

# Impact of UV irradiation on disinfection by-product formation and speciation from post-chlorination of dissolved organic matter

Ziming Zhao, Madhumita B. Ray, Ted Mao and Wenjun Sun 

## ABSTRACT

The objective of this study was to investigate the effects of combined low-pressure ultraviolet (UV) irradiation and chlorination on the formation of disinfection by-products (DBPs) from different dissolved organic matter (DOM) as DBP precursors. Commercially available humic acid (HA), extracellular organic matter (EOM) from green algae, cyanobacteria, and diatom, namely *Scenedesmus quadricauda* (SQ), *Merismopedia* sp. (Msp), and *Phaedactylum tricornutum* (PT), were used as the sources of DOM. The DBP formation increased with increasing total residual chlorine; EOM from PT presented the highest formation potential followed by HA, Msp, and SQ. The low dosage of 40 mJ/cm<sup>2</sup> UV irradiation is insignificant to change the DBP formation from HA and SQ; however, it decreased the DBP formation from bromide-containing EOM of PT and promoted the DBP formation from EOM of Msp at various total residual chlorines. The DBP formation of each DOM correlated well with total residual chlorine. The maximum DBP formation potential (DBPFP) reduction of 42.25 and 13.75% for haloacetic acid formation potential (HAAFP) and trihalomethane formation potential (THMFP) was obtained at the UV irradiation dosage of 300 mJ/cm<sup>2</sup> for EOM of PT. However, for the EOM derived from Msp, a maximum increase of 58.1 and 51.1% for HAAFP and THMFP was observed after UV-chlorination.

**Key words** | algal organic matter, disinfection by-products, extracellular organic matter, humic acid, ultraviolet (UV) irradiation

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## HIGHLIGHTS

- Disinfection by-product (DBP) formation increased linearly with total residual chlorine from 0.28 to 1.02 mg/L.
- Ultraviolet (UV) irradiation did not significantly alter the DBP formation from humic acid and algal organic matter (AOM) of *Scenedesmus quadricauda*.
- The haloacetic acid formation potential (HAAFP) and trihalomethane formation potential (THMFP) decreased by 42.25 and 13.75%, respectively, under the UV irradiation dosage of 300 mJ/cm<sup>2</sup> for AOM of *Phaedactylum tricornutum*, while the maximum increment of 58.1 and 51.1% for HAAFP and THMFP were observed from the AOM of *Merismopedia* sp.

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## INTRODUCTION

Ultraviolet (UV) irradiation has been increasingly applied in water treatment plants to inactivate a wide range of waterborne pathogens, such as *Cryptosporidium* (Rochelle *et al.* 2004) and *Giardia lamblia* (Shin *et al.* 2010), which are resistant to chlorination (Ye *et al.* 2018). It has also been an alternative treatment for the removal of small concentrations of organics including the taste and odor compounds (Chen *et al.* 2018). However, the main disadvantage of UV disinfection is that no residual inactivation capacity of UV radiation is left once the treated water is supplied in the distribution system (Wang *et al.* 2014). Many treatment plants use post-chlorination or chloramination to address this for maintaining water quality. However, depending on the dissolved organic carbon (DOC) content in the UV-treated water, the formation of disinfection by-product (DBP) is a concern of post-chlorination. Algal organic matter (AOM), a combination of extracellular organic matter (EOM) and intracellular organic matter (IOM), is an important source of dissolved organic matter (DOM) in surface water, which is a source of the precursors of many harmful DBPs (Hua *et al.* 2017; Hua *et al.* 2019). EOM is not removed effectively during coagulation and flocculation processes in water treatment plants (Lin *et al.* 2018) and, consequently, produces a variety of DBPs during chlorination. UV radiation at 254 nm is well known to be effective to inactivate waterborne pathogens and degrade organics by the process of photolysis (Shah *et al.* 2011). Being a complex mixture of carbohydrate, sugar, lipid, and protein, DOM is susceptible to be degraded during UV disinfection. The complex mixture of intermediates subsequent to photolysis may cause the formation of harmful products after chlorination. It is reported that UV irradiation can alter the characteristics of DOM, such as molecular weight and hydrophobicity/hydrophilicity (Buchanan *et al.* 2005; Chen *et al.* 2018). Although it is not significant, an increase of low molecular fraction and chlorine demand was observed after UV treatment (Choi & Choi 2010); consequently, more chloroform was formed (Buchanan *et al.* 2005; Liu *et al.* 2006). It has been also reported that UV irradiation can effectively

