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Research Paper

The Removal of N-nitrosodimethylamine, Trihalomethane, and Halonitromethane Precursors by RO Membrane from Water

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Abstract: Reverse osmosis (RO) has been proven to be effective for removing disinfection by-products (DBPs) precursors from different water sources during conventional water treatment processes. However, polymeric RO membranes have the potential to leach DBP precursors, which can introduce a bias in the true performance evaluation of these membranes during bench, pilot, and full-scale operations. This study first examined the NDMA precursor leaching potential of one commercially available virgin RO membrane. Following a cleaning procedure, the efficiency of the RO membrane was tested for the rejection of N-nitrosodimethylamine (NDMA), trihalomethane (THM), and halonitromethane (HNM) precursors from surface water and wastewater treatment plant effluents. While the leaching potentials of RO membranes for NDMA precursors were up to 370 ng/L, the leaching potential of membrane's decreased as the filtered volume of water increased. In the tested water samples, the RO rejection efficiencies ranged from 78 to 91%, 81 to 97%, and 63 to 78% for NDMA, THM, and HNM precursors, respectively. The results also showed that the background water quality did not have a considerable influence on the rejection of NDMA, HNMs, and THM precursors by the RO membrane. While the correlation between NDMAFP, HNMFP and THMFP, and total dissolved nitrogen (TDN) removals were weak (r^2 ranged from 0.02 to 0.3), the removals of DBPFP were correlated well with DOC removals (r^2 ranged from 0.6 to 0.89).

Keywords: *DBP precursors, Leaching, Disinfection By-products*

Introduction

High-pressure reverse osmosis (RO) membranes have emerged as the preeminent technology for the rejection of salts, natural organic matter, and emerging contaminants from water sources. During manufacturing processes, polymer-based RO membranes are, in general, made from either cellulose acetate or polysulfone coated with aromatic polyamides through a method called phase inversion, where the selected polymers are dissolved in water-soluble solvents. The manufacturing process involves in use of varying types of polymers, additives, and surface-coating materials. During membrane filtration, loosely bound chemicals and additives may be released into the permeate and serve as a precursor for emerging contaminants (Ersan *et al.*, 2015).

Although chemical disinfectants (such as chlorine, chloramine, ozone, and permanganate) have been used to kill harmful pathogens in drinking water supplies, these oxidants can react with precursors (organic matter, bromide [Br^-], iodide [I^-], and other anthropogenic pollutants) and result in the occurrence of unintended disinfection by-products (DBPs) in the treated waters. Trihalomethanes (chloroform, dichlorobromomethane, dibromochloromethane, bromoform) and haloacetic acids (monochloroacetic acid, dichloroacetic acid, trichloroacetic acid, monobromoacetic acid, and dibromoacetic acid) are among the major and regulated DBPs of chlorination (U.S. EPA, 2006). To mitigate the formation of regulated DBPs (THMs and HAAs), the water reclamation facilities have been switching to alternative oxidation strategies such as ozone, chlorine dioxide, UV photolysis, and chloramine. Among these, chloramine is one of the most used disinfectants in water reclamation facilities in the US. Although the formation of THMs and HAAs is minimized during chloramination of finished waters, this can facilitate the formation of other problematic unregulated DBPs, such as halonitromethanes (HNMs), haloacetonitriles (HANs), and nitrosodimethylamine (NDMA) (Chen & Valentine, 2006; Ersan *et al.*, 2016; Krasner *et al.*, 2006). While NDMA is reported as a potential human

