Online monitoring of *N*-nitrosodimethylamine for the removal assurance of 1,4-dioxane and other trace organic compounds by reverse osmosis

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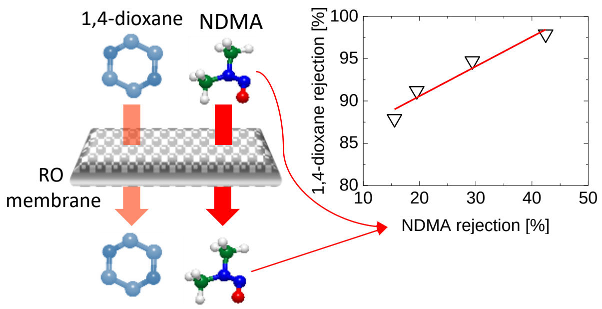


Online monitoring of *N*-nitrosodimethlyamine (NDMA) ~~in~~ during reverse osmosis (RO) treatment was ~~identified~~ effective for ensuring the removal of trace organic chemicals, particularly 1,4-dioxane.

Abstract

Public health protection and cost effectiveness of potable reuse can be improved by providing reliable water quality assurance for removal of trace organic compounds (TOrCs) by reverse osmosis (RO) membrane. This study evaluated the effectiveness of online monitoring of *N*-nitrosodimethlyamine (NDMA) removal by RO system to ensure the removal of low molecular weight TOrCs. Among TOrCs, the main focus was placed on 1,4-dioxane due to the limited information for RO. Laboratory-scale experiments showed that the rejection of 1,4-dioxane by two commercial RO membranes — ESPA2 and HYDRA (98 and 99%, respectively) — was higher than that of NDMA (57 and 81%, respectively). Pilot-scale experiments using a treated wastewater identified a strong linear correlation between 1,4-dioxane and NDMA rejection over a range of feed temperature. Pilot-scale results also demonstrated the applicability of NDMA a conservative performance indicator for 46 other TOrCs at two different RO feed temperatures. These results suggest that online monitoring of NDMA in RO feed and permeate can allow for ensuring the removal of larger TOrCs, which could provide additional protection of public health in potable reuse.

Keywords: *N*-nitrosodimethlyamine; 1,4-dioxane; trace organic compounds; potable reuse; reverse osmosis.

1 INTRODUCTION

In response to frequent and severe drought, the use of advanced treatment processes to reclaim wastewater for augmenting drinking water supply, also known as potable reuse, has been increasingly adopted in many countries and regions of world.[1] High quality reclaimed water is typically produced through conventional wastewater treatment followed by several layers of advanced treatment processes including microfiltration/ultrafiltration (UF), reverse osmosis (RO), and advanced oxidation process (AOP).[2] Among these advanced treatment processes, RO is a critical physical barrier to remove trace organic compounds (TOrCs) such as disinfection by-products, endocrine disrupting compounds, and pharmaceuticals and personal care products that are ubiquitous in reclaimed wastewater.[3-7] Thus, monitoring the integrity of the RO process is essential during potable water reuse operation. In particular, much of the recent attention has been given towards two specific TOrCs namely *N*-nitrosodimethlyamine (NDMA) and 1,4-dioxane.[8-11] The former is a disinfection by-product occurring ubiquitously in reclaimed wastewater,[9, 12] while the latter is a common industrial solvent often accidentally released into the sewer and the environment.[13] Both NDMA and 1,4-dioxane are probable carcinogenic and thus are regulated in potable water reuse applications. The occurrences of NDMA in RO permeate intended for potable water reuse have occasionally been reported[14, 15] at above the NDMA notification levels (10 ng/L) by the authority in California, USA.[16]

