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Contaminants of Concern in Potable Reuse Membrane Treatment: Evaluating Occurrence of Contaminants of Emerging Concern in MF/RO Treatment of Primary Effluent in a Novel Water Recycling Process

Research and Development Office
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Acronyms and Abbreviations

°C	degrees Celsius
Cc	concentration of CECs measured in concentrate, ng/L
Cf,	concentration of CECs measured in feed, ng/L
Cp	concentration of CECs measured in product, ng/L
BOD	biochemical oxygen demand
BP	by-products
CCWRF	Carbon Canyon Water Recycling Facility
CEC	contaminants of emerging concern
DBP	disinfection by-product
DEET	diethyl toluamide
DPR	direct potable reuse
EC	specific conductance
First phase	1-stage the RO process
GC-MS	gas chromatography mass spectrometry
gfd	gallons per square foot per day
g/mol	grams per mole
gpm	gallons per minute
HPI	hydrophilic
HPO	hydrophobic
I	ionic charged
IAP	independent advisory panel
IEUA	Inland Empire Utilities Agency
IPR	indirect potable reuse
kPa	kilopascal
L	liter
LC-MS	Liquid chromatography mass spectrometry
N	neutral
M_c	mass flux of CECs calculated in concentrate
M_f	mass flux of CECs calculated in feed
M_p	mass flux of CECs calculated in permeate
MF	microfiltration
mg/L	milligrams per liter
mgd	million gallons per day
mm	millimeters
MRL	method reporting limits
MW	molecular weight
ng/min	nanograms per minute
NDMA	N-nitrosodimethylamine
NF	nanofiltration
ng/min	nanograms per minute
ng/L	nanograms per liter
NWRI	National Water Research Institute
PCP	personal care product
PFOA	Perfluorooctanoic acid

PFOS	Perfluorooctane sulfonate
PVDF	polyvinylidene fluoride
Q_c	concentrate flow rate, L/min
Q_f	feed flow rate, L/min
Q_p	product flow rate, L/min
Reclamation	Bureau of Reclamation
RO	reverse osmosis
Second phase	2-stage RO process
TDS	total dissolved solids
TIN	total inorganic nitrogen
TN	total nitrogen
TOC	total organic carbon
TSS	total suspended solids
WWTP	wastewater treatment plant
µm/cm	micrometers per centimeter
µg/L	micrograms per liter

Executive Summary

This study experimented with the novel approach of using a microfiltration (MF) and reverse osmosis (RO) treatment train to treat the effluent of a primary settling tank at the Inland Empire Utility Agency in Chino, California. The innovative microfiltration (MF) and reverse osmosis (RO) treatment train generates a water source without secondary treatment and can still remove many CECs. This study showed the viability of eliminating secondary treatment and efficiently preparing wastewater for reuse through this novel treatment train.

The potential effect of some contaminants of emerging concern (CEC) on public health and the environment has urged water managers to more actively investigate strategies that remove, neutralize and/or destroy these compounds not only from drinking water but also as part of a wastewater treatment process effluent. Primary treatment is currently unable to eliminate all substances; therefore, it is usually followed by secondary treatment.

The MF/RO treatment train to treat the effluent from a primary settling tank was at the Inland Empire Utility Agency in Chino, California. The pilot used polyvinylidene fluoride hollow-fiber MF modules as pretreatment for an RO skid, which used Hydranautics ESPA2 membranes in a two-stage configuration with a feed capacity of 6 gallons per minute (gpm). In this pilot configuration, researchers monitored the removal of 38 of the most prevalent CECs through the MF/RO process.

To investigate how operating the RO process at two fixed recovery rates of 55% and 80% would affect the performance of the MF/RO membranes, researchers applied different fluxes (8, 10, 12 and 14 gallons per square foot per day [gfd]) and evaluated the removal of CECs in 1-stage and 2-stage RO configurations.

The occurrence of CECs in the MF influent, MF effluent, RO permeate, and RO concentrate were analyzed and studied. In the first phase (1-stage the RO process), flux of 14 gfd showed a better inorganics rejection (95.2%) when compared with those to other fluxes. Meanwhile, in the second phase (2-stage RO process), the flux of 12 gfd showed a better inorganics rejection (93.7%) when compared with to the other fluxes. Although concentrations of CECs slightly decreased in the RO permeate as the flux has increased, statistical analysis showed no significant differences between different fluxes in terms of CEC rejection.

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Introduction

The water industry is increasingly implementing recycled water projects to respond to current demands and challenges, such as water shortages, that the world faces today. To develop future water supplies that remain sustainable in dry years, water managers and their communities will rely on reclamation plants and their abilities to make wastewater a viable source of potable water.

Several options exist to beneficially reuse water. Indirect potable reuse (IPR) is one method of creating high-purity product water with reduced energy inputs and economic costs (Rodriguez et al. 2009). In this process, municipal wastewater is treated through a conventional treatment train (including aerobic biological treatment), processed through membrane technology, and then discharged directly into groundwater or surface water sources, which act as an environmental buffer (Leverenz et al. 2011).

Another method is direct potable reuse (DPR). This process entails full advanced treatment and can directly deliver water to a potable water treatment plant's supply without any environmental buffer. With that being said, regulations on implementing DPR are still in the premature stages of development.

Today, water managers are incorporating newly developed tertiary treatment processes to their IPR or DPR treatment trains to produce higher-quality water, especially as an opportunity for water reuse. However, whatever treatment method they select, these managers still face two distinct challenges that must be addressed: (1) contaminants of emerging concern (CEC) in wastewater, especially in the concentrate stream that is typically disposed of into the environment and (2) the relatively high energy consumption per volume of product water of these advanced processes.

One of the key issues related to water reuse is the occurrence of CECs (Romeyn et al. 2016). Prime examples of emerging contaminants include personal care products (PCP), endocrine-disrupting compounds, and pharmaceuticals; in particular, an expanding list of pharmaceuticals are now being found ubiquitously in the environment (Focazio et al. 2008, Loos et al. 2009, Silva et al. 2011, González et al. 2012, and Osorio et al. 2012). Compared to the feed stream, reject streams contain elevated concentrations of CECs (Zorita et al. 2009, Jelic et al. 2011, and Gracia-Lor et al. 2012). Recent innovations in water analysis methods, primarily in gas chromatography mass spectrometry (GC-MS) and liquid chromatography mass spectrometry (LC-MS), have allowed the water industry to develop a more comprehensive understanding of contaminants. At the least, some CECs have been found to present potential risks to water supplies due to their physiochemical properties, such as poor degradability and high water solubility (Knepper et al. 1999). These properties allow CECs to pass through most common filtration steps including some membrane treatment processes.

Membrane processes are well used in water treatment, including IPR and DPR processes, given their ability to produce stable and desired effluent quality (Reith and Birkenhead 1998, Qin et al. 2004, and Ravazzini et al. 2005). Membrane technologies such as microfiltration (MF) and reverse osmosis (RO) produce desired effluent water quality by separate organic compounds, total dissolved solids, and microorganisms from the feed and producing a permeate free of most contaminants and salts (Hofman et al. 1997, Rautenbach et al. 2000, Lee and Lueptow 2001, Radjenović et al. 2008, and Shivajirao 2012).

MF technologies have been found to filter out only a few emerging organic contaminants (Yoon et al. 2006, Kowalska 2008, and Sahar et al. 2011). However, RO processes have proved highly effective at removing a wide range of emerging contaminants and the resulting treated water can be used for more exigent purposes (Snyder et al. 2007, Calderón- Preciado et al. 2011, and Huang et al. 2011). A treatment train of MF followed by RO offers a way to bypass secondary and tertiary treatment and Reduce CECs from product water.

As of yet, no comprehensive study has evaluated the occurrence and concentration of CECs in all streams produced by IPR and DPR-type treatment trains. Most research pertains to the water product produced by such systems since membranes are known to effectively reduce most CECs.

The objectives of this study are to (a) evaluate the occurrence of CECs in MF/RO treatment of a WWTP's primary effluent and (b) demonstrate the effectiveness of MF/RO in treating primary effluent as a novel water recycling process.

Materials and Methods

Contaminants of Emerging Concerns

A CEC list was created according to an exhaustive study that identified the most common CECs in the literature. The top 38 common CECs were carefully selected and categorized by type. Table 1 presents a summary of the CECs examined in this study and their properties. The list consists of chemicals with high frequencies of occurrence and health risks.

Table 1. Selected and Examined CECs

Type	Compound	MW g/mol	Charge pH 7.0 (mV)[a]	pKa[a]	Log K_{ow}	Hydro class[b]	Chemical Formula	Reference
Analgesics/anti-inflammatories	Acetaminophen	151	0	9.46	0.34	HPI-N	$C_8H_9NO_2$	(Yamamoto et al. 2009)
	Diclofenac	296	-1	4	4.5	HPO-I	$C_{14}H_{11}Cl_2NO_2$	(Carballa et al. 2008)
	Ibuprofen	206	-1	4.85	3.5-4.91	HPO-I	$C_{13}H_{18}O_2$	(Lin et al. 2006)
	Naproxen	230	0	4.19	3.2	HPO-N	$C_{14}H_{14}O_3$	(Carballa et al. 2008)
	Salicylic acid	138			2.26	HPO	$C_7H_6O_3$	(Moffat et al. 2011)
	Primidone	218	0	11.5	0.9	HPI-N	$C_{12}H_{14}N_2O_2$	(Moffat et al. 2011)
Antibiotic	Amoxicillin	365	-0.33	3.23	0.87	HPI-I	$C_{16}H_{19}N_3O_5S$	(Jones et al. 2002)
	Azithromycin	749	1.8		4.02	HPO-N	$C_{38}H_{72}N_2O_{12}$	(McFarland et al. 1997)
	Ciprofloxacin	331			0.4	HPI	$C_{17}H_{18}FN_3O_3$	(Wick et al. 2009)
	Sulfamethoxazole	253	-1	6.16	0.89	HPI-I	$C_{10}H_{11}N_3O_3S$	(Carballa et al. 2008)
	Trimethoprim	290	0.6	7.16	0.91	HPI-N	$C_{14}H_{18}N_4O_3$	(Moffat et al. 2011)
Beta-blockers	Atenolol	266	1	14.08	0.16	HPI-N	$C_{14}H_{22}N_2O_3$	(Vieno et al. 2007)
	Propranolol	259			3.48	HPO	$C_{16}H_{21}NO_2$	-
Lipid regulators	Gemfibrozil	250	0	4.42	4.77	HPO-N	$C_{15}H_{22}O_3$	-
Psychiatric drugs	Carbamazepine	236	0	15.69	2.45	HPO-N	$C_{15}H_{12}N_2O$	(Carballa et al. 2008)
	Diazepam	285	0	2.92	2.82	HPO-N	$C_{16}H_{13}ClN_2O$	(Sangster 1997)
	Fluoxetine	309	0		4.05	HPO-N	$C_{17}H_{18}F_3NO$	(Moffat et al. 2011)
Hormones	Estrone	270	0	10.3	4.1	HPO-N	$C_{18}H_{22}O_2$	(Carballa et al. 2008)
	Testosterone	288	0		3.32	HPO-N	$C_{19}H_{28}O_2$	(Hansch et al. 1995)
	17-β-Estradiol	604	0		3.9-4.0	HPO-N	$C_{40}H_{60}O_4$	(Carballa et al. 2008)
	Progesterone	314	0		3.87	HPO-N	$C_{21}H_{30}O_2$	(Hansch et al., 1995)
Antiseptics	Triclosan	290	-0.14	7.68	4.8	HPO-I	$C_{12}H_7Cl_3O_2$	(Moffat et al. 2011)
Contrast media	Iopromide	791	0	11.1	-2.33	HPI-N	$C_{18}H_{24}I_3N_3O_8$	-
Psychostimulants	Caffeine	194	0		-0.07	HPI-N	$C_8H_{10}N_4O_2$	(Hansch et al. 1995)
Component of plastics	Bisphenol A	228	0	9.78	3.32	HPO-N	$C_{15}H_{16}O_2$	-
Drugs of abuse	Cotinine	176	0		0.07	HPI-N	$C_{10}H_{12}N_2O$	(Li et al. 1992)
Pesticides	Diethyl toluamide (DEET)	191	0		2.2	HPO-N	$C_{12}H_{17}NO$	(Moffat et al. 2011)

