

# Impact of a severe rain event on C- and N-DBP precursor removal using IEX

K. Doederer, Z. Ilieva and J. Keller

## ABSTRACT

During disinfection, dissolved organic matter (DOM) is the major precursor to form disinfection by-products (DBPs), which may be of potential human health concern. Previous research focused on waters of continental climates and less on subtropical environments. However, water sources in subtropical climates are regularly impacted by major rain events during the summer months. This study evaluated the C- and N-DBP precursor removal capacity of two conventional ion exchange (IEX) resins and one magnetic ion exchange (MIEX) resin with a raw water at normal conditions and impacted by a heavy rain event. The rain event introduced 3 mg C/L total organic carbon (TOC) comprised mainly of low to medium molecular weight organics. All three resins were able to remove TOC and DBP precursors (>66%) but being less efficient in reducing turbidity (3–48%) and colour (9–24%). The resin with the smallest bead size was affected the most by the increased medium MW DOM loading resulting in DOM and C-DBP precursor removal performance losses of 10% and 22%. When applied as a pre-treatment for coagulation, MIEX was more efficient in DBP precursor control than coagulation in addressing the additional organic and DBP precursor loading after a heavy rain event.

**Key words** | C-DBP, N-DBP, IEX, MIEX

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## INTRODUCTION

Dissolved organic matter (DOM) present in drinking water sources can negatively impact the water quality by introducing colour, taste and odor, promoting biological growth in distribution systems and leading to increased disinfectant demand. The reaction between DOM and a disinfectant, e.g. chlorine or chloramines, results in the formation of potentially harmful disinfection by-products (DBPs). DOM in the water originates from soils and plants of the terrestrial watershed or from biological growth and decomposition of algae and macrophytes within lakes or reservoirs (Dotson *et al.* 2014). DOM has been shown to contain around 50% dissolved organic carbon (DOC) by weight and much less dissolved organic nitrogen (DON), typically ranging from 0.5% to 10% by weight (Westerhoff & Mash 2002). While DOC has been identified to be the major precursor for carbonaceous DBPs (C-DBPs), DON can be an important

precursor for the nitrogenated DBPs (N-DBPs). N-DBPs are among the most geno- and cyto-toxic DBPs identified in drinking water research to date (Plewa *et al.* 2008).

In drinking water treatment, ion exchange (IEX), in particular the magnetic ion exchange (MIEX) process, has increasingly been used for DBP precursor control in North America, Europe, and Australia. In contrast to these continental climates, DBP precursor loading represented by DOC and DON concentrations was found to be lower as compared to surface water sources in a subtropical climate (Lyon *et al.* 2014). Furthermore, severe rain events occurring in the subtropics in the summer months can lead to heavy runoffs, resulting in a shift in both quantity and composition of DOM in the raw water.

The aim of this study was to provide a view on the treatment efficiency of ion exchange and its combination with

coagulation on C- and N-DBP precursor removal while removing DOM from a sub-tropical water source under (i) normal and (ii) heavy rain affected conditions. MIEX has previously been shown to be an efficient pre-treatment technology to remove DOM compared to conventional treatment by means of coagulation and filtration (Mergen *et al.* 2008). The combination of MIEX and coagulation incorporates the benefit of MIEX preferentially removing organics of low and high molecular weight (MW) and coagulation targeting high MW organics and turbidity. Although some studies have investigated combined treatment for trihalomethane (THM) and haloacetic acid (HAA) precursor removal in surface waters (Chow *et al.* 2002; Drikas *et al.* 2003; Fabris *et al.* 2008), its performance in C- and N-DBP precursor removal on rain impacted sub-tropical surface water has not been well documented.

## MATERIAL AND METHODS

### Water samples

Raw water for this study was collected at the inlet of one water treatment plant located in South East Queensland (SEQ) (Australia). Sampling of the raw surface water occurred after a major rain event, with the water body impacted by a total of 280 mm of rain with 83 mm, 87 mm and 110 mm in the span of three days (20/02–22/02/15). The second water sample was taken 3 months after the rain event (20/05/15), representing ‘normal’ raw water conditions.

