

Contaminants of emerging concern in reverse osmosis brine concentrate from indirect/direct water reuse applications

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ABSTRACT

Water shortage is becoming more common due to droughts and global population increases resulting in the increasing popularity of water reuse to create new water sources. Reverse osmosis (RO) membrane systems are popular in these applications since they can produce drinking water quality effluent. Unfortunately, RO systems have the drawback of generating concentrate streams that contain contaminants rejected by the membrane including chemicals of emerging concern (CECs). CECs are chemicals such as hormones, steroids, pesticides, pharmaceuticals, and personal care products that are used for their intended purpose and then released into wastewater. CECs are believed to be detrimental to aquatic wildlife health and pose an unknown human health risk. This research gathered the existing knowledge on CEC presence in concentrate, available proven concentrate treatment methods, their CEC removal abilities, and current CEC regulations. It was found that 127 CECs have been measured in RO concentrate with 100 being detected at least once. The most potent treatment process available is UV/H₂O₂ as it offers the highest removal rates for the widest range of chemicals. The less expensive process of ozone/biologically activated carbon offers slightly lower removal abilities. This comprehensive report will provide the groundwork for better understanding, regulating and treating concentrate stream CECs.

Key words | brine treatment, contaminants of emerging concern, reverse osmosis, reverse osmosis brine, reverse osmosis concentrate, water reuse

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INTRODUCTION

Water reuse is becoming increasingly popular as an answer to water shortage throughout the world. Limited water supplies are over-exerted and further polluted by the growing population reliant upon them.

Reverse osmosis (RO) has become a popular treatment process in water reuse as it produces very high quality effluent. The membranes used in RO are capable of removing contaminants that pass through other treatment processes making it likely that their use will continue to increase. Unfortunately, the RO process produces a brine concentrate stream that contains elevated concentrations of the contaminants removed by the membrane.

A constituent of this concentrate is chemicals of emerging concern (CECs). These are chemicals such as hormones, steroids, pesticides, pharmaceuticals, and personal care

products that are present in wastewater. They are used for their intended purpose and then released into wastewater. These compounds have been found to have a detrimental health impact on aquatic life and pose an unknown, although likely minimal, health risk to humans. (Snyder *et al.* 2008).

Concentrate from RO systems in coastal areas is often discharged into the ocean without treatment. The CECs that are present in concentrate are at elevated concentrations making its discharge most dangerous to organisms directly exposed to the flow. There is also concern about these CECs existing in the receiving waters further away from plant outfalls.

The purpose of this research was to gather the existing knowledge about which CECs are present in brine, the available brine treatment methods, their CEC removal abilities, and the current CEC regulations. The research began by

compiling a list of the known CECs in RO brine. This database includes the minimum, maximum and average CEC concentrations given in a comprehensive list. The data presented were gathered from previous research as well as questionnaires sent to various treatment plants with RO systems used for water reuse applications. A study was then conducted to determine the treatment methods which are most effective at reducing the risk posed by the chemicals in this database. This includes the strengths and weaknesses of each system. Research concluded by determining the regulations in place for CECs around the world.

The information presented in this article is the current knowledge on the topic of CECs in RO concentrate. It will aid in understanding, regulating and treating the chemicals to help protect aquatic life and public health.

BACKGROUND

Knowledge on brine CEC removal is contained in many individual reports with no centralized database of knowledge. A literature review was conducted to gather this knowledge. The results are presented in four main sections: (1) the known health issues posed by CECs; (2) the regulations that have been imposed for CECs present in treatment plant effluent; (3) the CECs that have been found in RO brine; and (4) the treatment methods being researched for removing CECs.

ISSUES POSED BY EFFLUENT CECs

The presence of CECs was first discovered in surface water sources and wastewater in the United States and Europe in the 1960s. Concerns about potential environmental risks came about in 1999 when the presence of pharmaceuticals in a river was connected to the feminization of fish living downstream from a wastewater treatment plant. Around the same time, the non-steroidal anti-inflammatory drug diclofenac was attributed to the renal failure of vultures. This poisoning has been blamed for a decline of more than 95% of the bird's population in India since the 1990s. Public awareness became even more heightened when a study found that organic wastewater contaminants, including CECs, were present in at least 111 of 139 streams tested. While it has been found that CEC levels in the environment are usually only trace concentrations due to dilution effects, their chemical persistence, microbial resistance and synergistic effects are still unknown. There has

also been evidence that even low concentrations can have adverse effects on aquatic life (Deegan *et al.* 2011).

Pharmaceutical use has become widespread as drug manufacturers continually develop new medicines to treat an increasing number of health issues. A large proportion of society is aging and relying more heavily on these new drugs. This is becoming an issue since the human body only absorbs a proportion of the medicine and then releases the unused proportion in waste that ends up in wastewater treatment plants. It is believed that 30–90% of medicine taken by the population enters unchanged into treatment plants (Watkinson *et al.* 2007). This flow is a problem as conventional treatment plants are not designed to remove these contaminants. It has been reported that a wastewater treatment plant in Rubí, Spain that serves 125,550 people (which produces an average daily flow rate of 22,000 m³ per day) discharges approximately 300 g (0.66 lbs) of pharmaceuticals every day (Radjenovic *et al.* 2006).

Testing has shown that typical removal rates of CECs in wastewater treatment plants are only 60–90% (Carballa *et al.* 2004). This incomplete elimination adds further reason for concern on the impact of plant effluent on the environment. The impact of effluent CECs was proven by a 2003 study that detected changes in the structure and function of algal communities that were in receiving streams (Watkinson *et al.* 2007).

Wastewater treatment plants in many coastal communities discharge their effluent into the ocean exposing sea life to CECs. A study conducted in 2006 found that marine flatfish living near treatment plant outfalls were being affected by endocrine disruptors in the flow. Male fish displayed elevated levels of plasma vitellogenin and estradiol. Other studies have also shown that fish exposed to ethinylestradiol levels measured in the environment exhibited altered reproduction abilities due to the estrogenic effects of the chemical (Vidal-Dorsch *et al.* 2012).

