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Formation potential of disinfection byproducts during chlorination of petroleum hydrocarbon-contaminated drinking water

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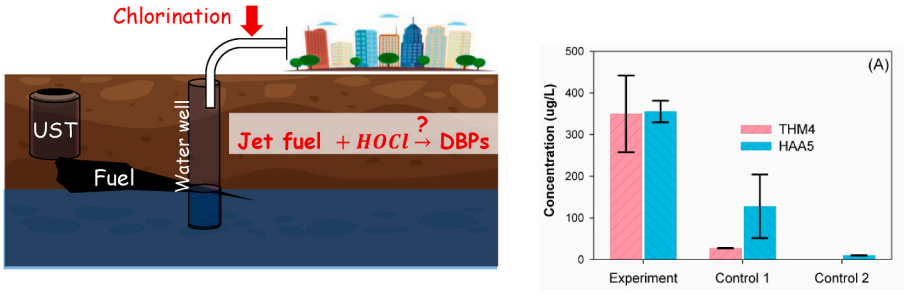
HIGHLIGHTS

- Regulated DBPs were produced when jet fuel petroleum hydrocarbons reacted with free chlorine.
- Chloroform was the most abundant regulated THM compound, followed by bromodichloromethane and dibromochloromethane.
- Monobromoacetic acid was the most abundant regulated HAA, followed by dibromoacetic acid and monochloroacetic acid.
- A large diversity of unregulated DBPs, including a complex group of high-molecular-weight compounds, were also detected.

GRAPHICAL ABSTRACT



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ABSTRACT

Recent leaks of underground fuel storage tanks in the Pearl Harbor region have led to direct release of un-weathered petroleum hydrocarbons (PHCs) into drinking water sources, which then directly underwent chlorination disinfection treatment. Since the control of disinfection byproducts (DBPs) traditionally focuses natural organic matters (NOM) from source water and little is known about the interactions between free chlorine and un-weathered PHCs, laboratory chlorination experiments in batch reactors were conducted to determine the formation potential of DBPs during chlorination of PHC-contaminated drinking water. Quantitative analysis of regulated DBPs showed that significant quantities of THM4 (average 3,498 μg/L) and HAA5 (average 355.4 μg/L) compounds were formed as the result of chlorination of un-weathered PHCs. Amongst the regulated DBPs, THM4, which were comprised primarily of chloroform and bromodichloromethane, were more abundant than HAA5. Numerous unregulated DBPs and a large diversity of unidentified potentially halogenated organic compounds were also produced, with the most abundant being 1,1-dichloroacetone, 1,2-dibromo-3-chloropropane, chloropicrin, dichloroacetonitrile, and trichloracetonitrile. Together, the results demonstrated the DBP formation potential when PHC-contaminated water undergoes chlorination treatment. Further studies are needed to confirm the regulated DBP production and health risks under field relevant conditions.

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1. Introduction

Releases of petroleum hydrocarbons (PHCs) during drilling, processing, transportation, storage, and final usage continue to cause frequent environmental pollution and significant human health challenges (Todd et al., 1999; Guo et al., 2022). PHC contamination of drinking water sources, including surface waterbodies and groundwater aquifers (Ossai et al., 2020), represents the most direct and consequential route of human health exposure. To assess human health risks from PHCs in drinking water sources, studies have traditionally focused on indicator compounds (e.g. BTEX and PAHs) and fuel additives (such as MTBE), which have documented adverse human health effects. In the past two decades, increasingly more efforts have been made to understand the human health risks associated with total petroleum hydrocarbons (TPHs), which encompass a wider range of PHCs as well as their degradation products in the environment (ITRC, 2018).

