

## Relating water quality monitoring to disinfection by-product formation in four pilot treatment streams

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### ABSTRACT

With increasing focus on the health implications from disinfection by-products (DBP) in drinking waters, appropriate monitoring parameters are required that can give rapid information on the potential of a treated water to form DBPs. Given that the most effective strategy for the reduction of concentrations of DBPs is the removal of their precursor, natural organic matter, parameters that relate to organic concentration and character are the most likely candidates. A project utilising four different treatment procedures applied to common source water was evaluated for DBP formation over three characteristic water quality periods and the link to key monitored parameters evaluated. Results showed that DBP formation was independent of the source water quality or treatment technology applied but was correlated to treated water dissolved organic carbon, and more strongly to the UV absorbance at 254 nm.

**Key words** | DBP, pilot plant, trihalomethanes, UV absorbance, water quality parameters

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### INTRODUCTION

Water treatment plants have traditionally been run with aesthetic quality improvements as the drivers of treatment, though with greater understanding of the effects of water-treatment derived chemicals on human health, the emphasis has progressively shifted towards undesirable contaminants present in the source water and those formed through treatment and disinfection processes. Of all source water contaminants, natural organic matter (NOM) is the most common and is usually present in the highest concentration (Gjessing *et al.* 1998). NOM is responsible for the majority of the coagulant demand in conventional treatment systems and the dissolved organic carbon (DOC) remaining after treatment represents the majority of the disinfectant demand in most chlorinated systems (Rodriguez *et al.* 2003; Warton *et al.* 2006). Consequently, the importance of NOM monitoring in modern drinking water treatment cannot be overstated.

Trihalomethanes (THMs) and haloacetic acids (HAAs) are the largest groups of disinfection by-products (DBPs) on a weight basis (Karanfil *et al.* 2008), and have been identified

as both the most common and most investigated halogenated DBPs found in drinking water (Mancini *et al.* 2005). Chloro- and bromo-substituted THMs consist of four chemical species – chloroform, dichlorobromomethane, dibromochloromethane and bromoform – and the sum of these can be referred to as THM4. There are a total of nine HAA species containing chlorine and bromine: chloro-, dichloro-, and trichloroacetic acid; bromo-, dibromo-, and tribromoacetic acid; and bromochloro-, bromodichloro-, and dibromochloroacetic acid (Cowman & Singer 1996). In most industrialised countries, including Australia, some classes of DBPs are regulated including THMs, chloral hydrate and selected HAAs (Rodriguez & Serodes 2005). However, more stringent DBP regulations are now also being promulgated for other chlorination DBPs, including further HAAs, haloacetonitriles and inorganic DBPs (e.g. bromate and chlorate), due to growing evidence-based health concerns of their potential reproductive, carcinogenic and mutagenic effects (Richardson *et al.* 2007; Hebert *et al.* 2010; Itoh *et al.* 2011).

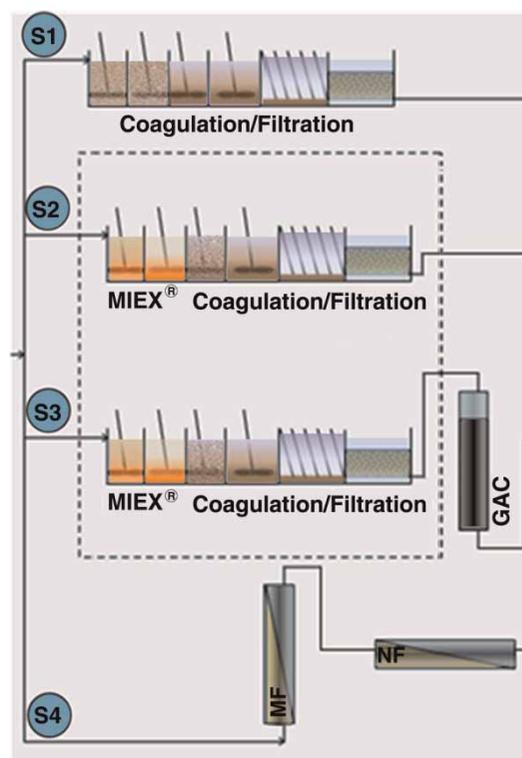
When chlorine is applied to water, the concentration and speciation of THM and HAA formation is influenced by multiple physicochemical factors, including disinfectant dose, pH, temperature and the concentration of precursors (organic and inorganic) present. In general, higher THM and HAA concentrations are expected at higher levels of these parameters (Twort *et al.* 2000; Sadiq & Rodriguez 2004), with the exception of HAA concentrations increasing at lower pH values (Rodriguez & Serodes 2005). Studies conducted on the correlation between THMs and HAAs in Canada, Spain, Finland and the UK have found a range of correlation coefficients varying depending on the water distribution systems and the specific by-products. Total THMs and HAAs correlated between 0.5 and 0.9 and in some cases HAA levels exceeded THM concentrations by three to four times (Williams *et al.* 1997; Villanueva *et al.* 2003; Malliarou *et al.* 2005). Different formation mechanisms exist between THMs and HAAs, which needs to be taken into account in chlorination strategies.

