO-RO.

Lastly, a key objective of the study was to establish the conceptual cost and physical footprint requirements for the two technologies to assess the feasibility given the technical success of the pilots. Planning-level capital, operation & maintenance (O&M), and life-cycle costs for theoretical 10- (45,461) and 20-mgd (90,922 m^3/d) RO concentrate influent treatment systems were developed for each treatment technology.

MATERIALS AND METHODS

OCWD AWPF for potable reuse

The AWPF treats municipal secondary-treated wastewater supplied by the Orange County Sanitation District (OC San) with a treatment train consisting of MF or UF, RO, and UV/H_2O_2 AOP to produce high-quality purified water. Detailed information about the treatment process is described in Gu *et al.* (2021). RO concentrate (ROC) from the AWPF (hereafter referred to as 'plant ROC') was used as the feed water to the CCRO and FO-RO pilot units. A slipstream of plant ROC was plumbed to continuously supply the feed tank to the CCRO pilot and to directly feed the FO-RO pilot. A chloramine residual between 3 and 5 mg/L as total chlorine was maintained in the plant RO feed.

CCRO pilot system

The CCRO system (DuPont/Desalitech ReFlex Max LP, Newton, MA) (as previously described by Gu *et al.* (2021)) pilot tested for recovery of water from the plant ROC. The pilot uses a pressure vessel (PV) with three 8 (O.D.) × 40 in. (201 × 1,016 mm) spiral wound RO elements (DuPont Filmtec BW30 XFRLE-400/34). The CCRO system recirculates its own

concentrate back to the feed over a defined cycle time before the concentrate is isolated in a side-conduit (a pressure vessel without membranes) and purged to waste without breaking pressure. The cyclical system allows for higher recovery to be achieved. Schematics that show the flow of RO concentrate during closed-circuit and plug-flow modes are included in Supplementary Figures S2-1 and S2-2.

FO-RO pilot system

The FO-RO pilot system (described in Szczuka *et al.* (2020)) is an automated unit that monitors conductivity, temperatures, pressures, and flow rates online. RO concentrate was fed directly into the pilot system. A pump was used to lower feed pressure and manage feed flow. The system used an FO module (stacked flat sheet design) that consisted of two FO elements (Porifera PPFO-100, San Leandro, CA) (membrane area: 14 m^2) (Supplementary Figure S1). The RO component of the FO-RO pilot consisted of two pressure vessels (61 mm I.D. × 1,016 mm) in series with a seawater spiral wound RO element (DuPont Filmtec SW30-2540, Edina, MN) (membrane area: 2.7 m^2). A sodium chloride recirculating draw solution created the osmotic driving force and dye markers (fluorescein dye in the first 2 months or uranine dye in the final month) were added to monitor the feed outlet and permeate. Because some salt in the draw solution loop will be 'lost' (or diffuses) through the semi-permeable FO and RO membranes, a sodium chloride solution (171.1 g/L) was added to the draw solution periodically as make-up. To maintain purity of the draw solution, a portion of the solution must be intermittently drained as 'blow down' (set at 0.25% of the permeate flow as ~3.0 mL/min).

The pilot operated at an average feed flow rate of 3.67 L/min and the RO permeate flow rate of 1.21 L/min. The regenerated draw pressure was controlled by the RO pump within the draw loop to maintain approximately 0.21 bar draw overpressure compared to the FO feed pressure which was 0.34–0.90 bar during most of the pilot testing. The RO membrane was operated at 17.9–31.0 bar.

Chemical and microbial water quality evaluation

The chemical water quality of the permeate produced by the CCRO and FO-RO pilot units was characterized by four sampling events in 2019 (2/21, 4/15, 6/25, and 9/25). For sampling events No. 2 and 3, plant ROC (common feed water to both pilots) water quality was characterized to calculate percent removal. Due to matrix and cost limitations, no CCRO or FO-RO concentrate samples were analyzed. Pilot feed, concentrate, and permeate electrical conductivities (EC) were measured online to ensure mass balance. The sampling locations are shown in Figure 1. To characterize the average chemical composition of the CCRO feed and permeate, a sampling scheme accounting for the cyclic nature of the CCRO was used