Additional human health risks may also arise when PHCs in the contaminated water undergo reactions during drinking water treatment and distribution processes. Chlorination is a commonly used disinfection process to control microbial pathogens and to reduce microbiological risks in drinking water, with the main adverse effect being the formation of disinfection byproducts (DBPs) (Rook, 1976). The regulated DBPs, which are also most commonly detected, include trihalomethanes (or THM4, including chloroform, bromodichloromethane (BDCM), dibromochloromethane (DBCM), and bromoform) and haloacetic acids (HAA5, including monochloroacetic acid (MCAA), dichloroacetic acid (DCAA), trichloroacetic acid (TCAA), monobromoacetic acid (MBAA), and dibromoacetic acid (DBAA)). Many studies have reported significant adverse health effects of the regulated DBPs; for example, THM concentrations in drinking water were linked to cancers in bladder, kidney and colon (Cantor et al., 1978; Sun et al., 2021). However, studies on DBP formation have traditionally focused on natural organic matter (NOM) in source water (Bond et al., 2009), while no studies on DBP formation from chlorination of PHC-contaminated water have been reported in the literature.

Recently, several major fuel spill incidents in the Pearl Harbor region on the island of Oahu, Hawaii, have led to direct contamination of groundwater aquifers by PHCs released from underground storage tanks. Some of the PHC contaminants entered groundwater wells without the usual weathering and degradation processes in the groundwater aquifer. For example, On Dec. 10, 2021, 140,000 ppb of total petroleum hydrocarbon diesel was detected in groundwater well water samples, which is 350 times of the local drinking water environmental action level (HDOH, 2021). Since the well water is typically directly chlorinated and then pumped into the distribution systems for consumption, there was a possibility of direct encountering between free chlorine and free products. Previous studies reported that free chlorine can react with diverse organic compounds, including aliphatic and aromatic olefinic hydrocarbons (Li et al., 2020), elevated NOM in shale gas wastewater (Parker et al., 2014; Huang et al., 2019), polycyclic aromatic hydrocarbons (PAHs) (Liu et al., 2020), and result in increased formation of DBPs and other chlorinated organics.

The objective of this study was to conduct chlorination experiments and determine whether un-weathered PHCs from jet fuels can react with free chlorine to produce regulated DBPs. Laboratory batch reactors containing both un-weathered PHCs and free chlorine were compared with two sets of control reactors that either contain only free chlorine or only PHCs. Samples from the reactors were analyzed to detect and quantify the regulated THM4 and HAA5 compounds, and results from the reactors were compared to determine the impact of PHCs on the formation of regulated DBP. The production of unregulated DBPs and other potential halogenated organic compounds from chlorination of PHCs was also examined.

2. Materials and methods

Standards and chemicals. The standards used in this experiment were prepared from dilutions of commercially available certified reference materials. For THM analysis, a chlorinated disinfectant by-product mix containing 14 DPBs at concentrations of 2000 μg/L (SPEX CertiPrep; Metuchen, NJ.) was used. For HAA analysis a haloacetic acid standard mix containing 6 HAAs at concentrations of 2000 μg/L was used (Restek; Centre County, PA). All chemicals used in this study were ACS grade or higher, and a pesticide-grade methyl-*t*-butyl ether (MTBE; Thermo Scientific, MA) with a purity of 99% or greater was used. The precursor JP-5 fuel was provided by NAVFAC Hawaii.

Chlorination experiments. Batch experiments were conducted in serum bottles (60 mL) capped with Teflon-lined stoppers and sealed with aluminum caps. Three sets of reactors (all in triplicate) were prepared, including the Experiment reactors (2.7 mM JP-5 jet fuel plus 27 mM free chlorine), Control 1 reactors (27 mM free chlorine only), and Control 2 reactors (2.7 mM jet fuel only). Each reactor contained 60 mL of synthetic freshwater (Smith et al., 2002) buffered at pH 7.0 with 10 mM phosphate buffer. Additional 0.0088 mM bromide was added to the synthetic freshwater to simulate the typical bromide concentrations in Hawaii groundwater.