### Treatment

#### IEX

The conventional IEX resins were chosen to be macroporous or of gel type, both possessing polyacrylic backbones. The

polyacrylic, as compared to the polystyrenic backbone, was shown to achieve higher DOM removals within gelular resins (Tan *et al.* 2005). Additionally, good DOM removal performances were attributed to the combination of macropores and the increased swelling properties of a resin (Bolto *et al.* 2002) (Table 1).

The optimal resin dose was determined by increasing the dose in increments of 5 mL/L. The optimal resin dose was selected as such when a 5 mL/L increase resulted in less than a 0.3 mg/L decrease in total organic carbon (TOC). The concentration chosen as optimal will be referred to as Point of Diminishing Return (PODR). To simulate full-scale treatment, multiple loading testing was performed for the MIEX resin. An equivalent of 250–1000 bed volumes (BV) of treated water was achieved by the resin doses tested. Although MIEX reacts quickly (<15 min), the contact time was chosen to be 30 minutes due to the slower kinetics of the conventional IEX resins.

#### Combined treatment: MIEX → alum

Combined treatment was performed, coagulating MIEX pre-treated water using coagulant doses of 20 and 40 mg/L as  $\text{Al}_2(\text{SO}_4)_3 \cdot 14\text{H}_2\text{O}$ . Two litres of MIEX treated water dosed with alum were stirred at 100 rpm. After 1 minute of rapid mixing, the stirrer speed was reduced to 20 rpm for 15 minutes followed by 30 minutes of settling time.

#### DBP precursor removal

DBP precursor removal was tested by applying free chlorine to the treated water, buffered with 1 mM phosphate buffer at pH 7. Free chlorine was dosed to achieve a 2 mg/L as  $\text{Cl}_2$  residual after 24 hours of reaction time. Samples were incubated headspace free, in the dark at room temperature. The chlorine demand was determined in preliminary tests.

**Table 1** | Properties of tested resins as per manufacturer. NA = not available

| Resin    | Type        | Structure   | Size ( $\mu\text{m}$ ) | Capacity (meq/mL) | Water content (%) |
|----------|-------------|-------------|------------------------|-------------------|-------------------|
| IEX a    | Gel         | Polyacrylic | 550                    | 1.25              | 57–64             |
| IRA-958  | Macroporous | Polyacrylic | 630                    | >0.8              | 66–72             |
| MIEX-DOC | Macroporous | Polyacrylic | 180                    | 0.44              | NA                |

## Analytical methods

Sample bottles were prepared by detergent and acid washed, followed by deionized and MilliQ water rinse prior to oven-drying at 105 °C. Preparation of standard dilutions and calibration curves was carried out within the laboratory immediately prior to work-up of the aqueous samples. All reagents were analytical grade. Dilution water, if required, was obtained from a MilliQ Advantage system.

Neutral-extractable DBPs, including four trihalo-methanes (THMs), six iodinated THMs (I-THMs), four haloacetonitriles (HANs), two halo ketones (HKs), two halo-nitromethanes (HNMs), and chloral hydrate (CH) were measured by utilizing a salted microextraction from water with subsequent analysis by gas chromatography and electron capture detection (GC/ECD) (Weinberg *et al.* 2002). Chlorine was quenched when samples were collected using L-ascorbic acid at double stoichiometric concentration.

pH was measured with a Mettler Toledo SevenEasy meter, and turbidity with a HACH portable turbidity meter. Newly acquired formazine turbidity standards were used to calibrate the instrument. UV<sub>254</sub> absorbance and colour at 475 nm were measured by a Cary 50 UV spectrometer in a 1 cm quartz cuvette. Alkalinity was measured according to Standard Method 2320 (APHA 1995). TOC was measured with a Shimadzu TOC-L TOC analyser with a TNM-L total nitrogen analyser unit and ASI-L autosampler. Except for turbidity measurement, all samples were pre-filtered through 1.2 µm glass fibre filters (GFF) before analysis. GFF were pre-rinsed with the sample. GFF filters were chosen instead of the commonly used 0.45 µm membrane filters in order to avoid leaching and/or sorption of organic carbon, which has been found to interfere with UV<sub>254</sub> measurements (Karanfil *et al.* 2003).