reduce DBP formation with low UV absorbance of DOM (Chen *et al.* 2018). Previous studies have been carried out under a relatively high UV dosage of 14–1,000 J/cm<sup>2</sup> for the water with high DOC in the range of 5–17.4 mg/L (Buchanan *et al.* 2006; Goslan *et al.* 2006; Liu *et al.* 2006; Choi & Choi 2010). However, most drinking water treatment plants in North America operate with  $\leq 5$  mg/L DOC (standard in most jurisdictions) and the recommended UV dose for disinfection in drinking water treatment in North America is 40 mJ/cm<sup>2</sup> (Sharvelle *et al.* 2017), which is much lower than the UV dosages tested in the literature. In addition, the literature on UV photolysis of EOM from algal species followed by chlorination under control conditions is rather limited. It was reported that UV pretreatment enhanced the formation of nitrogenous DBP (N-DBP) during the subsequent chlor(am)ination of EOM, especially dichloroacetonitrile (Zhang *et al.* 2016a). Several studies were conducted on UV irradiation followed by the chlorination of humic acid (HA) in water (Lee & Hur 2014; Zhang *et al.* 2016a, 2016b; Ye *et al.* 2018). It was found that UV irradiation did not alter the specific DBP formation potential (DBPFP) significantly based on the DOM characteristics, which indicated that most of the DBP precursors could not be removed by photodegradation (Lee & Hur 2014; Zhang *et al.* 2016a). With the increasing events of harmful algal blooms all over the world, a control study on the photolysis of AOM followed by chlorination is required to determine the effect of algal matter on drinking water treatment processes, which is the objective of this study.

In our previous study, the DBP formation of both EOM and IOM of four algae and two cyanobacteria were determined (Zhao *et al.* 2020). However, intracellular materials are only released under stress conditions and certain treatment conditions such as preoxidation. Hence, IOM was not included in this study, and only EOM from three types of species, green algae, cyanobacteria, and diatom, namely *Scenedesmus quadricauda* (SQ), *Merismopedia* sp. (Msp), and *Phaedactylum tricorutum* (PT), were used as the sources of DOM. In addition, only carbonaceous DBP

(C-DBP) was determined in the present study, as the concentration of N-DBP is much lower due to the release of a small amount of intracellular materials during the growth stage (Hua *et al.* 2020). Humic substances are widely found in freshwater, and HAs constitute the major fraction of natural organic matter (NOM) (Levchuk *et al.* 2018; Zhang *et al.* 2018) impacting drinking water treatment processes. The main purpose of including HA in the current study was to compare the impact of HA with EOM on DBP formation after UV irradiation/chlorination.

## MATERIALS AND METHODS

### Algal cultivation

The three freshwater species, SQ, Msp, and PT, were purchased from the Canadian Phycological Culture Centre (CPCC) at Waterloo University (Waterloo, ON, Canada). They were cultivated in 2 L conical flasks using a specific medium (high salt medium for SQ, BG-11 for Msp, and F/2 for PT) for each species at  $23 \pm 2$  °C with intermittent illumination (3,000 lx) at a light/dark cycle of 16/8 h. All solutions were prepared from reagent-grade chemicals and Milli-Q water, except the medium F/2, which was diluted by synthetic seawater prepared by Instant Ocean sea salt (Instant Ocean Company, USA). The growths of algae were monitored by cell counting using a hemocytometer (LW Scientific, USA) under a microscope (ZEISS, Germany). Algae and cyanobacterial cultures were harvested during the stationary growth phase based on the previous study (Wang *et al.* 2012). HA with an average molecular weight of 39.098 kDa was purchased from Alfa Aesar (Thermo Fisher Scientific, USA).

### EOM extraction and characterization

EOM of the species was extracted by centrifuging algal cell suspension at 3,700 rpm for 15 min. The supernatant was filtered with 0.45  $\mu\text{m}$  hydrophilic acrylic copolymer filter (Pall Corp., USA) to obtain EOM. DOC of EOM was determined by a Shimadzu TOC-V<sub>CPN</sub> analyzer (Shimadzu, Japan) with an ASI-L auto-sampler, and the detection limit was 0.1 mg/L on filtered samples. Glucose was used as the standard of

DOC at a concentration of 2–20 mg/L to obtain the DOC standard calibration curve. Three replicates were measured, and an average was reported with the coefficient of variance less than 2%.