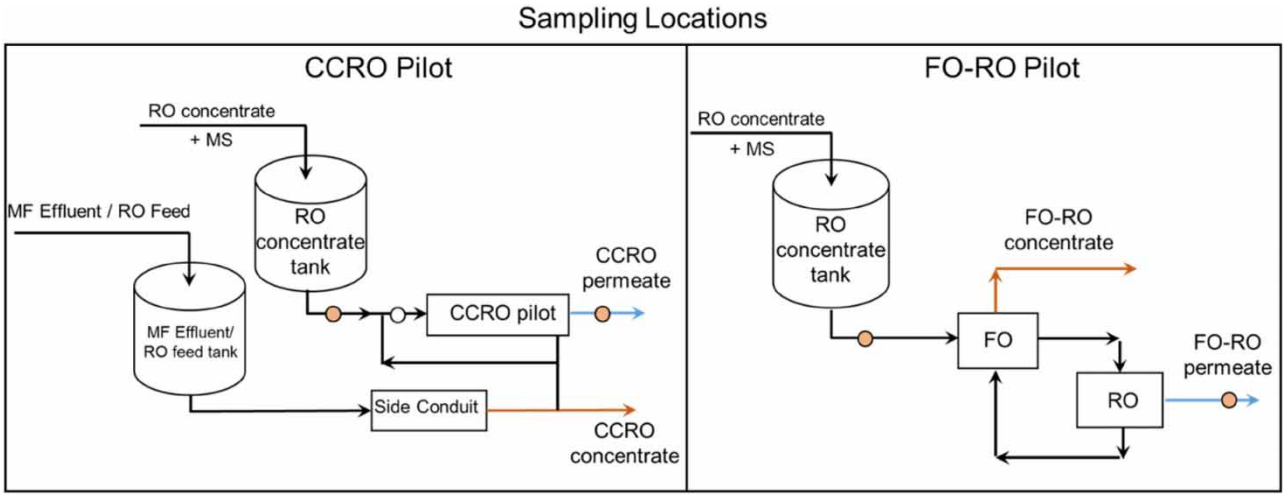


Figure 1 | Schematic of CCRO and FO-RO pilot units with sampling locations for chemical water quality assessment and native microbial enumeration (orange circles) and additional CCRO sampling location for MS coliphage challenge test (white circle). For the challenge test, MS coliphage (abbreviated 'MS' in the diagram) was spiked into RO concentrate tank. 'RO concentrate' in the diagram refers to concentrate generated by the plant primary RO which served as feed water to the pilots. Please refer to the online version of this paper to see this figure in colour: https://dx.doi.org/10.2166/wrd.2023.002.

(details in Supplementary Information S1). The permeate of the FO-RO did not vary with time but was sampled with the same approach to ensure consistency with CCRO.

Dissolved chemicals measured in the feed and permeate from each pilot unit included: general water quality (e.g., total organic carbon, pH, EC); general minerals; trace metals; nitrogen and phosphorus compounds (e.g., ammonia, nitrate, orthophosphate); selected regulated compounds from California Title 22 and drinking water quality regulations; DBPs (e.g., total trihalomethanes [TTHMs], haloacetic acids [HAA5], NDMA); CECs (e.g., 1,4-dioxane, pharmaceuticals), and excitation–emission matrix (EEM) spectroscopy (Horiba Aqualog, Irvine, CA) as an indicator of organic character. The full analytes list is included in Supplementary Table S1. To minimize cost, several Title 22 constituents were excluded from the sampling plan based on the review of historical plant data, as they are not usually detected in plant RO feed. Analysis was completed by the OCWD Philip L. Anthony Water Quality Laboratory or by Weck Laboratories (City of Industry, CA).

Microbial indicators commonly selected for potable water reuse were measured in the CCRO and FO-RO feed and permeate to characterize native microbiota and assess removal. Grab samples from plant ROC, CCRO permeate, and FO-RO permeate were collected on the same day (8/26, 9/25, and 10/23) and analyzed by EPA standard microbial detection methods. Microbial targets included total coliform, *Escherichia coli*, enteric viruses, and MS and SOM coliphage (Table 1). *Cryptosporidium* oocysts and *Giardia* cysts (approximately 4–6 μm and 5–18 μm in size) are expected to be efficiently rejected by size exclusion during MF/UF treatment at the treatment plant and not expected in the feed water to the pilots. Therefore, MS and SOM coliphage were measured in feed and permeate water as surrogates due to their smaller size at approximately 25 nm (Mann *et al.* 2019; Worley-Morse *et al.* 2019). Chlorine was quenched with sodium thiosulfate (50 mg/L) and confirmed by *N,N*-diethyl-*p*-phenylenediamine (DPD) method with a handheld Hach colorimeter (Pocket Colorimeter II, Loveland, CO) prior to microbial analysis.

For quality assurance, the rate of MS coliphage decay due to sampling and shipment was evaluated prior to pilot sampling (details in Supplementary Information S2). MS coliphage decay during overnight shipment was found to be negligible (i.e., no decay was observed).

Male-specific (MS) virus challenge test

Three MS coliphage spiking events were completed to evaluate the performance of the CCRO and FO-RO pilot units. A solution of 10^8 PFU/mL of MS coliphage concentration was prepared by adding MS coliphage concentrate (10^{11} PFU/mL, IEH-BioVir Laboratories, Modesto, CA) to a 400-gal or 250-gal feed tank for CCRO and FO-RO, respectively, and manually mixed for ~20 min before pilot startup. For all samples, MS coliphage was enumerated using EPA Method 1602, which has a detection limit of 0.1 PFU/mL. Grab samples from the FO-RO pilot unit were collected after an initial mixing and equilibration period of ~40 min after startup, and from the CCRO pilot after the initial startup and one full cycle. Duplicate samples were collected from the pilot sampling locations indicated in Figure 1. For CCRO, a feed sampling port was added to obtain a blend of plant ROC and the recirculated CCRO concentrate. CCRO feed and permeate samples were obtained at the start and end of one treatment cycle to compare MS coliphage removal at different times within the cycle.

All grab samples from the pilots were immediately dechlorinated with 50 mg/L of sodium thiosulfate to avoid MS coliphage degradation, chilled on ice, and shipped to MSU for analysis. The log reduction value (LRV) was calculated from the concentration of MS coliphage after treatment (permeate) and before treatment (plant ROC).