Most advanced water treatment plants for potable reuse applications are equipped with AOP-based post treatment to ensure adequate removal of NDMA and 1,4-dioxane in addition to disinfection requirements. Photolysis by UV irradiation is sufficient for the decomposition of NDMA,[17] while reactive free radicals (e.g. $HO^{\bullet}$ and $Cl^{\bullet}$) generated by AOP are necessary to oxidize 1,4-dioxane. As a result, 1,4-dioxane removal has been to benchmark AOP performance as an indicator for the removal of other TOrCs in California, USA. For potable reuse application, AOP is required to achieve a minimum 0.5-log (69%) removal of 1,4-dioxane by California Office of Administrative Law.[18] Since 1,4-dioxane is an industrial solvent, its occurrence in wastewater is site specific and is often associated with accidental release in the wastewater catchment. 1,4-dioxane concentration as high as 100 µg/L has been reported in treated wastewater while a lower concentration has been reported in the RO feed.[19, 20] It is not possible to directly validate 1,4-dioxane in a full scale plant due to its intermittent occurrence of 1,4-dioxane in wastewater.

In addition to 1,4-dioxane, a reliable surrogate performance indicator to monitor the rejection of TOrCs by RO can also improve treatment efficiency and reliability. In the context of membrane integrity monitoring, a surrogate indicator is required to satisfy three criteria for practical implementation. These criteria include: (a) ubiquitous occurrence in the source water (i.e. RO feed), (b) online monitoring capability and (c) similar behaviour with the target or can provide a conservative estimate. To date, common surrogate performance indicators (e.g. conductivity or total organic carbon (TOC) rejection) for RO can only be applied to monitor the pathogen rejection at a significantly reduce log removal credit and is not useful to monitor TOrC removal.[21]

The authors[22] have recently developed a very fast, sensitive, and reliable analytical technique for quantifying NDMA concentration in reclaimed water online. NDMA analysis is based on high-performance liquid chromatography followed by photochemical reaction and chemiluminescence detection.[23] This technique is highly sensitive and can quantify NDMA in RO feed water at 3 ng/L. NDMA, the smallest among TOrCs regulated in potable reuse, is ubiquitous in secondary treated effluent and is formed as a by-product of chloramination.[24-26] A recent study by the authors[27] has also demonstrated that NDMA can be used as potential surrogate for monitoring the rejection of six TOrCs by RO.

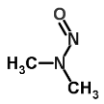
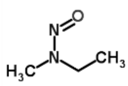
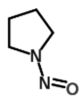
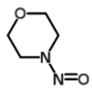
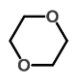
The objective of this study was to evaluate the potential of online monitoring of NDMA in RO feed and permeate to ensure the removal of many TOrCs, which is highly relevant to potable reuse. A particular focus was placed on the removal of 1,4-dioxane by RO due to its limited knowledge. Through laboratory-scale and pilot-scale experiments, the correlation between the rejection of NDMA and 1,4-dioxane as well as other 46 TOrCs was evaluated.

2 MATERIALS AND METHODS

2.1 Chemicals

Certified analytical grade solutions of *N*-nitrosamines – NDMA, *N*-nitrosomethylethylamine (NMEA), *N*-nitrosopyrrolidine (NPYR), and *N*-nitrosomorpholine (NMOR) – were purchased from Ultra Scientific (Kingstown, RI, USA). These solutions were used to prepare working stock solution containing *N*-nitrosamines in pure methanol at 1 µg/mL of each compound. Analytical grade 1,4-dioxane was purchased from Wako Pure Chemical Industries (Osaka, Japan). A working stock solution containing 1000 µg/mL 1,4-dioxane was also prepared in pure methanol. Physicochemical properties of four *N*-nitrosamines and 1,4-dioxane are displayed in **Table 1**.

Table 1: Physicochemical characteristics of the selected *N*-nitrosamines and 1,4-dioxane.

