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Special issue:
**Sustainable water management
for green infrastructure**

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Simultaneous removal of dissolved organic matter and bromide from drinking water source by anion exchange resins for controlling disinfection by-products

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ABSTRACT

Anion exchange resins (AERs) with different properties were evaluated for their ability to remove dissolved organic matter (DOM) and bromide, and to reduce disinfection by-product (DBP) formation potentials of water collected from a eutrophic surface water source in Japan. DOM and bromide were simultaneously removed by all selected AERs in batch adsorption experiments. A polyacrylic magnetic ion exchange resin (MIEX[®]) showed faster dissolved organic carbon (DOC) removal than other AERs because it had the smallest resin bead size. Aromatic DOM fractions with molecular weight larger than 1600 Da and fluorescent organic fractions of fulvic acid- and humic acid-like compounds were efficiently removed by all AERs. Polystyrene AERs were more effective in bromide removal than polyacrylic AERs. This result implied that the properties of AERs, i.e. material and resin size, influenced not only DOM removal but also bromide removal efficiency. MIEX[®] showed significant chlorinated DBP removal because it had the highest DOC removal within 30 min, whereas polystyrene AERs efficiently removed brominated DBPs, especially brominated trihalomethane species. The results suggested that, depending on source water DOM and bromide concentration, selecting a suitable AER is a key factor in effective control of chlorinated and brominated DBPs in drinking water.

Introduction

Chlorination is one of the most widely used disinfection process for inactivating microbes in water treatment plants (WTPs). Chlorine also reacts with dissolved organic matter (DOM) and bromide present in water, leading to formation of disinfection by-products (DBPs) such as trihalomethanes (THMs) and haloacetic acids (HAAs) (Liu et al., 2011; Zhang et al., 2011; Tian et al., 2013). The most effective way to control DBP formation is to remove precursors before chlorination. Coagulation, activated carbon

adsorption, ultrafiltration and nanofiltration membrane processes target DOM removal, but these processes could not effectively remove bromide in previous studies (Singer and Bilyk, 2002; Ates et al., 2009; Kristiana et al., 2011; Xu et al., 2013), causing an increase in the bromide to dissolved organic carbon (DOC) ratio (Br/DOC), and preferential formation of brominated DBPs in treated waters. Although reverse osmosis membrane can achieve the simultaneous removal of DOM and bromide in a well-designed one unit process, the implementation of this technology in WTPs is usually restricted by high cost and complicated operation. The simultaneous removal of DOM and bromide prior to chlorination process is of crucial importance to control DBP formation and to simplify treatment process.

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The use of anion exchange resin (AER) is an alternative technology to simultaneously remove DBP precursors and bromide (Hsu and Singer, 2010; Walker and Boyer, 2011; Xu et al., 2013) in WTPs owing to its benefits of being easy-to-handle, high efficiency, and low cost and having a simple regenerating practice using a chloride salt (Neale and Schfer, 2009). The AERs used in WTPs are either polystyrene or polyacrylic AERs. Polystyrene AERs are more hydrophobic than polyacrylic AERs; as a result, polyacrylic AERs tend to have a higher water content and more open structure (Boyer and Singer, 2008). The simultaneous removal of DOM and bromide in water solution by AERs has been investigated by some researchers. Hsu and Singer (2010) investigated DOM and bromide removal in simulated surface waters and found that polyacrylic AER, i.e. MIEX[®], was more effective in removing DOM, whereas polystyrene resins were more effective for bromide removal. Walker and Boyer (2011) and Xu et al. (2013) investigated the simultaneous removal of DOM and bromide from surface water by MIEX[®], which has the potential to remove both DOM and bromide, but the relationship between reductions of DOM, bromide and THM species had not been investigated. However, there has been no study on the effects of various properties of AERs on the removal efficiencies for DOM and bromide and the reduction of DBP formation potential in surface water. Therefore, the objectives of this study were: (1) to evaluate the effect of AER properties on bromide and DOM removal from surface water, (2) to evaluate the performance of AERs on the reduction of the DBP formation potential. To our knowledge, this is the first study on the influence of AER properties on the removal of DOM, bromide and associated THM species after chlorination. The results of this study were aimed to be used to design an AER process for the effective removal of bromide and DOM to control the occurrence of DBPs in drinking water.