JP-5 jet fuel was added to the Experiment reactors and Control 2 reactors to reach a final concentration of 2.7 mM. JP-5 molar concentrations were calculated by using average molar weights reported for JP-5 (Luning Prak et al., 2019). The particular type of jet fuel was selected to simulate the recent spills in the Pearl Harbor region (USEPA, 2023). The free chlorine stock solution was prepared using a sodium hypochlorite solution, and standardization of the free chlorine stock solution was performed using a PerkinElmer UV-VIS spectrophotometer by measuring the absorbance of OCl^- at 292 nm ($\varepsilon_{292nm} = 362\ M^{-1}\ cm^{-1}$) (Kumar and Margerum, 1987). Sodium hypochlorite was then added to the Experiment reactors and Control 1 reactors to reach a free chlorine concentration of 27 mM. For the Experiment reactors, this resulted in a 1:10 M ratio between PHCs and free chlorine (Li et al., 2020). All reactors were then subjected to vigorous mixing in dark for 48 h at room temperature (25°C). The chlorination experiment was stopped by adding ammonium chloride (45.9 mM), achieving a 1.7:1 M ratio of ammonium chloride: free chlorine (Moore et al., 2021). The reactors were then stored at 4 °C until DBP extraction.

DBP extraction. The chlorination experiment described above was repeated twice, once for the extraction and quantification of THM4 and once for HAA5. Extraction and quantification of THM4 followed the U.S. EPA Method 551.1 (USEPA, 1995). Pesticide-grade MTBE was used as the solvent for THM4 extraction. After extraction, the solvent phase was transferred to autosampler vials for gas chromatography-electron capture detection (GC-ECD) analysis. Extraction and quantification of the regulated HAA5 followed the EPA method 552.3 (USEPA, 2003).

DBP detection and quantification. All DBP measurements were performed on a Thermo Scientific Trace 1300 GC-ECD. For the analysis of THM4, a Rxi-624Sil column (30 m × 0.32 mm x 1.8 μm; Restek; Pennsylvania, US) was used. The injection volume was set to 2 μL. The carrier gas was helium, set to constant flow at 1.8 mL/min, with an injector temperature of 200 °C and a detector temperature of 290 °C. The oven temperature program began at 35 °C (hold 0.5 min), to 95 °C at 30 °C/min (hold 2 min), to 260 °C at 30 °C/min (hold 1 min), to 320 °C at 40 °C/min (hold 5 min). For HAA analysis, a Rtx-CLPesticides column (30 m × 0.32 mm x 0.32 μm; Restek; Pennsylvania, US) was used. The injection volume was set to 1 μL.The carrier gas was set to constant flow at 1.8 mL/min, with an injector temperature of 210 °C and a detector temperature of 290 °C. The oven temperature program began at 35 °C (4 min hold), then increased to 250 °C at 15 °C/min and hold for 5 min.

The MTBE solvent blank showed a small peak at the same elution time as chloroform, which however was significantly smaller than the chloroform peak detected in the samples and was subtracted during the analysis. All equipment used for the experiments and analysis were

either glass or stainless-steel. The glassware was acid-washed in 5 % sulfuric acid and rinsed with MTBE prior to use. Stainless-steel spatulas were solvent rinsed using MTBE. The calibration standards for THM4 (SPEX; New Jersey, US) also contained standards for eleven unregulated DBPs (1,1,1-trichloroethane, 1,1-dichloroacetone, 1,2-dibromo-3-chloropropane, 1,2-dibromoethane, carbon tetrachloride, chloropicrin, dibromoacetonitrile, dichloroacetonitrle, tetrachloroethene, trichloroacetonitrile, trichloroethene), which therefore were also quantified using the same method. The THM4 standards were prepared at five different concentrations of 10 μg/L, 20 μg/L, 50 μg/L, 75 μg/L, and 100 μg/L of each analyte in artificial freshwater medium and extracted using the same extraction procedure as described above. The calibration standards for HAA5 (RESTEK; Pennsylvania, US) with concentrations of 50 μg/L, 100 μg/L, 150 μg/L, 200 μg/L, and 250 μg/L were also prepared in artificial freshwater medium and then subjected to extraction and subsequent methylation. The calibration standards for HAA5 also included the unregulated bromochloroacetic acid, which was also quantified simultaneously. All calibration curves exhibited R^2 values greater than 0.99.