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**Corresponding: E-Mail: mersan@asu.edu; Phone: +1-732-208-1326* carcinogen and has been detected mainly in chloraminated waters (Russell et al., 2012), HANs and HNMs have higher cyto- and genotoxic potency than the regulated THMs and HAAs (Plewa et al., 2004). Although NDMA has been listed in the toxic pollutant category, there is no federal maximum contaminant level (MCL) has been issued for drinking water. Though, some of the states, i.e., California, has been issued notification limit of 10 ng/L for NDMA (CDPH, 2014). On the other side, there is no health-based advisory level or regulation exists for HANs and HNMs. Because of their pervasiveness in engineered and aquatic systems, alternative strategies for controlling their formation have been critical in protecting public health from the harmful effects of these contaminants. Several treatment techniques, such as activated carbon, anion exchange resins, enhanced coagulation, and nanofiltration (NF) and reverse osmosis (RO) membranes, have been used at the conventional scale to control the occurrence and formation of DBPs (Beita-Sandí *et al*., 2016; Boyer and Singer, 2005; Ersan et al., 2016; Gan et al., 2013; Krauss *et al*., 2010; Miyashita et al., 2009; Schmidt and Brauch, 2008; Snyder *et al*., 2007; Uyak *et al*., 2007). Among these, RO membranes have been a proven technology in water and wastewater treatment, seawater desalination processes, and brackish water treatment due to their ability to remove organic constituents from water, such as organic matter, bacteria, viruses, and harmful trace organics. However, previous research studies have reported NDMA precursors leach from membranes, anion exchange resins, and various materials (gaskets, rubber seals, etc.) (Ersan et al., 2015; Gan et al., 2013; Morran *et al*., 2011; Teefy *et al*., 2011). For example, in one of our previous studies, we demonstrated that nanofiltration (NF) membranes can leach from 130 to 450 ng/L of NDMA precursors regardless of their polymer type or manufacturer (Ersan et al., 2015). Therefore, the possibility of DBP precursor leaching from RO membranes raises questions about the true performance evaluation of these membranes for the removal of NDMA during bench, pilot, and full-scale applications. As a result, it is necessary to investigate whether polymeric RO membranes also contain precursors of NDMA, and then perform a DBP removal test after the precursors have been removed from the RO membranes.

The main goals of this study are to (i) examine the leaching of NDMA precursors from RO membranes and (ii) investigate the rejection of unregulated (i.e., NDMA and HNMs) and regulated (THMs) precursors by RO membranes from municipal WWTPs effluents and surface water sources. To the best of our knowledge, this is the first study reporting NDMA precursor leaching potential from RO membranes and providing a true evaluation of NDMA, THM, and HNM precursor removal efficiency of RO membranes.

Materials and Methods

Leaching experiments

A commercially available composite polyamide CPA2 (Hydranautics) RO membrane was used during the experiments (Table 1). All filtration experiments were conducted at 25 ± 1 ºC under constant operating pressure of 500 psi using SEPA II crossflow filtration cell described elsewhere (Ersan et al., 2016), and a schematic diagram was presented in Figure S1. The surface area of the membrane coupon was 140 cm^2. 500 ml of sample was withdrawn from the effluent side of the membrane system at the predetermined distilled and deionized (DDI) water volume to surface area ratios (L/m^2), and NDMA formation potential test (NDMAFP) was carried out.

Table 1. Characteristics of RO Membrane

Designation Manufacturer Polymer type	Molecular weight cutoff (Da)	NaCl Rejection (%)	pH range (25 ^{0}C)	Typical flux (m^3/day)
Composite Polyamide	300-500	99.7	2-10	37.9

Filtration Experiments

The water samples were collected from treated effluents of surface water sources (SW-A and SW-B) and wastewater treatment plants (WWTP-A and WWTP-B). The collected samples were passed through a pre-rinsed 0.2μm Whatman Polycap TC 150 capsule filter and characterized for ultraviolet absorption (254nm), dissolved organic carbon (DOC), total dissolved nitrogen (TDN), conductivity, ammonia (NH_4^+), nitrate (NO_3^-), bromide (Br^-), and pH. The formation potential (FP) concentrations of disinfection by-products (DBPs), nitrosodimethylamine (NDMA), halonitromethane (HNM; trichloronitromethane, TCNM), and trihalomethanes (trichloromethane, bromodichloromethane, dibromochloromethane, and tribromomethane) were measured according to slightly modified standard methods, discussed below. The water qualities of studied water sources are given in Table 2.

The removal efficiency, R (%) was calculated as shown in *eq 1.*

$$R = \left(1 - \frac{Cpermeate}{Cfiltrate}\right) * 100 \qquad (1)$$

where $C_{permeate}$ and $C_{filtrate}$ are the concentration of permeate and feed, respectively.