1 Materials and methods

1.1 Water sample

The water sample was collected in November 2011 from Lake Inba-numa in Chiba Prefecture, Japan, which is a representative eutrophic lake in Japan and a source for

water supply. The water sample was collected in a cleaned plastic container and then delivered to the laboratory. After filtration through 0.45 µm hydrophilic PTFE membrane filters (Millipore, JHWP09025), the filtered water was stored in brown glass bottles at 4°C to minimize changes of character, which is hereafter referred to as raw water and used in all the experiments. The water quality of the raw water are as the following: pH 9.1, DOC concentration of 2.71 mg/L, ultraviolet absorbance at 254 nm (UV₂₅₄) of 0.062 cm⁻¹, specific ultraviolet absorbance at 254 nm (SUVA₂₅₄) of 2.28 L/(m·mg), bromide concentration of 0.125 mg/L, and chloride concentration of 23 mg/L. Due to algal bloom, the water pH was alkaline and DOC was moderately high, while a rather high concentration of bromide may be due to anthropogenic pollution, because the Br and Cl ratio in the raw water (Br/Cl = 0.0054) was higher than in sea water (Br/Cl = 0.0035) (Stumm and Morgan, 1996).

1.2 Anion exchange resins

The characteristics of the five AERs used in this study are shown in **Table 1**. The AERs were selected based on their different structures (i.e. gel or macroporous) and materials (i.e. polyacrylic or polystyrene) for comparison purposes. To remove impurities from as-received AERs, they were preconditioned according to published procedures (Humbert et al., 2005; Phetrak et al., 2012), except for MIEX[®]. MIEX[®] was rinsed with Milli-Q water to wash away the fine particles and to confirm that there was no release of organic matter. All AERs, conditioned in the chloride form, were stored in Milli-Q water until use.

1.3 Adsorption of DOM and bromide by AERs

Kinetic exchange experiments were conducted based on the bottle-point method. A preconditioned AER was placed in each of a set of 100 mL flasks containing 60 mL of raw water to achieve an AER dose of 2 g/L. The adsorption experiments were conducted at 25°C with horizontal shaking at a rate of 200 r/min. The sampling time was set at 0, 5, 10, 15, 30, 60 and 120 min. At the end of each contact time, water samples were filtered through a pre-rinsed 0.45 µm PTFE filter (Dismic 45HP, Advantec, Japan) to remove AERs before DOM and anion analyses. Each adsorption experiment was performed in triplicate. The formation of DBPs after chlorination for 24

Table 1 Characteristics of AERs used in this experiment

Resin	Type	Structure	Material	Particle size (µm)	Capacity (meq/mL)	Manufacturer
IRA400	Type I	Gel	Polystyrene	300	1.40	Rohm & Haas
IRA910	Type II	Macroporous	Polystyrene	460	1.10	Rohm & Haas
MIEX [®]	Type I	Macroporous	Polyacrylic	150–180	0.52	Orica
IRA458	Type I	Gel	Polyacrylic	500	1.25	Rohm & Haas
Purolite	Type I	Macroporous	Polyacrylic	300–1200	0.80	Purolite

hr, such as trihalomethane formation potential (THMFP) and haloacetic acid formation potential (HAAFP), were examined for the AER-treated waters with 30-min contact time.