Industrial Compound	1,4 Dioxane	88			-0.27	HPI	$C_4H_8O_2$	(Hansch et al. 1995)
By Products (BPs)	N-Nitrosodimethylamine (NDMA)	74			-0.57	HPI	$C_2H_6N_2O$	(Hansch et al. 1995)
	N-Nitrosodiethylamine	102			0.48	HPI	$C_4H_{10}N_2O$	(Hansch et al. 1995)
	N-Nitrosomorpholine	116			-0.44	HPI	$C_4H_8N_2O_2$	(Hansch et al. 1995)
Antianxiety	Meprobamate	218	0		0.7	HPI-N	$C_9H_{18}N_2O_4$	(Hansch et al. 1995)
Flame retardant	TCEP	250	0		1.4	HPO-N	$C_9H_{15}O_6P$	-
	TCPP	327	0		2.59	HPO-N	$C_9H_{18}Cl_3O_4P$	(Miti 1992)
	TDCPP	430	0		3.65	HPO-N	$C_9H_{15}Cl_6O_4P$	(Miti 1992)
Statins	Atorvastatin	558			6.36	HPO	$C_{33}H_{35}FN_2O_5$	(Moffat et al. 2011)
Opioid	Methadone	309			3.93	HPO	$C_{21}H_{27}NO$	(Hansch et al. 1995)
Perfluorinated organic compounds	Perfluorooctane sulfonic (PFOS) acid	500			-1.08	HPI	$C_8HF_{17}O_3S$	(Krop and Voogt 2008)

MW = molecular weight
g/mol = grams per mole
[a]ACS 2015, ChemAxon 2015.
[b]Hydrophobicity class: HPI, hydrophilic; HPO, hydrophobic; N, neutral; and I, ionic charged.

WWTP and the Pilot Project

The study was performed at the Carbon Canyon Water Recycling Facility (CCWRF) in Chino, California. CCWRF provides primary treatment by preliminary screening and grit removal, primary clarification, secondary treatment consisting of aeration basins and clarification, tertiary treatment consisting of filtration and disinfection using chlorine, and finally dechlorination. The plant is designed to treat an annual average flowrate of 11.4 million gallon per day (mgd) (IEUA 2014).

In collaboration with IEUA, this pilot project researched the effects of sending primary treated effluent directly to MF and followed by RO, thus bypassing secondary and tertiary treatment. Figure 1 shows the schematic of the treatment train used in this study, as well as the locations of sampling points for CECs.

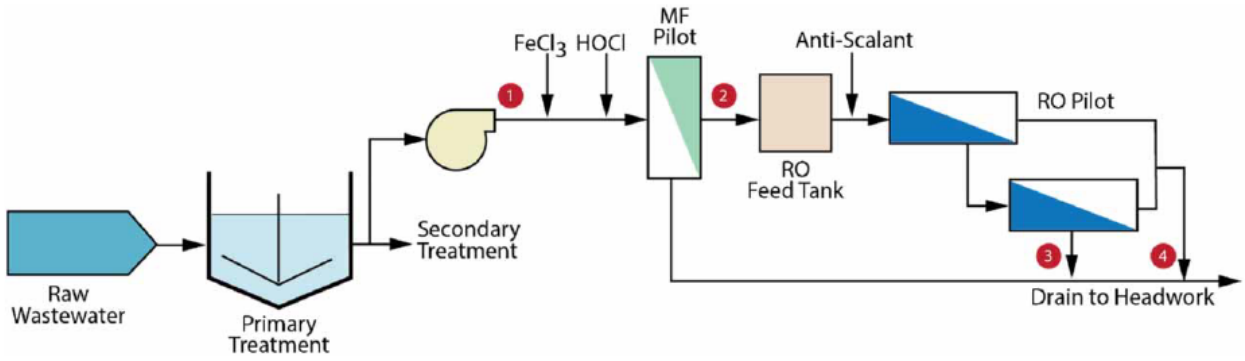

Figure 1. Pilot system with sampling points, 1 (Primary effluent), 2 (MF permeate), 3 (RO concentrate), and 4 (RO permeate).

The scope of the project involved demonstrating the feasibility of this innovative treatment train, especially in reducing CECs, and displaying a mass balance of CECs between the feed, product, and concentrate streams. A future benefit of this treatment train would be decreased energy consumption for overall treatment, given that the most energy-intensive component of a conventional treatment train is associated with the secondary biological treatment processes (Raucher and Tchobanoglous 2014).

Raw Wastewater

The pilot project used primary effluent wastewater from the CCWRF as feed to the MF and RO system. Table 2 shows the wastewater characteristics that the IEUA reported in 2012.

Table 2. Influent Wastewater Characteristics at Carbon Canyon Water Recycling Facility

Constituent	Unit	Minimum	Average	Maximum
Specific Conductance	µm/cm	903	1,048	1,184
pH	units	6.9	7.1	7.2
Total Organic Carbon (TOC)	mg/L	166	246	334
Biochemical Oxygen Demand (BOD)	mg/L	308	451	627
Total Suspended Solids (TSS)	mg/L	228	390	730
Total Dissolved Solids (TDS)	mg/L	509	538	559
Ammonia-Nitrogen (NH_3-N)	mg/L	29.2	34.1	45.8
Total Inorganic Nitrogen (TIN)	mg/L	29.7	31.7	33.1
Total Nitrogen (TN)	mg/L	46.0	53.3	59.6
Boron	mg/L	0.2	0.3	0.3
Chloride	mg/L	100	116	132
Fluoride	mg/L	0.2	0.2	0.3
Sulfate	mg/L	35	45	53
Total Hardness, as $CaCO_3$	mg/L	169	198	250
Arsenic, Total Recoverable	µg/L	<10	<10	<10
Cadmium, Total Recoverable	µg/L	<10	<10	<10
Chromium, Total Recoverable	µg/L	<10	<10	<10
Copper, Total Recoverable	µg/L	40	63	80
Lead, Total Recoverable	µg/L	<20	<20	<20
Mercury, Total Recoverable	µg/L	<0.5	<0.5	<0.5
Nickel, Total Recoverable	µg/L	<10	<10	<10
Selenium, Total Recoverable	µg/L	<20	<20	<20
Silver, Total Recoverable	µg/L	<10	<10	<10
Zinc, Total Recoverable	µg/L	120	195	280
Free Cyanide (Aquatic)	µg/L	<2	<3	4
Bis (2-ethylhexyl) phthalate	µg/L	12	12	13

Source: IEUA 2011-12, from 2012 data (IEUA 2011-12 and IEUA 2014).
µm/cm = micrometers per centimeter
mg/L = milligrams per liter
µg/L =micrograms per liter

MF System

The MF pilot system was a fully automated membrane system designed and maintained by PALL Corporation. Its operational parameters were measured continuously at ten-minute intervals and automatically recorded. Total feed flow rate into the MF unit was 25 gpm. Average flux during the course of experiments was 13 gfd.

The pilot unit included a hot water heater and chemical pumps for automatic enhanced flux maintenance cleans that were carried out every 24 hours. The system was equipped with two new UNA-620A hollow-fiber MF modules, each of which contained 538 square feet of active membrane surface area and operated in outside-to-inside filtration mode. The membrane was a polyvinylidene fluoride (PVDF) hollowfiber type with a nominal pore size of 0.1 µm. PVDF fibers are known for having high mechanical and chemical resistance.

Ferric chloride and chlorine (bleach) were injected directly into the feed stream (i.e., the WWTP's primary effluent), which then fed the MF pilot skid at a target concentration of 20 parts per million (ppm) and 1.5 ppm, respectively. Bleach was added to create a chloramine residual to reduce microbial growth on the membranes. While further testing would be valuable to find the optimum dosage for the coagulant, the present configuration was adequate in demonstrating the benefit of adding ferric chloride to the process. The two membranes were operated in parallel to provide a suitable flow rate to the downstream RO pilot.

RO System

The RO pilot system was the Membrane Evaluation Research Unit 5 (MERU5) skid owned by the Bureau of Reclamation's (Reclamation) Yuma Area Office. MERU5 has up to three stages; however, for this study, only 1-stage and 2-stage were used. 1-stage consisted of two 4-inch pressure vessels, each containing three RO ESPA2-4040 elements, while 2-stage consisted of two 2.5-inch pressure vessels, each containing three RO ESPA2-2540 elements.

The MF effluent was used as feed water to the RO and contained inorganics, dissolved organic constituents, and a trace level of suspended materials that could potentially precipitate on the membrane surface. A feed spacer 34 millimeters (mm) thick in the ESPA2 membrane was used to prevent colloidal fouling and increase the effectiveness of membrane cleaning. Furthermore, by using these membranes, precipitation and the costs of additional cleaning were minimized.

An antiscalant, Vitec 1400, was dosed into the RO feed stream at a concentration of approximately 3 mg/L to prevent inorganic scale from forming on the membrane's surface.