Table 2. Characterization of selected water sources

Parameters	WWTP-A	WWTP-B	SW-A	SW-B
UV_{254nm}	0.083	0.047	0.283	0.059
DOC (mg/L)	5	1.99	7.62	3.01
$SUVA_{254}$ (L/mg/m)	1.7	2.4	3.7	2.0
TDN (mg/L)	8.5	8	0.78	0.83
Conductivity (µs/cm)	572	80.1	48	44.9
NH_4^+ (mg/L)	0.02	0.04	0.01	0.02
Ca^{2+} (mg/L)	18.5	4.8	4.1	5.0
NO_3 (mg/L)	38	35	3	4
Br^- (µg/L)	50	30	<MRL*	18
pH	7.8	7.3	7.1	7.2
NDMAFP (µg/L)	1049	164	179	106
HNMFP (µg/L)	36	13	17	10
THMFP (µg/L)	218	136	1325	206

*Bromide MRL= 10 µg/L

Analytical methods

To ensure that all the precursors were converted to their respective DBPs, NDMAFP, HNMFP, and THMFP tests were performed with an excess of oxidants. The details of the analytical methods used in this study were provided in the literature (Ersan et al., 2016). Briefly, 10 mM phosphate buffered was used to buffer the samples at pH 7.8 before the addition of the oxidants. For NDMAFP, 100 mg/L (as Cl_2) of chloramine was spiked to both raw water and treated source water samples and reacted for 5 days. For THMFP, 50 mg/L of chlorine was added to the samples and hold for 5 days. For HNMFP test, the samples were spiked with ozone followed by chlorine. Ozone stock solution (~20-30 mg O_3/L) was produced using a lab-scale ozone generator (Model GTC-1B, Griffin Technics, NJ). The samples were initially ozonated at a mass ratio of 1:1 (O_3:DOC) for 5 min. Then, the samples were reacted with 50 mg/L of Cl_2 for 24h. After 24h, the oxidant residuals were measured, and residual Cl_2 was quenched at a 1:1 molar ratio with sodium thiosulfate before the analysis.

NDMA samples were extracted according to USEPA Method 521. (USEPA, 2004). Before the solid phase extraction, N-nitrosodimethylamine-d6 (NDMA-d6) was added as a surrogate compound to the quenched samples. Later, the extracted samples were spiked with an internal standard, N-nitrosodi-n-propylamine-d14 (NDPA-d14) and measured by Varian GC 3800-MS/MS equipped with an RTX-5MS (Restek 30 m, 0.25 mm, 0.25 mm) column.

The analysis of THM and HNM samples was performed according to USEPA Method 551.1 with minor modifications. A liquid-liquid extraction (LLE) procedure was followed for the extraction of the samples in accordance with the USEPA 551.1 method. Briefly, samples were transferred into extraction vials (50mL) which was followed by transferring 3 ml of MtBE and 10g of overnight (at 105 °C) dried sodium sulfate (Na_2SO_4) into the vials, respectively. The extraction vials were then shaken at 300 rpm for 30 minutes. After 30 minutes, the samples were placed on a flat surface for 10 minutes to allow for phase separation. MtBE phase was extracted and analyzed on an Agilent 6890 GC outfitted with a DB-1 column (J&W Scientific 30 m x 0.25 mm x 1 µm) and an electron capture detector (ECD). The details on the analytical methods are summarized in Table S1.

Results and Discussion

NDMA Precursor Leaching Potential of RO Membrane

The NDMAFP leaching potential of the CPA2 RO membrane is shown in Figure 1 as a function of the DDI water volume passed through the membrane. The initial NDMAFP leaching potential in the RO permeate was up to ~313 ng/L, which gradually decreased as the passed volume of water increased up to 1070 L/m^2. Even after flushing the membrane with 1070 L/m^2 of water, the leaching potential was still well above (60 ng/L) the background NDMAFP concentration (DDI= 12 ng/L). In our laboratory, we also tested NDMA precursor leaching potential of a point of use under sink RO membrane. The results showed that the initial permeate NDMA leaching potential from the membrane was 370 ng/L (Figure S2). Like the CPA2 RO membrane, the leaching potentials decreased with the increasing volume of water passed. This indicates the importance of flushing home-type RO filters for the initial operation to eliminate the exposure of residual chemicals from these filters. In another study, authors reported NDMA precursor leach from nanofiltration (NF) membranes (Ersan et al., 2015). The authors examined the leaching potential of NDMA precursors from NF membranes made from various polymeric materials (i.e., polyamide [PA], cellulose acetate [CA], and polypiperazine amide [PPA]) and manufactured by various companies. Their findings indicated that membrane leaching was not limited to a single membrane type or manufacturer. This may indicate that the precursors may be originated from manufacturing impurities (i.e., dimethyl formamide [DMF] or other chemical additives) that remained in the membrane structure after the manufacturing process. Two confirming results, from this study and the previous study, indicate that the leaching potential was significantly reduced after passing a large amount of water per surface area (m^2) of the membrane. From the application point of view, although it is likely to reduce the precursor leach from the membranes, this may raise a concern about water and energy consumption at the initial startup of these systems. According to Schreiber et al. (2007), the addition of free chlorine reduced NDMA formation by deactivating its precursors (Schreiber and Mitch, 2007). Thus, in this study, we investigated the effectiveness of chlorine on the deactivation of NDMA precursors from RO membranes. As discussed in our previous study, the RO membrane was first flushed with 1 mg/L (as Cl_2) chlorine-exposed DDI water. As a result, the leaching potential from the RO membrane was significantly reduced and reached levels comparable to those observed in DDI water (Figure 1).