DBP identification. GC-MS was used to identify some of the unknown compounds detected at higher retention times. All measurements were performed on a Thermo Scientific Trace 1300 GC coupled to an ISQ_LT mass spectrometer. The column as well as injector and oven conditions were kept similar to what was used for THM4. A 1 μL splitless injection was performed with a splitless time of 1 min. The ion source was maintained at 315°C and the transfer line at 300°C. The mass range scanned was from 35-450 with a dwell time of 0.2s. Mass spectra of unknown compounds were searched in the NIST MS library for tentative identification.

Data analysis. ANOVA was used to compare the amount of DBPs produced amongst the three groups of reactors, while Post Hoc Tukey-HSD test was performed to compare individual groups. All statistical analyses were performed in the *R* environment.

3. Results and discussion

Formation of regulated DBPs. Large quantities of regulated DBPs (THM4 and HAA5) were formed in the Experiment reactors that contained both PHCs and free chlorine (Fig. 1). The total THM4 concentrations in the Experiment reactors averaged at 3,498 μg/L (range: 2066–3915 μg/L, and std dev. = 921 μg/L), while the total HAA5 concentration averaged at 355.4 μg/L (range: 321.1–412.1 μg/L, and std dev. = 25.7 μg/L). In contrast, the Control 1 and Control 2 reactors either produced significantly lower concentrations of DBPs (ANOVA, $P < 0.01$) or no detection at all.

In the Control 1 reactors which contained free chlorine but no spiked PHCs, significantly smaller quantities of THM4 (range: 269–278 μg/L, and std dev. = 3.7 μg/L) and HAA5 (range: 19.7–344.6 μg/L, and std dev. = 76.3 μg/L) than the Experiment reactors were detected. The formation of a small amount of THM4 and HAA5 in the Control 1 reactors was likely caused by the reaction of free chlorines with residual organic matters present in the materials used in the experiments, which was also observed by other studies where low levels of DBPs in chlorination experiments were also reported in the controls (Sun et al., 2019; Jiang et al., 2022). Nevertheless, the amounts of DBPs produced in the Experiment reactors were exceeding the small amount of DBPs detected in the Control 1 reactors by 13 and 6 folds for THM4 and HAA5, respectively. The post-hoc test confirmed the difference between the Experiment and Control 1 reactors was statistically significant ($P < 0.01$). In the Control 2 reactors, which contain only PHCs but no free chlorine, no THM4 was detected, and only trace amounts of three regulated HAA5 compounds (TCAA, DCAA, and DBAA) were detected in one of the triplicate reactors. Additional analysis of the PHC source (i.e. jet fuel JP-5) dissolved in the solvent MTBE confirmed that no THM4 or HAA5 were present in the jet fuel itself (data not shown).

Comparison between the Experiment reactors and the two sets of Control reactors clearly indicates that the presence of PHCs and free chlorine was responsible for the elevated concentrations of regulated DBPs. Previous laboratory experiments that focused on NOM chlorination have typically reported DBPs in the range of 1–100 μg/L (Yang et al., 2013; Zhao et al., 2016). The high levels of regulated DBPs detected in this experiment are likely the result of the high concentration of PHCs used in the chlorination experiment, as the concentration of organic carbon is an important factor in the formation of DBPs (Chang et al., 2001; Kim et al., 2002). Many of the experiment conditions used in this study were also conducive for DBP formation, including high concentration of free chlorine (Hua and Reckhow, 2007), bromide concentration (Chang et al., 2001), pH (Yang et al., 2015) and reaction time (Doederer et al., 2014). Overall, the molar conversion ratio (i.e. concentration of DBPs versus PHCs) were 0.91% for THM4 and 0.087% for HAA5.