1.4 Analytical methods

DOC concentration and UV_{254} were measured by a non-purgeable organic carbon method using a TOC-5000A analyzer (Shimadzu Corp., Kyoto, Japan) and UV/Vis spectrophotometer (U-2000, Hitachi, Ltd., Tokyo, Japan), respectively. $SUVA_{254}$ (L/(m·mg)) is an indicator of aromatic moieties in surface water, which is defined as UV_{254} divided by DOC concentration. A Metrohm 861 ion chromatography analyzer (Metrohm, Switzerland) was used to measure the concentrations of anions.

The molecular weight (MW) distribution of DOM was analyzed through the use of a Protein Pak 60A° (Waters Corp., USA) column and LC-10ADvp liquid chromatography pump connected to an SPD-M10Avp diode array detector at 254 nm (Shimadzu Corp., Kyoto, Japan). Details of high-performance size exclusion chromatography (HP-SEC) analysis are given by Phetrak et al. (2012).

Spectrofluorimetric analysis was performed using a spectrofluorimeter (F-4500, Hitachi) equipped with a xenon lamp. Three-dimensional fluorescence excitation-emission matrix (3D F-EEM) spectra were collected at excitation (Ex) and emission (Em) spectra in a wavelength range of 220–550 nm, which can be used to identify fluorescent organic compounds present in water samples. The spectra of Milli-Q water were recorded as the blank.

THMFP and HAAFP were analyzed according to the Japanese Standard Methods for the Examination of Water. Chlorination was performed by adding sodium hypochlorite into samples buffered at $pH\ 7.0 \pm 0.2$, followed by 24 hr incubation in the dark at 20°C. The residual free chlorine was maintained at 1–2 mg/L. The reactions were immediately quenched with sodium ascorbate prior to analysis for THMs and HAAs. Four THMs (chloroform (TCM), dibromochloromethane (DBCM), bromodichloromethane (BDCM) and bromoform (TBM)) were analyzed using a purge and trap concentrator (Aqua PT 5000J, Teledyne Tekmar, OH, USA) coupled to a gas chromatography mass spectrometry system (GC/MS-QP2010 Plus; Shimadzu Corp., Kyoto, Japan) and five HAAs (chloroacetic acid (CAA), dichloroacetic acid (DCAA), trichloroacetic acid (TCAA), bromoacetic acid (BAA) and dibromoacetic acid (DBAA)) were analyzed using liquid-liquid extraction and chemical derivatization, followed by analysis with a HP Hewlett Packard Agilent 6890 GC System gas chromatograph equipped with a Hewlett Packard/Agilent HP 5973 mass selective detector.

2 Results and discussion

2.1 DOM and bromide removals by AERs

The time courses of the reduction of DOM (in terms of DOC and $SUVA_{254}$) are shown in Fig. 1. AERs re-