## RESULTS AND DISCUSSION

### Effect of heavy rainfall on raw water quality

The raw water used for this study was taken from a surface water reservoir serving a drinking water treatment plant in SEQ, Australia. Due to the subtropical climate in SEQ, heavy rain events mainly occur in the warmer summer months. This study managed to capture a rain event with a

**Table 2** | Water characteristics during normal conditions and after a heavy rain event

| Parameter         | Unit                        | Normal | Rain  |
|-------------------|-----------------------------|--------|-------|
| pH                | (-)                         | 7.5    | 6.6   |
| TOC               | (mg C/L)                    | 13.8   | 16.8  |
| TON               | (mg N/L)                    | 0.54   | 0.70  |
| UV <sub>254</sub> | (cm <sup>-1</sup> )         | 0.745  | 0.819 |
| Alkalinity        | (mg/L as aCO <sub>3</sub> ) | 30     | 4     |
| Bromide           | (mg/L)                      | 0.08   | 0.04  |
| Turbidity         | (NTU)                       | 4      | 26    |
| SUVA              | (L/mg.m)                    | 5.3    | 4.2   |

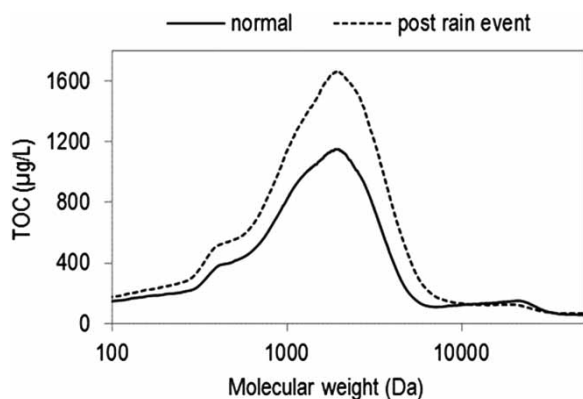
total of 280 mm from February 20–22/2/2015 (Bureau Of Meteorology Australia). Table 2 summarizes the water characteristics of the raw water after a heavy rain event and at ‘normal’ conditions. Basing on historical data process engineers at the WTP suggested a timeframe of generally 2 to 3 months for TOC levels to return to normal levels after a severe rain event. Therefore, the sample representing normal conditions was taken 3 months after the rain event.

Turbidity spiked from 4 to 26 NTU, which is typical for a heavy rain event. On the other hand, a dilution effect was observed for bromide and alkalinity, reducing levels from 0.08 to 0.04 mg/L and from 30 to 4 mg/L as CaCO<sub>3</sub>, respectively. Stormwater runoff from heavy rainfall can introduce organic matter into the water body, in this case leading to an increase of raw water TOC by 3 mg C/L peaking to 17 mg C/L. Using size exclusion chromatography with organic carbon detection (HPLC-SEC-OCD), the TOC was found to be made up of low molecular weight (MW) DOM at 550 Da, medium MW DOM between 700 and 2,800 Da and high MW biopolymers >20 kDa (Figure 1). As seen, the major fraction of TOC introduced by the heavy rain event mainly consisted of medium to low MW organic matter, with the major part comprised of building blocks and humic substances.

Total organic nitrogen (TON), which can act as an important N-DBP precursor, was measured at 0.6 mg/L during normal conditions but increased by 23% after the heavy rain event.

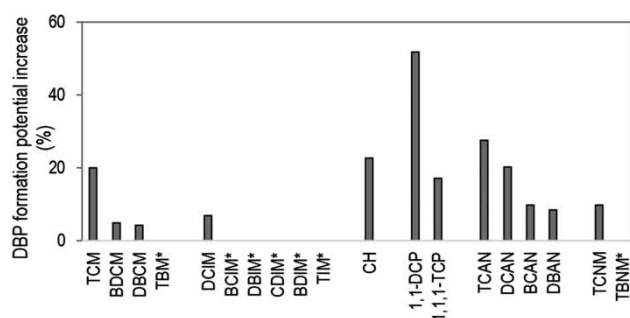
### Impact of rain on DBP precursor presence in raw waters

The impact of the rain event on the introduction of DBP precursors in the raw water was investigated by performing



**Figure 1** | SEC chromatogram of the raw water during normal conditions and after a heavy rain event.

DBP formation potential (FP) tests using free chlorine. The tests revealed that the water post rain event had a higher propensity to form DBPs, i.e. more precursors were available for reaction with chlorine and to be removed by IEX treatment. While DBP FPs were greater for all DBPs in the rain impacted waters, the greatest effect was seen for the solely chlorinated DBPs (Figure 2), including chloroform, chloral hydrate (CH), haloketones (HK) and tri- and dichloroacetonitrile (TCAN, DCAN), except for trichloronitromethane (TCNM), whose precursor load was increased by more than 20%. The increase in C-DBP FP is in accordance with a 16% higher TOC level in the rain impacted water.