Name	NDMA	NMEA	NPYR	NMOR	1,4-dioxane
Structure					
Molecular formula	$C_2H_6N_2O$	$C_3H_8N_2O$	$C_4H_8N_2O$	$C_4H_8N_2O_2$	$C_4H_8O_2$
Molecular weight [Da]	74.1	88.1	100.1	116.1	88.1
pKa at pH8[1]	3.5	3.4	3.3	3.1	Not ionized
Log *D* at pH8[1]	0.04	0.40	0.44	-0.18	-0.09

[1] Chemicalize (https://chemicalize.com)

In addition, 46 TOrCs frequently detected in municipal wastewater were also investigated **(Table S1)**. A stock solution was prepared from analytical grade chemicals to contain 100 µg/mL of each of these compounds in pure methanol. In this study, TOrCs are categorised as neutral (≤50% ionised) or charged (≥50% ionised) compounds at pH 6.5 which is the feed solution pH in this study **(Table S1)**. These charged TOrCs can be further classified as positively or negatively charged or zwitterions. Neutral TOrCs can also be further classified as hydrophilic (log $D < 2$) or hydrophobic (log $D \geq 2$) according to their Log D value at pH 6.5 (log D is the logarithm base 10 of the apparent water-octanol distribution coefficients at a specific pH).[28, 29]

A secondary effluent was further treated by ultrafiltration (UF) and used for all laboratory and pilot scale RO experiments in this study. This UF-treated secondary effluent had a pH of 6.6±0.1.

2.2 Laboratory-scale RO system and experiments

Two commercial thin-film composite polyamide RO membranes – namely ESPA2 and Hydrapro®501 – were provided by Hydranautics (Oceanside, CA, USA). The ESPA2 is a low pressure membrane for water reuse applications. The HYDRApro®501 (HYDRA) is designed for industrial applications where the feed stream can be at a high temperature (condensate water) or contain proteins (e.g. for protein recovery), surfactants (e.g. laundry wastewater recycling), and even aggressive chemicals (e.g. chemical recovery).

The rejection of four *N*-nitrosamines and 1,4-dioxane by RO was evaluated using a laboratory-scale RO system **(Fig. S2)**. The RO system was operated by recirculating the feedwater and permeate at a permeate flux of 20 L/m^{2}h, 40 mL/min cross-flow rate, and 20 °C feedwater temperature. The concentration of NDMA and 1,4-dioxane in the RO feedwater were 500 ng/L and 500 µg/L, respectively. Prior to feed and permeate sample collection (in amber vials) for TOrC analysis, the system was stabilised for at least 1 h. Sample volumes for *N*-nitrosamines and 1,4-dioxane were 1.5 and 100 mL, respectively.

2.3 Pilot-scale system and experiments

Pilot validation was performed using an RO system equipped with one 4-in. spiral-wound ESPA2 element (Hydranautics, Oceanside, CA, USA) **(Fig. S3)**. This element contained 7.43 m^2 of membrane. The RO system was operated at a permeate flux of 20 L/m^2h and system recovery of 20%.

Two separate pilot-scale experiments were conducted. The first experiment was conducted using a UF-treated wastewater containing NDMA and 1,4-dioxane for 7.5 h. It has been established that the rejection of hydrophilic and neutral chemicals such as *N*-nitrosamines reach a steady state condition within 1 h;[30] thus, the impact of the short experimental period on their rejection is negligible. From 0 to 2 h, the concentration of NDMA and 1,4-dioxane in the RO feedwater was incrementally increased from zero to about 150 ng/L and 100 µg/L, respectively. The feedwater temperature was adjusted between 15 and 33 °C. RO feedwater and RO permeate were continuously fed to two separate online NDMA analysers. The second experiment was performed using a UF-treated wastewater containing 46 TOrCs. The system was operated over 46 h prior to the sample collection to ensure that their adsorption had reached the steady state condition, and thus, minimise the effect of adsorption of hydrophobic TOrCs to RO membrane on their rejection. TOrCs were introduced to the feedwater to obtain 45 µ/L of each compound. The feedwater temperature was adjusted at 20 °C. RO feed and permeate samples were collected in 500 mL glass bottles for the analysis of TOrCs.