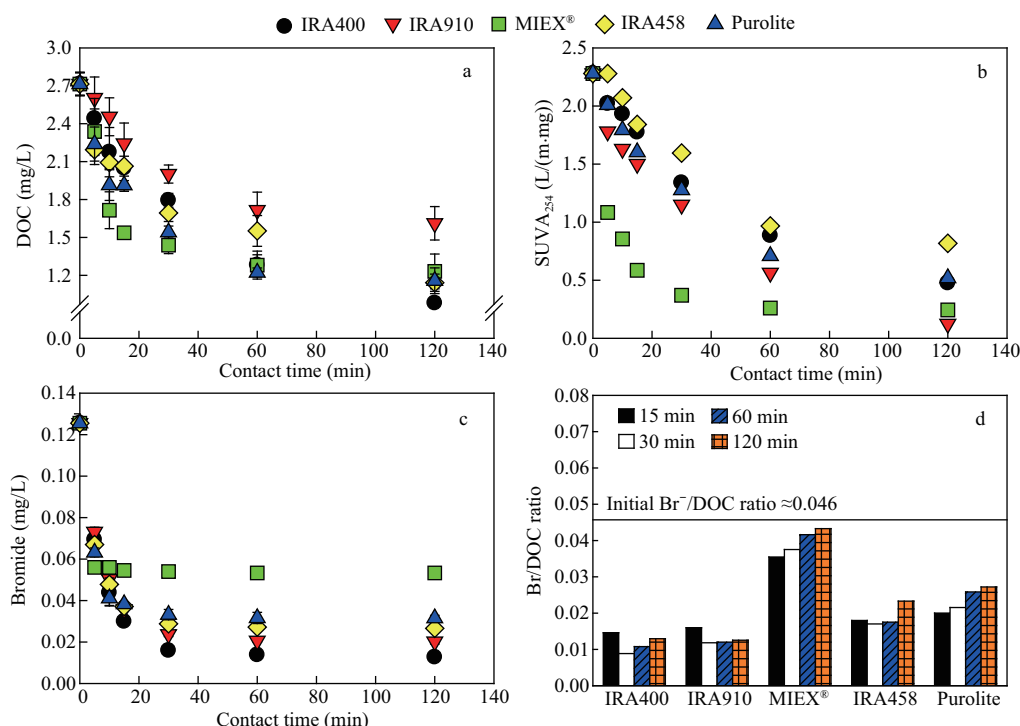


Fig. 1 Reduction of DOM and bromide with contact times. (a) DOC, (b) $SUVA_{254}$, (c) bromide (Br) and (d) Br/DOC ratio.

duced DOC from the initial concentration of 2.71 mg/L to 0.98–1.61 mg/L at 120 min, which corresponded to DOC removal rates of 41%–64% (**Fig. 1a**). The trend of DOC reduction by polystyrene IRA910 was relatively smaller than for other AERs. For example, at 30 min contact time, 2.00 mg/L of DOC remained after treatment by IRA910, whereas it was 1.43–1.80 mg/L in water treated by IRA400, MIEX®, IRA458 and Purolite. This result was similar to the previous result conducted by our group (Phetrak et al., 2012). The distinctively different trends in DOC reduction by AERs could be attributed to the uniqueness of AER properties. MIEX®, having the smallest resin size, exhibited the fastest DOC reduction at 30 min among the other AERs. A smaller resin size provides a larger solid-liquid interface between the AER and surrounding water, resulting in a faster DOC exchange rate (Humbert et al., 2012). Greater UV₂₅₄ reduction rates than DOC reduction rates were observed for all of the tested AERs, which resulted in decreasing trends of SUVA₂₅₄ with contact time (**Fig. 1b**). The decreases in SUVA₂₅₄ indicated that DOM having aromatic moieties, which absorb UVA₂₅₄, was preferentially removed by the AERs. In addition, the SUVA₂₅₄ value of MIEX® decreased markedly from 2.28 L/(m·mg) at start-up to 0.37 L/(m·mg) at 30 min of contact time, which was lower than those of the other AERs, indicating the higher affinity of MIEX® for aromatic moieties than the other AERs (Singer and Bilyk, 2002; Rokicki and Boyer, 2011; Walker and Boyer, 2011; Xu et al., 2013). The preferential aromatic moieties removal by MIEX® was possibly due to the iron oxide incorporated into the resin (Shuang et al., 2013).

The change of bromide concentration by various AERs is also shown in **Fig. 1c**. Bromide reduction rates were apparently faster than DOC reduction rates, leading to the lowest bromide concentrations occurring at about 30 min for all of the AERs. During the course of bromide exchange by AERs, the removal rates varied between 58% and 90% for the AERs. The polystyrene AERs

(IRA400 and IRA910) were more effective in bromide removal (removal rates 84%–90% at 120 min) than the polyacrylic AERs (MIEX®, IRA458, and Purolite; 58%–79%), which is similar to the results reported by Hsu and Singer (2010). Among the polyacrylic AERs, MIEX® showed the lowest reduction of bromide, while MIEX® reached the steady state bromide concentration within the shortest time (5 min) compared to the other AERs. This implied that MIEX® had the highest affinity for DOM not simply from the ion-exchange capacity or the polyacrylic material, but possibly due to its unique structure. Since the DOC removal by MIEX® was the highest but the bromide removal was the lowest, the highest Br/DOC ratio was obtained (**Fig. 1d**). A similar result of a high Br/DOC ratio was found with nanofiltration membranes, which were more effective in removing DOC than bromide (Lainé et al., 1993; Ates et al., 2009). Nonetheless, polystyrene AERs showed smaller Br/DOC ratios in the treated waters than those of polyacrylic AERs because they had greater removal rates for bromide than DOC, which could be useful in controlling the formation of brominated DBPs.