The most important DBP precursors are hydrophobic acids (humic substances) which are ubiquitous in natural aquatic reservoirs as DOC and constitute over 80% of the total organic matter in fresh waters (Mattsson *et al.* 1998; Leenheer 2004; Fabris *et al.* 2008). In NOM molecules, the functional groups that absorb in the UV region ( $\lambda < 400$  nm) are substituted aromatic groups, including mono- and poly-substituted phenols and various aromatic acids (Nikolaou & Lekkas 2001). Moreover, chromophores responsible for UV absorbance are likely to have conjugated double bonds and unbonded electrons like those associated with oxygen, sulphur and halogen atoms (Gallard & von Gunten 2002). NOM reactions with halogens (e.g. chlorine, bromine and iodine) have been reported to occur via oxidation (i.e. cleaving of carbon-carbon double bonds) and/or substitution through the replacement of a functional group by a halogen molecule. Hence a measure of UV absorbance that targets general aromatic structures (in the range of 250–280 nm) has been suggested for characterisation of NOM in water sources (Westerhoff *et al.* 2004). The most widely used method to quantify NOM reactivity in DBP formation is by absorbance of ultraviolet light at 254 nm ( $UV_{254}$ ) and specific UV absorbance (SUVA) (Roccaro & Vagliasindi 2009). Hence the monitoring of NOM either directly or via surrogate parameters can be seen as an important part of effective water quality management.

A project evaluating four different drinking water treatment pilot streams over a 2-year period provided the opportunity to evaluate the relationship between organic water quality parameters, such as  $UV_{254}$  and DOC, against the subsequent disinfectant demands and DBP formation. In the context of three defined water quality periods, the impact on disinfection parameters resulting from four treatment processes of varying DOC removal efficiency from common source water were compared and correlated.

## MATERIAL AND METHODS

The source water for the investigation was from the inlet to the Mt Pleasant water treatment plant (WTP) in South Australia. This water is sourced from the River Murray via the Mannum to Adelaide pipeline that provides one of the major drinking water supplies for metropolitan Adelaide. Details of the process streams are shown in Figure 1 and are described below.



**Figure 1** | Schematic of pilot treatment streams applied (dashed section indicates main WTP treatment).

### Stream 1: conventional treatment

The conventional treatment process comprised coagulation/flocculation/dual media filtration utilising an upstream clarifier pilot plant with a perspex filter column. The coagulant employed was aluminium sulphate (alum) as  $\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$ . Coagulation pH of between 6.0 and 6.5 was maintained through addition of caustic (sodium hydroxide, analytical grade) or sodium bicarbonate buffering (food grade), depending on source water alkalinity. In addition, a coagulant aid, either anionic polyacrylamide (LT20, BASF, Australia) or high molecular weight cationic polymer (LT425, BASF, Australia), was dosed downstream of the coagulant. This process was selected as a baseline/control as it represents the most widely applied drinking water treatment process employed in Australia. The primary coagulant dose set point was driven by changes in settled water turbidity to maintain  $\leq 1$  NTU at the head of the filter. As this was measured via grab sampling after treatment, it represents a feedback control strategy, similar to most operating WTPs.

### Stream 2: MIEX/conventional coagulation

This treatment comprises high rate magnetic ion-exchange contact (MIEX DOC<sup>®</sup> process, Orica, Australia) for DOC removal coupled with coagulation/flocculation/dual media (sand/anthracite) filtration. Although the coagulation stage may remove additional DOC, the dosing strategy targets clarification (turbidity removal), a function that ion-exchange cannot achieve. The primary coagulant used is aluminium sulphate (alum) as  $\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$ ; however, additional coagulant aids, LT22 and LT425 (BASF, Australia), were also dosed periodically during coagulation as required.