Table 1 | Microbial targets for water quality assessment

Microbial target	Detection method	Analytical laboratory
Total coliform *E. coli*	IDEXX Colilert Quanti-Tray 2000 (SM9223B)	OCWD
Male-specific coliphage (MS)	EPA 1602 double-agar overlay	Michigan State University (MSU) Water Quality and Environmental Microbiology Laboratory
Somatic coliphage (SOM)		
Enteric viruses	EPA 1615 cell culture only	IEH-BioVir Laboratories

UV-AOP treatment suitability study

UV-AOP treatment suitability tests were carried out with a single-lamp pilot UV reactor (Trojan Technologies, ON, Canada) under conditions that simulated the advanced oxidation performance of the full-scale reactors at the plant. The reactor (316 stainless steel, 6.82 cm I.D., 9.78 L in volume) is equipped with the same 257 Watt low-pressure high-output mercury amalgam lamp, quartz sleeve, and ballast of the UV-AOP facility. The RO permeate (908 L) from the CCRO and FO-RO pilot units was collected in separate polyethylene chemical totes. These source waters were gravity fed through 15.4 m of vinyl tubing to the pump that delivered the feed water (22.7 L/min) to the UV reactor and flow to waste. The UV dose was estimated at 850 ± 50 mJ/cm^2 to match the advanced oxidation of 1,4-dioxane (0.5-log) and consumption of hydrogen peroxide (10–13%) achieved by the full-scale UV-AOP in the presence of 3 mg/L of H_2O_2.

For the UV-AOP test, NDMA (Sigma-Aldrich) and 1,4-dioxane (certified ACS, Fisher) were spiked into the RO permeate produced from each pilot unit as follows: NDMA 500 ng/L, 1,4-dioxane at 15 μg/L (Test #1) and 25 μg/L (Test #2). Hydrogen peroxide (30% w/w; Sigma-Aldrich) was diluted with deionized water to a concentration of 3% (w/v) and delivered by a peristaltic pump (Masterflex; Cole-Parmer, Vernon Hills, IL) into a static inline mixer (Koflo Model 1-80-4-4-9.1; Cary, IL) to achieve a targeted concentration of 3 mg/L in the feed water to UV reactor.

Duplicate feed water (UVF) and product water (UVP) paired samples were collected in 2.5-L amber glass bottles. Immediately after collection, source water was transferred to 1-L amber glass bottles for NDMA analysis that contained 100 mg sodium thiosulfate to quench the residual chlorine (chloramines). NDMA was measured by a modified EPA 521 solid-phase extraction method. 1,4-Dioxane was measured by the modified EPA 524.2 method utilizing a purge-and-trap GC/MS technique (Yoo *et al.* 2002, 2003). Total chlorine (Method 10070) and monochloramine (Method 10171) were measured by colorimetry with a DR6000 spectrophotometer (Hach, Loveland, CO). UV percent transmittance (UVT) was measured at 254 nm. Hydrogen peroxide was measured by the titanium oxalate method (Sellers 1980; USP_Technologies 2023).

Cost estimate methodology

Planning-level capital, O&M, and life-cycle costs for 10- (45,461) and 20-mgd (90,922 m^3/d) systems were developed for the two treatment alternatives as Class 5 cost estimates. The expected accuracy of the Class 5 cost estimates is 50% over the estimate to 30% under the estimate. Capital costs consist of all items that would be constructed/purchased for the evaluated alternatives. The direct cost of each process was based on vendor-quoted information and on recent experience from historical relevant projects. General factors such as site work (including grading, paving, yard pipes), electrical, and instrumentation costs were added to the direct cost. A project contingency of 30% was included to account for undefined elements and potential risks. General conditions, contractor overhead and profit, sales tax, and engineering, management, and legal expenses were also factored into the total construction cost. The O&M cost estimates include power, labor, chemicals, membrane replacement, and an allowance for equipment replacement cost. In addition, preliminary plant layouts were developed to determine space requirements for the alternatives.

RESULTS

Permeate water quality assessment: chemical constituents

Selected permeate quality parameters for CCRO and FO-RO (average of four water sampling events), and primary RO permeate (ROP) (March–September 2019) are shown in Figure 2. The complete water quality and chemical constituent removal data of the two pilots are provided in Supplementary Information S3.

The permeate from the CCRO and FO-RO pilots had higher constituent concentrations compared to the plant ROP, which is expected considering the much more challenging and lower quality RO concentrate feed to the pilots. However, the CCRO and FO-RO permeates generally met permit limits for the plant final product water (FPW), except for certain parameters discussed below.

The average FO-RO permeate total dissolved solids (TDS) concentration at 202 ± 101 mg/L was much higher than the average CCRO permeate TDS of 50.5 ± 28.9 mg/L. Both are higher than the 6-month average plant ROP TDS of 17.0 ± 8.0 mg/L. However, they are still well below the FPW permit TDS requirement of 500 mg/L. For the FO-RO, the greater TDS is believed to be primarily due to the high salt concentration of the draw solution serving as the RO feed (EC of ~37 mS/cm), indicating ~99% salt removal across the RO membrane (Szczuka *et al.* 2020). In addition, the RO system in the FO-RO pilot was not operated at ideal design parameters especially in terms of surface velocity, hence a commercial-scale FO-RO system is expected to provide better permeate EC under similar operating conditions.