2.2 DOM characterization

DOM in raw water and treated water was characterized by HP-SEC with a UV₂₅₄ detector and 3D F-EEM to better understand the fractions of DOM removed by the AERs. The MW distributions of aromatic DOM present in raw water and after treatment by MIEX® and IRA458 are shown in **Fig. 2**. The chromatograms showed that a majority of aromatic DOM in raw water were in the MW range of 700–4000 Da, corresponding to humic compounds (Jarusutthirak and Amy, 2007). The AER treatment was shown to be an effective method to remove a wide range MW of aromatic DOM, with especially high removal rates for aromatic DOM greater than 1000 Da. However, different abilities of aromatic DOM removal from raw water were observed between MIEX® and IRA458. Aromatic DOM greater than 1600 Da was completely removed by

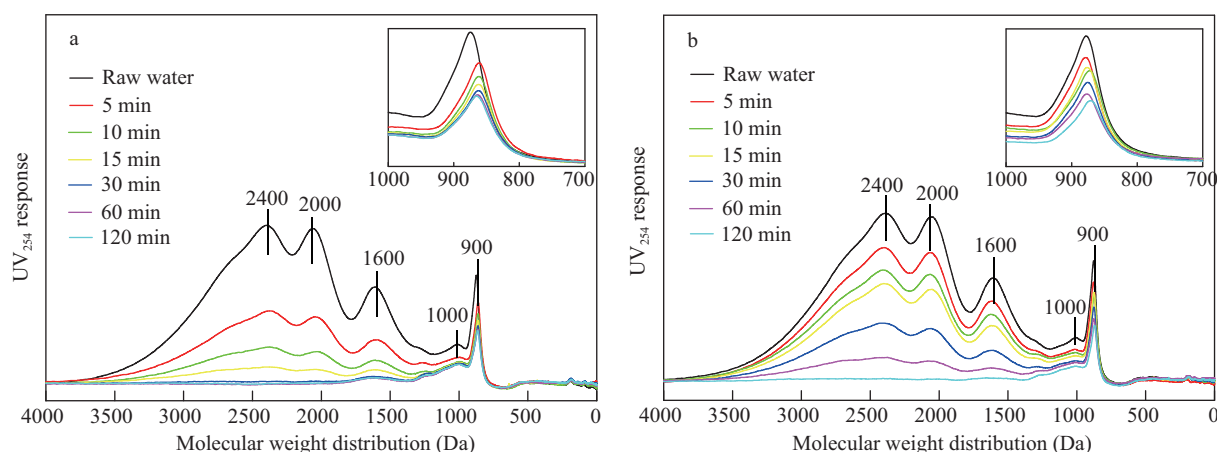


Fig. 2 HP-SEC chromatogram of aromatic DOM presented in raw water and AERs treated water in different contact time at AER dose of 2 g/L. (a) MIEX® and (b) IRA458.

MIEX[®] at 30 min, but this DOM fraction still remained after the 60-min contact time with IRA458. This was explained by the fact that the macroporosity, small resin size and iron oxide content of MIEX[®] provided greater DOM accessibility and more solid-liquid interfaces, resulting in a more rapid removal of aromatic DOM greater 1600 Da than IRA458 and other AERs (data not presented). However, aromatic DOM smaller than 1000 Da remained after treatment by either MIEX[®] or IRA458, indicating that DOM in this smaller-MW fraction was not easily removed by the AERs, which is consistent with the finding by Humbert et al. (2005).