Mass Balance

The mass balance was calculated following the method used by Gao et al. (2012). The average mass flow of each compound was calculated by multiplying the sum of the CEC concentrations in the permeate and concentrate with corresponding average flows in the influent (Equations 1 - 3):

$$M_f = Q_f \times C_f \quad (1)$$

$$M_c = Q_c \times C_c \quad (2)$$

$$M_p = Q_p \times C_p \quad (3)$$

M_f, M_p and M_c (ng/min) are the mass flux of CECs calculated in the influent, permeate and concentrate streams, respectively. Q_f, Q_p and Q_c (L/min) represent feed, product and concentrate flows, respectively. C_f, C_p and C_c (ng/L) are the

average concentrations of CECs measured in the feed, permeate and concentrate flows, respectively.

The discrepancy in the mass balance of CECs compounds can be calculated and presented as M_{disc}. To estimate the mass of CECs that is lost due to the membranes' uptake, Equation 4 is used

$$M_{disc} = M_f - M_p - M_c \quad (4)$$

The mass balance discrepancy, in percentage, is calculated using Equation 5:

$$R_{disc} = \frac{M_{disc}}{M_f} \times 100 \quad (5)$$

Experimental Procedure

The RO feed tank (500 gallons) was filled with MF permeate at a constant flow rate of 10 gpm. To start the RO process, the pressure of the feed water was gradually increased along with the pump speed. After the target pressure (i.e., 300 kilopascal [kPa]) was achieved, the RO feed valve was gradually opened. At the same time, the concentrate valve and permeate valves were fully opened and initiated. By increasing and decreasing the pump rate and controlling the flow rate of the concentrate valve, the target flow rate in the permeate can be achieved. To achieve the target flux and recovery rate, permeate and concentrate flow rates were calculated and set in the RO unit by changing the set points of the feed valve, concentrate valve, and the feed's pump rate.

The experiments in this study were performed in two phases. In the first phase, only 1-stage RO was operated, using 4-inch elements with a fixed recovery rate of 55%. This recovery rate was selected to evaluate whether recovery has any significant effect on the membranes' ability to reject CECs. The total membrane surface area used in this phase was 510 square feet (ft^2). Four different fluxes of 8, 10, 12, and 14 gfd were selected and targeted under the constant recovery rate of 55%.

In the second phase of the study, 1-stage and 2-stage RO membranes were operated. Again, 1-stage RO used 4-inch elements, while 2-stage used 2.5-inch elements to achieve a recovery rate of 80%. Four different fluxes of 8, 10, 12, and 14 gfd were selected and targeted under the constant recovery rate of 80%. The total membrane surface area used in this set of experiments was 660 ft^2.

Antiscalant with a concentration of 3 mg/l was added to the RO feed stream before the high-pressure pumps. Ferric chloride ($FeCl_3$) and chlorine (HOCl) were injected directly into the primary effluent stream that fed the MF pilot skid at a target concentration of 20 ppm and 7 ppm, respectively.

Permeate and concentrate from the RO unit were collected for sampling, and the streams were blended and sent to the common drain line to the WWTP's headworks. The duration for each flux test ranged from three to five hours. After each test condition, the RO pilot was flushed with RO permeate. Each test was done in a different day. Permeate and concentrate samples for each test run were collected no sooner than three hours after the start of testing to allow the RO system to stabilize. RO samples were taken when permeate and concentrate conductivity were constant for at least for an hour with no feed temperature variations.

The sample volume was 8 liters (L) for organic compounds analysis and 2.5 L for inorganic compounds analysis. Prepared amber glass (for organic compounds) and polynutrients and poly-metals (for inorganic compounds) bottles were used for sampling. Bottles contained sodium thiosulfate and ascorbic acid (for organic analysis) and phosphoric acid, sulfuric acid, and nitric acid (for inorganic analysis) as preservatives. Samples were chilled to below 4 degrees Celsius (°C) on ice or frozen gel packs and delivered to the local, certified laboratory on the same day. All CEC and inorganic analyses were performed at this location.

Due to limited resources, the MF feed (i.e., primary effluent) and MF product (i.e., RO feed) were sampled and analyzed for CECs only once during the study. According to CCWRF, the water chemistry of raw sewage to the plant does not have significant variations over extended periods of time. However, a municipal WWTP can experience daily variations in its feed water's water chemistry.

Analytical Method

The collected samples were then shipped in the same day to the Weck Laboratories, Inc. in City of Industry, California. Methods 8270, 1694, and 1625 provided quantitative data on the suite of 38 CECs being investigated for this research. These methods involved online pre-concentration followed by liquid chromatograph separation and series mass spectrometry (LS-MS-MS) with electrospray ionization in positive and negative modes. Samples were pre-concentrated using a previously developed direct online extraction/analysis method (Haghani et al. 2009) to achieve low-ng/L method reporting limits (MRL). The test methods used for the MRL for the subject 38 CECs ranged from 1 to 2,500 ng/L.

Results and Discussion

Occurrence and Removal of Inorganics in the MF

CECs originate from industrial and domestic products such as pesticides, PCPs, preservatives, surfactants, flame retardants, and perfluorochemicals. These contaminants are also excreted by humans in the form of human waste that contains pharmaceutical residues or steroidal hormones. CECs also surface as

chemicals formed during wastewater and drinking water treatment, known as disinfection by-products (DBP).

Table 3 shows an analysis of the primary effluent (i.e., MF feed) and MF permeate (i.e., MF effluent) for inorganics compounds. The data in the 'MF feed' column was obtained from the reports provided by CCWRF (IEUA 2014). As expected, the MF process does not remove dissolved inorganic constituents; however, it is excellent at removing suspended materials, which reduces the concentration of certain organic compounds represented by BOD and TOC.

Table 3. Characteristics of Primary Effluent and MF Permeate

Compound	Unit	MF Feed	MF Permeate
Chloride	mg/L	116	120
Sulfate as SO_4	mg/L	45	45
Ammonia as N	mg/L	34.1	53
Phosphorus as PO_4	mg/L	NA	11
Barium	mg/L	NA	0.0090
Calcium	mg/L	NA	42.8
Magnesium	mg/L	NA	10.2
Silica as SiO_2	mg/L	NA	18
Sodium	mg/L	NA	80
Bicarbonate Alkalinity as HCO_3	mg/L	NA	380
Alkalinity as $CaCO_3$	mg/L	250	320
Total Dissolved Solids (TDS)	mg/L	538	510
Total Suspended Solids (TSS)	mg/L	390	ND
Total Organic Carbon (TOC)	mg/L	246	74
Biochemical Oxygen Demand (BOD)	mg/L	451	140
Specific Conductance (EC)	µm/cm	1,048	1,097
pH		7.1	6.8

(NA: Not Analyzed, ND: Not Detected)

Considering the CECs molecular weights (MW) listed in Table 1, the MF process is unlikely to significantly or meaningfully remove CEC micropollutants from the primary effluent. After all, MF is generally used as pretreatment for particulate matter reduction and water stabilization and to avoid fouling of the RO membranes, which creates optimal operating conditions for the RO process.

Acetaminophen was the most abundant compound with a concentration of 130 µg/L in primary effluent (e.g. MF feed) as displayed in Table 4. Acetaminophen was followed by other analgesics, anti-inflammatories, lipid regulators gemfibrozil and bezafibrate, and the betablocker atenolol. High concentrations of acetaminophen and caffeine have also has been reported in similar studies: Yang et al. (2011) reported acetaminophen and caffeine concentrations of about 100 µg/L in an advanced wastewater reclamation plant located in Gwinnett County, Georgia. They found a high ibuprofen concentration

of approximately 10 µg/L and carbamazepine concentration of approximately 1 µg/L, which are similar to the numbers that were observed in this study's MF feed analysis.

The presence of by-products (BP), such as NDMA, is particularly important in places where a treatment plant's effluent is used for IPR. Chlorinating wastewater leads to relatively high concentrations of BPs. In fact, NDMA formation can exceed 100 ng/L during the chlorination of secondary wastewater effluent (Najm and Trussell 2017), whereas chlorination of surface waters typically results in the formation of less than 10 ng/L of NDMA (Najm and Trussell 2017).

NDMA results from chlorination due to the slow reaction of monochloramine with dimethylamine, which ultimately forms an unsymmetrical dimethylhydrazine intermediate (Choi and Valentine 2002 and Mitch and Sedlak 2002). There were no N-nitroso compounds found in the primary effluent. However, the MF process uses chloramines as a disinfectant, which are formed by adding chlorine bleach to naturally occurring ammonia in the primary effluent. Therefore, after the MF process, N-nitroso compounds were detected in the laboratory analysis. Other studies have shown that chlorination using hypochlorite results in approximately an order of magnitude less NDMA than what is formed through chlorination using monochloramine (Mitch and Sedlak 2002).

As for perfluorooctane sulfonate (PFOS), the compound was not detected in the MF feed but found in the MF permeate. This result could be due to an inaccuracy in the laboratory analysis or a possible transformation of fluorosulfonamides, such as FOSE and FOSA, to PFOS. This observation has been reported in other studies as well (Schultz et al. 2006, Sinclair and Kannan 2006, and Loganathan et al. 2007). However, in prior studies, the average PFOS concentration formed was 4 ng/L; in this study, the PFOS concentration was 270 ng/L.

In general, conventional wastewater treatment techniques (e.g., trickling filtration, activated sludge, anaerobic digestion, and chlorination) have been reported to have little effect on PFOS removal (Schultz et al. 2006 and Sinclair and Kannan 2006) given that microbial communities cannot metabolize PFOS (Key et al. 1998 and Hollingsworth et al. 2005). In some cases, PFOS concentrations were greater in the WWTP effluent as compared with those in the influent (Schultz et al. 2006 and Sinclair and Kannan 2006). This suggests microbial transformation (Schultz et al. 2006, Sinclair and Kannan 2006, and Loganathanet al. 2007) of fluorosulfonamides (e.g., sulfonamidoethanol [FOSE] and perfluorooctane sulfonamide [FOSA]) to PFOS (Tomy et al. 2004 and Xu et al. 2004), the transformation of fluorotelomer alcohols to perfluorooctanoic acid (PFOA), or the release of residual PFOX from the solid phase. The RO process has been reported to be effective in removing PFOSs.

RO Pilot Operation Data

As mentioned before, this study consisted of two phases: the first phase was an MF process followed by a 1-stage RO process using 4-inch ESPA2-4040 elements with a target recovery rate of 55%. The second phase was an MF process followed by a 2-stage RO process using 4-inch ESPA2-4040 and 2.5-inch ESPA-2540 elements with a recovery rate of 80%. For both phases, four different fluxes of 8, 10, 12 and 14 gfd were selected and targeted.