**Figure 2** | Percentage increase of DBP formation potentials comparing normal and post rain event raw waters. TCM = trichloromethane, BDCM = bromodichloromethane, DBCM = dibromochloromethane, TBM = tribromomethane, DCIM = dichloriodomethane, BDCM = bromochloriodomethane, DBIM = dibromochloromethane, CDIM = chlorodiodomethane, BDIM = bromodiodomethane, TIM = triiodomethane, CH = chloral hydrate, 1,1-DCP = 1,1-dichloropropanone, 1,1,1-TCP = trichloropropanone, TCAN = trichloroacetonitrile, DCAN = dichloroacetonitrile, BCAN = bromochloroacetonitrile, DBAN = dibromoacetonitrile, TCNM = trichloronitromethane, TBNM = tribromonitromethane, \* = below  $<0.1 \mu\text{g/L}$ .

Organic nitrogen can contain N-DBP precursors, which upon reaction with chlorine may form N-DBPs (Bond *et al.* 2015; Shah & Mitch 2011). N-DBPs investigated included TCAN, DCAN, bromochloroacetonitrile, dibromoacetonitrile, TCNM and tribromonitromethane. The rain event introduced 0.16 mg N/L of TON, which may have contributed to the additional FP for the HANs and TCNM. On the other hand, brominated THM and HAN FPs did increase by less than 10%, which is only half of their chlorinated analogues. While more DOM was introduced by the rain water runoff, the higher water volume resulted in a dilution effect for the bromide present leading to a 50% lower concentration.

### Impact of rain on DOM removal by IEX

DOM removal of the raw waters treated by the two conventional resins and the MIEX-DOC resin at different resin doses was evaluated by means of TOC removal. In the range of 5–20 mL/L, which was an achieved equivalent of 250–1,000 bed volumes (BV), an increase in TOC removal effectiveness with resin dose can be observed for both water conditions (Figure 3). Under normal water conditions, MIEX reached the PODR at a dose of 10 mL/L (500BV) and IEX a and IRA-958 at 20 mL/L (250 BV). All three resins could effectively remove TOC, with IRA-958 reducing the TOC to 4.1 mg C/L, IEX a to 3.4 mg C/L, and MIEX to 3.1 mg C/L. MIEX could remove TOC to a similar extent to the IEX resins at half of the resin dose due to its three times smaller bead size, which provides greater surface area.

However, when treating the raw water after a heavy rain event MIEX, IEX a and IRA-958 reached identical PODRs compared to normal conditions at 10 mL/L and 20 mL/L, respectively. Comparing the results only at the optimal resin doses, the TOC removal performance of IEX a appeared to be unaffected by the change in water matrix. IRA-958 showed a slight improvement from 70% to 77%, while MIEX TOC removal performance decreased from 78% to 68%. The difference in TOC removal performance of the MIEX and IRA-958 might be explained by the greater loading of humic substances in the rain-affected water (Figure 1). Increasing water content of conventional IEX resins has previously been positively correlated with the removal of humic substances (Cornelissen *et al.* 2008). IRA-958