Figure 3 shows the 3D F-EEM of raw water and EEM volumes (accumulated fluorescence intensities) of the individual regions in 3D F-EEM spectra of raw water and AER treated waters, which was computed using the fluorescence region integration proposed by Chen et al. (2003). The position of Ex-Em of each region followed Chen et al. (2003) and Nguyen and Roddick (2013). Region I (Ex/Em: 220–270/280–330) and region II (Ex/Em: 220–270/330–380) corresponded to aromatic proteins such as tyrosine- and tryptophan-related compounds, respectively. Region III (Ex/Em: 220–270/380–550), region IV (Ex/Em: 270–440/280–380), and region V (Ex/Em: 270–440/380–550) corresponded to fulvic acid-like compounds, soluble microbial products (SMPs) and humic acid-like compounds, respectively. The result demonstrated that the raw water contained predominantly humic and fulvic acid-like compounds, which were efficiently removed by all of the tested AERs. However, all AERs showed marginal removal of tyrosine, tryptophan and SMPs, which was consistent with findings reported by Cornelissen et al. (2008) and Nguyen and Roddick (2013). The reasonable explanation for higher removal of fulvic and humic acid-like compounds by AERs is that these compounds are negatively charged at basic pH (pH = 9) and thus can be easily removed by ion exchange mechanism. The greatest removal of humic acid-like compounds (71%) and fulvic acid-like compounds

(55%) were found for MIEX[®] at a dose of 2 g/L for 30 min, while they were also removed by other AERs in the range of 43%–56% for humic acid-like compounds and 32%–43% for fulvic acid-like compounds.

2.3 Control of disinfection by-products by AERs

The concentrations of THM and HAA species in the raw water and treated water by AERs at the dose of 2 g/L for 30 min are shown in **Table 2**. The total THMFPP in raw water was higher than the total HAAFP, which was in agreement with results reported by Marhaba and Van (2000) and Ates et al. (2009). Chloroform (47%) was the dominant THM species in raw water, followed by BDCM (35%), DBCM (17%) and TBM (1%). For HAAFP in raw water, TCAA was the most dominant species (56%), followed by DCAA (33%), whereas DBAA, CAA and BAA were found in small proportion lower than 7%. This indicated that DBP precursors in the raw water contributed to the formation of brominated THM species rather than the formation of brominated HAAs. This is consistent with the work by Ates et al. (2009) using reservoir water. In addition, the relatively large proportion of brominated THM species of raw water can be explained by the relatively high concentration of bromide in Lake Inba numa. All of the tested AERs showed efficient total THM removal at 52%–77% and total HAA removal of 47%–89%. The highest total THMFPP and HAAFP removals were achieved by MIEX[®] because it had the greatest DOC removal at 30 min compared with the other AERs. MIEX[®] efficiently removed chlorinated THMs, whereas the removal of brominated THMs (i.e. DBCM and BDCM) was lower than those of the other AERs because of the low removal rate of bromide by MIEX[®]. Polyacrylic resins such as IRA458 and Purolite were capable of removing both chlorinated THMs (36%–42%) and brominated THMs (DBCM: 62%–77%; BDCM: 67%–69%). Interestingly, polystyrene AERs showed higher brominated THM removal rates (85%–92% for DBCM and 78%–81% for BDCM) than chlorinated