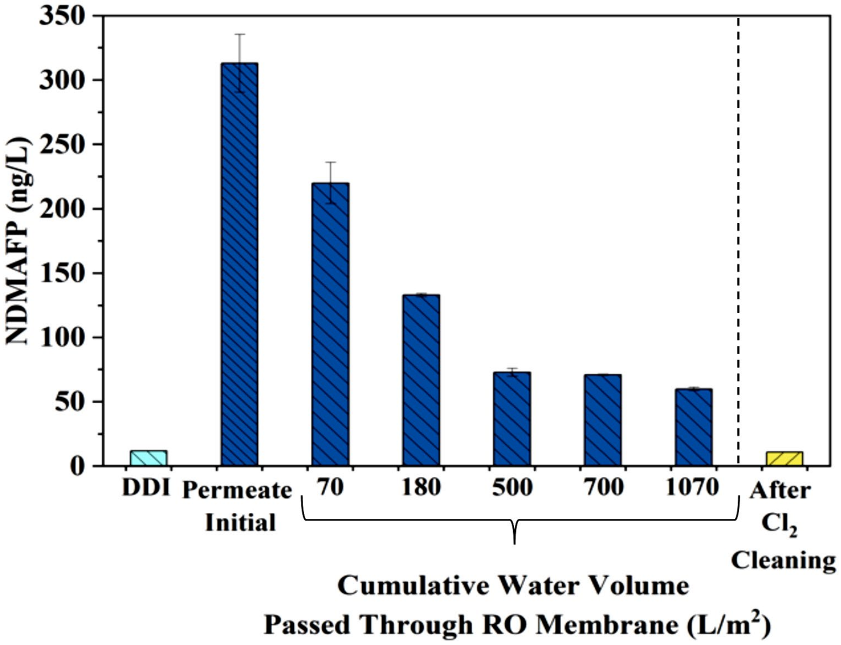

Figure 1. NDMA leaching potentials of RO Membrane

The current study demonstrated that NDMA precursors leach from RO membranes; therefore, it is critical to monitor such precursor leaching in laboratory-scale studies as well as industrial and home-type membrane applications. Previous research studies evaluated NDMA precursor leaching potentials of various polymeric materials and chemicals (i.e., resins and coagulant aids)(Gan et al., 2013; Teefy et al., 2011). The results showed that using these polymeric-based chemicals and materials during water treatment increased the occurrence of NDMA precursor concentrations in treated water effluents, ranging from low levels (20 ng/L) to very high levels (600 ng/L). The reported NDMA precursor leaching concentrations in the previous studies were comparable to the concentrations found in our study

(60-313 ng/L). The results from these studies suggest that it is likely to wash out the residual precursors after long operating hours. However, special attention should be given to the formation of NDMA at the startup period of membrane systems if chloramine is used as the primary oxidant.

Because of the proven leaching potential of NDMA precursors from RO membranes, we used a cleaning procedure described in a previous study (Ersan et al., 2016, 2015) to eliminate NDMA precursor leaching from RO membranes and thus make a true evaluation of RO membranes for removing NDMA precursors from different water sources.