UV absorbance at 254 nm (UV<sub>254</sub>) was obtained using a UV/Vis spectrophotometer (Model 3600, Shimadzu, Japan) in the range of 200–300 nm with a 1 cm quartz cuvette. The specific UV absorbance (SUVA, L/mg·m) is widely used for characterizing the aromaticity of NOM and predicting its DBPFP in water treatment. SUVA is defined as the UV absorbance at 254 nm normalized by the overall organic loading in terms of DOC (mg/L), which represents the average absorptivity at 254 nm from all the dissolved organics compounds (Hua *et al.* 2015). The initial DOC of the EOM was kept below 5 mg/L, as this is the typical DOC that many drinking water treatment plants experience (Mash *et al.* 2014; Yang *et al.* 2015; Guo *et al.* 2016; Fu *et al.* 2017; Xu *et al.* 2017).

### UV irradiation

The UV irradiation was performed in a bench-scale collimated beam enclosing a low-pressure UV lamp and a collimated tube with a nonreflective inner surface (Trojan Technologies, Canada) (Hu 2016). UV intensity was monitored and calibrated by ILT1400 radiometer (International Technologies, USA) and SED 240SEL detector at 254 nm. The UV intensity of 0.156 mW/cm<sup>2</sup> and UV dosages of 0–300 mJ/cm<sup>2</sup> were applied to 50 mL water samples containing algal matter in a Petri dish and placed under the UV lamp using constant stirring at room temperature. UV doses were calculated by multiplying the measured intensity and exposure time (Chen *et al.* 2018). The initial pH of 8 was adjusted with 0.01 M H<sub>2</sub>SO<sub>4</sub> or 0.1 M NaOH and borate buffer.

### Post-chlorination and DBP quantification

The chlorination of water samples after UV irradiation treatment was conducted immediately in headspace-free amber glass bottles containing a calculated amount of hypochlorite-buffer solution and incubated under dark at ambient temperature ( $23 \pm 1$  °C) for a specific time. The chlorination of the water sample was performed following the uniform formation condition (UFC) method (Summers

et al. 1996) determining DBPFP. Ammonium chloride was added to quench the residual chlorine (Kristiana et al. 2014) in water to obtain the trihalomethane formation potential (THMFP) and haloacetic acid formation potential (HAAFP) following the method of USEPA551.1 (Munch & Hautman 1995) and EPA552.3 (Domino et al. 2003). The free and total residual chlorine was measured following DPD method 8167 using a HACH DR5000 and a UV-Vis spectrophotometer (HACH Company, USA). The formation of four major THM<sub>4</sub>, including trichloromethane (TCM), tribromomethane (TBM), bromodichloromethane, and dibromochloromethane, were extracted with methyl *tert* butyl ether (MTBE) via liquid-liquid extraction following the method of USEPA 551.1 (Munch & Hautman 1995). The six HAA<sub>6</sub>, bromochloroacetic acid (BCAA), dichloroacetic acid, monochloroacetic acid, trichloroacetic acid (TCAA), monobromoacetic acid, and dibromoacetic acid (DBAA), were extracted with MTBE, methylated with acid methanol following the modified USEPA method 552.3 (Domino et al. 2003). The DBPs were determined by gas chromatography supplemented with electron capture detector GC-ECD (Shimadzu GC-2014) with a BPX5 capillary column (30 m × 0.25 mm ID, 0.25 μm film thickness). The method detection limits for HAAs and THMs were determined using the lowest concentration of each standard solution in the range of 0.02–0.16 μg/L for

HAAs and 0.05–0.63 μg/L for THMs. DBPFP (μg/mg C) were obtained by dividing the concentration of DBP (in μg/L) by the DOC (in mg/L). For better clarification, the sequence of the entire experimental scheme is shown in Figure 1.