Tests on CEC levels at treatment plant outfalls show an environmental impact risk. Concentration levels between 0.0004 and 0.0009 µg/L of a wide range of contaminants have been detected. Several recalcitrant CECs including gemfibrozil, oxybenzone and sulfamethoxazole have even been detected well away from the outfalls (Vidal-Dorsch *et al.* 2012).

CECs have also been detected in drinking water sources in the United States. DEET has been found to occur most frequently and is not readily removed by standard drinking water treatment processes. Carbamazepine and dilantin are also frequently detected in raw water and have shown some persistence in treatment processes. Atrazine has

repeatedly been found to have the highest concentration in raw waters with concentrations reaching 571 ng/L (Snyder *et al.* 2008).

No cases of human health impacts attributed to CECs in treatment plant effluent were discovered in the course of this study. The minimal exposure of humans to effluent CECs limits the potential danger they pose. Despite this, groundwater contamination from chromium VI in Hinkley, California in 1993 and water contamination warnings in several cities on the US East Coast in early 2014 show the concern that exists on this topic.

Regulations on CECs

No regulations specifically on RO concentrate CECs were found during the course of this study. Regulations on wastewater treatment plant CECs and drinking water CECs have begun to be established.

The United States does not have nationwide regulations on CECs in wastewater treatment plant effluent. Permits issued by the National Pollution Discharge Elimination System (NPDES) focuses on basic contaminants and not trace organic compounds (Audenaert *et al.* 2014).

The Environmental Protection Agency (EPA) has currently established primary drinking water regulations for approximately 90 contaminants ranging from coliform and viruses to heavy metals and disinfectants. Many of these have been found to be discharged from treatment plants. Even though this list is fairly large, there are still a significant number of contaminants for which regulations have not been implemented. These can be found in the EPA's Contaminant Candidate List. This list is currently on version 3 and contains 104 chemicals and 12 microbiological contaminants. The EPA uses this list to further its research on the listed constituents to determine whether they need to be regulated. Even with its large database and continued research, the EPA itself does not have the jurisdiction to impose regulations on the chemicals it deems hazardous. States are left with this role in determining which chemicals need to be regulated (EPA 2012).

California is the one state setting the standard in CEC regulation by using the EPA's 2012 *Guidelines for Water Reuse* to institute regulatory policies. Beginning in 2009, California's State Water Resource Control Board (SWRCB) implemented a new recycled water policy which allowed for extensive research on the risks posed by CECs and recommendations of how CECs should be monitored. The findings were published in the report *Monitoring Strategies for Chemicals of Emerging Concern (CECs) in*

Recycled Water – Recommendations of a Scientific Advisory Panel. Standards were established in this report on which chemicals should be monitored and what acceptable concentrations of each should be. The influence of this publication can be seen in California as every California Regional Water Quality Control Board report on treatment plants consists of a requirement for a special study on plant effluent CECs including a plan for continual monitoring of CEC concentrations along with their frequency (Anderson *et al.* 2010).

International regulation of CECs varies with each country's environmental agencies. The European Union, Germany and Switzerland have established lists of chemicals known to cause environmental harm while pushing for their discontinued use. Switzerland has also proposed an indicator list consisting of carbamazepine, diclofenac, sulfamethoxazole, benzotriazole and mecoprop (Kazner 2011).

The regulations in Europe are leading to the installation of ozonation systems to protect the environment. A push is also underway to gather monitoring data in order to set a regulatory framework. Watch lists (similar to those by the EPA but focusing on plant discharge) are under development to aid in environmental protection. Switzerland is the leader in this work as they are the first country to set some nationwide regulations and indicator compounds (Audenaert *et al.* 2014).

Many other nations follow the World Health Organization's (WHO) guidelines on the maximum allowable concentrations of chemicals in drinking water. These recommendations are found in the chemical fact sheet in the WHO's 'Guidelines for Drinking-Water Quality' report. The fact sheet also provides the acceptable daily intake, recommended treatment processes, occurrence, and limit of detection concentrations.

CECs present in concentrate

There is limited knowledge on the CECs that are present in RO concentrate. Running tests to measure CEC concentrations is an expensive and time-consuming task meaning that even advanced research studies are limited in the number of CECs they can test for at one time. Cross-referencing multiple studies has helped build a more comprehensive list but more experiments need to be conducted to expand this knowledge.

Fewer CECs have been found in RO concentrate than in plant influent. Treatment systems commonly used before RO systems have been shown to remove CECs with varying degrees of success. It has been found that personal care

products such as tonics and musks are well-removed in primary treatment with most of the remainder removed during biological treatment (Carballa *et al.* 2004). Several pharmaceuticals including ibuprofen, naproxen and sulfamethoxazole are largely removed during biological treatment (Carballa *et al.* 2004). Some plants also then employ filtration systems that offer some additional removal of certain CECs. Even with these multiple barriers, however, removal rates remain at unsatisfactory levels. The chemicals that remain after these treatment methods are then subjected to the RO membrane.

RO membranes are known to offer high removal rates of CECs while yielding very high quality water. Unfortunately, the process concentrates the removed contaminants into a brine concentrate stream. This concentrate flow has increased toxicity levels that require additional treatment to alleviate. Even the common practice of dilution through mixing the concentrate flow with other plant effluent provides no net improvements in environmental safety (Snyder *et al.* 2007).

A compilation of the CECs and their concentrations measured in RO concentrate by studies around the world was assembled into a single table as part of this study. It was found that a total of 127 CECs have been measured in RO concentrate with 100 CECs being present at detectable levels. Several of these CECs were detected in over ten separate measurements. These include atenolol, caffeine, carbamazepine, diclofenac, gemfibrozil, ibuprofen, naproxen, sulfamethoxazole, triclosan, trimethoprim, TCEP, and DEET. All are commonly used in society so it is not surprising to find them in the concentrate. A summary of these results is shown in Tables 1 and 2.