### Stream 3: MIEX/conventional coagulation/granular activated carbon

The third treatment stream comprised the product water from stream 2 (described above) with the addition of two parallel granular activated carbon (GAC) filters utilising Calgon F400 in packed bed columns with gravity fed empty bed contact times (EBCT) of approximately 14 min

at 125 L/h per column. The filtrate from both columns was blended into a common storage.

### Stream 4: microfiltration/nanofiltration

Dual membrane filtration consisted of microfiltration (MF) pre-treatment for particulate removal using a single submerged hollow fibre module (Memcor CMF-S system, USA) followed by a single FILMTEC NF 270-4040 spiral wound nanofiltration (NF) membrane (DOW Chemical Company, USA). The MF system operated at 1,000 L/h, achieving 75% permeate recovery. The NF system operated in cross-flow configuration at 43% permeate recovery, producing 325 L/h. Nominal pore size for the MF is reported as 0.2  $\mu\text{m}$  with the molecular weight cutoff for the NF of 270 Daltons, achieving high levels of DOC removal with significant reduction in hardness.

### Analyses

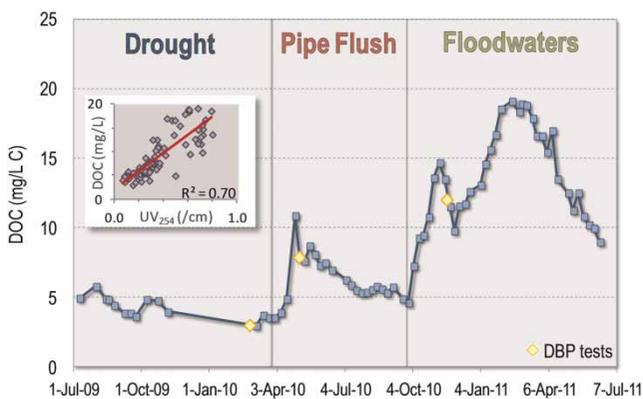
Samples for DOC and  $\text{UV}_{254}$  analyses were filtered through 0.45  $\mu\text{m}$  pre-rinsed membranes.  $\text{UV}_{254}$  was measured through a 1 cm quartz cell and colour through a 5 cm cell using an Evolution 60 Spectrophotometer (Thermo Scientific, USA). DOC was measured using a Sievers 900 Total Organic Carbon Analyser (GE Analytical Instruments, USA). Chlorine demand was determined by dosing with a saturated chlorine solution and measurement of residual free chlorine following 7 days' contact time. Chlorine demand is defined as the concentration difference between the dosed amount and titrated residual. Bromide was determined by ion chromatography using Standard Method 4110 (APHA *et al.* 2007). THM4 and chloral hydrate were analysed by headspace gas chromatography with electron capture detection according to Standard Method 6232C (APHA *et al.* 2007). All nine chloro- and bromo-substituted haloacetic acids (HAA9) were analysed according to Standard Method 6251B (APHA *et al.* 2007). The bromide incorporation factor (BIF), a measure of DBP speciation shifts, was calculated using Equation (1) from Chang *et al.* (2001), where the concentrations of THMs were in molar units ( $\mu\text{mol/L}$ ).

$$\text{BIF} = \frac{\text{CHBrCl}_2 + 2\text{CHBr}_2\text{Cl} + 3\text{CHBr}_3}{\text{TTHM}} \quad 0 \leq \text{BIF} \leq 3 \quad (1)$$

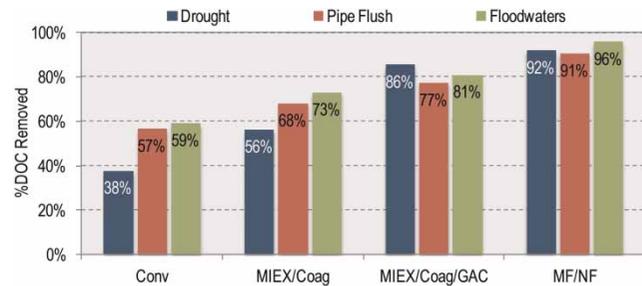
## RESULTS AND DISCUSSION

Four pilot treatment processes were operated, treating the same source water between July 2009 and June 2011. During that time, water quality changes were experienced, largely as a result of two major events. After a period of extended drought where river inflows were minimal and source water quality was relatively stable, an operational change (supply pipeline flush) and an environmental change (arrival of floodwaters from eastern states (Queensland, New South Wales and Victoria) of Australia) occurred. These are denoted as ‘Pipe Flush’ and ‘Floodwaters’ periods, respectively.