## RESULTS AND DISCUSSION

### Residual chlorine with a variation of chlorine dosage

From preliminary chlorine demand, the water sample spiked with each DOM stock solution was dosed with different free chlorine concentrations to establish the chlorine demand curve for each specific water matrix. The total residual chlorine with a variation of dosage under identical incubation and measurement conditions is shown in Figure 2. It can be seen that the EOM and HA solution demonstrated different total residual chlorine profiles with chlorine dose. A linear equation with the highest correlation coefficient of 0.9985 can well fit the total chlorine of SQ in the experimental range. However, unlike the SQ, a longer lag phase occurred for Msp before total residual chlorine increased gradually following an exponential pattern. The PT and HA showed a similar increase pattern;

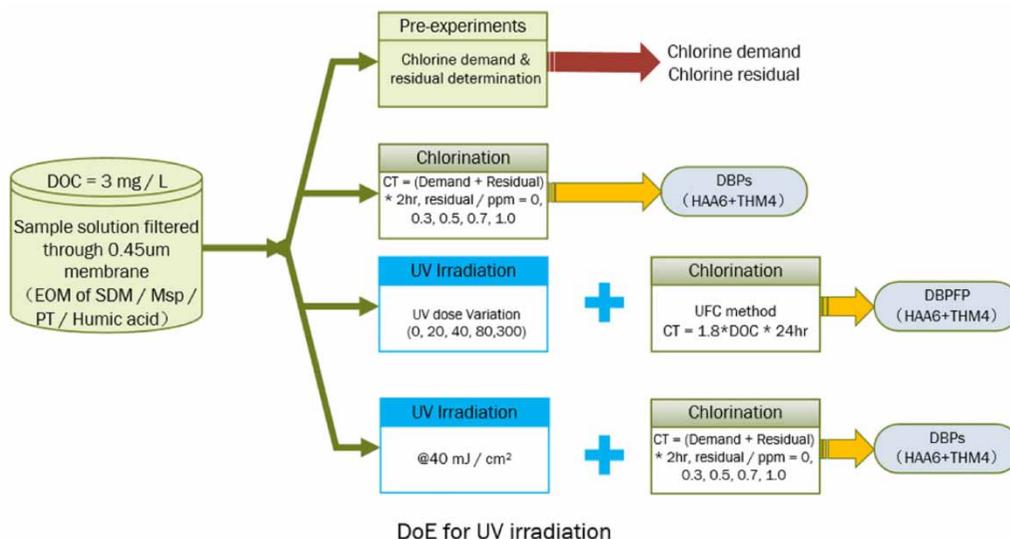
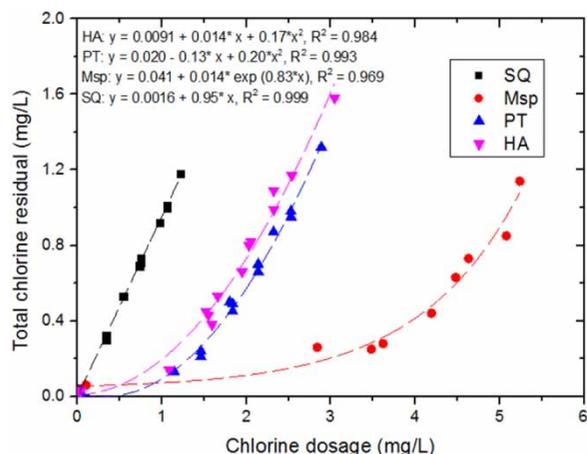


Figure 1 | Experimental sequence showing UV irradiation and chlorination of DOM.



**Figure 2** | Total residual chlorine with a variation of chlorine dosage after 2 h incubation in dark at room temperature for DOM solution with initial DOC of  $3.0 \pm 0.2$  mg/L.

thus, a quadratic polynomial equation can fit the data with the correlation coefficient of 0.993 and 0.984 for PT and HA, respectively.