2.4 Analytical techniques

The *N*-nitrosamine concentration was determined by HPLC-PR-CL.[31] Sample volumes into the HPLC-PR-CL were 20 μL for UF-treated wastewater (i.e. RO feedwater) and 200 μL for RO permeate. For samples collected during laboratory-scale experiments, an auto-sampler was used for the *N*-nitrosamine analysis. The method detection limits (MDLs) of NDMA, NMEA, NPYR and NMOR for a 200 μL injection volume were 0.3, 0.7, 1.4 and 0.8 ng/L, respectively. The MDLs of NDMA, NMEA, NPYR and NMOR for a 20 μL injection volume were 2.7, 6.3, 7.7 and 11.8 ng/L, respectively. For pilot-scale experiments, two online NDMA monitoring systems were configured with two HPLC-PR-CL instruments, each of which was equipped with a six-port valve **(Fig. S4)**.[27] Concentrations of 1,4-dioxane were determined by headspace (HS) solid-phase micro-extraction followed by gas chromatography (GC) and mass spectrometry (MS) using an Agilent G1888/6890/5973 HS-GC-MS with a VF-624 ms column (Agilent Technologies, Palo Alto, CA, USA). 1,4-Dioxane-d8 was used as the surrogate standard. The detection limits of 1,4-dioxane was 2 μg/L. Concentrations of TOrCs were determined using a method previously reported in literature.[32] This method involves solid phase extraction followed by analytical quantification using an ultra-performance liquid chromatography equipped with atmospheric pressure ionization and tandem mass spectrometer.

3 RESULTS AND DISCUSSION

3.1 Role of molecular size for the rejection of N-nitrosamines and 1,4-dioxane

The four *N*-nitrosamines investigated here and 1,4-dioxane are neutral and hydrophilic. Thus, their rejection was governed mostly by size interaction.[28] As expected, the rejection of these neutral and hydrophilic *N*-nitrosamines by both ESPA2 and HYDRA RO membranes increased with increasing molecular weight. It is noteworthy that 1,4-dioxane rejection (i.e. >98%) was markedly higher than that of NDMA **(Fig. 1a)**. Indeed, 1,4-dioxane rejection by the HYDRA and ESPA2 RO membranes (98 and 99%, respectively) was higher than NMEA rejection (which is also better rejected by RO than NDMA) **(Fig. 1a)** despite their identical molecular weight (88.1 Da) **(Table 1)**. Our results are consistent with a previous study by Schoonenberg Kegel et al.[33] who also reported higher rejection of 1,4-dioxane (96%) than that of NDMA (74%) by an RO membrane.

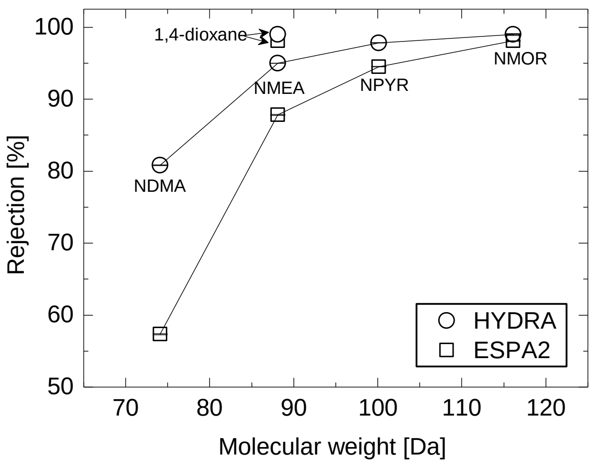


Fig. 1 – Rejection of 1,4-dioxane and four *N*-nitrosamines by RO membranes as a function of their molecular weight at the laboratory scale (permeate flux = 20 L/m^2h, feed temperature = 20.0 ± 0.1 ºC). Values reported here are the average and ranges of duplicate analytical results.