A majority of the CECs found were pharmaceuticals. The rest belonged to other common chemical categories. The CEC category distribution is depicted in Figure 1.

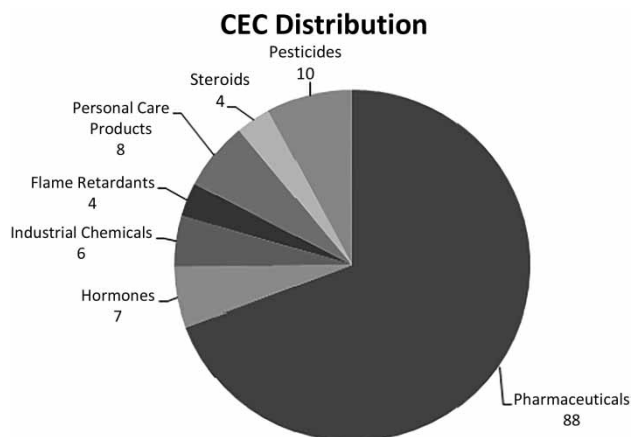


Figure 1 | CEC category distribution.

CONCENTRATE STREAM TREATMENT METHODS

A wide range of treatment methods have been studied for removing concentrate stream CECs. The unique physical and chemical properties of the CECs were found to have a large impact on their removal by each treatment method. The findings of this report are summarized in the following sections.

Discharge reduction

The single most effective method to prevent CECs in the environment is reducing their presence in wastewater. Proper prescriptions by doctors and reduced source discharges by pharmaceutical companies are key steps being investigated. Phasing out the most dangerous medicines is also a move being researched (Ternes & Joss 2006).

Coagulation/flocculation

Coagulation/flocculation is a simple method of injecting chemicals that gather together charged particles into floc that can be settled or filtered out. Alum and ferric chloride (FeCl_3) have been tested as viable coagulant chemicals. The process is popular as alum has been found to offer a 42% removal rate of dissolved organic carbon (DOC) while ferric chloride has DOC removal rates of 52% (Dialynas *et al.* 2008).

The effectiveness of CEC treatment has been found to be much lower. A jar test conducted on the removal of diclofenac, clofibrac acid, bezafibrate, carbamazepine and primidone using iron(III) chloride found no impact on the pharmaceutical levels was made by the flocculant. These results were also found when the experiment was conducted on a full-scale system (Ternes *et al.* 2002).

Despite these poor results, further research is needed to determine the effectiveness of different coagulation/flocculation chemicals at removing CECs since their low cost and ease of operation are encouraging their widespread implementation to treat other contaminants.

Activated carbon adsorption

Activated carbon (AC) is a process that has long been used in water treatment. It involves the adsorption of contaminants into the pores on activated carbon pieces. It is often used in powder (PAC) or granular (GAC) forms. AC systems can be arranged in different configurations that include

Table 1 | Concentrations of pharmaceutical CECs measured in RO concentrate

Chemicals	Chemical uses	Number of tests	Number of detects	Number of unmeasurable detects	Number of non-detects	Minimum concentration (µg/L)	Maximum concentration (µg/L)	Average concentration (µg/L)	Standard deviation
Pharmaceuticals									
4-AAA	Analgesic metabolite	2	2	0	0	11.847	15.569	13.708	2.632
5,5-diphenylhydantoin	Anticonvulsant	1	1	0	0	0.145	0.145	0.145	–
Acebutolol	Beta blocker	1	1	0	0	0.760	0.760	0.760	–
Acetaminophen	Analgesic	8	7	0	1	ND	0.150	0.041	0.050
Amoxicillin	Antibiotic	4	1	0	3	ND	21.887	21.887	–
Atenolol	Beta blocker	13	13	0	0	0.465	49.739	9.051	16.743
Atorvastatin	Statin	1	0	0	1	ND	ND	ND	–
Azithromycin	Antibiotic	6	6	0	0	1.400	343.244	115.432	139.508
Bacitracin	Antibacterial	3	0	0	3	ND	ND	ND	–
Bezafibrate	Fibrate	2	2	0	0	0.500	0.583	0.542	0.059
Bisoprolol	Beta blocker	1	1	0	0	0.940	0.940	0.940	–
Butalbital	Barbiturate	1	1	0	0	0.298	0.298	0.298	–
Caffeine	Stimulant	14	14	0	0	0.025	50.000	6.949	15.275
Carbamazepine	Anti-epileptic	26	26	0	0	0.112	7.266	1.666	1.807
Carisoprodol	Muscle relaxant	1	1	0	0	1.905	1.905	1.905	–
Cefaclor	Antibiotic	3	0	0	3	ND	ND	ND	–
Celiprolol	Beta blocker	1	1	0	0	1.800	1.800	1.800	–
Cephalexin	Antibiotic	3	0	0	3	ND	ND	ND	–
Chlortetracycline	Antibiotic	3	0	0	3	ND	ND	ND	–
Cimetidine	Anti-histamine	2	1	0	1	ND	6.438	6.438	–
Ciprofloxacin	Antibiotic	2	1	0	1	ND	0.430	0.430	–
Clarithromycin	Antibiotic	4	4	0	0	0.800	36.800	23.691	16.725
Clindamycin	Antibiotic	2	0	0	2	ND	ND	ND	–
Clopidogrel	Blood thinner	3	3	0	0	4.509	5.852	5.201	0.672
Codeine	Opiate	4	4	0	0	0.673	2.031	1.362	0.605
Dehydronifedipine	Human plasma metabolite	1	1	0	0	2.105	2.105	2.105	–
Diazepam	Anti-epileptic	4	4	0	0	0.007	0.774	0.533	0.356
Diclofenac	Anti-inflammatory	12	12	0	0	0.001	1.500	0.436	0.441
Dilantin	Anti-epileptic	9	9	0	0	0.270	1.753	0.779	0.484
Doxycycline	Antibiotic	3	0	0	3	ND	ND	ND	–

(continued)