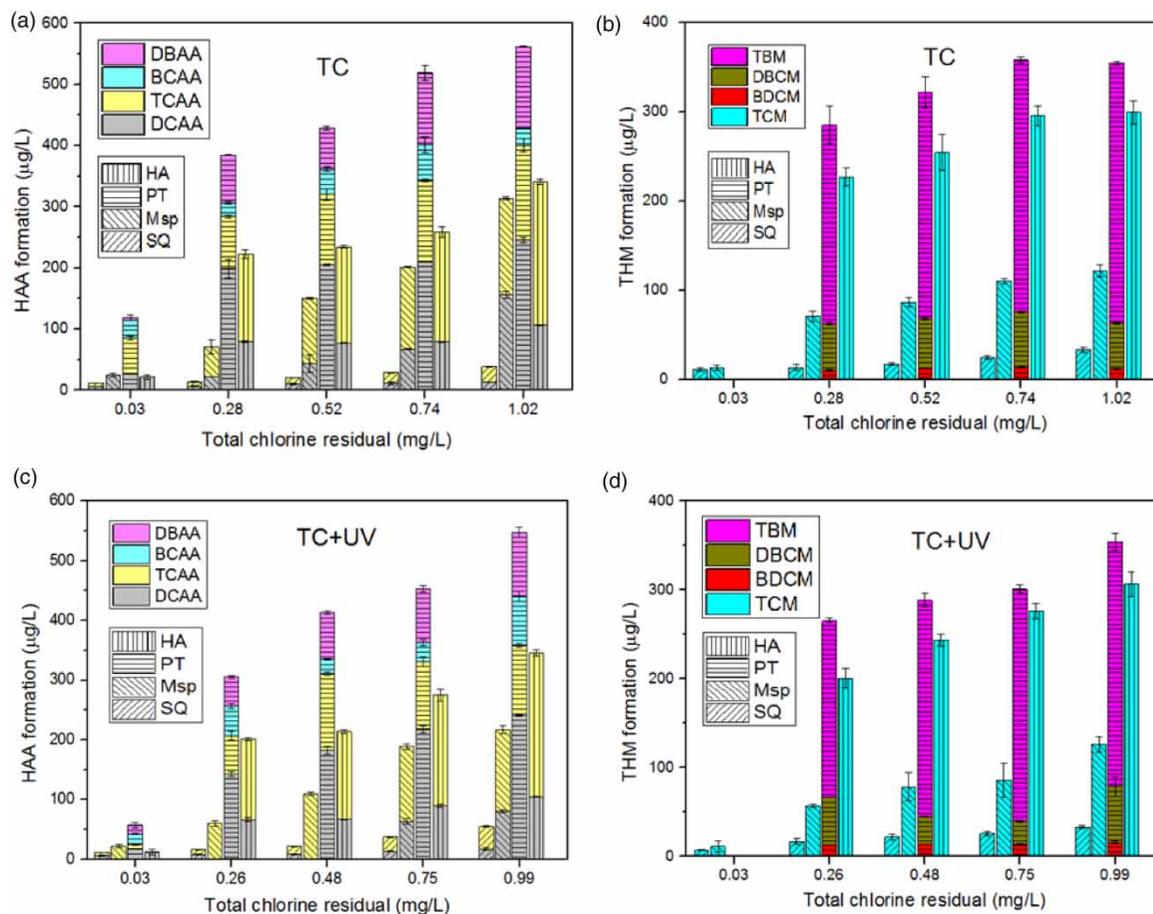
It has been recognized that chlorine demand associates with organic matter and other reactive compounds remaining in the treated water, and many parameters including DOC concentration,  $UV_{254}$ , and inorganics such as iron, manganese, and ammonia may influence the chlorine demand (Roth & Cornwell 2018). It is interesting to see that although each DOM solution was dosed at a similar initial DOC of 3 mg/L, different DOM showed different chlorine demand, with Msp showing the highest chlorine demand followed by PT and HA. SQ presented the lowest chlorine demand probably due to its relatively lower SUVA value of 0.26, and the highest chlorine demand of Msp might result due to the presence of nitrate ions in the cultivation media BG-11 (Xu *et al.* 2012; Zhang *et al.* 2014) and up to 45% proteins content in its chemical composition (Zhao *et al.* 2020). DOM from PT presented the similar trend and comparable amount of chlorine demand to HA, even though with much less SUVA value of 0.74 than that of 3.18 from HA (Ye *et al.* 2018). This could be due to high organic-N content in PT, which provides sufficient amino groups for organic chloramine formation (Zhang *et al.* 2016a). Since many water treatment plants keep a total residual chlorine of around 1–1.5 mg/L (Roth & Cornwell 2018), different chlorine dosages will be required for different algal matter.

### Effect of total residual chlorine on DBP formation

According to the results of the chlorine demand test presented above, the calculated amount of free chlorine was added into water samples of each DOM for the following DBP formation tests. DBP formation was evaluated for the water samples before and after UV irradiation to evaluate the influence of UV disinfection on the DBP formation under different total residual chlorine after incubation in dark for 2 h at room temperature as the post-chlorination process.