For the monitoring of treatment efficiency, the most relevant parameter is DOC which is a quantitative measure that has direct connection to both coagulant and disinfectant demand (Ratnaweera *et al.* 1999; Rodriguez *et al.* 2003; Warton *et al.* 2006). Source water DOC (Figure 2) increased rapidly within short intervals in May and December 2010, consistent with the onset of changing water quality periods. DOC concentrations peaked between February and March 2011 when the highest levels of aquatic organic matter arrived with the flood waters. DOC removal (Figure 3) improved with each successive water quality period (‘Pipe flush’ and ‘Floodwaters’) for the conventional coagulation and MIEX/coagulation treatment trains. This improving DOC removal indicates that in addition to the increasing concentration, the additional organic material was also characteristically treatable, producing similar treated water quality despite the considerable concentration increases. The membrane treatments (MF and NF) maintained removal



**Figure 2** | Raw water DOC (mg/L C) at Mt Pleasant WTP, July 2009 to June 2011. Inset: relationship of DOC to UV absorbance at 254 nm.

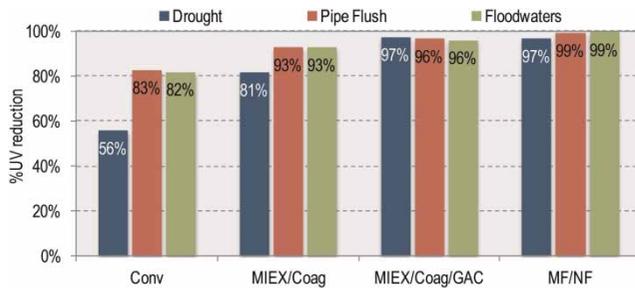


**Figure 3** | Average DOC removed by each treatment technology. Conv = conventional coagulation; MIEX/Coag = MIEX adsorption and coagulation; MIEX/Coag/GAC = MIEX, coagulation and GAC adsorption; MF/NF = microfiltration plus nanofiltration.

efficiencies throughout the period of influent floodwaters indicating that neither increased concentration nor changes in the character of the NOM affected the ability of the NF membrane to function effectively. For the MIEX/coagulation/GAC stream, the diminishing adsorption capacity of the GAC was counteracted by the improving performance of the MIEX treatment for DOC removal.

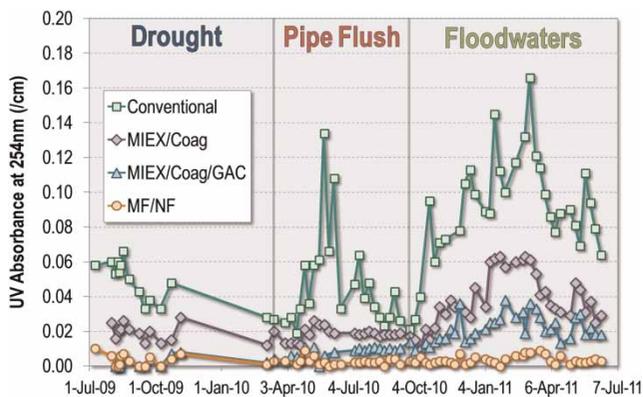
UV absorbance measurement at 254 nm has long been applied for semi-quantification and characterisation of organic matter (Edzwald *et al.* 1985; Amy *et al.* 1987; Korshin *et al.* 1999) and as a surrogate for the characteristic functionality of DOC that is involved in coagulation mechanisms and ion-exchange, as well as the greatest reactivity with disinfectants (Chow *et al.* 2006; Korshin *et al.* 2009). Although UV absorbance at a single wavelength does not provide comprehensive information of the character of organics (Del Vecchio & Blough 2004), it is operationally relevant in terms of representing the treatable fraction of the DOC. Because water treatment processes are typically more selective towards removal of UV absorbing compounds due to their susceptibility to treatment mechanisms (Chow *et al.* 2006; Korshin *et al.* 2009), UV<sub>254</sub> can overestimate the overall actual DOC removal (Figure 4). For source water monitoring, however, this can be seen as a beneficial characteristic, with UV absorbance providing a closer link with organic matter treatability. For this water source, there was a moderate linear relationship ( $R^2 = 0.70$ ) between source water DOC and UV<sub>254</sub> (Figure 2, inset) due to notable differences in the timing and response at the onset of water quality periods with the UV<sub>254</sub> increasing sooner than DOC and with greater amplitude.