The RO pilot unit had a capacity of 19.5 L/min-33.6 L/min of feed from the RO feed tank, and the pressure was variable between 470 kPa and 1,000 kPa to obtain the mentioned fluxes with 55% and 80% recoveries for phase one and phase two, respectively. Antiscalant with a concentration of 3.02 mg/L was added to the RO feed stream before the high-pressure pumps. Permeate and concentrate from the RO unit were collected for sampling, and the remainder of the streams was blended with the RO concentrate and directed to the common drain line to the plant's headworks.

Recovery of the membranes was derived from Equation 6:

$$Recovery\ (\%) = \frac{Volume\ of\ the\ permeate}{Volume\ of\ the\ feed} \times 100 \quad (6)$$

Flux (J) is the volume of permeate (V) collected per unit membrane area (A) per time (t) as shown in Equation 7:

$$J = \frac{V}{At} \quad (7)$$

Removal of Inorganics through the RO process

The RO system's performance was evaluated in terms of the permeate's pollutant concentrations and the membrane rejection. The rejection of the RO membrane was calculated using Equation 8:

$$Rejection\ (\%) = \left(\frac{C_f - C_p}{C_f}\right) \times 100 \quad (8)$$

where C_f, mg/L, is the feed concentrations and C_p, mg/L, is the permeate concentration.

Table 4 and Table 5 show all removal rates at the different fluxes tested. For the first phase of this study, conductivity rejection was found to be 92.8%, 90.0%, 93.3% and 93.5% for fluxes of 8, 10, 12 and 14 gfd, respectively. For the second phase, conductivity rejection was 85.5%, 89.1%, 91% and 91.5% for fluxes of 8, 10, 12 and 14 gfd, respectively.

These findings matched expectations: increasing flux slightly decreases the salt concentration in the permeate. This is because the salt leakage across the membrane remains fairly constant. At higher flux rates, the mass of salt passing across the membrane is blended with more permeate than at lower flux rates, resulting in a lower conductivity product stream. The only exception to this condition was when applying flux of 8 gfd in the first phase. With that being said, this relatively high salt-removal rate could simply be an error since this was the first data point collected in this pilot study and the experiment was not mature enough for data collection.

For the first phase of this study, 94.1%, 90.0%, 93.4% and 93.5% of chloride rejections were achieved with fluxes 8, 10, 12 and 14 gfd, respectively. For the second phase, 85.8%, 90.8%, 92.1% and 92.9% of chloride rejections were achieved with fluxes 8, 10, 12 and 14 gfd, respectively. These results align with what was explained earlier about conductivity removal.

More than 98.2% of sulfate rejection was obtained with all fluxes in both phases. In addition, calcium removal was 97.7% with all fluxes for both phases, while average sodium removal was 89.4% for the first phase and 86.9% for the second phase with all fluxes.

Higher rejection of di- and multivalent ions could be explained by the size of multivalent ions, which is larger than monovalent ones, and by their charge effect, which is consistent with results reported in past literature. An increase in an anion charge leads to an increase of electrostatic interactions with membranes.

Phosphate rejection was more than 99.2% for all conditions in the first phase; but, in the second phase, phosphate was not efficiently rejected. This result cannot be explained when the removal is compared with other ions.

Overall, in the first phase (i.e,. 1-stage RO), the flux of 14 gfd showed better rejection values for most inorganic compounds compared with those of other fluxes. Similar results were found for the second phase, which ultimately means that increasing flux improves the permeate quality.

However, results show that 1-stage RO with a 55% recovery rate had a better removal rate of CECs when compared with 2-stage RO with an 80% recovery rate. These results align with those of other studies. As the concentration gradient of contaminants increases across the membrane at higher recovery rates, the overall removal efficiency for various compounds decreases.

Table 4. Occurrence of the Target CECs through the Process with Different Fluxes in Phase One with One Stage RO

				Flux 8 gfd			Flux 10 gfd			Flux 12 gfd			Flux 14 gfd		
Compound	**Unit**	**Primary Effluent**	**MF Permeate**	**Permeate Conc.**	**Concentrate Conc.**	**Removal%**	**Permeate Conc.**	**Concentrate Conc.**	**Removal%**	**Permeate Conc.**	**Concentrate Conc.**	**Removal%**	**Permeate Conc.**	**Concentrate Conc.**	**Removal%**
1,4-Dioxane	µg/l	ND	0.7	ND	1.9	100	ND	2	100	ND	2.2	100	ND	2.1	100
N-Nitrosodiethylamine	ng/l	ND	6	ND	4	100	ND	ND	100	ND	23	100	ND	2.7	100
NDMA	ng/l	ND	8.1	7.6	15	6.2	28	42	0	11	18	0	10	20	0
N-Nitrosomorpholine	ng/l	ND	12	ND	45	100	6.7	47	44.2	2.1	14	82.5	2.6	44	78.3
Propranolol	ng/l	0.013	0.058	0.0028	ND	95.2	4.4	73 [a]	0	2.7	2.6[d]	0	3.8	130[b]	0
PFOS	ng/l	ND	270	7.8	300	97.1	26	590	90.4	20	580	92.6	13	ND	95.2
17-b-Estradiol	ng/l	43	51	ND	80	100	1.5	32	97.1	ND	11	100	ND	46	100
Estrone	ng/l	33	160[a]	ND	180 [a]	100	ND	78	100	ND	110 [a]	100	ND	57	100
Progesterone	ng/l	5.2	26	ND	28	100	ND	93	100	ND	49	100	1.1	73	95.8
Testosterone	ng/l	4.2	110	ND	230 [a]	100	4.1	260 [a]	96.3	3.7	180 [a]	96.6	3.6	260 [a]	96.7
Bisphenol A	ng/l	310	170	39	360 [a]	77.1	21[c]	820	87.6	16[c]	520 [a, b]	90.6	35	470 [a, b]	79.4
Diclofenac	ng/l	200	160 [a]	12	220 [a]	92.5	48	540	70	39	580 [a]	75.6	46	600	71.3
Gemfibrozil	ng/l	4200[a]	3900	82	12000 [a]	97.9	90	6100 [a]	97.7	74	5500 [a, b]	98.1	70	6000 [a]	98.2
Ibuprofen	ng/l	15000[a]	12000[a]	690 [a]	13000 [a]	94.3	2300 [a]	34000 [a]	80.8	1800 [a]	33000 [a]	85	2500 [a]	43000 [a]	79.2
Iopromide	ng/l	1700[a]	1300	ND	3600 [a]	100	ND	ND	100	ND	6.1	100	16	25	98.8
Naproxen	ng/l	19000[a]	8900 [a]	580 [a]	8800 [a]	93.5	1900 [a]	12000 [a]	78.7	1300	12000 [a]	85.4	1500 [a]	14000 [a]	83.1
Salicylic Acid	ng/l	95000[a]	54000	8400[a, b]	880	84.4	100	240	99.8	61 [a]	260	99.9	1900	950	96.5
Triclosan	ng/l	1600[a]	120	5.3	280 [a]	95.6	4.1	180 [a, b]	96.6	8	310 [a, b]	93.3	8.7	220 [a, b]	92.8
Acetaminophen	ng/l	130000[a]	27000[a, b]	22000[a]	67000 [b]	18.5	22000 [a]	18000 [a]	18.5	10000 [a]	6000 [a]	63	11000 [a]	10000 [a]	59.3
Amoxicillin	ng/l	6400	2400[b]	41	25 [b]	98.3	ND	ND	100	ND	ND	100	51	980[b]	97.9
Atenolol	ng/l	3500[a]	2500[a]	210 [a]	4600 [a]	91.6	260 [a]	4400 [a]	89.6	160 [a]	3300 [a]	93.6	210 [a]	4200 [a]	91.6
Atorvastatin	ng/l	280	470[a]	14	1500 [a, b]	97	17	820 [a]	96.4	16	1100 [a, b]	96.6	18	640 [a]	96.2
Azithromycin	ng/l	1400	780	ND	210	100	ND	ND	100	ND	ND	100	ND	37 [a, c]	100
Caffeine	ng/l	84000[a]	26000[a, b]	4500 [a]	32000 [a, b]	82.7	5300 [a]	130000 [a, b]	79.6	4400 [a]	30000 [a, b]	83.1	3800 [a]	31000 [a]	85.4

				Flux 8 gfd			Flux 10 gfd			Flux 12 gfd			Flux 14 gfd		
Compound	**Unit**	**Primary Effluent**	**MF Permeate**	**Permeate Conc.**	**Concentrate Conc.**	**Removal%**	**Permeate Conc.**	**Concentrate Conc.**	**Removal%**	**Permeate Conc.**	**Concentrate Conc.**	**Removal%**	**Permeate Conc.**	**Concentrate Conc.**	**Removal%**
Carbamazepine	ng/l	170	200[a, b]	5.9	43[b]	97.1	12	260[a]	94	8.6	76	95.7	8.6	220[a, b]	95.7
Ciprofloxacin	ng/l	1500	570[b]	43	15[b]	92.5	32[c]	790[a, c]	94.4	70[c]	860[a, b, c]	87.7	65[c]	ND	88.6
Cotinine	ng/l	1700[a]	1300[a]	51	2900[a, b]	96.1	130[c]	340[a, c]	90	61[c]	1700[a]	95.3	79[c]	2400[a]	93.9
DEET	ng/l	2100[a]	1800[a]	150[a]	6300[a, b]	91.7	120	3800[a]	93.3	100	3900[a]	94.4	110	4500[a]	93.9
Diazepam	ng/l	ND	6.4	ND	12[b]	100	ND	10	100	ND	13	100	ND	16	100
Fluoxetine	ng/l	62	8.6	1	ND	88.4	ND	52[a]	100	1	18	88.4	1.1	16[b]	87.2
Meprobamate	ng/l	72	19	3.2	47[b]	83.2	13	20	31.6	7.2	24	62.1	16	370[a]	15.8
Methadone	ng/l	27	30	ND	3.7[b]	100	ND	ND	100	ND	ND	100	ND	ND	100
Primidone	ng/l	200[b]	38[b]	6	6.4[b]	84.2	ND	18	100	ND	ND	100	ND	ND	100
Sulfamethoxazole	ng/l	1800[a]	920[a]	37	3000[a, b]	96	43	2600[a]	95.3	34	3600[a]	96.3	38	1200[a]	95.9
TCEP	ng/l	290	280[a]	15[c]	230[b]	94.6	25	710[a]	91.1	23	810[a]	91.8	20	460[a]	92.9
TCPP	ng/l	920[a]	760[a]	46[c]	1200[a, b]	93.9	86[c]	2800[a]	88.7	110[c]	1700[a]	85.5	71[c]	3000[a]	90.7
TDCPP	ng/l	370	190[a]	14	1600[a, b]	92.6	15[c]	1700[a]	92.1	18[c]	2800[a]	90.5	20[c]	1100[a]	89.5
Trimethoprim	ng/l	680[a]	590[a]	35	1200[a, b]	94.1	59	1000[a]	90	53	1000[a]	91	35	1100[a]	94.1

ND: Not Detected

[a] The concentration indicated for this analyte is an estimated value above the calibration range.