3.2 Online monitoring of NDMA for 1,4-dioxane removal

The potential of online monitoring of NDMA as a surrogate indicator for 1,4-dioxane rejection by RO was evaluated at the pilot scale by identifying the correlation between their rejection at various feedwater temperatures. In response to the changes in feedwater temperature between 15 to 33 °C, in the RO permeate, conductivity increased from 10 to 18 µS/cm, NDMA concentration increased from 85 to 123 ng/L, and 1,4-dioxane concentration increased from 2 to 11 µg/L **(Fig. 2)**. The increase in solute permeation due to increasing temperature led to a decrease in the rejection of conductivity, NDMA and 1,4-dioxane from 98.8% to 97.9%, from 29% to 16% and from 95% to 88%, respectively.

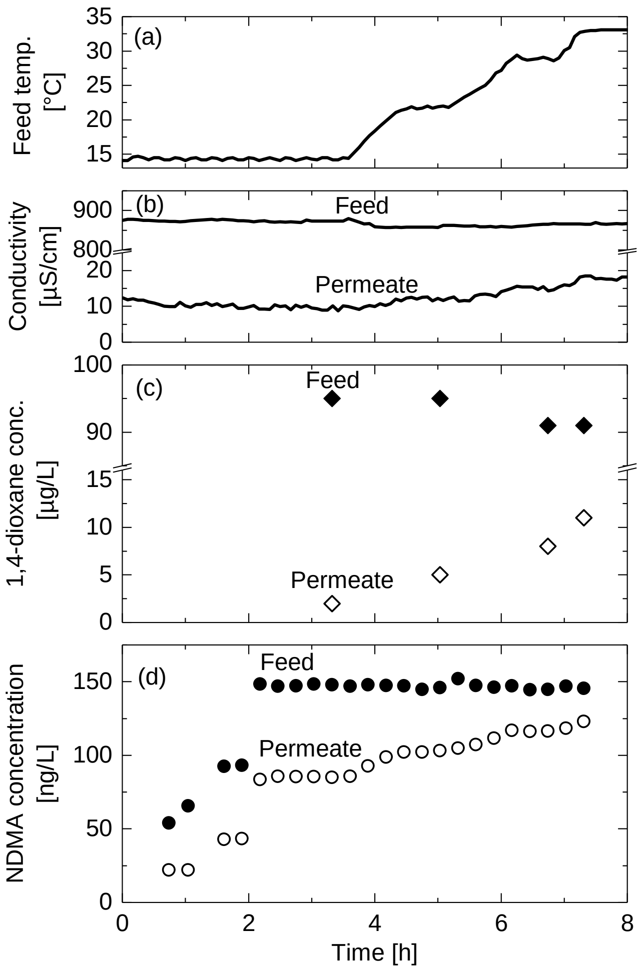


Fig. 2 – The effects of changes in (a) feed temperature on (b) conductivity, (c) 1,4-dioxane and (d) NDMA concentrations during the system operation using the UF-treated wastewater by ESPA2 RO membrane at the pilot scale (permeate flux = 20 L/m^{2}h).

Data from **Fig. 2** were also used to evaluated the correlation between the rejections of 1,4-dioxane and NDMA as well as conductivity as potential surrogate indictors. Pilot-scale data show a strong correlation (R^2 = 0.96) between conductivity and 1,4-dioxane rejection **(Fig. 3a)**. Nevertheless, conductivity rejection only varied in a very narrow range (97.9–98.8%), which was much narrower than the range of changes in 1,4-dioxane rejection (88–98%). A high correlation (R^2 = 0.92) was also obtained between NDMA rejection and 1,4-dioxane rejection **(Fig. 3b)**. The variation in NDMA rejection was over a broad range (16–43%) when 1,4-dioxane rejection varied from 88% to 98%. Results in **Fig. 3** suggest that, compared to conductivity, NDMA is a more sensitive surrogate indictor, one that can adequately indicate changes in separation performance due to variation in operating conditions. By contrast, conductivity rejection is not significantly affected by operating conditions. The successful pilot-scale demonstration confirms potential for using online monitoring of NDMA rejection to continuously ensure 1,4-dioxane rejection by RO for potable water reuse. However, further validations focusing on the effect of membrane variety (e.g. high rejection RO membranes) and long-term changes (e.g. membrane fouling, chemical cleaning and membrane aging) are still necessary prior to the implementation in the full scale.