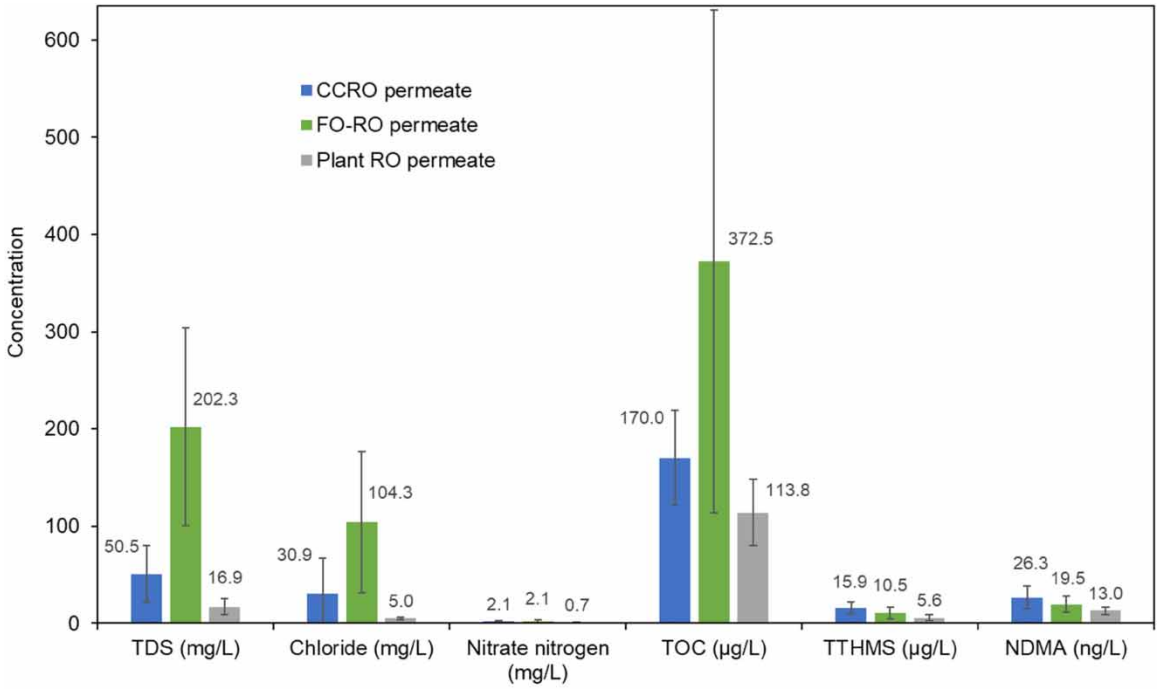


Figure 2 | Concentration of selected key permeate water quality parameters (average ± std. dev., $n = 4$, 2019) for CCRO (blue bars) and FO-RO (green bars) pilots. For plant RO permeate (gray bars), values shown are the average from March through September 2019. Please refer to the online version of this paper to see this figure in colour: https://dx.doi.org/10.2166/wrd.2023.002.

The average FO-RO permeate chloride (Cl^-) level was 104 ± 72.5 mg/L Cl^-, higher than the average CCRO permeate concentration of 30.9 ± 35.7 mg/L Cl^-. Both are higher than the 6-month average plant ROP concentration of 5.0 ± 0.98 mg/L Cl^-. The FO-RO permeate concentration was above the FPW permit requirement of 55 mg/L Cl^-. Nitrate nitrogen (NO_3-N) levels in the FO-RO and CCRO permeates were low (2.1 ± 1.7 and 2.1 ± 0.9 mg/L), though higher than the plant ROP (0.70 ± 0.15 mg/L), but still below the FPW permit requirement of 3 mg/L NO_3-N.

The average FO-RO permeate TOC was 372 ± 259 µg/L, and the average CCRO permeate TOC was 170 ± 49 µg/L, which are low but slightly higher than the 7-month average plant ROP TOC of 114 ± 34 µg/L. Average TOC removal was ~99.4% for FO-RO and ~99.6% for CCRO (Supplementary Figures S12 and S13), corresponding to greater than 2-log, similar to the RO plant removal. Except for the FO-RO pilot permeate sample on 9/25/2019, the pilot permeates are below the plant FPW permit requirement of <0.5 mg/L (500 µg/L) of TOC. In a theoretical full-scale application, TOC would be further reduced, perhaps significantly, through blending with plant ROP prior to UV-AOP treatment.

TTHMs in the CCRO and FO-RO permeates were higher than plant RO permeate but still well below the FPW permit requirement of 80 µg/L. NDMA concentrations in FO-RO pilot permeate (19.5 ± 8.1 ng/L) and CCRO pilot permeate (26.3 ± 11.7 ng/L) exceeded the 10 ng/L California State Water Resources Control Board (CASWRCB) monitoring trigger level for FPW that is applicable to potable reuse plants (CASWRCB 2018). The corresponding estimated NDMA removal for the CCRO pilot was 82.5% and for the FO-RO pilot was 90.0% (as shown in Supplementary Figure S10, for the sampling date on 6/25). While the NDMA concentration in the plant RO permeate also exceeded this level (13.0 ± 4.0 ng/L), it should be noted that, as with the main plant RO facility, residual NDMA in the CCRO and FO-RO permeates would be removed by UV-AOP. NDMA is degraded primarily through direct photolysis (Lee *et al.* 2005) with a small contribution by HO radical-mediated oxidation (Lee *et al.* 2007). NDMA was further evaluated as part of the UV-AOP treatability study.

The FO-RO pilot showed slightly better removal capability for VOCs compared to CCRO. This is likely due to the double-membrane barrier design of the FO-RO and lower recovery and permeate flux compared to the CCRO pilot single-membrane system and higher permeate flux. Higher permeate flux can lead to slightly lower rejection of organics due to exacerbated concentration polarization at the membrane surface (Xie *et al.* 2014; Quay *et al.* 2018). Both pilots were effective in removing CECs (Supplementary Information S3).