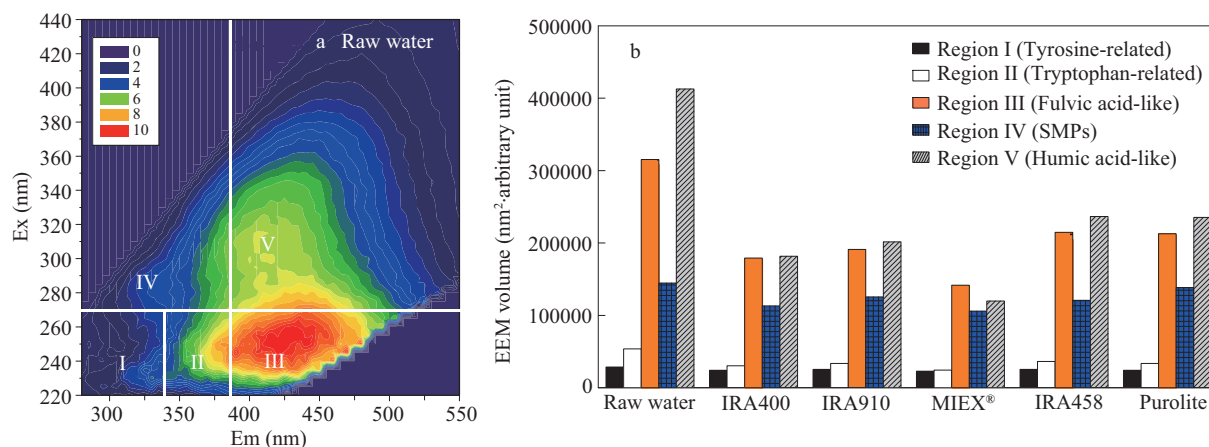


Fig. 3 (a) 3D F-EEM spectra of raw water and (b) fluorescent EEM volumes of raw water and treated waters by AERs at the dose of 2 g/L for 30 min.

Table 2 Formation of THM and HAA species in raw water and treated waters from AERs

Water sample	THM species (μg/L)				HAA species (μg/L)				
	TCM	DBCM	BDCM	TBM	CAA	DCAA	TCAA	BAA	DBAA
Raw water	36	13	27	1	2	15	25	0	3
DBP _{raw water} (%)	47	17	35	1	4	33	56	0	7
AERs treated water									
IRA400	19	1	5	0	0	8	13	0	0
IRA910	21	2	6	0	0	8	15	0	0
MIEX [®]	7	4	7	0	0	3	2	0	0
IRA458	23	3	9	0	0	8	16	0	0
Purolite	21	5	11	0	0	7	12	0	0

AER dose of 2 g/L and 30 min contact time.

THM removal rates (42%–47%) due to their greater bromide than DOC removal rates. MIEX[®] was also effective in the removal of chlorinated HAAs including DCAA (80%) and TCAA (92%), whereas other AERs showed removal rates of DCAA and TCAA of 47%–53% and 36%–52%, respectively. In addition, CAA and DBAA, of the HAA species, were not found after treatment by all the AERs. The findings of this study suggest that polystyrene AERs removed bromide to a greater extent than DOC, resulting in a lower Br/DOC ratio and decreasing the relative occurrence of brominated THMs, while MIEX[®] was effective in removing DOC, showing a substantial removal of chlorinated DBPs.

3 Conclusions

This study reported that differences in AER properties affected the removal of DOM and bromide from surface water, and consequently the reduction of THM species after chlorination. A polyacrylic macropore-type resin, MIEX[®], showed the most effective removal of DOC, possibly due to the small bead size, but polystyrene AERs were more effective in bromide removal than the polyacrylic resins. All selected AERs showed preferential removal of DOM with MW greater than 1600 Da and of fulvic acid-like and humic acid-like compounds of the fluorescent DOM fractions. The AER treatments significantly lowered the DBPFP levels, with total THMFP removal rates of 52%–77% and total HAAFP removal rates of 47%–89%. As for MIEX[®], although bromide removal was relatively lower than those by other AERs, the highest DOC removal at 30 min led to a significant reduction of chlorinated DBPs. On the other hand, polystyrene AERs decreased the relative occurrence of brominated THM species in raw water. The results of this study suggest that AERs are able to remove both DOM and bromide simultaneously. Depending on the DOM characteristics and bromide concentration of source waters, we should select a suitable AER to meet the goal of both total and

brominated DBPs in treated water.

Acknowledgments

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