Under the chlorination conditions adopted, the total DBP formation increased with the increasing total residual chlorine as shown in Figure 3. HAA formation was higher than THM formation, and the diatom PT produced the highest amount of both types of DBP followed by the others in the order:  $PT > HA > MSP > SQ$ . The different DBP formation among various EOMs may be attributed to their different SUVA values (Zhao *et al.* 2020), where the average SUVA values of EOMs were 0.74, 0.55, and 0.26 for PT, MSP, and SQ, respectively. The similar results were also found by Hua *et al.* (2020), where high SUVA precursors produced high levels of C-DBPs. DBPs formed from HA were less than those of EOM from PT, with its relatively higher SUVA value of 3.18. Bromide ions may react with EOM of PT faster when both bromide and free chlorine are present; in this circumstance, chlorine reacts preferentially as an oxidant, while bromide reacts as a substituting agent (Chen *et al.* 2018).

Since PT can also be marine algae, its medium was diluted by synthetic seawater prepared by commercial Instant Ocean sea salt, which contains  $94.12 \mu\text{g/L}$  bromide in the final DOM solution. Thus, significant amounts of brominated DBPs were formed after chlorination. This is in agreement with an earlier work (Zhao *et al.* 2020), where both IOM and EOM of PT showed the highest DBP formation. Since bromide is frequently observed in natural and marine water, it was found to be more active with DOM during chlorination (Chen *et al.* 2018). It can alter the DBP speciation composition and promote brominated DBP formation, which might be attributed to the reaction between bromide ion and free chlorine to form hypobromous acid/hypobromite ion (Liu & Croue 2016; Jiang *et al.* 2018). An earlier study also demonstrated the increased



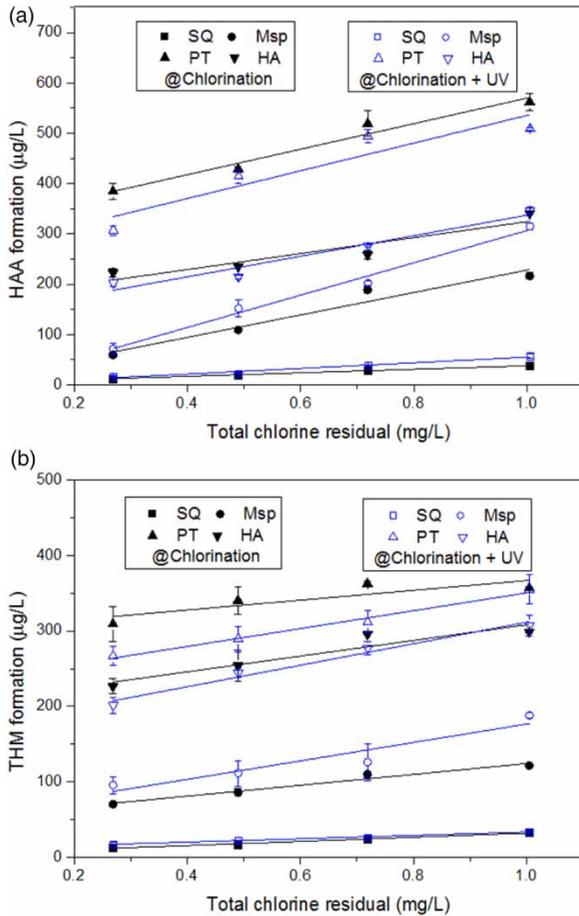
**Figure 3** | DBP formation with a variation of residual chlorine (a) and (b), 40 mJ/cm<sup>2</sup> UV irradiation followed by chlorine dose (c) and (d) after 2-h incubation in dark for DOM spiked water sample with initial DOC of 3.09 ± 0.19 mg/L.

brominated total organic halogen after the UV irradiation/chlorination process (Zhao *et al.* 2011).

For the other DOM, DCAA and TCAA were the dominant HAA species for SQ, Msp, and HA with the total HAA formation of 38.23 ± 1.25, 313.56 ± 6.05, and 340.82 ± 9.29 µg/L, respectively, when the total chlorine residual was around 1.0 mg/L. It was noted that the DCAA/TCAA from each EOM followed a similar trend that was obtained with extracted HA and fulvic acid from surface water (Hua *et al.* 2017). In this study, TCAA formed was less than DCAA, indicating that hydroxyl groups account for a major proportion in the chemical structure of EOM (Reckhow & Singer 1985). However, HA formed more TCAA than DCAA, which demonstrated that the oxidizable functional groups (e.g. conjugated system) are abundant in the chemical structure of HA; it is reasonable considering its higher SUVA value of 3.18.