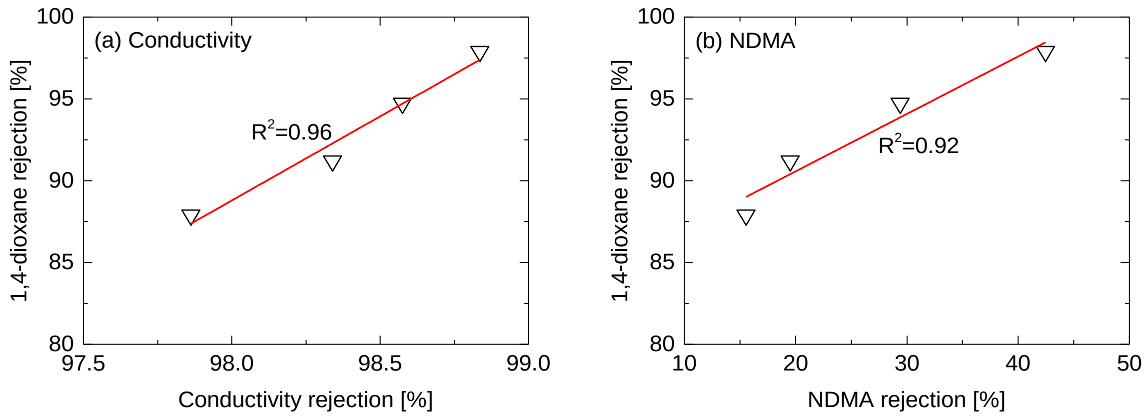


Fig. 3 – Correlation between 1,4-dioxane rejection and (a) conductivity rejection and (b) NDMA rejection by ESPA2 RO membrane at pilot-scale operation.

3.3 Online monitoring of NDMA for other 46 TOrCs

In addition to other *N*-nitrosamines and 1,4-dioxane, the potential use of online monitoring of NDMA as a surrogate indicator for TOrCs was evaluated by comparing the rejection of NDMA and that of 46 TOrCs at pilot-scale operation **(Fig. S5)**. Similar to the results reported in **Fig. 2**, the rejection of neutral TOrCs at 20 °C increased with increasing molecular weight **(Fig. 4a)**, indicating that their rejection was mainly governed by a size exclusion mechanism.

A similar trend in TOrC rejection was observed at an elevated feed temperature of 30 °C **(Fig. S6)**. A plot of the "minimum projection area", which is the area of the compound projected with the minimum plane of its circular disk **(Fig. S7)**, revealed a better correlation in rejection for hydrophilic TOrCs **(Fig. 4b)**. It is clear that a minimum projection area of approximately 20 $Å^2$ is the critical boundary for determining the permeation of TOrCs through the ESPA2 RO membrane.

Among all TOrCs investigated here, NDMA has the lowest rejection by RO; thus, NDMA is a conservative surrogate indicator. All neutral TOrCs were rejected at greater than 98% with only a few exceptions. These exceptions included one small hydrophilic TOrC (acetaminophen, 94%) and two hydrophobic TOrCs (triclosan, 92%; and triclocarban, 68%). Acetaminophen was the smallest pharmaceutical selected in the study; thus, it is reasonable to attribute the low rejection to a molecular size interaction. The low rejection of triclosan and triclocarban could be due to their adsorption to the polymeric RO membrane surface. Triclosan and triclocarban are relatively large in molecular size (MW = 290 and 316 Da, respectively) but are also very hydrophobic (Log D = 4.93 and 4.95, respectively) compared to all other TOrCs selected here. Hydrophobic interaction between these compounds and the membrane polymeric matrix can lower their rejection.[34, 35] Due to adsorption, these chemicals can accumulate at the membrane surface, and subsequently result in more diffusion through the membrane active skin layer. The low rejection of hydrophobic TrOCs has been reported with polyamide RO membranes.[36-39] Thus, it is important to include these two TOrCs when validating a surrogate indicator for TOrC rejection.