PFAS (EPA Method 537.1), including PFOA (73.0 ng/L) and PFOS (66.0 ng/L) (detection limit: 0.00092 ng/L), were detected in the plant RO concentrate sample (6/25) but not in the permeate samples for both pilots (Supplementary Table S6). Similarly, 1,4-dioxane was detected in the plant ROC sample (8.5 μg/L for 6/25) (detection limit: 0.50 μg/L) but not in the permeate samples for the pilots (Supplementary Table S6).

EEMS results (Supplementary Information S4) showed both treatment processes were able to remove most soluble microbial products and proteins, fulvic acid-like compounds, and humic acid-like compounds from the plant ROC above 99.5% for both pilots. The FO-RO pilot showed slightly higher removal (0.25% on average) than the CCRO pilot.

Microbial water quality assessment

In general, the pilot feed water was observed to be relatively free of coliform indicators. One exception was a CCRO feed grab sample taken on 9/25, where 3 MPN/100 mL of total coliform was detected. Aside from this total coliform result, both SOM coliphage and *E. coli* were not detected.

In contrast, MS coliphage was detected in all CCRO and FO-RO feed water grab samples, which ranged from 4.2 to 94 PFU/mL and 8.2 to 92 PFU/mL, respectively. Finally, all permeate grab samples from the CCRO and FO-RO pilots were free of all microbial indicators: total coliform, *E. coli*, SOM, and MS coliphage were below detection limits as shown in Table 2.

MS coliphage removal challenge tests

MS coliphage challenge tests were performed on the CCRO and FO-RO pilot units by seeding the feed tanks with a target of 10^8 PFU/mL in three separate events after 1 h of pilot operation to ensure steady conditions. For CCRO, MS coliphage removal was assessed at the beginning and end of a treatment cycle (Figure 3(a) and 3(b)). Throughout all three events (10/8, 10/30, and 11/8), MS coliphage removal from pilot feed and the blended feed (recirculated feed) were consistent with an average of 5.2-log removal. At the end of the cycle, when the feed is more concentrated, an average of 4.3-log removal was observed, suggesting an average of 0.9-log reduction in performance compared to the beginning of the cycle (Table 3) which may be within the uncertainty of the measurement.

Permeate samples for the first two FO-RO challenge tests resulted in non-detect values, while Event 3 resulted in a 270 PFU/mL detection of MS coliphage corresponding to 6-log removal of the virus (Figure 3(c)). Given the double-membrane barrier and >8-log removal of MS coliphage observed in the first two events (estimated from the detection limit of MS coliphage for permeate samples at <0.1 PFU/mL), this result may indicate loss of membrane integrity, but with substantially high performance as removal remained very high. Without further testing of membrane integrity, it is unknown whether the lower LRV of approximately 6.0 log is representative of normal operation (Table 3).

UV-AOP treatment suitability

CCRO and FO-RO permeate water quality

UV-AOP pilot tests were completed on 6/24/2019 and 8/26/2019. The general water quality of the CCRO and FO-RO permeate feed water to the pilot UV reactor is shown in Table 4. The FO-RO permeate had an order of magnitude higher chloride

Table 2 | Native microbial assessment for CCRO and FO-RO pilot feed and permeate waters

	Sample date	CCRO feed	CCRO permeate	Sample date	FO-RO feed	FO-RO permeate
MS-2 coliphage (PFU/mL)	8/26/19	19	<0.1	9/25/19	94	<0.1
	9/25/19	94	<0.1	10/23/19	8.2	<0.1
	10/23/19	4.2	<0.1	11/6/19	55	<0.1
Somatic coliphage (PFU/mL)	8/26/19	<0.1	<0.1	9/25/19	<0.1	<0.1
	9/25/19	<0.1	<0.1	10/23/19	<0.1	<0.1
	10/23/19	<0.1	<0.1	11/6/19	0.2	<0.1
Total coliform (MPN/100 mL)	8/26/19	<0.1	<1.0	9/25/19	<1.0	<1.0
	9/25/19	3.1	<1.0	10/23/19	<1.0	<1.0
	10/23/19	<0.1	<1.0	11/6/20	<1.0	<1.0
E. coli (MPN/100 mL)	8/26/19	<0.1	<1.0	9/25/19	<1.0	<1.0
	9/25/19	<0.1	<1.0	10/23/19	<1.0	<1.0
	10/23/19	<0.1	<1.0	11/6/19	<1.0	<1.0

Values with a (<) symbol represent the limit of detection for their respective methods.