Table 1 | continued

Chemicals	Chemical uses	Number of tests	Number of detects	Number of unmeasurable detects	Number of non-detects	Minimum concentration (µg/L)	Maximum concentration (µg/L)	Average concentration (µg/L)	Standard deviation
Enrofloxacin	Antibiotic	2	0	0	2	ND	ND	ND	–
Erythromycin	Antibiotic	14	11	3	0	D	7.984	1.332	2.255
Erythromycin-H ₂ O	Antibiotic	3	0	3	0	D	D	D	–
Famotidine	Anti-histamine	4	4	0	0	0.000	3.958	2.500	1.881
Fenofibric acid	Receptor agonist	2	2	0	0	0.800	1.480	1.140	0.481
Fluoxetine	Antidepressant	8	8	0	0	0.017	1.143	0.202	0.382
Furosemide	Diuretic	2	1	0	1	ND	2.522	2.522	–
Gemfibrozil	Cholesterol reducer	21	21	0	0	0.000	11.997	2.436	3.412
Glibenclamide	Antidiabetic	1	1	0	0	0.016	0.016	0.016	–
Hydrochlorothiazide	Diuretic	3	3	0	0	0.214	27.000	15.321	13.718
Hydrocodone	Analgesic	7	7	0	0	0.015	0.541	0.270	0.174
Ibuprofen	Anti-inflammatory	13	13	0	0	0.033	21.250	2.953	6.146
Indometacin	Anti-inflammatory	1	1	0	0	0.895	0.895	0.895	–
Iohexol	X-ray contrast media	4	4	0	0	2.400	71.300	31.236	29.021
Iomeprol	X-ray contrast media	2	2	0	0	0.386	3.900	2.143	2.485
Iopamidol	X-ray contrast media	1	1	0	0	2.626	2.626	2.626	–
Iopromide	X-ray contrast media	11	10	0	1	ND	7.000	1.365	2.480
Ketoprofen	Anti-inflammatory	1	1	0	0	0.429	0.429	0.429	–
Lidocaine	Anesthetic	1	1	0	0	0.418	0.418	0.418	–
Lincomycin	Antibiotic	3	1	0	2	ND	0.057	0.057	–
Lorazepam	Anti-epileptic	3	3	0	0	2.880	6.473	5.176	1.994
Lovastatin	Statin	1	0	0	1	ND	ND	ND	–
Mefenamic acid	Anti-inflammatory	1	1	0	0	0.001	0.001	0.001	–
Meprobamate	Tranquilizer	10	10	0	0	0.780	7.972	2.457	2.283
Metoprolol	Beta blocker	7	7	0	0	0.033	8.553	2.056	2.935
Metronidazole	Antibacterial	2	2	0	0	7.447	8.247	7.847	0.566
Monensin	Antibiotic	1	0	0	1	ND	ND	ND	–
Nadolol	Beta blocker	3	3	0	0	0.000	1.014	0.667	0.578
Nalidixic acid	Antibiotic	2	2	0	0	0.085	0.189	0.137	0.074
Naproxen	Anti-inflammatory	18	18	0	0	0.000	9.223	1.374	2.223
Nicotine	Receptor antagonist	2	2	0	0	0.912	5.683	3.298	3.374

(continued)

Table 1 | continued

Chemicals	Chemical uses	Number of tests	Number of detects	Number of unmeasurable detects	Number of non-detects	Minimum concentration (µg/L)	Maximum concentration (µg/L)	Average concentration (µg/L)	Standard deviation
Nifedipine	Calcium channel blocker	1	1	0	0	0.273	0.273	0.273	–
Norfloxacin	Antibiotic	1	1	0	0	0.120	0.120	0.120	–
Ofloxacin	Antibacterial	6	5	0	1	ND	25.309	9.526	9.793
Oleandomycin	Antibiotic	1	1	0	0	0.010	0.010	0.010	–
Oxytetracycline	Antibiotic	3	0	0	3	ND	ND	ND	–
Paroxetine	Antidepressant	1	1	0	0	0.508	0.508	0.508	–
Penicillin G	Antibiotic	3	0	0	3	ND	ND	ND	–
Penicillin V	Antibiotic	3	0	0	3	ND	ND	ND	–
Pentoxifylline	Blood thinner	1	1	0	0	0.000	0.000	0.000	–
Primidone	Anti-epileptic	5	5	0	0	0.320	0.905	0.551	0.236
Propranolol	Beta blocker	4	4	0	0	1.050	7.200	4.246	2.602
Propyphenazone	Anti-inflammatory	2	2	0	0	0.156	0.258	0.207	0.072
Ranitidine	Anti-histamine	4	3	0	1	ND	8.240	5.825	2.216
Roxithromycin	Antibiotic	1	1	0	0	0.150	0.150	0.150	–
Salbutamol	Bronchodilator	4	4	0	0	0.198	0.889	0.588	0.310
Salinomycin	Antibacterial	3	0	0	3	ND	ND	ND	–
Sotalol	Beta blocker	4	4	0	0	0.022	6.196	3.409	2.818
Sulfadiazine	Antibiotic	1	1	0	0	0.472	0.472	0.472	–
Sulfamerazine	Antibiotic	1	0	0	1	ND	ND	ND	–
Sulfamethazine	Antibacterial	2	1	0	1	ND	0.635	0.635	–
Sulfamethizole	Antibiotic	1	0	0	1	ND	ND	ND	–
Sulfamethoxazole	Antibiotic	24	23	0	1	ND	8.638	3.085	2.640
Sulfasalazine	Antibiotic	2	1	0	1	ND	0.045	0.045	–
Sulfathiazole	Antibiotic	3	0	0	3	ND	ND	ND	–
Tetracycline	Antibiotic	3	0	0	3	ND	ND	ND	–
Theobromine	Stimulant	1	1	0	0	1.010	1.010	1.010	–
Theophylline	Bronchodilator	1	1	0	0	0.753	0.753	0.753	–
Timolol	Beta blocker	1	1	0	0	0.018	0.018	0.018	–
Triclosan	Antibacterial	14	14	0	0	0.008	3.371	0.923	1.249
Trimethoprim	Antibacterial	14	14	0	0	0.000	1.172	0.512	0.394
Tylosin	Antibiotic	1	1	0	0	0.005	0.005	0.005	–
Venlafaxine	Antidepressant	1	1	0	0	0.333	0.333	0.333	–