The response of the treatments to water quality changes can be observed from examination of the treated water UV



**Figure 4** | Average absorbance at 254 nm (/cm) removed by each treatment technology. Conv = conventional coagulation; MIEX/Coag = MIEX adsorption and coagulation; MIEX/Coag/GAC = MIEX, coagulation and GAC adsorption; MF/NF = microfiltration and nanofiltration.

absorbance (Figure 5). During the drought period, the removal of UV absorbing components from the low DOC water supply was established, demonstrating the performance advantages of the multi-treatment streams versus conventional coagulation. Throughout the pipe flush and floodwater periods, the increased variability of the source water presented challenges to the control of the coagulation resulting in periods of sub-optimal removal. In the pipe flush period, the capacity of the GAC in the ‘MIEX/Coag/GAC’ stream began to diminish, resulting in a divergence from the previous similarity of UV absorbance reduction with the high efficiency ‘MF/NF’ stream, although it still represented a considerable reduction over the ‘MIEX/Coag’ stream. By the floodwater period, the capacity of each of the streams to treat high DOC water was challenged, with the conventional and ‘MIEX/Coag’ streams demonstrating elevated UV absorbance during the peak inlet concentration. The ‘MIEX/Coag/GAC’ and ‘MF/NF’ were also affected by the floodwaters but to a lesser degree,



**Figure 5** | Treated water UV absorbance (/cm at 254 nm) at Mt Pleasant WTP, July 2009 to June 2011.

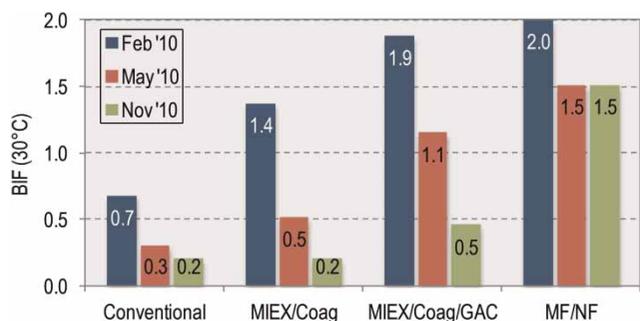
highlighting the benefits of multi-barrier treatments in buffering treated water quality changes during challenging water quality periods.

For the formation of DBPs, it is often the character of the DOC rather than the concentration that is more relevant to the rate of formation and final concentrations attained. One of the benefits of improved treatment is that the variability in DBP formation due to source water quality changes is reduced. This was observed through the four applied treatments with the conventional coagulation stream showing the highest variability in DOC and consequently DBP formation (Table 1). Advanced multi-stage treatments such as the MF/NF (stream 4) showed consistently low THM formation, regardless of seasonal variation in the source water. Similarly, Bond *et al.* (2010) found that selective removal of DBP precursors using coagulation, MIEX contact or ion-exchange was not possible and high level DOC removal using NF was required to reduce DBP formation. Although the addition of GAC adsorption (stream 3) further improves DOC removal above the capacity of the MIEX and coagulation treatment (stream 2), the data suggest a diminishing GAC adsorption capacity for THM precursors over the investigated duration. This can be expressed through the difference in THM formation ( $\Delta\text{THM}_4$ ) between streams 2 and 3 during the drought period (76  $\mu\text{g/L}$ ) and flood inflow period 9 months later (39  $\mu\text{g/L}$ ). Badawy *et al.* (2012) also found that virgin GAC filtration was highly effective in reducing DOC and also DBP formation; however, the effect of long-term operation and absorption saturation was not examined. Haloacetic acid and chloral hydrate concentrations were diminished more effectively than THM<sub>4</sub> with increasing levels of treatment, suggesting a greater degree of HAA precursor removal by the MIEX ion-exchange process. The dual-stage membrane process was successful in removing almost all precursors throughout the three water quality periods.

Although treatment technologies such as MIEX adsorption and nanofiltration were capable of removing some bromide in addition to DOC (Table 1), removal of additional DBP precursors by some of the treatment streams, and the complementary reduction in chlorine demand, increased the degree of undesirable bromine substitution into DBPs. This was demonstrated through increasing BIFs (Figure 6). Notably, the MIEX/Coag/GAC stream produced similar values to the MF/NF

**Table 1** | 7-day chlorine demand and total trihalomethane (THM4) formation for treatments of Mt Pleasant source water at 30 °C; BIF = bromine incorporation factor; CH = chloral hydrate