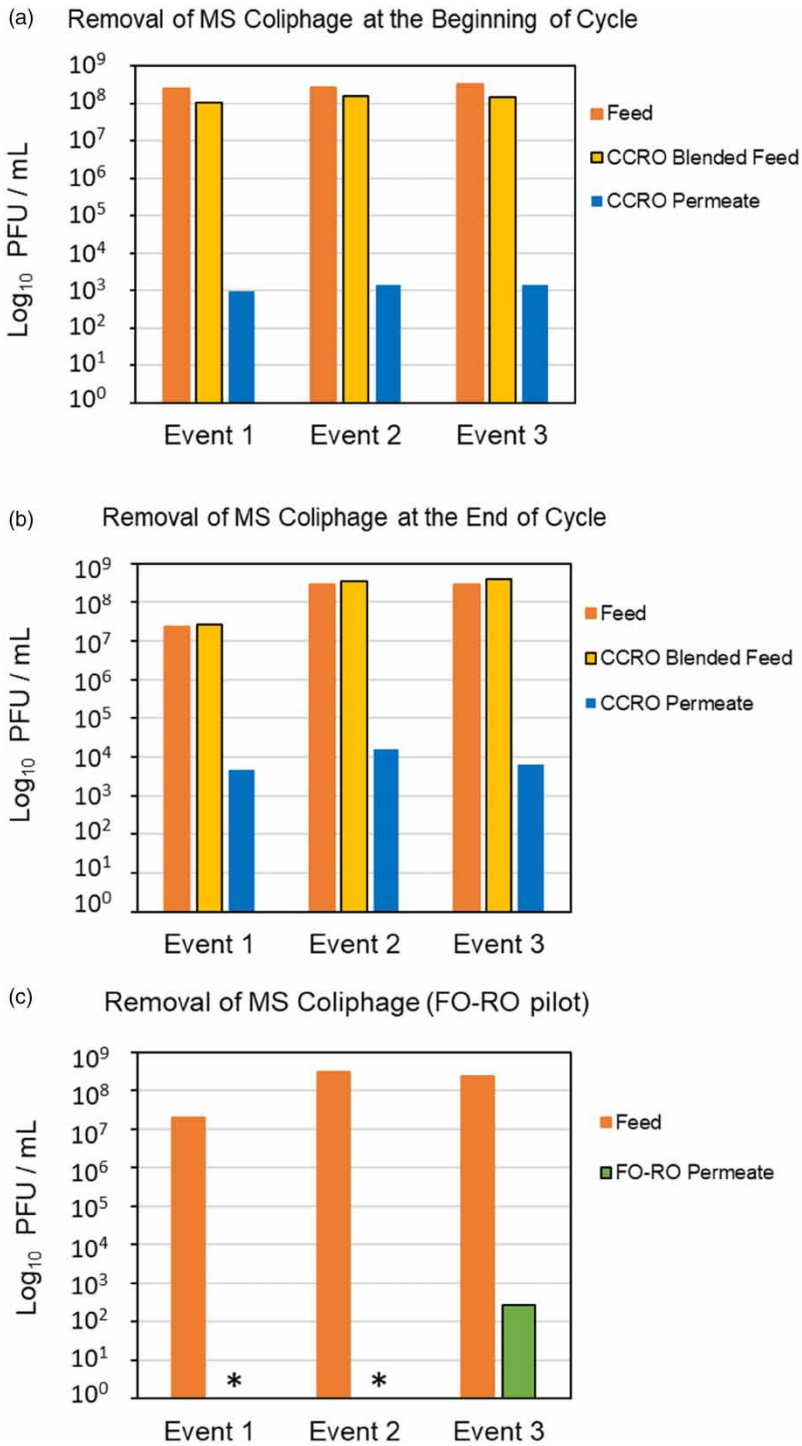


Figure 3 | MS coliphage log removal challenge test of CCRO and FO-RO pilots. MS coliphage removal was evaluated at the beginning (a) and end (b) of the CCRO cycle, and separately also for the FO-RO (c). All grab samples were measured with experimental replicates ($n = 2$), and the averages are shown. Events 1, 2, and 3 correspond to 10/8, 10/30, and 11/12/2019, respectively. Asterisks (*) represent values below the method limit of detection (<0.1 PFU/mL).

concentration than the CCRO permeate, which was related to the FO draw solution. The TOC concentration was also twice as high in the FO-RO permeate. The chloride concentration in the FO-RO permeate was significantly higher when the second test was conducted due to FO membrane fouling issues. Bicarbonate, nitrite, and natural organic matter (NOM) can have a

Table 3 | MS coliphage log removal values from challenge tests performed on CCRO and FO-RO pilots

	Event 1	Event 2	Event 3
CCRO (Beginning of cycle)			
CCRO feed	5.4	5.3	5.4
CCRO blended feed	5.1	5	5
CCRO (End of cycle)			
CCRO feed	3.7	4.3	4.7
CCRO blended feed	3.7	4.4	4.8
FO-RO			
FO-RO feed	8.3[a]	9.5[a]	6

[a]Permeate samples were non-detect for MS coliphage. The method limit of detection (0.1 PFU/mL) was used to estimate log removal.

Table 4 | General water quality of CCRO and FO-RO permeates treated by UV-AOP suitability testing

	6/24/19		8/26/19	
Constituent	**CCRO**	**FO-RO**	**CCRO**	**FO-RO**
Chloride (Cl^-), mg/L	14.6	126	13.2	465
Bicarbonate (HCO_3), mg/L	10.3	14.3	8.8	12
Bicarbonate (as $CaCO_3$) (HCO_3Ca), mg/L	8.5	11.8	7.2	9.8
Ammonia (NH_3-N), mg/L	0.4	2.0	<0.1	2.2
Nitrite nitrogen (NO_2-N), mg/L	<0.002	<0.002	<0.002	<0.002
Nitrate nitrogen (NO_3-N), mg/L	3.23	2.23	2.79	2.65
Total chlorine, mg/L	1.6	1.5	1.3	1.3
UVT@254 nm, %T	98.5	99.0	98.7	98.7
pH	6.2	6.4	6.1	6.5
Total organic carbon (TOC), mg/L	0.16	0.33	0.18	0.39

negative impact when present at high concentration, reducing efficiency of the AOP (Kwon *et al.* 2020; Lee *et al.* 2020; Zhang *et al.* 2022). Chloride can shift the balance of hydroxyl radical formation toward the formation of the chlorine dimer ($Cl_2^{\bullet-}$) radical that reacts more slowly with 1,4-dioxane (Patton *et al.* 2018).