Composition of regulated DBPs. Individual THM4 and HAA5 compounds were detected at different concentration levels in the Experiment reactors (Fig. 2). The most abundant regulated THM compound was chloroform (1722 ± 262 μg/L in the triplicate reactors),

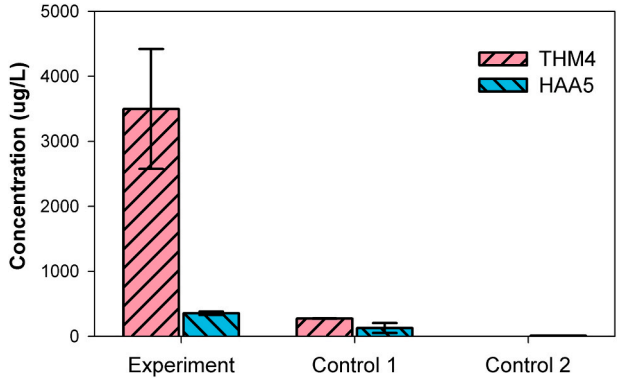

Fig. 1. Total concentrations of regulated THM4 and HAA5 in the Experiment (PHCs + HOCl), Control 1 (HOCl only), and Control 2 (PHCs only) reactors.

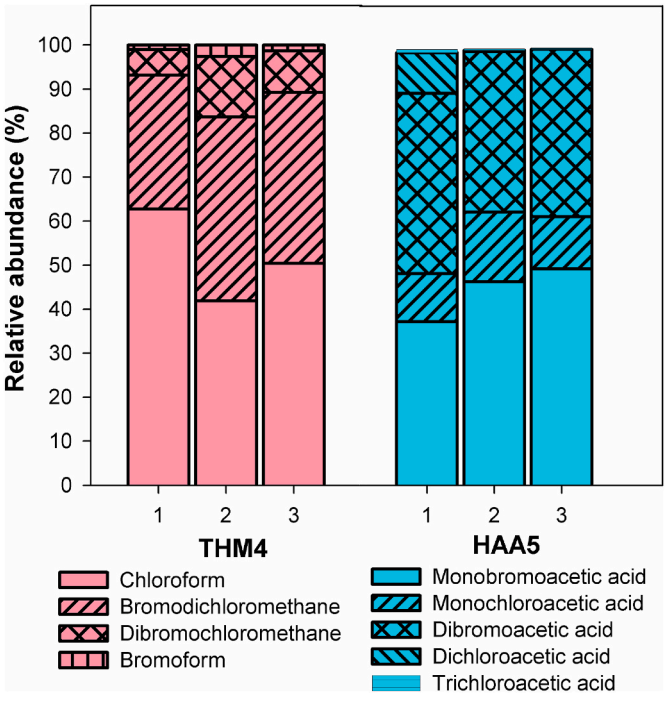

Fig. 2. Composition of the regulated THM4 and HAA5 compounds detected in the triplicate Experiment (PHCs + HOCl) reactors.

accounting for an average of 53.1% (std dev. = 8.0%) of total THM4 compounds detected. The next highest concentration was detected for BDCM (1344 ± 467 μg/L, 36.2 ± 4.7%), followed by DBCM (370 ± 167 μg/L, 9.2 ± 2.8%), and bromoform (163 ± 35 μg/L, 1.5 ± 0.6%). Amongst the regulated HAA, 5 compounds were detected in the Experiment reactors. MBAA (159.1 ± 29.1 μg/L) was the most dominant HAA, accounting for 44.6 ± 5.2% of total HAA5 compounds detected. DBAA accounted for 38.8 ± 1.8%, and MCAA accounted for 13.0 ± 2.2%, while dichloro- and trichloroacetic acid were only detected in smaller quantities and in two of the three Experiment reactors.

Several previous studies reported a similar abundance pattern for THM4 (chloroform > BDCM > DBCM > bromoform) by chlorination of NOM (Bond et al., 2009; Yang et al., 2011). Chloroform was previously reported as the most abundant THM when compared to other THM4 compounds (Lu et al., 2009), which was also the most abundant THM4 in this study. The detection of brominated DBPs in this experiment also corresponds to previous studies (Chang et al., 2001), and was due to the introduction of bromide to simulate the level detected in Hawaii's groundwater. Several epidemiological studies identified greater health risks stemming from brominated DBPs over corresponding chlorinated DBPs (Sharma et al., 2014).