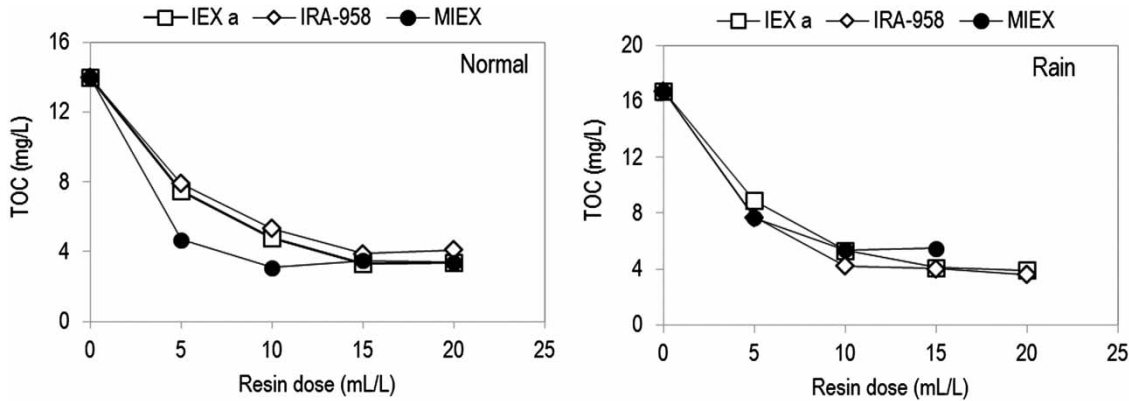


Figure 3 | TOC concentrations before and after IEX treatment at normal and after rain event conditions.

generally possesses a high water content (66–72%), which also is the greatest of both conventional resins tested. Conversely, for the MIEX resin the increased humics content in the water may have led to size exclusion/blockage, which resulted in the decreased TOC removal. Literature has shown that DOM with 2,000–5,000 Da can quickly saturate or block the resin pores (Mergen *et al.* 2008).

### Impact of rain on DBP precursor removal

Figure 4 shows the C- and N-DBP precursor removal with the two different raw water matrices. C-DBPs include the four trihalomethanes, chloral hydrate, 1,1-dichloropropanone and 1,1,1-trichloropropanone. All three resins showed a better C-DBP precursor removal performance at

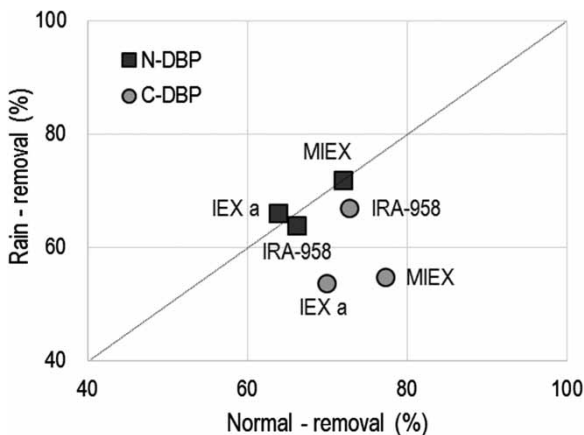


Figure 4 | C- and N-DBP precursor removal by IEX a, b and MIEX-DOC. C-DBPs combine THMs, iodinated-THMs, HKS, CH and N-DBPs combine haloacetonitriles, halonitromethanes.

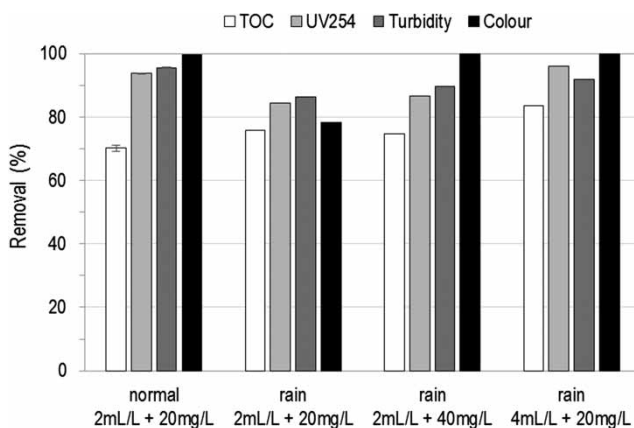
normal conditions compared to the rain event. When treating the rain affected water with MIEX, C-DBP precursor removal was reduced by 22%. This was likely due to the blockage of the resin exchange sites, preventing the small DBP precursors from interacting. Removal of DOM by MIEX has been shown to be water specific. Hydrophobic water (specific UV absorbance, SUVA >4 L/mg.m) dominated by high and medium MW DOM demonstrated lower DOM removal efficiencies caused by pore blockage of the resin (Mergen *et al.* 2008). The larger MW DOM blocking the resin prevents smaller organics from interacting with the resin and their subsequent removal. Low MW DOM has been shown to play an important role in forming DBPs (Hua & Reckhow 2007). On the other hand, C-DBP precursor removal was not reduced when using IRA-958. IRA-958 has the greatest resin size and therefore is less prone to blockage due to medium MW organic matter. N-DBP precursor removal by all three resins appeared not to be affected by the change in raw water composition.