Removal of NDMA and 1,4-dioxane from the CCRO and FO-RO permeates by UV/H_2O_2 AOP

Currently, the CASWRCB recycled water regulations for advanced treatment require testing to demonstrate 0.5-log removal of 1,4-dioxane by an AOP for permitting (CASWRCB 2018). Both UV-AOP pilot tests yielded removal of NDMA near 1.4-log and removal of 1,4-dioxane near 0.5-log that was comparable to the historical performance of the full-scale UV-AOP that treats the RO permeate (Table 5). Additional discussion of the UV-AOP testing results with respect to the comparison of CCRO, FO-RO, and the full-scale plant UV-AOP performance is provided in Supplementary Information S5.

Cost and physical footprint evaluation

As part of the feasibility assessment, a preliminary cost and footprint evaluation of CCRO and FO-RO concentrate treatment technologies was prepared for theoretical full-scale installation at the plant to treat a portion (10- (45,461) or 20-mgd (90,922 m^3/d)) of the RO concentrate produced by the primary RO system.

The design of the CCRO system is based on multiple 1-mgd (4,546 m^3/d) CCRO trains. Each train consists of 80 primary PVs, each holding five 201 × 1,016 mm RO elements (with a membrane area of 14,864 m^2) along with 40 side-conduit PVs. The proposed CCRO system is intended for treating the primary RO concentrate, while the side-conduit would be filled with primary RO feed.

Table 5 | NDMA and 1,4-dioxane log removal value for UV/H_2O_2 AOP reactor treatment of the CCRO and FO-RO pilot permeates

Date	Pilot unit	NDMA Log removal	1,4-Dioxane Log removal
6/24/19	CCRO	1.38	0.61
		1.43	0.65
	FO-RO	1.38	0.46
		1.40	0.54
8/26/19	CCRO	1.45	0.75
		1.46	0.70
	FO-RO	1.28	0.35
		1.29	0.40

The design of the FO system also drew from pilot testing. A 45,461-m^3/d FO system would incorporate 70 FO membrane stacks, with each stack having a membrane area of 1,486 m^2. The combined FO permeate and draw solution (high salinity sodium chloride) would be treated by a conventional seawater RO system to produce FO-RO permeate. The 90,922-m^3/d configuration would essentially replicate the design of the 45,461-m^3/d system. Schematics and conceptual site layout for the hypothetical FO-RO and CCRO system design are included in Supplementary Figures S19–S26. Additional discussion is provided in Gu *et al.* (2020).

From the information provided by the two vendors, and the assumptions used to establish the building dimensions, it should be possible to accommodate both technologies on the OCWD site at both treatment capacities. The estimated footprint requirements for the FO-RO facilities at 10 mgd (1,486 m^2) and 20 mgd (2,230 m^2) scale are smaller than the CCRO facilities at the same scale (10 mgd: 2,415 m^2 and 20 mgd: 4,785 m^2). The side-conduit accounts for 44% of the CCRO footprint. However, the CCRO system would be expected to recover more potable water compared to the FO-RO system, at approximately 17,763 and 35,122 m^3/d for the 10- and 20-mgd (feed water flow rate) CCRO systems, respectively, compared with 13,238 and 26,477 m^3/d for the 10- and 20-mgd FO-RO systems.

Recovery of the CCRO system was assumed to be 61.0% based on pilot testing described by Gu *et al.* (2021). At this recovery, a full-scale CCRO system would increase overall plant recovery from 85 to 88% or 91% for both systems, respectively (via adding the CCRO permeate flow to the plant primary RO permeate flow). The projected overall plant recovery differs depending on whether the CCRO system is fed 45,461 or 90,922 m^3/d of primary RO concentrate because treating a larger flow rate of the available primary RO concentrate (87,064 m^3/d) produces more permeate, and thus, the 'overall' recovery is greater.

The FO-RO system is expected to achieve a recovery of 35% based on pilot testing as previously described by Desormeaux *et al.* (2019) and Gu *et al.* (2020). With a full-scale system, the overall plant recovery would thus be expected to increase from 85 to 87.3% or 89.5% for the two sizes, respectively, via adding the FO-RO permeate flow to the plant primary RO permeate flow.

Based on the cost estimates received from the vendors, the FO-RO system has a lower capital cost compared to the CCRO system. It should be noted that the CCRO vendor provided a market list price rather than a competitive bid price, which is typically lower. Additionally, the equipment cost estimate provided by the FO-RO vendor was significantly lower than expected, considering it includes both FO membrane stacks and the conventional high-pressure RO system (Figure 4).

For O&M cost, based on information provided by the vendors, electrical power cost estimates, and pilot chemical usage, the CCRO system is expected to have a lower annual O&M cost (as shown in Figure 4). The impacts of membrane fouling were considered in the cost estimates that were made for electrical power and chemical consumption. Chemical costs were estimated based on the observed cleaning frequency from pilot testing. For CCRO, six cleanings per year per train were assumed. For FO-RO, 2 cleanings per year per train for the RO, and 17 cleaning events per year per train for the FO were assumed. The FO-RO system has ~3 times higher annual chemical cost compared to CCRO, which is mainly due to draw solution usage that accounts for 33.2% of its annual chemical cost, and more frequent chemical cleaning of the FO membranes. For electrical power (pumping cost), an average operating pressure was chosen to provide an average between clean and fouled membrane conditions. The specific energy consumption of the technologies based on the pumps was estimated to be 0.6 kWh/m^3 for the CCRO system and 0.76 kWh/m^3 for the FO-RO system.