[b] Low internal standard recovery possibly due to matrix interference. The result is suspect.

[c] Blank contamination. The analyte was found in the associated blank as well as in the sample.

[d] The original extraction and/or analysis of this sample yielded QC recoveries outside acceptance criteria. It was re-extracted/re-analyzed after the recommended maximum hold time.

Table 5. Occurrence of the Target CECs Along the Process with Different Fluxes In Phase Two With Two Stage RO

				Flux 8 gfd			Flux 10 gfd			Flux 12 gfd			Flux 14 gfd		
Compound	**Unit**	**Primary Effluent**	**MF Permeate**	**Permeate Conc.**	**Concentrate Conc.**	**Removal%**	**Permeate Conc.**	**Concentrate Conc.**	**Removal%**	**Permeate Conc.**	**Concentrate Conc.**	**Removal%**	**Permeate Conc.**	**Concentrate Conc.**	**Removal%**
1,4-Dioxane	µg/l	ND	0.7	ND	2.3	100.0	ND	4.4	100.0	ND	4.9	100.0	ND	4.5	100.0
N-Nitrosodiethylamine	ng/l	ND	6	3.1	12	48.3	ND	ND	100.0	ND	20[b]	100.0	ND	11[b]	100.0
NDMA	ng/l	ND	8.1	17	56	0.0	17	75	0.0	21	66	0.0	16	68	0.0
N-Nitrosomorpholine	ng/l	ND	12	2.8	24	76.7	3.8	74	68.3	3	13	75.0	2.4	20	80.0
Propranolol	ng/l	0.013	0.058	5.5	6.1[a]	0.0	ND	ND	100.0	6.2	ND	0.0	1.9	ND	0.0
Perfluorooctane sulfonate (PFOS)	ng/l	ND	270	ND	ND	100.0	9.2	ND	96.6	7.9	ND	97.1	7.7	ND	97.1
17-b-Estradiol	ng/l	43	51	ND	ND	100.0	ND	ND	100.0	ND	ND	100.0	ND	ND	100.0
Estrone	ng/l	33	160[a]	ND	2.9	100.0	ND	4.4	100.0	ND	2.2	100.0	ND	2.3	100.0
Progesterone	ng/l	5.2	26	ND	2.3	100.0	ND	3.8	100.0	ND	2.7	100.0	ND	2.9	100.0
Testosterone	ng/l	4.2	110	2.8	5.7	97.5	2	10	98.2	1.4	5.7	98.7	1.6	6.3	98.5
Bisphenol A	ng/l	310	170	43[c]	1100[a, b]	74.7	27[c]	1100[a, b]	84.1	19[c]	1200[a, b]	88.8	13	660[a, b]	92.4
Diclofenac	ng/l	200	160[a]	8.8	1100[a, b]	94.5	10	1800[a, b]	93.8	6.3	1300[a, b]	96.1	5.3	1300[a, b]	96.7
Gemfibrozil	ng/l	4200[a]	3900	100	12000[a]	97.4	56	13000[a, b]	98.6	42	11000[a, b]	98.9	43	14000[a, b]	98.9
Ibuprofen	ng/l	15000[a]	12000[a]	1000[a]	130000[a, b]	91.7	460[a]	100000[a, b]	96.2	260[a]	60000[a, b]	97.8	300	81000[a, b]	97.5
Iopromide	ng/l	1700[a]	1300	34	480[b]	97.4	93	640[a, b]	92.8	14	280[b]	98.9	29	520[b]	97.8
Naproxen	ng/l	19000[a]	8900[a]	800[a]	63000[a, b]	91.0	440[a]	70000[a, b]	95.1	280[a]	40000[a, b]	96.9	250	48000[a, b]	97.2
Salicylic Acid	ng/l	95000[a]	54000	3000[a]	190000[a, b]	94.4	2200[a]	11000[a, b]	95.9	1600[a]	21000[a, b]	97.0	1400	20000[a, b]	97.4
Triclosan	ng/l	1600[a]	120	200[a]	710[a]	0.0	260[a]	610[a]	0.0	340[a]	660[a]	0.0	440	560[a]	0.0
Acetaminophen	ng/l	130000[a]	27000[a, b]	15000[a]	86000[a, b]	44.4	16000[a]	42000[a, b]	40.7	14000[a]	140000[a, b]	48.1	14000	420000[a, b]	48.1
Amoxicillin	ng/l	6400	2400[b]	43	ND	98.2	31	ND	98.7	13	ND	99.5	14	ND	99.4
Atenolol	ng/l	3500[a]	2500[a]	270[a]	7000[a]	89.2	150[a]	6100[a]	94.0	200[a]	8000[a, b]	92.0	130	12000[a]	94.8
Atorvastatin	ng/l	280	470[a]	1.6	2100[a]	99.7	3.5	9300[a]	99.3	1.6	2600[a]	99.7	2.3	4400[a]	99.5

				Flux 8 gfd			Flux 10 gfd			Flux 12 gfd			Flux 14 gfd		
Compound	**Unit**	**Primary Effluent**	**MF Permeate**	**Permeate Conc.**	**Concentrate Conc.**	**Removal%**	**Permeate Conc.**	**Concentrate Conc.**	**Removal%**	**Permeate Conc.**	**Concentrate Conc.**	**Removal%**	**Permeate Conc.**	**Concentrate Conc.**	**Removal%**
Azithromycin	ng/l	1400	780	180[b]	ND	76.9	ND	ND	100.0	180[b]	ND	76.9	ND	ND	100.0
Caffeine	ng/l	84000[a]	26000[a, b]	3200 [a]	9400 [a]	87.7	2600 [a]	15000 [a]	90.0	2600 [a]	11000 [a]	90.0	19000	14000 [a]	26.9
Carbamazepine	ng/l	170	200[a, b]	12	250 [a, b]	94.0	7.6	80[b]	96.2	6.7	120 [b]	96.7	6.3	92 [b]	96.9
Ciprofloxacin	ng/l	1500	570[b]	57[c]	76	90.0	16[c]	180	97.2	210	140	63.2	8.8	120	98.5
Cotinine	ng/l	1700[a]	1300[a]	42	2900 [a]	96.8	47	1500 [a]	96.4	31	1200 [a, b]	97.6	34	3200 [a, b]	97.4
DEET	ng/l	2100[a]	1800[a]	120	4500 [a]	93.3	81[c]	5100 [a]	95.5	68[c]	4400 [a]	96.2	32	3000 [a]	98.2
Diazepam	ng/l	ND	6.4	ND	8.1	100.0	ND	9.3	100.0	ND	13	100.0	ND	13	100.0
Fluoxetine	ng/l	62	8.6	1.5	ND	82.6	ND	ND	100.0	ND	ND	100.0	ND	9.8 [b]	100.0
Meprobamate	ng/l	72	19	120	3800 [a]	0.0	ND	18	100.0	150 [a]	3000 [a]	0.0	7.5	100	60.5
Methadone	ng/l	27	30	1.9	ND	93.7	ND	ND	100.0	ND	ND	100.0	ND	ND	100.0
Primidone	ng/l	200[b]	38[b]	5.1	67[b]	86.6	2.5	11[b]	93.4	2.8 [b]	40	92.6	3	23 [b]	92.1
Sulfamethoxazole	ng/l	1800[a]	920[a]	65	4800 [a]	92.9	37	5000 [a]	96.0	20	4000 [a]	97.8	22	5500 [a]	97.6
TCEP	ng/l	290	280[a]	670 [a]	880 [a]	0.0	ND	2300 [a]	100.0	4800 [a]	4400 [a]	0.0	1100	6500 [a]	0.0
TCPP	ng/l	920[a]	760[a]	72[e]	1100 [a, e]	90.5	68[e]	1800 [a, e]	91.1	80 [e]	4300 [a, e]	89.5	41	2800 [a, e]	94.6
TDCPP	ng/l	370	190[a]	13	810 [a]	93.2	14	800[a]	92.6	15	1600 [a]	92.1	8.4	890 [a]	95.6
Trimethoprim	ng/l	680[a]	590[a]	57	1900 [a]	90.3	1.1	1900[a, **b**]	99.8	92	1500 [a]	84.4	6.1	1800 [a]	99.0

ND: Not Detected

[a] The concentration indicated for this analyte is an estimated value above the calibration range.

[b] Low internal standard recovery possibly due to matrix interference. The result is suspect.

[c] Blank contamination. The analyte was found in the associated blank as well as in the sample.

[d] The original extraction and/or analysis of this sample yielded QC recoveries outside acceptance criteria. It was re-extracted/re-analyzed after the recommended maximum hold time.

[e] The recovery of this analyte in the CCV's was over the control limit. Sample result is suspect.

[f] The sample was originally analyzed within holding time. However, it required a dilution and the re-analysis was performed after the recommended holding time had expired

Removal of CECs through the RO Process

CECs were studied in the permeate and concentrate streams at fluxes of 8, 10, 12 and 14 gfd, with recovery rates of 55% and 80%. Pressure-driven separation membranes are effective barriers in rejecting these pollutants (Gur-Reznik et al. 2011). In particular, studies have shown that RO is effective in removing compounds that have MWs of greater than approximately 200 g/mol (Sedlak and Pinkston 2003). The majority of the target CECs have MWs between 100 and 560 g/mol, except a few such as iopromide, azithromycin, NDMA, and 1,4 dioxane, which have MWs of 791, 749, 74 and 88 g/mol, respectively. Compounds with lower MWs exhibited much lower removal by RO.

Figure 2 shows the effects of CECs' MW on their removal in the first and second phases of this study, as well as expected high removal rates. Figure 2also shows that both phases experienced sharp drop-offs in removal efficiency for compounds with MWs of 300 g/mol or less.