The yield of chlorinated DBPs decreased with the increasing bromide concentration, while it led to the increase of brominated DBPs. However, in the current study, bromide concentration is constant at 94.12 µg/L as stipulated by the growth media. Based on the previous studies, the formation of BCAA and DBAA was still lower than that of TCAA and DCAA when the solution was spiked with high bromide concentration up to 200 µg/L (Liu *et al.* 2018). However, after adding bromide up to 400 µg/L, the formation of DBAA and BCAA was higher than that of TCAA and DCAA. Our study indicates that bromide concentration of 94.12 µg/L might not change DBP speciation considerably. Dong *et al.* (2021) also showed that the formation of DCAA was higher than BCAA and DBAA, when they spiked the solution with 100 µg/L bromide (Dong *et al.* 2021).

It should be noted that TCM was the only THM species formed from SQ, Msp, and HA. TBM (Figure 3(b)) was the



**Figure 4** | The comparison of DBP formation and the correlation with total residual chlorine of DOM spiked water sample between chlorination and UV irradiation of 40 mJ/cm<sup>2</sup> followed by chlorination after 2-h incubation in dark.

most abundant species of THM for bromide-containing water of PT. It was interesting to find that even Msp showed the highest chlorine demand (Figure 2), the DBP formation was much lower than PT and HA. Overall, except SQ, the DBP formation by the other three DOM (i.e., Msp, PT, and HA) always exceeded the DBP limit of 80 µg/L for HAAs and 100 µg/L for THMs as regulated by the guidelines for Canadian drinking water quality (Wang *et al.* 2015b).

The effect of UV radiation at 40 mJ/cm<sup>2</sup>, a typical dosage used for disinfection, caused a slight decrease in DBP formation after chlorination, although the effect was insignificant (Figure 3). The comparison of the DBP formation between with and without UV irradiation is shown in Figure 4, and the regression coefficients are presented in Table 1. There was no increase in DBP formation after UV radiation for SQ and HA (Figure 4). This is reasonable as much higher dosage is required for effective photolysis of trace concentration of organics (micropollutants) (Chowdhury *et al.* 2020). However, there is a small amount of DBP formation changes for PT and Msp, where both HAA and THM formation decreased for PT, but with a small increase for Msp.

Table 1 shows the regression coefficients between each of the HAAs/THMs and total residual chlorine. The strong correlations (0.936–0.999) found between HAAs/THMs and total residual chlorine indicated that experimental conditions were well controlled and total residual chlorine as a variable showed robust correlations with DBP formation from each DOM. Similar results were found in a previous study (Dyck *et al.* 2015).

**Table 1** | Regression parameters of total residual chlorine and DBP formation

	SQ		Msp		PT		HA	
	TC	TC + UV	TC	TC + UV	TC	TC + UV	TC	TC + UV
<b>HAAs</b>								
Intercept	3.06	−2.06	−15.41	5.2	316.17	259.16	165.65	131.94
Slope	34.82	56	321.18	222.82	253.56	275.32	158.68	204.3
R <sup>2</sup>	0.999	0.984	0.995	0.975	0.984	0.936	0.94	0.976
<b>THMs</b>								
Intercept	4.46	10.69	52.24	53.8	301.49	230.91	204.08	168.28
Slope	27.53	22.18	71.98	122.21	65.21	115.12	103.92	42.58
R <sup>2</sup>	0.993	0.996	0.984	0.953	0.958	0.971	0.943	0.99

## Effect of UV irradiation on DBP formation

To further investigate the DBPFP of each DOM with a variation of UV dosage, a comparison was made under identical experimental conditions (the UFC method), and the results are shown in Figure 5. The EOM from PT presents the highest specific DBPFP up to 232  $\mu\text{g}/\text{mg C}$  for HAAFP and 138.79  $\mu\text{g}/\text{mg C}$  for THMFP. TBM species dominated more than an average of 56.28% of THMs at each UV dosage, compared with around 24% brominated acetic acid formed, which indicated that TBM is easier to be formed in the presence of bromide ions. It is clear to see that with the increase of UV dosage, both HAAFP and THMFP decreased, with the maximum reduction of 42.25 and 13.75% for HAAFP and THMFP at a UV irradiation dosage of 300  $\text{mJ}/\text{cm}^2$ , respectively. The