Overall, assuming that the capital cost is funded over a 30-year loan period at a fixed annual interest rate of 5%, the unit cost of water (i.e., accounting for both capital and O&M costs) is expected to be similar and in the range of \$0.91 to \$1.12/m^3,

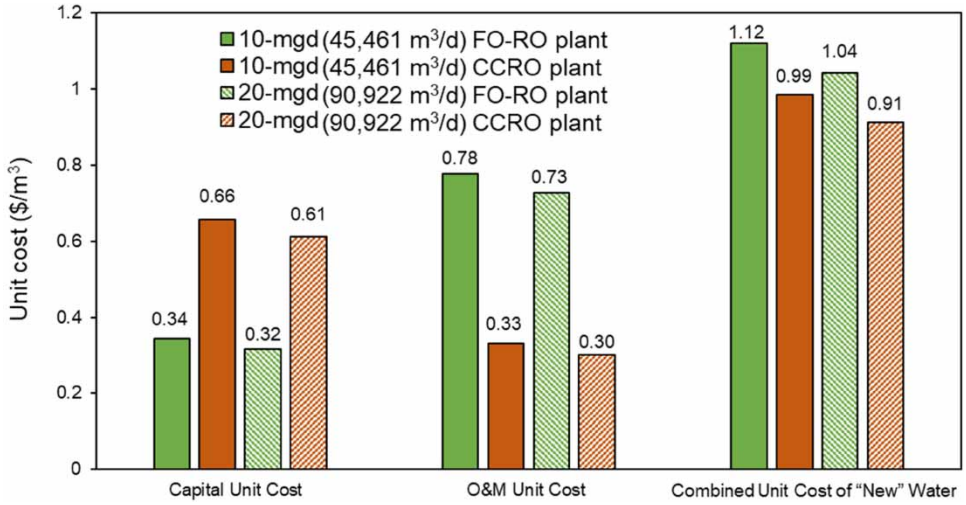


Figure 4 | Estimated capital, O&M, and combined total unit cost ($/m^3 of permeate) for producing additional water using 10-mgd and 20-mgd (45,461 or 90,922 m^3/d) CCRO and FO-RO systems.

depending on the design flow rate of RO concentrate that is treated and the technology used. For the CCRO process, the total unit cost is expected to be between $0.91 and $0.99/m^3 for systems treating 20-mgd and 10-mgd of RO concentrate, respectively. For the FO-RO process, the total unit cost range is expected to be similar but slightly higher at between $1.04 and $1.12/m^3 for systems treating 20-mgd and 10-mgd of RO concentrate, respectively (Figure 4). In comparison, the current total cost for the plant feed water treated with MF, RO, UV-AOP, and post-treatment stabilization is approximately $0.69/m^3. Thus, not surprisingly, there is a greater cost to 'squeeze out the last drop of water'.

If the goal is to compare the footprint and costs of two technology options that produce the same amount of additional permeate instead of the same treatment capacity, Supplementary Figures S27 and S28 can be used to extrapolate the necessary information. For instance, if both a CCRO and FO-RO system generates 6 mgd (22,712 m^3/d) of additional permeate, the extrapolated combined cost would be $0.97/m^3 (CCRO) and $1.07/m^3 (FO-RO). In addition, the CCRO system would occupy a larger footprint (~3,259 m^2) than the FO-RO system (~2,198 m^2).

This cost estimate does not include any savings from avoiding plant ROC disposal, as OCWD's disposal cost is zero due to the partnership (joint potable reuse project) with OC San. For other projects with concentrate disposal costs, the reduction in primary concentrate discharge (accounting for discharge of new technology concentrate, e.g., CCRO, FO-RO concentrate), will improve the economics of adding concentrate recovery as a 'fourth stage'.

CONCLUSIONS

Both CCRO and FO-RO pilot treatments of primary RO concentrate yielded high-quality permeate with low inorganic, organic, and microbiological constituent concentrations. All permeate grab samples from the CCRO and FO-RO pilot units were devoid of native microbiological indicators, such as total coliform, *E. coli*, and SOM and MS coliphage; further, challenge testing with MS coliphage demonstrated high log removals. The double-membrane barrier of FO-RO is likely responsible for higher observed MS coliphage log removal during challenge testing, as well as greater removal for certain chemical constituents that were native to the feed water (plant ROC).

At the full scale, the permeate from either recovery technology could be combined with the current plant primary RO permeate before being treated with UV-AOP to boost total water output. Permeate from CCRO and FO-RO was suitably treated by UV-AOP/H_2O_2 that yielded approximately 1.4-log and 0.5-log removal of NDMA and 1,4-dioxane, respectively, comparable to full-scale UV-AOP treatment of the plant RO permeate.

A recovery system with a feed flow rate of 10- or 20-mgd (45,461 or 90,922 m^3/d) treating plant RO concentrate by CCRO is projected to produce 17,763 or 35,122 m^3/d, respectively, compared to 13,238 or 26,476 m^3/d for FO-RO. The CCRO system is predicted to have a lower annual O&M cost, but higher capital cost, compared to FO-RO. The unit cost of water (accounting for both capital and O&M costs) is expected to be similar for the two technologies and in the range of $0.91 to $1.12/m^3

of water produced, depending on the flow rate of primary RO concentrate that is treated. Overall, the study determined that 'fourth stage' treatment of RO concentrate from municipal potable reuse to recover more purified water is feasible and reasonably economical using existing commercially available technologies.

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DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

CONFLICT OF INTEREST

The authors declare there is no conflict.

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