In this study, a significantly higher concentration of total THM4 than that of HAA5 was observed, which is a similar general trend compared to other studies (Padhi et al., 2019). Interestingly, the relative abundance of individual HAA5 compounds detected in this study showed different distribution than previous studies, as MBAA was more abundant than DBAA and DCAA. Previous studies typically reported di-HAAs and tri-HAAs as the primary HAAs observed (Padhi et al., 2019; Stefán et al., 2019; Zhang et al., 2020). This may be due to the different amounts of bromide involved in the reaction, the different precursors, and other environmental conditions (Chang et al., 2001).

Unregulated halogenated compounds or potential unidentified DBPs. Currently, EPA is implementing the stage 1 and stage 2 disinfectants and disinfection byproducts rules which regulate the amount of THM4 and HAA5 as well as bromate and chlorite, although the need for studying other halogenated compounds as potential DBPs has been long identified (Weinberg et al., 2002). There have been growing evidences indicating that the one- and two-carbon-atom DBPs, including the regulated THM4 and HAA5, only accounts for about 16% of cytotoxicity in disinfected drinking water (Mitch et al., 2023). Additional 11 unregulated halogenated compounds were analyzed in the THM sample preparations because the THM sample preparations permitted better chromatographical identification than the HAA sample preparations. Amongst the 11 halogenated compound targets, five were detected in the Experiment reactors, including 1,1-dichloroacetone, 1,2-dibromo-3-chloropropane, chloropicrin, dichloroacetonitrile, and trichloracetonitrile as unregulated potential DBPs (Fig. 3A). Most of these unregulated potential DBPs formed in the Experiment reactors were not formed in any of the control reactors (Fig. 3B), indicating that the formation occurred due to the presence of PHCs and free chlorine together.

These unregulated potential DBPs had an average total concentration of 386.2 μg/L (range: 241.7–505.4 μg/L, and std dev. = 104.4 μg/L) during chlorination of PHCs in the Experiment reactors, which was significantly less than the regulated THM4 and HAA5. Other studies also reported formations of unregulated DBPs, mainly haloacetaldehydes, haloacetonitriles, haloacetamides, halonitromethanes, and haloketones, and the detected quantities were also smaller than THM4 and HAA5 (Szczuka et al., 2017; Xu et al., 2020). The most abundant haloacetonitrile in previous reports was dichloroacetonitrile (Mian et al., 2018), which was also the most abundant one detected in this study (range: 80–149 μg/L, and std dev. = 126 μg/L). Haloacetonitriles have been reported to have higher toxicity compared to regulated DBPs (Liu et al., 2018). Dichloroacetone was also detected in notable amounts (range: 84–177 μg/L, and std dev. = 41 μg/L). Additionally, smaller quantities of chloropicrin (range: 12–20 μg/L, and std dev. = 4 μg/L), 1,2-dibromo-3-chloropropane (range: 4.6–7.6 μg/L, and std dev. = 1.5 μg/L), and trichloroacetonitrile (range: 3.8–5.4 μg/L, and std dev. = 0.9 μg/L) were formed in this study.