Table 2 | Concentrations of non-pharmaceutical CECs measured for in RO concentrate

Chemicals	Chemical uses	Number of tests	Number of detects	Number of unmeasurable detects	Number of non-detects	Minimum concentration (µg/L)	Maximum concentration (µg/L)	Average concentration (µg/L)	Standard deviation
Hormones									
Diethylstilbestrol	Estrogen	2	0	0	2	ND	ND	ND	–
Equilin	Estrogen	2	0	0	2	ND	ND	ND	–
Estradiol	Estrogen	11	10	0	1	ND	0.028	0.014	0.010
Estriol	Estrogen	3	1	0	2	ND	0.000	0.000	–
Estrone	Estrogen	7	7	0	0	0.021	0.612	0.139	0.210
Ethinylestradiol	Estrogen	5	3	0	2	ND	0.017	0.006	0.010
Testosterone	Androgen hormone	3	1	0	2	ND	0.000	0.000	–
Industrial chemicals									
4-n-octylphenol	Corrosion inhibitor	2	0	0	2	ND	ND	ND	–
4-tert-octylphenol	Corrosion inhibitor	3	2	0	1	ND	1.183	0.887	0.420
Bisphenol A	Plasticizer	7	5	0	2	ND	1.343	0.532	0.471
Nonylphenol	Plasticizer	3	1	0	2	ND	9.433	9.433	–
Para-chlorobenzene sulfonic acid	Chemical producer	2	0	0	2	ND	ND	ND	–
Tris-2-chloroethyl phosphate	Plasticizer	2	2	0	0	1.900	3.100	2.500	0.849
Flame retardants									
TCEP	Hydrochloride salt	12	12	0	0	0.426	5.810	1.416	1.489
T CPP	Flame retardant	1	1	0	0	3.033	3.033	3.033	–
TDCPP	Flame retardant	1	1	0	0	1.233	1.233	1.233	–
Tetrabromobisphenol A	Brominated flame retardant	2	0	0	2	ND	ND	ND	–
Personal care products									
Acesulfame potassium	Artificial sweetener	1	1	0	0	29.220	29.220	29.220	–
Aspartame	Artificial sweetener	2	0	0	2	ND	ND	ND	–
Cotinine	Tobacco alkaloid	1	1	0	0	0.423	0.423	0.423	–
Galaxolide	Synthetic musk	1	1	0	0	2.180	2.180	2.180	–
Musk ketone	Nitro-musk	1	1	0	0	0.329	0.329	0.329	–
Neotame	Artificial sweetener	2	1	0	1	ND	0.010	0.010	–
Oxybenzone	Sunscreen	4	4	0	0	0.017	0.061	0.029	0.021
Sucralose	Artificial sweetener	3	3	0	0	124.000	459.433	274.478	170.354

(continued)

Table 2 | continued

Chemicals	Chemical uses	Number of tests	Number of detects	Number of unmeasurable detects	Number of non-detects	Minimum concentration (µg/L)	Maximum concentration (µg/L)	Average concentration (µg/L)	Standard deviation
Steroids									
4-androstene-3	Testosterone enhancer	2	2	0	0	0.010	0.026	0.018	0.011
Androstenedione	Adrenal steroid	3	3	0	0	0.000	0.017	0.008	0.009
Epitestosterone	Natural steroid	2	0	0	2	ND	ND	ND	-
Progesterone	Endogenous steroid	5	4	0	1	ND	0.017	0.005	0.008
Pesticides									
Atrazine	Herbicide	3	3	0	0	0.000	0.017	0.011	0.009
clofibric acid	Herbicide	1	1	0	0	1.038	1.038	1.038	-
DEET	Insect repellent	15	15	0	0	0.061	3.000	0.730	0.809
Diaminochlorotriazine	Herbicide	1	1	0	0	0.035	0.035	0.035	-
Diuron	Herbicide	6	6	0	0	0.072	0.545	0.281	0.161
Linuron	Herbicide	2	2	0	0	0.027	0.063	0.045	0.026
Pentachlorophenol	Pesticide	2	0	0	2	ND	ND	ND	-
Phenylphenol	Preservative	2	0	0	2	ND	ND	ND	-
Quinoline	Plant alkaloid	1	1	0	0	0.385	0.385	0.385	-
Simazine	Herbicide	3	3	0	0	0.036	0.085	0.058	0.025

upflow-expanded bed, upflow-compacted bed, downflow-single stage and downflow-multistage. Efficient carbon utilization, costs, and operation procedures are some of the differences between these AC configuration systems (Rose *n.d.*).

Studies have found that GAC is capable of removing greater than 90% of most CECs, effluent organic matter (EfOM) and DOC (Snyder *et al.* 2007; Dialynas *et al.* 2008). Carbamazepine has shown high removal rates with GAC treatment while bezafibrate and diclofenac have shown slightly lower removal rates (Ternes *et al.* 2002). The high removal rates of most CECs are likely due to the high adsorption abilities of GAC. It has also been found to remove a wide range of the different fractions that compose DOC meaning it can be used to remove a broad range of contaminants (Dialynas *et al.* 2008). A mixture of sand and iron-coated GAC has also been found to be highly effective while allowing the reuse of previously waste iron in a beneficial manner (Joo 2014).

There are several issues with AC. Clofibrac acid has shown resistance to AC treatment (Ternes *et al.* 2002). Contact time is lengthy to achieve high removal rates meaning GAC beds must be large and PAC doses must be high. The carbon must be disposed of or regenerated once it has been spent. Disposal requires hazardous waste-like handling and thermal regeneration requires large amounts of energy. Removal of CECs is also heavily impacted by the levels of natural organic matter present in the wastewater. These issues make implementation of AC difficult (Snyder *et al.* 2007).