The control of NDMA precursors from RO membrane

Figure 2 depicts the removal efficiencies of the CPA2 RO membrane for NDMA precursors. When the formation potentials of NDMA were compared between feed and permeate formation potentials, 78-91% of NDMA precursors were removed by the RO membrane. These results suggest that the primary mechanism that controlled the rejection of precursors by the RO membrane was size exclusion. Other factors, such as the physicochemical properties of the precursors (i.e., hydrophilicity, hydrophobicity, charge, etc.), may also have an impact on the overall NDMA removal process.

Figure 2 shows that precursor removals from various sources occurred in the following order: WWTP-A > SW-A > SW-B > WWTP-B. Among the tested waters, municipal WWTP-A effluents had higher NDMA precursor removals than SW-A-B and WWTP-B effluents. This finding suggests that either relatively large precursors or particle-associated NDMA precursors were present in WWTP-A. (Mitch and Sedlak, 2004). The lower NDMA precursor removals observed in SW-A, SW-B, and WWTP-B can be attributed to the presence of small molecular size NDMA precursors in these sources. These findings imply that NDMA precursors derived from various sources and treatment facilities may differ in terms of molecular size (or possibly other properties), which affects the rejection efficiency of RO membranes.

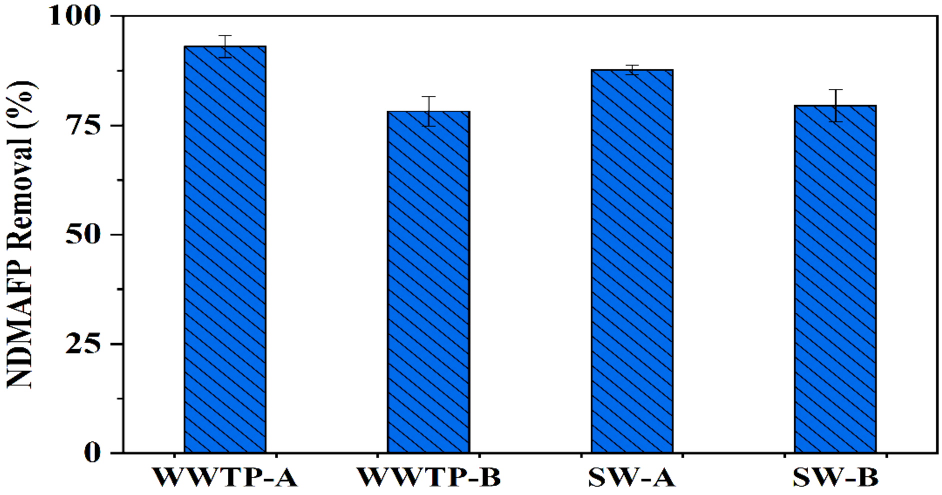

Figure 2. The removal efficiency of NDMA from RO membrane in different water sources

Furthermore, while it is preferable to measure the precursors directly rather than relying solely on FP tests, the structure of precursors is not well understood, limiting direct measurements of these precursors in different sources. However, bulk water parameters, such as DOC and TDN, can be used as surrogate parameters to predict DBPFP removals in different sources. Therefore, in this study, we correlated the removal of NDMAFP with DOC and TDN removals in these sources. Overall, the rejection of DOC and TDN by RO membrane in the studied source waters ranged from 87 to 95% and 79 to 93%, respectively (Figure 3). When the percent removal of NDMAFP was plotted versus the percent DOC and TDN removals (Figure S3), there was a good correlation between NDMAFP and DOC removals (r^2=0.60). However, the correlations between NDMAFP and DON removals (r^2=0.04) were significantly low.