### MIEX – coagulation combined treatment

IEX and MIEX could reduce TOC and DBP precursors by more than 60% even when treating rain impacted water. However, the raw waters were high in colour (~300 HU) and turbidity (~26 NTU) with IEX a, IRA-958, and MIEX only being able to remove turbidity by 48%, 47%, 3% and colour by 9%, 13%, 24%, respectively. The combination of MIEX with coagulation has previously been shown to successfully remove colour and turbidity while adding the

benefit of lowering MIEX and coagulant doses. When applying MIEX as pre-treatment before coagulation under normal conditions, similar TOC and UV<sub>254</sub> (>70%) removals were achieved using an 80% lower MIEX and 70% lower alum dose compared to MIEX or enhanced coagulation alone.

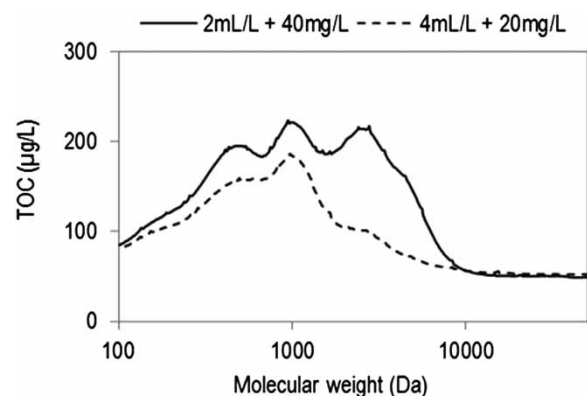
Utilizing MIEX as pre-treatment for coagulation combines the benefits of MIEX removing low to medium MW DOM and coagulation preferentially removing high MW OM. When applying the same MIEX and coagulant doses (2 mL/L + 20 mg/L) for treatment of rain affected water compared to normal conditions, combined TOC removal was only reduced by 5% and UV<sub>254</sub>, turbidity and colour removal by 9%, 10%, and 22%, respectively (Figure 5). Maintaining the MIEX dose while doubling the coagulant dose resulted in a complete removal of colour with no improvement of TOC, UV<sub>254</sub> and turbidity removal. On the other hand, when maintaining coagulant dose but doubling the MIEX dose, removal of all parameters improved. TOC removal increased from 71% to 84%, lowering the TOC in treated water below 3 mg C/L. The major component of the introduced TOC during the rain event was made up of medium and low MW organic matter. As coagulation preferentially removes high MW compounds, no TOC removal improvement with increasing coagulant dose was observed. On the contrary, MIEX preferentially removes medium MW compounds leading to greater TOC removal. This can be seen in Figure 6, where a greater MIEX dose is able to further reduce low to medium MW DOM in the range of 550 to 2,800 Da.



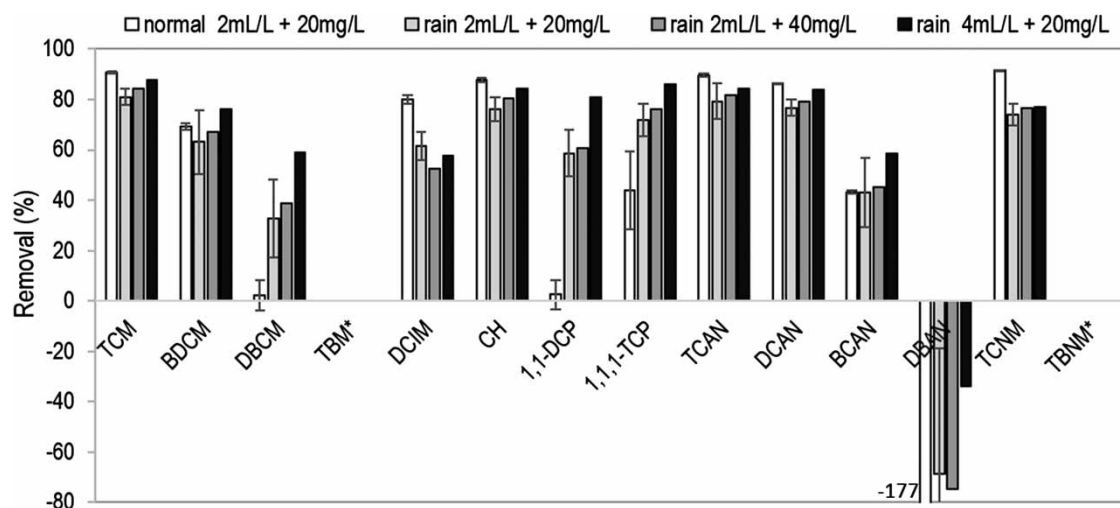
**Figure 5** | TOC, UV<sub>254</sub>, turbidity and colour removal by MIEX treatment followed by alum coagulation. Error bars indicate the absolute difference between duplicate experiments with the 'normal' water sample.