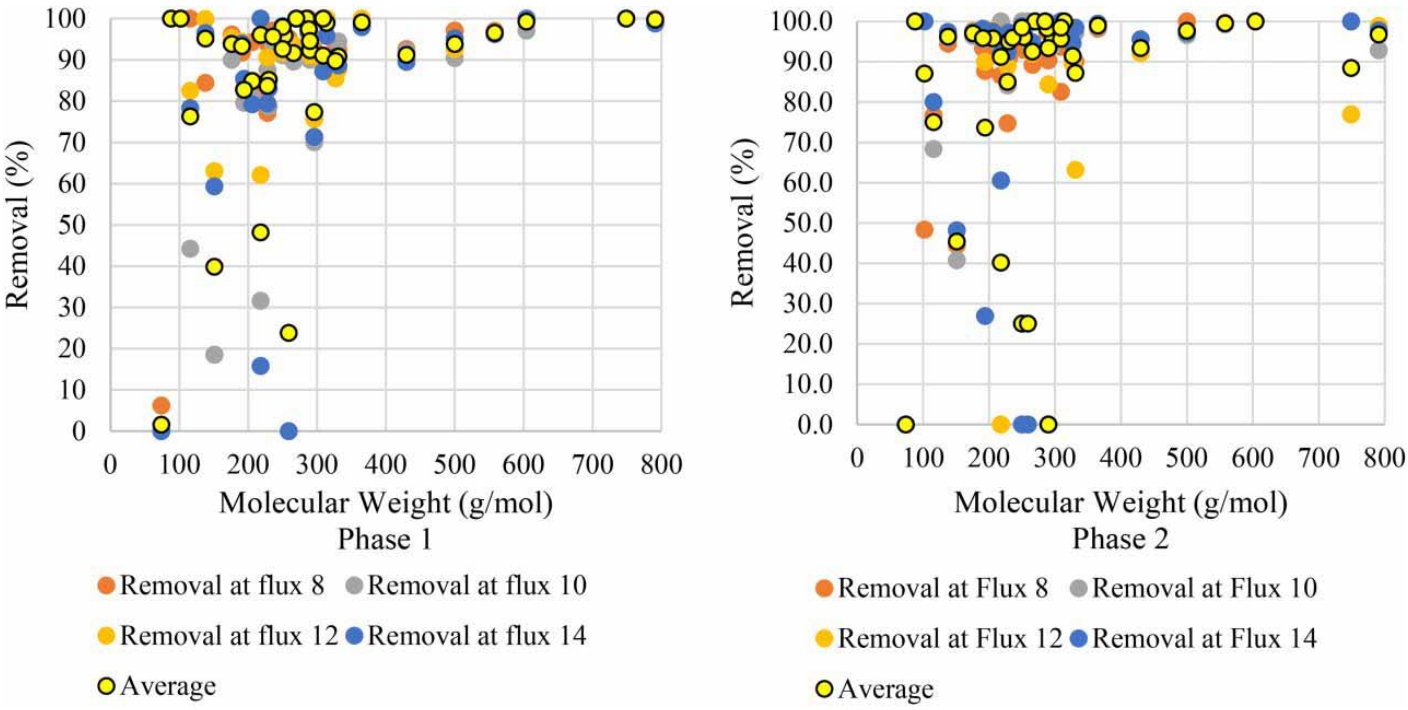

Figure 2. Effect of MW on the removal of CECs for phase one and phase two.

Table 4 and Table 5 show the concentrations of CECs in the RO feed (MF permeate), permeate, and concentrate for the first and second phases of this study, respectively. Correspondingly, Figure 3 presents the percent removal of different CECs in the RO permeate for all three of the mentioned analyses.

The data demonstrates the effectiveness of RO treatment in eliminating CECs in the RO permeate while operating at different flux rates. For the first phase, the average removal rates for the analyzed CECs with fluxes 8, 10, 12 and 14 gfd were

90.2%, 83.8%, 87.2% and 85.1%, respectively. For the second phase, the average removal rates at the same fluxes were 78%, 89.5%, 80.6% and 83%, respectively.

1-stage RO with a 55% recovery rate had an overall better removal rate of CECs when compared with 2-stage RO with an 80% recovery rate. This result aligns with those of past studies (Chellam and Taylor 2001). As mentioned earlier, when the concentration gradient of contaminants increases across the membrane at higher recovery rates, the overall removal effectiveness for various compounds decreases.

As can be seen in Figure 3, CECs with the lowest rejections were meprobamate, beta-blockers and BPs, which had 48.2%, 57.7% and 59.3% removal, respectively. CECs that were completely rejected were 1,4-dioxane and methadone. Similarly, more than 98% of hormones and gemfibrozil were rejected. Other CECs with high rejection rates were iopromide with 99.5% rejection and atorvastatin with 95.6% rejection. High rejection rates also occurred for some antibiotics such as amoxicillin, azithromycin, ciprofloxacin, sulfamethoxazole and trimethoprim. In addition, high removal efficiencies of 93% were observed for compounds such as caffeine, cotinine and DEET.

As mentioned before, NDMA was poorly removed with RO because of its low MW.

The concentration of 1,4 dioxane in the RO feed was lower than the notification level of 1 μg/L. And while 1,4 dioxane was not observed in the RO permeate, its concentration was higher than the notification level in the concentrate stream.

As expected, the compounds rejected during the RO treatment were concentrated to different degrees in the RO concentrate stream. In this study, the highest concentration in the concentrate was of acetaminophen at 130 μg/L at a flux of 8 gfd, and the lowest detectible concentration was of NDMA at 2.7 ng/L at a flux of 14 gfd.

The concentration of each compound in the concentrate stream was found for every test condition in phase one (i.e., with 55% recovery), and the results were compared against one another at the different fluxes. The highest concentrations of CECs were found at a flux of 8 gfd and 55% recovery. CECs with the highest concentrations were as follows: 1,4-dioxane, 17-b-estradiol, estrone, gemfibrozil, iopromide, acetaminophen, atenolol, atorvastatin, azithromycin, cotinine, DEET and trimethoprim. The reason for this could be the lower concentration gradient across the membrane in the low flux.

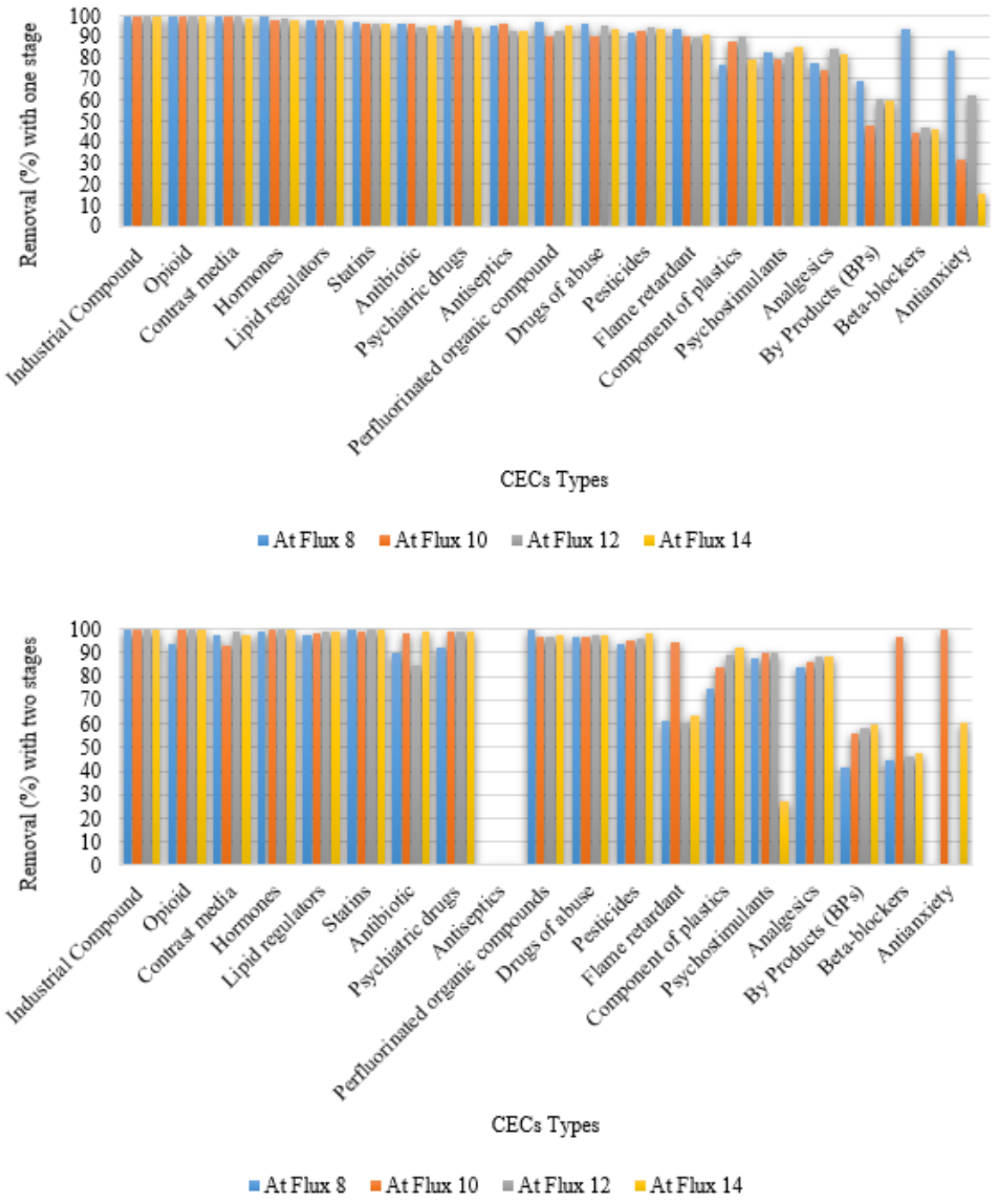

Figure 3. Removal of CECs with different fluxes for phase one and phase two.

The main drawbacks of using RO membrane processes are the costly disposal or treatment of the resulting RO concentrate and the potential environmental risks to aquatic ecosystems that receive the said concentrate (Perez-Gonzalez et al. 2012). Acceptable methods of waste disposal typically include discharge to waste treatment facilities, natural waters or an evaporation pond. Other methods to reduce the organic pollutant load of RO concentrate include advanced oxidation processes such as ozonation, fenton processes, photocatalysis and photooxidation, as well as sonolysis and electrochemical oxidation. However, the high cost of some of these technologies may limit their application (Perez-Gonzalez et al. 2012).

CECs and their associated degradates represent a challenge for regulators to establish human health-based criterion due to the limited scientific knowledge regarding acute and chronic health effects (Tchobanoglous 2015). In recognition of the lack of human health based criterion related to reuse water supply, the National Water Research Institute (NWRI) convened an independent advisory panel (IAP) to develop a list of recommended CECs, based on collective knowledge, to be considered as performance monitoring protocol for DPR systems (NWRI 2013). The IAP suggested risk-based human health criteria for the control of 13 CECs in DPR applications and the maximum concentration of those 13 CECs in the RO permeate for two phases of testing in this study is provided in Table 6.