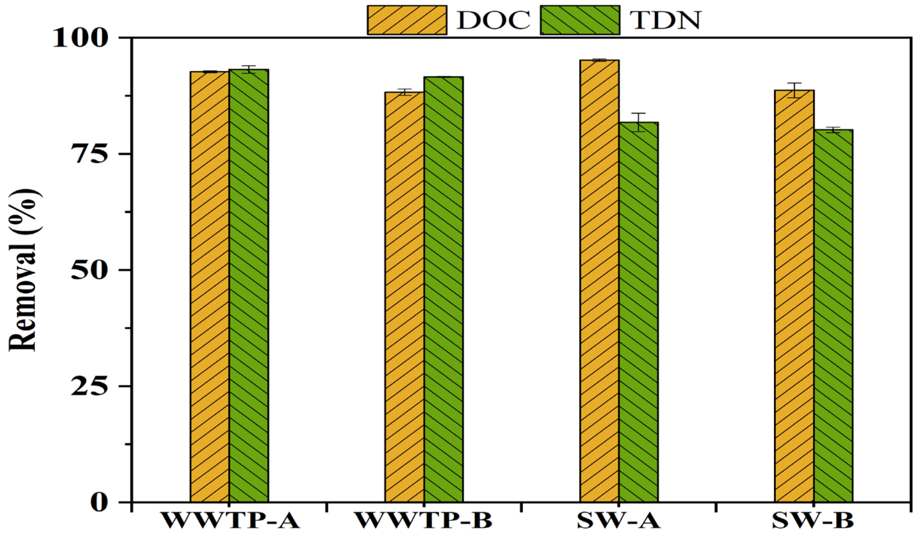

Figure 3. Percent removals of DOC and DON by RO membrane from different water sources

3.3. The control of HNM precursors from RO membrane

The removal efficiencies of HNMFP by CPA2 RO membrane from different water matrices are shown in Figure 4. When the feed and the permeate HNMFP concentrations were compared, HNMFP removal efficiencies by RO membrane ranged from 63 to 78%. As compared to NDMAFP removals, the removal of HNMFP by RO membrane was much lower than NDMAFPs. Lower efficiencies may be attributed to the smaller size of HNM precursors compared to NDMA precursors, resulting in lower rejection by the CPA2 RO membrane. On the other hand, the removal of HNM precursors by the CPA2 RO membrane may be also altered by the physicochemical characteristics of HNM precursors. According to Figure 4, precursor removals from different sources followed the order of SW-A > WWTP-B > WWTP-A >SW-B. Among all types of source waters, there was slightly higher HNM precursor removal observed for the SW-A source. This may be attributed to the presence of larger molecular weight HNM precursors in the SW-A source (Ersan *et al*., 2016).

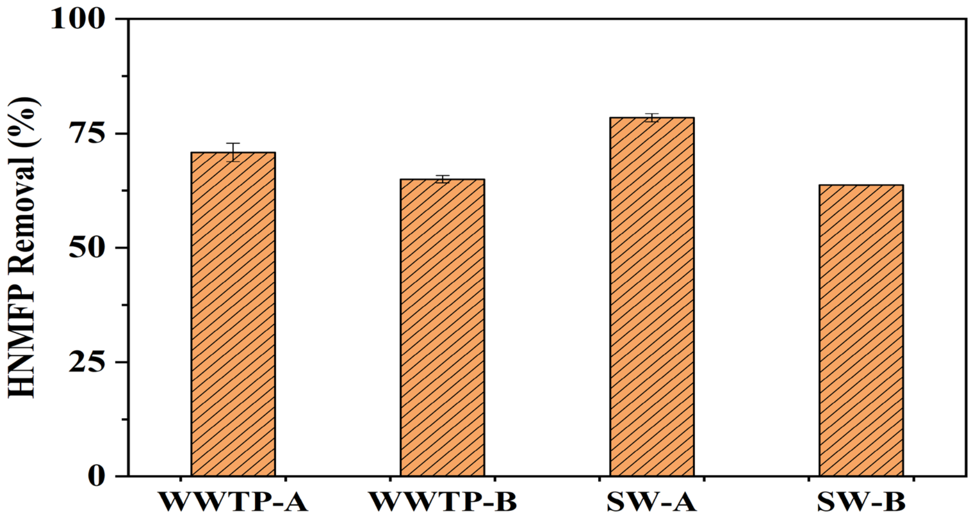

Figure 4. The removal efficiencies of HNM from RO membrane in different water sources

In addition, in Figure S4, the removal of HNMFP was plotted against DOC removals in all the sources. The results suggest that there was a good correlation between HNMFP and DOC removals (r^2= 0.89) for the tested water sources. On the other side, a weak correlation was obtained between HNMFP and TDN removals (r^2=0.02) (Figure S4). This may suggest that DOC can be used as a surrogate parameter to predict the removal of HNMFP from waters with similar water quality.

The control of THM precursors from RO membrane.