The effect of combined treatment on individual DBP precursor removal is shown in Figure 7. Doubling the coagulant dose while maintaining the MIEX dose after a heavy rain event did not result in additional DBP precursor removal. When doubling the MIEX dose but maintaining the coagulant dose, the solely chlorinated DBP precursor removals, including TCM, CH, TCAN, and DCAN, improved between 5% and 8% (as compared to 2 mL/L + 20 mg/L), resulting in similar DBP concentrations to during normal conditions. Precursor removal improvements were greater for the brominated THMs and HANs, ranging between 13% and 35%. This may be explained by the bromide removal capacity of the MIEX resin. Therefore, when adjusting the treatment to compensate for the impact of a heavy rain event in regards to C- and N-DBP precursor control, MIEX was more efficient than coagulation. The better performance of MIEX may be related to its capacity to remove low MW DOM.

As demonstrated by size exclusion chromatography (Figure 1) a significant amount of low MW organics was introduced during the rain event. Comparison with injected standards shows that some of the low MW DOM was around 550 Da. According to literature this fraction contains mainly low MW neutrals which include monooligosaccharides, alcohols, aldehydes, phenols and ketones (Huber *et al.* 2011). Many of the small organic compounds are precursors which effectively react with chlorine to produce DBPs. For example, methyl ketones can undergo further chlorine substitution and result in haloketone formation, while aldehyde oxidation can result in chloral hydrate formation (Trehay *et al.* 1986).



**Figure 6** | SEC chromatogram of rain affected raw water by MIEX and alum combined treatment.



**Figure 7** | Individual C- and N-DBP precursor removal by MIEX treatment followed by alum coagulation. Error bars indicate the absolute difference between duplicate experiments. \* = below  $<0.1 \mu\text{g/L}$ .

In addition to TOC, TON also can act as a DBP precursor. An important part of the TON content in water is amino acids, which are small molecules with a molecular weight less than 200 Da (Dotson *et al.* 2008). Chlorination of amino acids was shown to form nitriles, which with subsequent chlorine substitution can lead to dichloroacetonitrile formation (Trehy & Bieber 1981). Increasing the MIEX dose in comparison to the coagulant dose did result in greater HAN precursor removal. This may be related to the fact that enhanced coagulation alone could only remove 40%, while MIEX achieved 52% TON removal in the rain affected water.

## CONCLUSIONS

This study evaluated the impact of a major rain event on the raw water quality, DBP precursors and their removal by IEX and MIEX within a subtropical surface water. The major conclusions of this work include the following:

- A major rain event introduced organic matter (3 mg C/L TOC), mainly comprised of low and medium MW organics.
- Formation potentials for the solely chlorinated DBPs increased by more than 20% after the rain event. The impact for the brominated counterparts was less pronounced due to a dilution effect leading to 50% less bromide in the raw water.

- IEX and MIEX treatment were able to reduce TOC, C- and N-DBP precursors in highly coloured and high TOC subtropical surface water pre and post a heavy rain event.
- MIEX as pre-treatment for alum coagulation could remove TOC, colour and turbidity while reducing resin and coagulant doses and subsequently chemicals for pH adjustment.
- Increasing MIEX while maintaining coagulant dose was more efficient in addressing additional organic and DBP precursor loading after a heavy rain event.

MIEX is a registered trademark owned by Ixom Operations Pty Ltd.

## ACKNOWLEDGEMENTS

The authors would like to thank Seqwater staff for assisting with the sampling at the water treatment plants. We also acknowledge Lanxess and IXOM for providing IEX and MIEX-DOC resin samples and especially Anthony Gibson for fruitful exchanges.

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First received 8 June 2017; accepted in revised form 26 January 2018. Available online 13 February 2018