Table 6. DWQ SWRCB and NWRI Risk-Based Human Health Criteria

CEC	Criterion[a] (ng/L)	Phase one, RO permeate				Phase two, RO permeate			
		at Flux 8	at Flux 10	at Flux 12	at Flux 14	at Flux 8	at Flux 10	at Flux 12	at Flux 14
17-β-estradiol	5	ND	1.5	ND	ND	ND	ND	ND	ND
Estrone	320	ND	ND	ND	ND	ND	ND	ND	ND
Cotinine	1,000	51	130	61	79	42	47	31	34
Primidone	10,000	6	ND	ND	ND	5.1	2.5	2.8	3
Meprobamate	200,000	3.2	13	7.2	16	120	ND	150	7.5
Atenolol	4,000	210	260	160	210	270	150	200	130
Carbamazepine	10,000	5.9	12	8.6	8.6	12	8	6.7	6.3
TCEP	5,000	15	25	23	20	670	ND	4,800	1,100
DEET	200,000	150	120	100	110	120	81	68	32
Triclosan	50,000	5.3	4.1	8	8.7	200	260	340	440
$PFOS_b$	70	7.8	26	20	13	ND	9.2	7.9	7.7

[a]NWRI (2013).
[b]DWQ SWRCB (California Department of Water Quality State Water Resources Control Board).

In the electrostatic repulsion mechanism, rejection relies on relative charge interactions and not just on molecule size. Rejection of organics, colloids, and large molecules depends on the sieving parameter, solute, and pore size. Meanwhile, ionic components and lower MW organics are rejected due to charge interactions between membrane surfaces (Hilal et al. 2004).

Accordingly, CEC rejection could be the result of both size exclusion and the charge repulsion mechanism. Specifically, negatively charged compounds studied by Verliefde et al. (2007a, 2007b) were rejected more effectively than neutral and positive compounds. Berg et al. (1997) obtained similar results: charged organics were rejected at higher rates than noncharged organics of the same MW. Kimura et al. (2003) investigated the rejection of organic CECs categorized as DBPs and pharmaceuticals using polyamide nanofiltration (NF)/RO membranes in bench-scale filtration experiments.

This study found that charged compounds could be rejected by more than 90%, regardless of other physicochemical properties. Although the charge of the CEC compounds was not analyzed in this study, CECs such as diclofenac, ibuprofen, sulfamethoxazole, and triclosan, which are negatively charged, were rejected by more than 90% in both phases. In contrast, the rejection of noncharged compounds such as acetaminophen were found to be influenced mainly by their size. To assess the percent rejection of charged/ionic CECs, frequency distributions were plotted for observed RO rejection. Figure 4 shows the frequency of observed rejection for neutral and ionic/charged CECs. With few exceptions, charged/ionic CECs were rejected (<90%) by the RO membrane.

A membrane surface gains negatively charged properties usually due to the presence of sulfonic and/or carboxylic acid groups, which are deprotonated at neutral pH (Bellona et al. 2004 and Verliefde et al. 2007a). Different pH conditions will substantially change the membrane surface charge. Studies have revealed that increasing pH can also increase the negative surface charge of membranes; thus, higher rejections, especially for negatively charged compounds, can be expected (Childress and Elimelech 2000 and Tanninen and Nyström 2002).

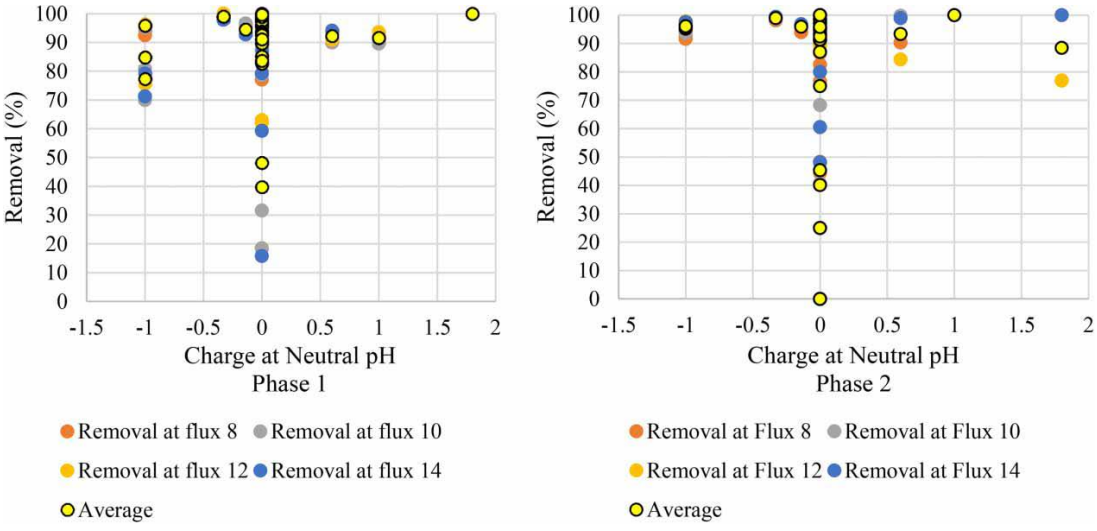

Figure 4. Effect of surface charge on the removal of CECs for phase one and phase two.

Mass Balance of CECs in the RO Process

Table 7 and Table 8 show the summary of the mass balance analysis using Equations (4) and (5). In an ideal situation with zero lab-analysis error, all M_{disc} values would be zero. With that being said, when calculating the mass of discrepancy using mass balance analysis via Equations (4) and (5), a positive M_{disc} value equates to a possibility of CECs accumulating within the system and being adsorbed to the solid phase.

Table 7. Mass Loss of CECs (M_{disc}) and Percent of Elimination Due to Sorption (R_{disc}) with 1-stage RO

Compound	Flux 8 gfd		Flux 10 gfd		Flux 12 gfd		Flux 14 gfd	
	M_{disc} (mg/d)	R_{disc} (%)	M_{disc} (mg/d)	R_{disc} (%)	M_{disc} (mg/d)	R_{disc} (%)	M_{disc} (mg/d)	R_{disc} (%)
Methadone	0.8	94.6	1.1	100.0	1.2	100.0	1.5	100.0
Primidone	0.9	83.8	1.1	78.8	1.6	100.0	1.8	100.0
Iopromide	-7.5	-20.5	45.7	100.0	53.8	99.8	62.0	98.5
Azithromycin	19.5	88.3	27.4	100.0	32.3	100.0	36.9	97.8
PFOS	3.8	50.0	-0.3	-2.9	0.1	0.7	12.7	97.4
Salicylic Acid	1380.8	90.5	1891.6	99.7	2231.8	99.7	2543.2	97.3
Ciprofloxacin	15.2	94.6	7.0	35.0	6.2	26.3	25.9	93.8
Estrone	2.3	51.0	4.4	78.2	4.6	69.5	6.5	83.8
Amoxicillin	66.8	98.6	84.3	100.0	99.5	100.0	93.3	80.3
N-Nitrosodiethylamine	0.1	71.0	0.2	100.0	-0.2	-69.8	0.2	79.6
Acetaminophen	-412.1	-54.0	241.6	25.5	778.0	69.5	796.6	60.9
17-b-Estradiol	0.5	31.7	1.3	70.4	1.9	90.4	1.5	59.0
Carbamazepine	5.0	89.0	2.7	38.6	6.7	80.8	4.6	47.7
Sulfamethoxazole	-11.5	-44.2	-9.3	-28.8	-28.8	-75.4	17.1	38.5
Caffeine	269.3	36.7	-1229.2	-134.6	425.2	39.5	476.7	37.9
Atorvastatin	-5.4	-40.6	3.3	20.1	-1.1	-5.6	8.2	36.1
Gemfibrozil	-38.7	-35.1	39.5	28.9	58.9	36.5	55.0	29.1
TCEP	4.8	61.2	-1.8	-18.2	-3.8	-32.7	2.9	21.5
Naproxen	134.0	53.3	87.7	28.0	118.5	32.1	83.4	19.4
Atenolol	10.7	15.2	13.8	15.7	39.3	38.0	23.1	19.1
Cotinine	0.3	0.7	37.8	82.8	21.3	39.5	8.1	12.8
Triclosan	-0.1	-4.1	1.3	31.1	-0.9	-18.2	0.7	12.8
Trimethoprim	1.4	8.1	3.9	18.8	4.9	19.9	3.5	12.1
Fluoxetine	0.2	93.4	-0.5	-170.1	0.0	0.8	0.0	8.5
Testosterone	0.3	9.0	-0.3	-7.6	1.2	25.6	-0.5	-9.1
Diazepam	0.0	18.4	0.1	30.2	0.0	10.0	0.0	-13.5
DEET	-29.0	-57.1	1.3	2.0	0.7	0.9	-14.7	-16.9
Progesterone	0.4	53.1	-0.5	-59.8	0.2	16.5	-0.4	-29.8
1,4-Dioxane	-3.6	-18.1	-6.8	-27.6	-11.4	-39.2	-12.3	-36.3
Bisphenol A	-0.2	-5.1	-7.3	-122.3	-2.9	-40.8	-3.0	-36.8
Ibuprofen	168.2	49.6	-156.4	-37.1	-150.1	-30.2	-430.6	-74.1
N-Nitrosomorpholine	-0.2	-63.2	-0.4	-105.6	0.2	38.6	-0.5	-78.4
NDMA	-0.1	-33.6	-0.9	-321.5	-0.2	-74.1	-0.3	-79.5
TCPP	6.0	27.9	-18.9	-70.8	-2.3	-7.2	-31.0	-84.4
Diclofenac	1.6	35.9	-3.8	-67.2	-4.9	-74.2	-6.7	-86.0
TDCPP	-14.5	-270.7	-20.3	-304.0	-43.9	-558.1	-15.5	-168.7
Meprobamate	-0.1	-17.2	0.1	15.4	0.2	22.9	-7.6	-830.4
Propranolol	0.0	97.3	-1.2	-60291.5	-0.1	-4478.8	-3.0	-105278.1

Table 8. Mass Loss of CECs (M_{disc}) and Percent of Elimination Due to the Sorption (R_{disc}) with 2-stage RO