The removal of THMFP from different sources by CPA2 RO followed the order of SW-A > WWTP-A ≈ SW-B > WWTP-B, as shown in Figure 5. For all the tested water sources, the removal of THMFP ranged from 86 to 97%. Overall, the RO removal efficiencies for THM precursors were higher than NDMA and HNM precursors. The results indicate that THM precursors are relatively larger than NDMA and HNM precursors. Among the water sources, RO removal efficiency for THM precursor from SW-A was the highest, which may be due to existing of larger size precursor molecules in this source. Previous studies found that NF membranes removed 41-98% of THM precursors (Ángeles et al., 2008; Chellam, 2000; Ersan et al., 2016; Golea et al., 2016; Lin et al., 2006), which is comparable with RO membrane results in the present study. In the literature, generally, the rejection of THM precursors was much higher than NDMA and HNM precursors due to the larger molecular size/weight of THM precursors (such as natural organic matter) than NDMA precursors, which has been linked to small molecular weight compounds of anthropogenic origin (Ersan *et al.*, 2016; Mitch & Sedlak, 2004).

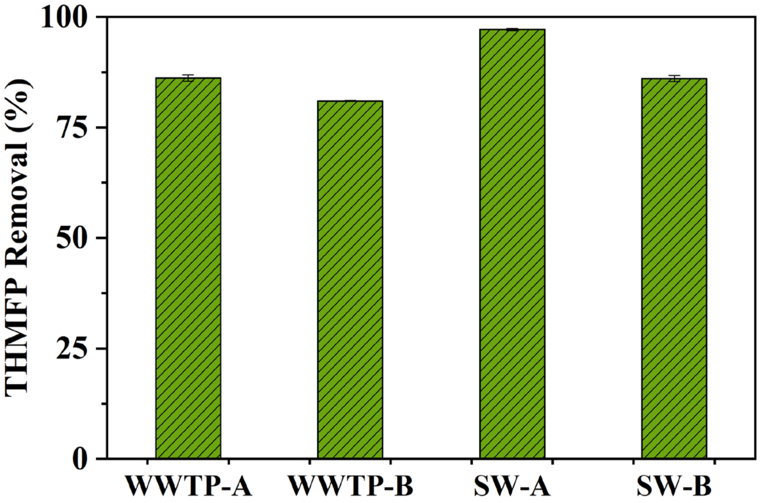

Figure 5. The removal efficiency of THM from RO membrane in different water sources

When the percent THMFP removal was plotted against the percent DOC and DON removals (Figure S5), there was a strong correlation (r^2=0.70), implying that the percent DOC removals can be used as a surrogate parameter for THM precursor removals. On the other hand, a weaker correlation (r^2=0.32) was observed between THM precursor and TDN removals.

Conclusions

In this study, we investigated the leaching of NDMA precursors from RO membranes as well as the removal of NDMA, HNM, and THM precursors from various types of water/background matrices. For the first time, NDMA precursor leach from RO membranes was reported. The leaching potentials from both CPA2 and under-sink RO membranes were up to 370 ng/L. These findings highlighted the importance of flushing/cleaning RO membranes before use in both scientific research and drinking water production to eliminate bias in RO unit performance evaluations. As a result, the CPA2 RO membrane was tested in this study after being cleaned with a low dose of chlorine. To the best of our knowledge, this is the first study in the literature to report the true efficiency of RO membrane at the initial operation for removing NDMA precursors from the surface and treated wastewater backgrounds. In addition to NDMAFP removals, the removal of other DBPs by RO membrane, HNM, and THM, was also investigated. Overall, the removal efficiencies for NDMA, HNM, and THM precursors in the studied sources ranged from 78 to 91%, 63 to 78%, and 81 to 97%, respectively. These results showed that among the tested background sources, THM precursor removals by CPA2 RO membrane were always higher than those of NDMA and HNM precursors. The background water characteristics of the water sources (SW vs WWTP effluent) did not have a considerable impact (within 10% when % removals in WWTP compared with SW) on the removal of NDMA, HNMs, and THM precursors. While DBPFP removals correlated well with DOC removals (r^2=0.6-0.89), there was a weak correlation with TDN (r^2=0.02-0.32) in this study. This could imply that the bulk parameters, such as DOC, can be used as surrogate parameter for predicting the removal of DBPFP by RO membranes.

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Conflicts of interest: *The authors declare no conflicts of interest.*

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