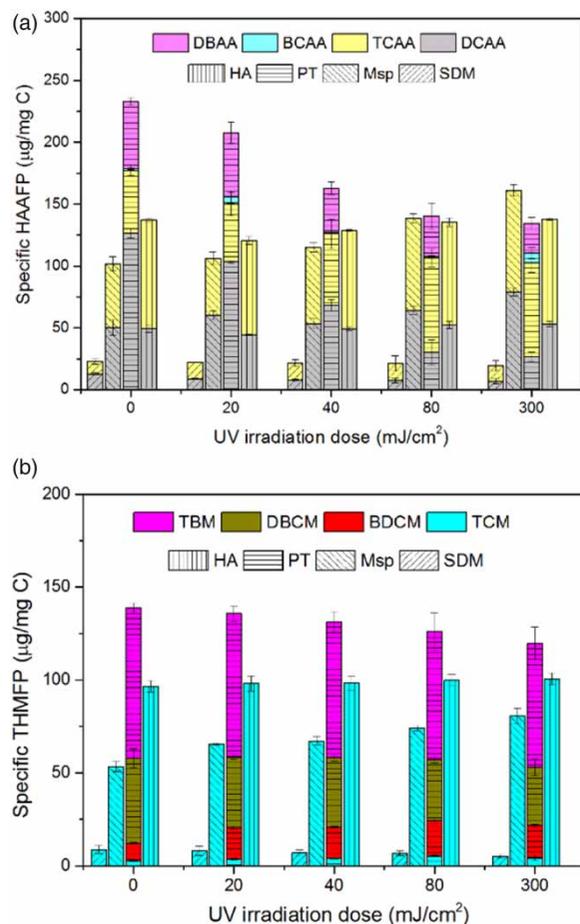


Figure 5 | Specific DBPFP: (a) HAAFP and (b) THMFP of DOM spiked water sample.

previous study presented that UV irradiation with a dosage of 396  $\text{mJ}/\text{cm}^2$  remarkably decreased HAA in DOM, which might result from the change of humic-like component and protein (Dong *et al.* 2019). In a different study, about 16.4% of THM reduction was found at a UV dosage of 100  $\text{mJ}/\text{cm}^2$  (Chen *et al.* 2018). This is probably due to the photolysis of DOM to alter the SUVA by UV irradiation (Hu 2016), thus decreasing the final formation of DBP. A similar trend was also observed from THMFP of SQ, even though the effect was not significant. Except PT, the brominated species did not feature among HAAs and THMs assayed for SQ, Msp, and HA during chlorination. It was noted that DBPFP of Msp increased slightly with the UV dosage increase, with the maximum formation of  $160.95 \pm 3.30$  and  $80.44 \pm 4.14$   $\mu\text{g}/\text{mg C}$  for HAAFP and THMFP, respectively, at a UV irradiation dosage of 300  $\text{mJ}/\text{cm}^2$ . This result was not consistent with the previous finding which might be due to different precursor properties of DOM. It was also reported that UV treatment might pose a source-specific effect (Zhang *et al.* 2016a), and TCAA formation was promoted by 19% at a low UV dose (Chen *et al.* 2018).

It was reported that UV irradiation can eliminate the aromatic, methyl, guaiacyl lignin group in HA and decrease the formation of TCM during subsequent chlorination. Conversely, UV irradiation can also activate the phenolic hydroxyl group and promote DCAA and TCAA formation (Wang *et al.* 2015a), and thus the final DBPFP of HA may not be changed significantly under moderate UV irradiation dosage.

## CONCLUSIONS

The investigation of DBPFP of selected DOM with a variation of total residual chlorine and UV irradiation was performed in this study. DBP formation was highly correlated with residual chlorine EOM from PT presented the highest formation potential followed by HA, Msp, and SQ. The comparison of DBP formation between with and without UV irradiation for each type of DOM indicates that 40  $\text{mJ}/\text{cm}^2$  UV irradiation may be insignificant to change the DBP formation from HA and SQ. However, it can decrease the DBP formation from bromide-containing

EOM of PT and can promote the DBP formation from EOM of Msp at various total residual chlorine. The maximum DBPFP reduction of 42.25 and 13.75% for HAAFP and THMFP was obtained at a UV irradiation dosage of 300 mJ/cm<sup>2</sup> for EOM of PT. However, for the EOM derived from Msp, a maximum increase of 58.1 and 51.1% of HAAFP and THMFP was observed. The results implied that further attention should be given to determine the effectiveness of UV irradiation on the degradation of algal matter.

## ACKNOWLEDGEMENTS

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## DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

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