Drought period							
25-Feb-10 7-day/30 °C	DOC (mg/L)	Bromide (mg/L)	Cl <sub>2</sub> demand (mg/L)	THM4 (µg/L)	THM BIF	HAA9 (µg/L)	CH (µg/L)
Conventional	2.03	0.19	6.45	196	0.7	168	31
MIEX/Coag	1.16	0.14	1.96	102	1.4	37	5
MIEX/Coag/GAC	0.45	0.17	0.78	26	1.9	6	< 1
MF/NF	0.17	0.15	0.42	14	2.0	2	< 1
Pipe flush							
3-May-10 7-day/30 °C	DOC (mg/L)	Bromide (mg/L)	Cl <sub>2</sub> demand (mg/L)	THM4 (µg/L)	THM BIF	HAA9 (µg/L)	CH (µg/L)
Conventional	4.53	0.12	8.40	260	0.3	237	55
MIEX/Coag	1.73	0.03	2.24	90	0.5	47	15
MIEX/Coag/GAC	0.51	0.08	1.01	35	1.1	21	3
MF/NF	0.21	0.08	0.48	16	1.5	2	< 1
Floodwaters							
19-Nov-10 7-day/30 °C	DOC (mg/L)	Bromide (mg/L)	Cl <sub>2</sub> demand (mg/L)	THM4 (µg/L)	THM BIF	HAA9 (µg/L)	CH (µg/L)
Conventional	4.58	0.04	6.56	199	0.2	198	41
MIEX/Coag	2.95	0.02	3.60	121	0.2	83	24
MIEX/Coag/GAC	2.06	0.08	2.28	82	0.5	40	13
MF/NF	0.25	0.04	0.22	8	1.5	< 1	< 1

**Figure 6** | Trihalomethane bromide incorporation factor through water quality periods.

during the early stages of pilot plant operation (February 2010), but by the disinfection testing of the third nominated water quality period (November 2010), the BIF was more closely related to the conventional and MIEX/coagulation streams. While the change in source water quality was also a factor, the diminishing BIF shown by the MIEX/Coag/GAC stream was more greatly influenced by the reducing

adsorption capacity of the GAC for DOC removal throughout the study. With the MF/NF stream, the consistently higher BIF was tempered by lower overall DBP concentrations formed, diminishing the significance of DBP speciation.

Comparison of the organic water quality parameters with DBP formation showed that THM, HAA and chloral hydrate concentrations were highly correlated with DOC and UV<sub>254</sub>, as well as the chlorine demand (Figure 7). This is consistent with the findings of a number of published investigations (Kitis *et al.* 2002; Zhang *et al.* 2011). As this correlation was found to hold across all three raw water quality periods and various treatment technologies, it indicated that the mechanism by which the water was treated was less important than the residual concentration of organic matter (DOC). Further refinement was achieved by monitoring the aromatic character using UV<sub>254</sub> (Chow *et al.* 2008). As bulk chlorine demand results from the influence of

	$R^2$ 30°C					
DOC	1					
UV	0.93	1				
Cl <sub>2</sub> Dem	0.83	0.88	1			
THM	0.83	0.87	0.98	1		
HAA	0.83	0.91	0.99	0.95	1	
CH	0.91	0.94	0.97	0.94	0.96	1
	DOC	UV	Cl <sub>2</sub> Dem	THM	HAA	CH

**Figure 7** | Correlation of organic parameters with 7-day Cl<sub>2</sub> demand (Cl<sub>2</sub> Dem) and DBPs; CH = chloral hydrate.

both concentration and character of NOM, it demonstrated the highest correlations with individual DBP formation. This is contrary to the findings of Bond *et al.* (2009) but may represent the improved potential for correlations of DBP formation with the characteristics of heterogeneous NOM in this study rather than the character of individual model compounds.

## CONCLUSIONS

From the investigation into long-term water quality data from four pilot treatment plants, a number of key points can be identified. DBP formation is dependent mostly on the resulting treated water organic concentration and character and largely independent of the treatment technology used to produce it. Bromide incorporation into THMs was consistently higher in the dual-membrane process (stream 4) compared with the coagulation and ion-exchange based treatments (streams 1–3) because of the low chlorine demand; however, actual DBP concentrations in stream 4 were very low which diminished the impact. The chlorine demand, which can represent a measure of organic matter reactivity, was strongly related to DBP concentrations for this water source. Additionally, UV absorbance at 254 nm provided an accurate representation of source water organic variation and was a better indicator of DBP formation (Ave.  $R^2 = 0.91$ ) than DOC (Ave.  $R^2 = 0.86$ ).

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