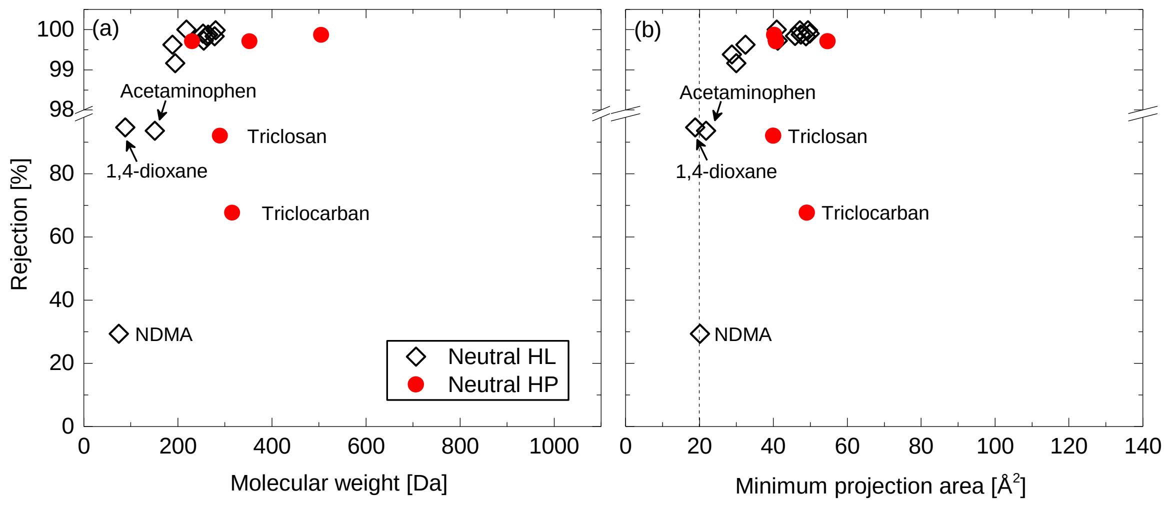


Fig. 4 – Rejection of NDMA, 1,4-dioxane and 17 neutral TOrCs by ESPA2 RO membrane as a function of their (a) molecular weight and (b) minimum projection area at the pilot scale treatment of UF-treated wastewater (permeate flux = 20 L/m^{2}h, feed temperature = 20–22 ºC).

It has been well demonstrated in the literature that the rejection of ionised compounds are well rejected by RO membranes.[28, 40] As expected, the rejection of most of the charged TOrCs by the ESPA2 RO membrane was high (>98 and >97%) at 20 and 30°C, respectively **(Fig. 5 and Fig. S8)**. Nevertheless, the rejection of one positively charged compound (ethenzamide, 96% at 20 °C) appeared to be lower than the other charged TOrCs presumably due to its small size (MW = 165 Da and minimum projection area = 30 Å) and positive charge. In fact, the rejection of positively charged TOrCs was generally lower than that of negatively charged TOrCs. Despite of the low rejection of some TOrCs, the low rejection can generally be explained by mechanisms related to size, charge or hydrophobic interactions. More importantly, the results here confirmed that NDMA is a conservative surrogate indicator for monitoring the rejection of all TOrCs selected in this study.