Despite these issues, it appears that GAC may have a place in RO concentrate treatment. The decreased flow rates may limit the amount of GAC required while still offering high CEC removal rates. Some studies recommend only seasonal use of GAC in order to remove CECs during periods when their levels are known to be high (Snyder *et al.* 2007).

Biologically activated carbon

A variation of activated carbon is biologically activated carbon (BAC). This involves the creation of a microorganism biofilter within a bed of granular activated carbon. The water that passes through this filter is subjected to a generally aerobic environment of activated bacteria that provide high removals of most CECs (Sundaram & Emerick 2010a, b).

BAC is frequently implemented as a polishing step after ozonation. The constituent molecules and increased bioactivity that are created during the ozone process promote the growth

of microbial communities. The microbial makeup of the biofilter has been found to develop and change for months after coming online due to variations in the system influent (Sundaram & Emerick 2010a, b).

BAC has the advantages of partial regeneration of the activated carbon by the microbial community, degradation of less biodegradable organics that are absorbed by the carbon and then exposed to the microbes, and increased levels of biological reaction rates due to a readily available food source adsorbed by the carbon. These factors are important as CECs are often less biodegradable organics (Ng *et al.* 2008).

Studies have also been conducted on pairing BAC with capacitive deionization (CDI). This links the high organic removal capabilities of BAC with the inorganic removal abilities of CDI. High removal rates for TOC have been found with this layout (Ng *et al.* 2008).

BAC systems have repeatedly shown consistent removal performance. The stability of the matured microorganism community allows for high removal rates of a wide range of organics, ozone byproducts, estrogenicity, CECs, and other associated toxicity. BAC treatment has also shown high removal levels of some problematic contaminants such as flame retardants, TCP, TCEP and NDMA but does not display the same effectiveness against 1,4-dioxane and benzophenone. This has been attributed to the chemical makeup of these molecules (Sundaram & Emerick 2010a, b).

Concern has existed about the ability of BAC to handle the high salinity of RO concentrate. Fortunately, studies have lessened this concern by proving that sudden changes in organic load and salinity do not have drastic negative effects on BAC performance (Lu *et al.* 2013). The microorganism community will simply adjust to the different water quality parameters. Further research is needed, however, to verify these findings.

Full-scale implementation of BAC systems has been hindered by uncertainties. It is unknown how the units perform over the course of their lifetimes, how to handle the spent carbon when the media must be replaced, and what is the fate of CECs removed by the system. These areas must be further researched before use of BAC systems becomes more common (Sundaram & Emerick 2010a, b).

There is some concern of coliform regrowth in the BAC unit. The filter bed provides the opportunity for total and fecal coliform levels to increase which may also indicate pathogen levels will increase. BAC effluent routinely exceeded the requirement of 2.2 MPN/100 mL while treating municipal wastewater for reuse projects. This will

likely also be the case when treating RO concentrate. It has been recommended to consider adding a downstream process such as UV disinfection to counteract this effect (Gerrity *et al.* 2011).

Ozonation

Ozone (O₃) is a powerful oxidant that is gaining popularity in water and wastewater treatment as it has been found to be more effective than chlorination and chloramination. It can be turned into an advanced oxidation process (AOP) through the addition of either peroxide (H₂O₂) or ultraviolet (UV) light.

The organic compounds subjected to ozonation are oxidized by reacting with molecular ozone and with hydroxyl radicals generated by ozone decomposition. This allows the process to remove a wide range of contaminants (Justo *et al.* 2013).

Studies have found the process does well at removing estrogenic activity and most CECs that do not have a high resistance to oxidation. Sulfamethazine, acetaminophen, and naproxen have consistently displayed high removal rates under this process. Studies have found that most CECs are removed at rates of 80–100% (Snyder *et al.* 2006; Abdelmelek *et al.* 2011; Justo *et al.* 2013). Carbamazepine, sulfamethoxazole, and trimethoprim have even been suggested as indicator compounds to measure the performance of ozone/peroxide treatment (Snyder *et al.* 2006).

It should be noted that despite requiring higher ozone doses, the ozonation of brine concentrate requires less ozone than treating the full plant flow. This makes it a more economical means of implementing ozonation (Benner *et al.* 2008).

Not all CECs are subject to high removal rates during ozone treatment. Meprobamate was found to be among the most resistant to ozone treatment. Atenolol and carbamazepine have also displayed removal rates below 80% (Justo *et al.* 2013). Atrazine, iopromide, ibuprofen, and TCEP have also shown resistance (Snyder *et al.* 2006). Removal of clofibric acid may also be an issue (Ternes *et al.* 2002).

Ozone systems are often followed by BAC units. The ozone breaks down many compounds into their constituents which provide a more readily degradable food source for the microbial community living in the BAC unit. This treatment train yields lower effluent CEC levels than either system can obtain on its own (Sundaram & Emerick 2010a, b).

Despite its effectiveness, ozonation has some drawbacks. Some of the most concerning are the increase in bioactivity in the effluent, inadequate removal of engineered

compounds such as flame retardants, and the formation of carcinogenic byproducts such as bromate and NDMA (Sundaram & Emerick 2010a, b). Residual DOC in the system effluent can further lead to byproduct formation when chlorine is introduced as a residual disinfectant (Snyder *et al.* 2006). There is also concern about the competition for the hydroxyl molecules between EfOM, the inorganic constituents, and the micropollutants of interest (Abdelmelek *et al.* 2011). It also does not leave a disinfecting residual in the effluent. These issues have limited the widespread implementation of ozonation.

The formation of bromate as a byproduct to the process causes concern. Studies have found that bromate formation can be limited by year-round peroxide addition, seasonal ammonia addition, and optimal ozone dosage. The dosage requirements and level of removals are water-specific. Peroxide addition interrupts bromate formation at the molecular level. A peroxide dose of 1:1 peroxide to ozone molar ratio has been found to help eliminate bromate formation. Ammonia converts bromide into bromamines which reduces the constituents available for the creation of bromate. An ammonia dose of greater than 1.0 mg N/L was found to reduce bromate formation. Ozone doses of 5 mg/L have been found to typically provide a good balance of CEC removal and bromate formation. A dose of 3 mg/L was found to yield lower CEC removal rates than desired while a dose of 7 mg/L yielded excessive bromate levels (Sundaram & Emerick 2010a, b).