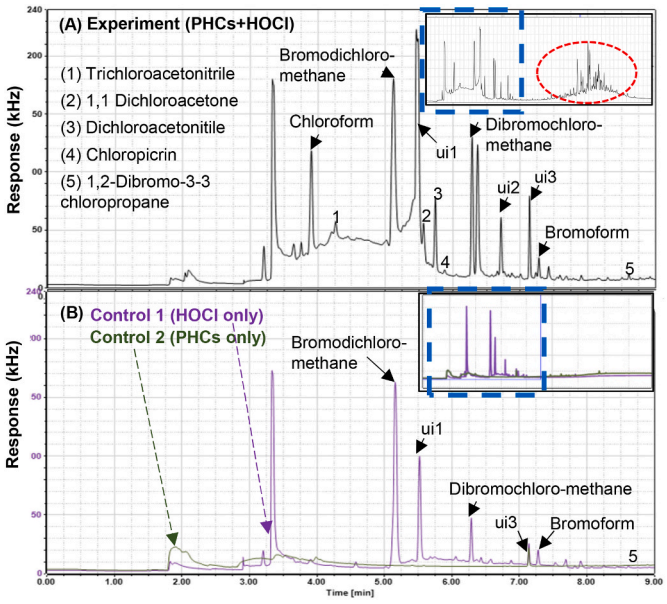

Fig. 3. GC-ECD chromatograms showing regulated THM4, other unregulated DBPs, and potential DBP peaks in the Experiment reactors (A) versus in the Control 1 and 2 reactors (B). The inserts show the full chromatographs that contain the sections shown in amplification (blue dashed boxes) and other unidentified potential DBP peaks (red dashed ovals). (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

Besides the unregulated DBPs that can be identified in this study, numerous others GC-ECD peaks were detected either only in the Experiment reactors or with significantly higher abundance in the Experiment reactors than in the Control reactors. For example, several large peaks were detected in the THM4 chromatograms with retention times close to those of identified DBPs (e.g. Fig. 3A and B, ui1, ui2, ui3). A large cluster of peaks with much longer retention times were also detected in the Experiment reactors (Fig. 3A; red dashed oval) but not in any of the Control reactors (Fig. 3B). These compounds are potentially high molecular weight halogenated organics as disinfection byproducts with uncharacterized human health impacts; similar observations were also made by previous studies on chlorination of humic substances (Zhang et al., 2005; Mitch et al., 2023). Some of these halogenated organics were tentatively identified by GC-MS through MS library searches. Compounds with library matches included 1-chloroundecane, 1-chloromethylnaphthalene, 1-chloromethyl-4-methylnaphthalene, 2,4-dibromo-1,3,5-trimethylbenzene, 2-bromo-4.6-di-*tert*-butylphenol, 2,7-dibromo-naphthalene, 1-bromo-4-methylnaphthalene, and 2,7-dibromo-1-methyl-naphthalene (data not shown).

3.1. Implications

Contamination of soil and groundwater aquifers by PHCs from point sources such as fuel storage leaks (USEPA, 2023) and major petroleum spills (Robertson, 1996; Fryirs et al., 2015) have frequently occurred before, yet direct introduction of PHCs into groundwater wells without weathering, as observed in the recent contamination events in the Pearl Harbor aquifer, is rare. Free chlorine is highly reactive and is known to react with various organic compounds to produce halogenated organic compounds, including regulated and unregulated DBPs (Deborde and Von Gunten, 2008; Gan et al., 2020). This study showed that elevated levels of regulated DBPs can form from chlorination of un-weathered PHCs in synthetic freshwater. This provide a strong indication of the risk of DBP production when un-weathered PHCs are subjected to

chlorination both before and within the drinking water distribution systems.

It should be noted that the experiments in this study were conducted at laboratory-controlled and optimized conditions to demonstrate the potential for DBP formation when PHCs encounters free chlorine. Further studies are needed to understand the DBP formation potential and health risks under field-relevant conditions including groundwater and chlorination chemistry and distribution system physicochemical and hydraulic parameters. Besides the regulated THM4 and HAA5, results of this study also indicated the presence of diverse halogenated organic compounds, unregulated potential DBPs, and other unidentified compounds that are potentially halogenated organics as DBPs with yet-unknown risks. The observation of a large number of unidentified GC-ECD peaks at higher retention times suggests the production of high-molecular-weight halogenated organic compounds as potential DBPs during the chlorination of PHCs, highlighting the need of further research for their identification and risk characterization in order to optimize engineering decision making and human health protection.

CRediT authorship contribution statement

Mandy-Tanita Brinkmann: Writing – original draft, Visualization, Formal analysis, Data curation. **Kexin Rong:** Methodology, Data curation. **Yuefeng Xie:** Writing – review & editing, Methodology. **Tao Yan:** Validation, Formal analysis, Conceptualization, Visualization, Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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