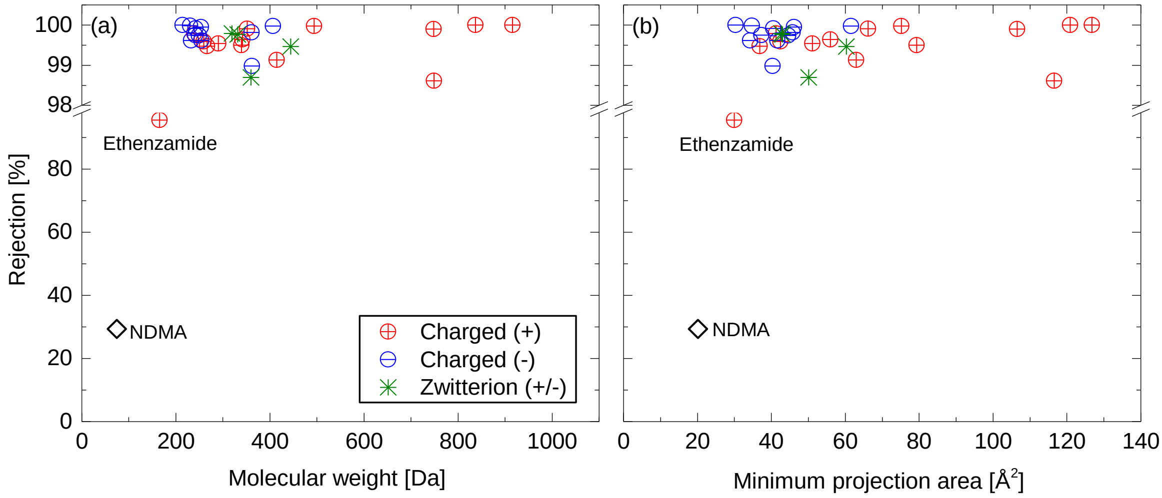


Fig. 5 – Rejection of NDMA and 29 charged TOrCs by ESPA2 RO membrane as a function of their (a) molecular weight and (b) minimum projection area at the pilot scale treatment of UF-treated wastewater (permeate flux = 20 L/m^{2}h, feed temperature = 20–22 ºC).

3.4 Implication to full-scale operation

NDMA meets all three key attributes for a good surrogate indicator for monitoring TOrC rejection by RO membranes. NDMA is ubiquitous in reclaimed water used as the feed solution to RO at well above the instrument detection limit (1–2 ng/L).[14, 41] Recent analytical development has resulted in a reliable and affordable technique for online NDMA monitoring at concentrations relevant to their occurrence in reclaimed water. This can allow for monitoring NDMA online to continuously ensure the removal of 1,4-dixoane, *N*-nitrosamines, and other TOrCs by RO during potable water reuse. However, this study used high NDMA concentrations (about 150 ng/L) in the RO feedwater; thus, further validation using reclaimed wastewater (with NDMA concentration in the typical range of 20–30 ng/L) at an advanced water treatment plant will be the scope of our future study.

4 CONCLUSION

Results from this study demonstrate the potential of using online monitoring of NDMA to ensure the removal of other TOrCs including 1,4-dixoane by RO in potable reuse applications.

A strong correlation between NDMA and 1,4-dixoane rejections was validated. In addition, NDMA rejection was lower than all TOrCs investigated in this study. In other words, a conservative result can be expected for NDMA as a surrogate indicator. Using NDMA as a surrogate indicator for monitoring the rejection of other TOrCs can allow water utilities to provide a higher removal credit for difficult-to-analyse compounds such as 1,4-dioxane. This study demonstrated that NDMA rejection by the HYDRA RO membrane at 81%, which could also provide the minimum rejection credit of 81% for 1,4-dioxane. This result is significant as the current removal credit by RO for 1,4-dixoane is zero since 1,4-dixoane does not occur continuously in the RO feed and it cannot be artificially introduced to the feed for validation.

5 ACKNOWLEDGEMENT

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