It is important to note that the addition of peroxide changes the dynamics of ozone treatment. Peroxide addition results in faster creation of hydroxyl (–OH) molecules which yields shorter reaction times while involving a wider range of contaminants. The downsides to this augmentation, however, include the higher cost of using an additive, ensuring no residual peroxide is released to the environment, and creating a weaker disinfection environment than ozone alone. It is recommended to avoid peroxide dosing unless reaction time and bromate formation govern system design and operation (Gerrity *et al.* 2011).

Ultraviolet light (UV)/hydrogen peroxide (H₂O₂)

The AOP of UV/H₂O₂ is a proven method that is common in many advanced treatment systems. It employs UV light to break down chemicals while also breaking peroxide down into powerful hydroxyl radicals that further degrade contaminants.

It is important to note that the power output of UV systems vary greatly. Less powerful UV disinfection systems are

not capable of achieving the energy output necessary to degrade CECs. More powerful units must also provide adequate exposure time to ensure proper removal rates. Systems capable of achieving CEC removal often apply over 20 times the UV dose used in a typical wastewater disinfection unit (Kim *et al.* 2008).

UV/H₂O₂ has been found to be superior to UV alone. The addition of peroxide allows for higher removal rates of a wider range of contaminants while using much lower UV doses. A study found that a UV dose of 2,768 mJ/cm² resulted in far lower CEC removal rates than a UV dose of 923 mJ/cm² combined with 7.8 mg/L of H₂O₂. The lower UV dose means less energy consumption helping the process become more cost-effective. The cost of the peroxide should also be factored into operating costs (Kim *et al.* 2008).

UV/H₂O₂ systems have been found to offer high removal rates of atenolol, diclofenac, and carbamazepine (Justo *et al.* 2013). This somewhat contrasts to ozone which displayed limited removal of these compounds. Tests have found it is capable of removing a broad range of CECs including ketoprofen, naproxen, metoprolol, clarithromycin and primidone at removal rates greater than 90% (Kim *et al.* 2008). This broad removal capability makes it a powerful technology.

Trimethoprim and paroxetine have been found to exhibit lower percentage removals (Justo *et al.* 2013). This has been attributed to their molecular structure. Norfloxacin and caffeine have also shown reduced removal rates (Kim *et al.* 2008). Research has also found that several chemicals including DEET and clarithromycin require higher UV doses even with the addition of peroxide in order to achieve removal rates above 90% (Kim *et al.* 2009).

A major drawback with UV/H₂O₂ systems is their operational costs. They have a high energy demand as the lamps require substantial amounts of energy to maintain proper operating parameters. Bulbs also require replacement when they burn out. UV/H₂O₂ systems have been found to be more costly to operate than ozone units (Pisarenko *et al.* 2012). Removal rates also appear to be affected by pH levels in the water receiving treatment (Canonica *et al.* 2008).

An important aspect of UV/H₂O₂ is its proven track record. Operating parameters have been researched heavily. The system also improves water quality using significantly less oxidant than ozone systems (Justo *et al.* 2013). These factors combined with the broad CEC removal abilities make UV/H₂O₂ the most potent system available for CEC removal in RO concentrate.

A promising form of UV treatment is high intensity UV from solar energy. This technology harnesses the power of the sun to treat waste flows with UV intensities much higher

than typical systems. The addition of H₂O₂ or ammonia is also an option. It has been found to offer very high removal rates of all contaminants but is still in the development phase. The systems are currently being developed by Focal Technologies, Inc. and are just starting to come to market. (E. Steinmeyer, personal communication, April 10, 2015).

Alternative advanced oxidation processes

Applying the power of advanced oxidation using other methods is also being studied. An emerging concept is electrical treatment of RO concentrate. Electro-oxidation, electrochemical treatment, electrodialysis and electrochlorination have been studied as viable alternatives to the more common AOPs. They use various combinations of electricity, metals, and (in some methods) the addition of chemicals to treat the water. The use of light and catalysts in photocatalysis and ultrasound in sonolysis are also options under investigation.

Electro-oxidation in particular has shown great promise. The salinity and chloride concentrations in concentrate help improve these systems' performance. Using boron-doped diamond electrodes to apply a current density of 100 A/m² has been found to yield removal rates greater than 97% for bisphenol A, hydrochlorothiazide, nicotine, atenolol, furosemide, and bezafibrate. Removal rates of 94% were also found for ofloxacin, fenofibric acid, 4-AAA, naproxen and gemfibrozil. Ibuprofen has been found to be the most resistant with removal rates of 70% (Pérez *et al.* 2010; Uriaga *et al.* 2013).

Limited research has been conducted on the CEC removal capabilities of the other systems. Most studies focus on their DOC removal capabilities with promising results. Removal rates of DOC as high as 50% were recorded making them good candidates for combining with other processes that have opposing strengths (Dialynas *et al.* 2008).

Several drawbacks are known for these systems. Processes that employ electrolytic oxidation require very long oxidation times making the process energy-consuming and expensive (Dialynas *et al.* 2008). Electro generated chlorine has been found to lead to the formation of harmful by-products such as trihalomethanes (THMs) and haloacetic acids (HAAs) which must be removed by a polishing treatment (Bagastyo *et al.* 2011). These issues result in the need for much more research.

Less common and emerging technologies

There are many other treatment methods that are less mainstream but still worth noting. Many are aimed at the goal of

making RO systems run at, or near, zero liquid discharge (ZLD) in order to avoid expensive discharge permits. These methods are separated into membrane-based and thermal-based technologies (Subramani & Jacangelo 2014).