Compound	Flux 8 gfd		Flux 10 gfd		Flux 12 gfd		Flux 14 gfd	
	M_{disc} (mg/d)	R_{disc} (%)	M_{disc} (mg/d)	R_{disc} (%)	M_{disc} (mg/d)	R_{disc} (%)	M_{disc} (mg/d)	R_{disc} (%)
17-b-Estradiol	1.2	100.0	1.6	100.0	1.9	100.0	2.2	100.0
Azithromycin	15.2	81.3	24.3	100.0	23.9	81.6	34.4	100.0
Methadone	0.7	94.9	0.9	100.0	1.1	100.0	1.3	100.0
Estrone	3.8	99.7	5.0	99.4	6.0	99.7	7.0	99.7
Amoxicillin	56.6	98.5	74.1	99.0	89.7	99.6	105.1	99.4
Progesterone	0.6	98.3	0.8	97.1	1.0	97.9	1.1	97.8
Testosterone	2.6	97.0	3.3	96.7	4.0	97.9	4.7	97.4
PFOS	6.5	100.0	8.2	97.3	9.9	97.7	11.6	97.1
Ciprofloxacin	12.2	89.4	16.2	91.4	14.0	65.7	23.7	94.3
Salicylic Acid	372.3	28.8	1559.8	92.7	1818.3	89.7	2145.0	90.1
Iopromide	28.3	90.9	34.2	84.4	46.2	94.8	51.5	89.9
Carbamazepine	3.4	71.4	5.5	88.9	6.4	85.1	7.7	87.8
Primidone	0.5	55.7	1.1	88.9	1.0	72.7	1.3	80.2
Fluoxetine	0.2	85.9	0.3	100.0	0.3	100.0	0.3	77.7
DEET	20.3	47.2	22.2	39.5	31.9	47.3	52.0	65.5
N-Nitrosodiethylamine	0.0	20.2	0.2	100.0	0.1	32.2	0.2	64.1
Diazepam	0.1	76.0	0.1	70.8	0.1	58.7	0.2	60.2
Cotinine	17.1	55.1	30.0	73.9	38.7	79.3	28.1	49.1
N-Nitrosomorpholine	0.1	43.2	-0.2	-49.2	0.3	58.1	0.3	47.3
Trimethoprim	4.4	31.1	6.5	35.1	7.9	35.9	10.2	39.1
Gemfibrozil	37.0	39.6	38.8	31.9	61.1	41.8	49.0	28.5
TCPP	11.8	64.9	10.7	45.3	-6.7	-23.5	7.5	22.4
Caffeine	517.5	83.2	651.8	80.4	814.0	83.4	187.5	16.4
Bisphenol A	-1.8	-43.1	-2.3	-42.7	-3.3	-52.5	1.2	16.2
TDCPP	0.6	13.7	0.6	9.5	-5.5	-77.6	0.3	3.7
Atenolol	22.8	38.2	36.0	46.2	26.8	28.5	0.8	0.7
Naproxen	-88.3	-41.4	-172.0	-62.0	20.3	6.1	-33.6	-8.6
Sulfamethoxazole	-1.0	-4.6	-3.6	-12.4	3.4	9.8	-8.0	-19.6
1,4-Dioxane	6.3	37.7	-5.7	-26.3	-11.1	-42.4	-8.0	-26.1
Ibuprofen	-321.9	-112.1	-263.7	-70.5	-15.4	-3.4	-184.3	-34.9
Meprobamate	-19.1	-4202.5	0.5	81.0	-26.7	-3740.1	-0.4	-42.7
Diclofenac	-1.3	-34.8	-6.5	-131.0	-4.1	-68.4	-4.4	-62.6
Atorvastatin	1.7	15.0	-43.7	-298.2	-2.3	-12.8	-17.4	-84.1
Acetaminophen	-34.9	-5.4	180.1	21.4	-473.8	-46.8	-3056.0	-256.9
N-Nitrosodi-methylamine (NDMA)	-0.4	-201.1	-0.6	-253.7	-0.8	-272.2	-0.9	-262.1
Triclosan	-4.2	-147.2	-6.6	-175.3	-10.7	-237.6	-18.9	-358.2
TCEP	-10.3	-153.5	-5.7	-65.0	-166.6	-1585.2	-92.3	-748.0
Propranolol	-0.1	-9578.8	0.0	100.0	-0.2	-8415.6	-0.1	-3175.9

Positive and negative results for M_{disc} (e.g. azithromycin and estrone for positive and NDMA and propranolol for negative) occurred for the following potential reasons. First, variations may have existed in the feed quality (i.e., CEC concentrations in feed). Because this study had limited resources to analyze CECs in the feed sample, it assumed that there were no feed chemistry variations and then analyzed one sample of primary effluent (i.e., MF feed) and one sample of MF permeate (i.e., RO feed), both of which were collected on the same day at the same time.

Another reason could be adsorption or desorption of CECs from the dissolved (i.e., aqueous) phase to the solid phase in the process. The solid phase in this study included the surface of the RO membrane, the concentrate, and permeate stream piping, and—most importantly—suspended and deposited micro-particles on the

concentrate side of the membrane. A negative value of M_{disc} in Table 7 and Table 8 represents desorption, and a positive value of M_{disc} represents adsorption.

Furthermore, positive and negative M_{disc} values could be attributed to lab measurement errors. Table 7 and Table 8 note varying laboratory procures such as "The concentration indicated for this analyte is an estimated value above the calibration range". Therefore, some level of error may have been introduced to the lab results. Measuring chemicals in the level of nanograms per liter can be a sensitive process that always comes with some uncertainties about quality control (i.e., result replicates).

Understanding the removal mechanism and relationships between controlling parameters in the RO system is key to optimizing CEC rejection. At the early stage of filtration in RO when the membrane is not ripe, the dominant mechanism for removal is the adsorption of nano-amounts of CECs into the membrane surface (Nghiem et al. 2004a and 2004b), until it reaches equilibrium. Preliminary removal could yield false results (Nghiem and Schäfer 2002). A cake develops on the surface of the membrane that decreases its pore size to below the nominal rating, thus improving removal (Nghiem et al. 2004a, 2004b, and Xu et al. 2014), but later develops fouling. In addition to the pore size decreasing, this improvement in removal could also be due to the enhanced adoption capacity of the solid phase (e.g., fouling biofilm).

The adsorption mechanism correlates with solute-solid hydrophobic interactions (Nghiem and Schäfer 2002 and Nghiem et al. 2002). Hydrophobic interaction between the solid phase, particularly the RO membrane, and solutes is one of RO's important rejection mechanisms. A membrane's hydrophobicity is typically characterized by its contact angle, whereas hydrophobicity of solutes can be correlated and quantified using the logarithm of the octanol-water partition ($\log K_{ow}$). Molecules with $\log K_{ow}$ greater than 2 are referred to as hydrophobic (Ying et al. 2004). Octanol and water partition coefficient values are determined as logs, the ratio of the concentration in the octanol phase against the concentration in the aqueous phase at adjusted pH, such that the predominant form of the compound is unionized. Figure 5 shows the effect of Log K_{ow} on the removal examined CECs for phases of the test.

Hydrophobic properties have an influence on the sorption mechanisms. For instance, strong hydrophobic compounds such as aromatic pesticides, non-phenylic pesticides, and alkyl-phthalates were highly rejected even by the lowest desalting membrane (Kiso et al. 2001). However, the retention decreases as the membrane is saturated and its ability for sorption is reduced. As studied by Braeken et al. (2005), hydrophobic molecules are rejected better than hydrophilic molecules after long-term operation.

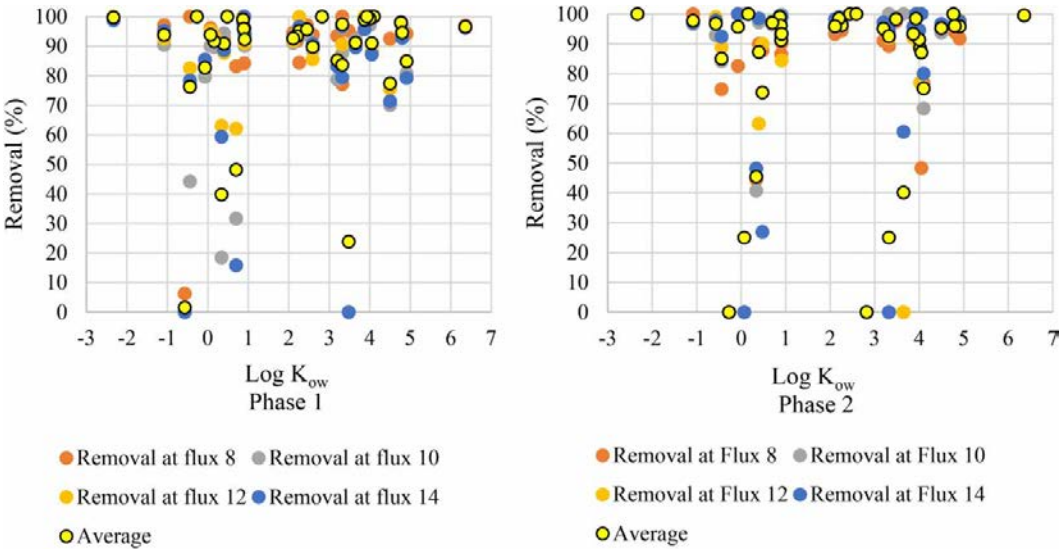

Figure 5. Effect of Log K_{ow} on the removal of CECs for phase one and phase two.

In this study, hormones which have values of log K_{ow} >2 (e.g., estrone and 17-β-estradiol, azithromycin, and methadone), adsorbed to the solid phase and potentially followed this pattern. See the mass balance calculation and the results in Table 7 and Table 8.

Conclusion

The potential effect of contaminants of emerging concern (CEC) on the public health and the environment has urged water managers to more actively investigate strategies that remove, neutralize and/or destroy these compounds not only from drinking water but also as part of a wastewater treatment process effluent. Primary treatment is currently unable to eliminate all substances; therefore, it is usually followed by secondary treatment.

However, the innovative MF/RO treatment train generates a water source without secondary treatment and can still remove many CECs. By analyzing the RO concentrate stream, this study showed the viability of eliminating secondary treatment and efficiently preparing wastewater for reuse through this novel treatment train.

This study investigated the removal of 38 different CECs at the pilot scale with different fluxes. In the first phase (1-stage RO), the flux of 14 gfd showed a better rejection value (95.2%) than rejection values from other fluxes. In the second phase (2-stage RO), the flux of 12 gfd showed a better rejection value (93.7%) than rejection values from other fluxes. Statistical analysis revealed that there is no significant difference between different fluxes.

The results showed that 1-stage RO with a 55% recovery rate had a better removal rate of CECs than 2-stage RO with an 80% recovery rate. As the concentration gradient of contaminants increased across the membrane at the higher recovery rate, the overall removal rate decreased for various compounds.

Azithromycin, hormones, carbamazepine, diazepam, gemfibrozil, atorvastatin, methadone, and iopromide were removed by RO in both phases. All these compounds have MW >200 g/mol and are also based on the log K_{ow}. All these CECs also have hydrophobic characteristics; therefore, the RO process was able to remove them efficiently. In contrast, NDMA, propranolol, acetaminophen, and meprobamate were the least effectively removed, given their low MW (less than 200 g/mol).

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