Membrane-based technologies apply membrane treatment under different conditions. These include chemical softening of brine concentrate, chemical precipitation during RO cleaning activities, and disc tube membranes. Most of these methods are intended to reduce the concentrate volume to levels that can be more easily discharged or recycled to the beginning of the plant. It is not known if these methods offer any CEC removal but it is unlikely. The reduction of brine flow would actually result in more concentrated CEC levels in the concentrate (Subramani & Jacangelo 2014).

Thermal-based technologies use mechanical or natural methods to evaporate concentrate flows. Multi-effect distillation, brine concentrators and crystallizers, wind-aided intensified evaporation, and spray dryers all fall under this category. They are well-proven at reducing concentrate volume but are often expensive and complex to operate. The solid residue that is left behind is pure concentrations of the contaminants in brine including CECs meaning the handling of this waste is an environmental issue (Subramani & Jacangelo 2014).

Emerging technologies are also being developed which have great potential but need much more research. These systems include forward osmosis (FO), membrane distillation, thermoionic process, and eutectic freeze crystallization. They promise to solve some of the problems of existing methods but will likely be faced with the same CEC handling issues (Subramani & Jacangelo 2014).

FO is one of the most promising emerging technologies. FO is an emerging treatment process that involves driving water through a semi-permeable membrane using the pressure difference of the osmotic pressure between the feed and draw solution. The water diffuses from the lower osmotic pressure of the feed solution to the higher osmotic pressure of the draw solution (Martinetti *et al.* 2009). Since the travel of the fluid in FO is natural osmosis it in turn requires less energy to operate compared to other methods. The FO process is commonly operated by injecting an easily removed solution of high concentration (such as ammonia) into the feed in order to generate the hydrostatic osmotic pressure gradients. This causes the fluid (which can be brine) to cross the semi-permeable membrane where the membrane filters out the unwanted constituents. The saline solution is then removed with an additional process such as slight heating to remove ammonia.

The effectiveness of FO has been tested by injecting 1 M of concentrate into the feed along with the use of a cellulose acetate asymmetric semi-permeable membrane. This resulted in a water flux of a high initial value of 8.9 gallons/ft²/day which declined to 6.0 gallons/ft²/day after 18 hrs (1 gallon = 3.785 liters; 1 square foot = 0.0929 square meters). The resulting removal of organic contaminants in the concentrate was approximately 76%. Further research is needed to determine how much of this removed organic contamination was CECs (Tang & Ng 2008).

The FO process has less membrane fouling than other membrane treatment methods. This results in less contaminated cleaning water to handle. Despite the high potential, difficulties in manufacturing of the FO membranes and lack of knowledge on the ideal operating parameters have limited its implementation (Subramani & Jacangelo 2014).

FO has been under development for many years but has not yet been developed into commercially viable systems. Many of these problems appear to be due to the membrane manufacturing issue. It has been considered that the lower flows involved in RO concentrate streams could allow smaller membranes to be used which would likely help alleviate some of the manufacturing issues.

Although tests are being conducted, there has not been substantial research done on concentrate treatment using FO to date. Even less is known on CEC removal in FO. Despite this lack of current knowledge, FO's ability to remove constituents using less energy than other treatment options means that it has the potential to gain widespread use in the future.

CEC disposal options

Even the best RO concentrate treatment options produce hazardous products. Handling these products is site specific. Most RO concentrate is mixed with other treatment plant effluent and discharged to a receiving body of water. Deep injection wells are another less common environmental discharge option (Subramani & Jacangelo 2014). The preferred ZLD practice is to treat the concentrate to a level where it can be reintroduced at the beginning of the treatment plant but no plants were found during this study that currently have this ability.

Treatment systems that produce solid residue (such as brine crystallizers and evaporation ponds) pose a unique issue. This residue requires disposal at landfills which can be an unpopular and potentially expensive option (Subramani & Jacangelo 2014). No mention of burning this residue was found during the course of this study even though this is sometimes seen as a valid disposal option for other waste materials.

CONCLUSIONS

There are substantial concentrations of a wide range of CECs in concentrate even though RO systems usually follow several other treatment methods. The CECs that make it through these processes are removed from the main flow by the RO membranes but are concentrated into concerning levels in the brine flow.

Some of the most frequently found CECs with the highest average levels should be considered as potential indicator compounds. These include the following ten CECs: the beta blocker atenolol, the anti-epileptic carbamazepine, the cholesterol reducer gemfibrozil, the anti-inflammatory ibuprofen, the X-ray contrast media iohexol, the antibiotic sulfamethoxazole, the anti-bacterial triclosan, the flame retardant component TCEP, the insect repellent DEET, and the herbicide diuron. Each represents a different family of CECs with unique properties. All ten of these have been found to be common and offer a large enough range to be used for determining treatment process efficiency. The hormones estrone, estradiol, and ethynylestradiol and the androgen hormone testosterone should also be monitored due to their frequent presence and endocrine disrupting abilities.

The research repeatedly found UV/H₂O₂ and ozone-BAC to be the most viable treatment options. UV/H₂O₂ has the benefit of being a well-studied system that is already commonly employed for drinking water and wastewater treatment. It was found to be effective at removing a broad range of CECs but has the implementation issue of high power requirements for the UV bulbs. Typical operating parameters are also well-documented but more research needs to be done to determine if high CEC levels need higher contact-time values.

Ozone-BAC is a treatment train that has shown promise at removing CECs and seems to be effective on a different subset of CECs than UV/H₂O₂. The main concern is the limited knowledge on the BAC operating parameters and a concern of bromate and NDMA formation in the ozone process. It is an important system to research, however, as the lower operational cost could be a major benefit.

Some of the other treatment methods work well for removing particular CECs but they do not have the high removal rates for a wide range of CECs that are offered by UV/H₂O₂ and ozone-BAC. These options may be better choices if cost is the main concern or there is a particular CEC of interest.

Many of the emerging technologies are very promising. They have the potential to offer very high CEC removal rates while having low operational costs. Unfortunately, too little information is currently known about these systems

for full-scale implementation. Much of this technology is at least 5–10 years from being considered as a viable alternative.

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