Desalination and Water Treatment
www.deswater.com
doi: 10.1080/19443994.2016.1195286

57 (2016) 28698–28704
December

Taylor & Francis
Taylor & Francis Group

Formation of disinfection by-products during chlorine dioxide pre-oxidation of chironomid larvae metabolites followed by chlorination

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Received 9 April 2015; Accepted 20 May 2016

ABSTRACT

The objective of this research was to investigate the formation of disinfection by-products (DBPs) during chlorine dioxide pre-oxidation of chironomid larvae metabolites followed by chlorination. The mode of action of chlorine dioxide/chlorine combination was clarified, including the impact of different factors on DBP formation. The results indicated that pre-oxidation suppressed the production of most DBPs such as trihalomethanes, haloacetic acids, haloacetonitriles, and haloketones, compared to when chlorination was used alone. The concentrations of trichloromethane (TCM), dichloroacetic acid (DCAA), trichloroacetic acid (TCAA), dichloroacetonitrile (DCAN), and 1,1-dichloro-2-propanone (1,1-DCP) decreased with prolonged pre-oxidation times, with the highest yields observed for DCAA and TCAA. The solution pH had a distinct influence on DBP formation, with the concentrations of DCAA, TCAA, DCAN, 1,1-DCP, and 1,1,1-trichloro-2-propanone (1,1,1-TCP) initially increasing and then decreasing with increasing pH. The maximum concentrations of DCAA and TCAA were observed at pH 7–8, whereas the TCM content increased continuously on increasing the pH up to 8–9. Other DBPs reached their maxima at pH 6–7. Regarding thermal effects, the formation of 1,1,1-TCP enhanced at higher temperatures, while the concentrations of DCAN, 1,1,-DCP, and TCM first increased between 10 and 20°C and then decreased between 20 and 30°C. The concentrations of DCAA and TCAA decreased from 10 to 20°C and then increased between 20 and 30°C.

Keywords: Chlorine dioxide; Chlorination; Combined disinfection; Chironomid larvae; Disinfection by-products

1. Introduction

Since the discovery of trihalomethanes (THMs) in chlorinated water in the 1970s, disinfection by-products (DBPs) have become the focal point of water treatment. Currently, more than 700 DBP species have been discovered [1]. As described in numerous reports, DBPs detected in drinking water can be classified as THMs, haloacetic acids (HAAs), haloacetonitriles (HANs), haloketones (HKs) chloropicrin

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(TCNM), chloral hydrate (CH), etc. [2]. Trichloromethane (TCM) is generally considered a non-genotoxic carcinogen, whose mechanism of action involves cytotoxicity and regenerative cell proliferation [3]. Trichloroacetic acid (TCAA) can cause bacterial mutation; the newly discovered DBP dichloroacetic acid (DCAA) is carcinogenic to rodents, dichloroacetonitrile (DCAN) leads to mutagenicity in bacterial assays [4], and some of the DBPs (e.g. HANs, HNMs, and HAcAms) have significantly higher cyto and genotoxicity than the regulated THMs and HANs [5]. Besides, 1,1-dichloro-2-propanone (1,1-DCP) and 1,1,1-trichloro-2-propanone (1,1,1-TCP) exhibit proven carcinogenic and mutagenic effects on rats [6].

Many factors that can affect the formation of DBPsFP during disinfection have been studied, including the reaction time, pH, temperature, disinfectant concentration, and precursor properties. The formation of stable THMs and HAAs is favored by increased reaction time and chlorine dosage [7]. The concentration of DCAN reaches a maximum and then decreases with increasing chlorine dosage, continuously decreasing with prolonged reaction time [8]. The amounts of 1,1-DCP and 1,1,1-TCP formed increase with increasing chlorine dosages. Increasing the reaction time leads to a continuous increase in the 1,1-DCP concentration, while that of 1,1,1-TCP shows an inverse dependence [9]. Higher pH reduces HAA, DCAN, and 1,1,1-TCP formation, but favors the formation of THM [2,10].

Previous studies have determined that the formation of DBPs depends primarily on the source water quality and the nature of treatment processes [11]. In the 1920s, chironomid larvae were detected in urban water supply systems [12]. Although there are no indications that these organisms pose a threat to public health, their presence is still not desirable because of the associated poor hygiene [13]. Many researchers have recently turned their attention to chironomid larvae, with the aim of controlling their presence during the water purification process. Formation of DBPs during chlorination of chironomid larvae is observed when water treatment is performed under harsher conditions [14,15], and the results show that increased reaction time, chlorine dosage, and temperature favor the formation of relatively stable DBPs.

Chlorine dioxide is often used as a disinfectant/oxidant in drinking water treatment, since it can effectively inactivate viruses, bacteria, and protozoan pathogens owing to its strongly oxidizing character and longer preserved activity in the system (US EPA). It is also employed for color and algae removal, and for taste and odor control [16,17]. Lykins and Griese together with Linder et al. found that oxidation with chlorine dioxide prior to chlorination could reduce the levels of THMs and total organic halogens (TOX) [18,19]. Therefore, chlorination or chloramination is often carried out after pre-oxidation with chlorine dioxide.

Consistent efforts have been directed to the determination of the identities and toxicities of various DBP species and their classes. Special attention was paid to the development of simulation models for the formation of THMs, HAAs, and HANs to control their occurrence. However, their formation during chlorine dioxide pre-oxidation and subsequent chlorination of chironomid larvae metabolite solutions is still unknown. Therefore, the objectives of this study were to investigate the characteristics of DBPsFP removal during the combined disinfection process, by considering the chlorine dioxide and chlorine dosage, pre-oxidation time, pH, and temperature.

2. Materials and methods

2.1. Chemicals used

All solutions were prepared using deionized water. HPLC-grade methanol, acetone, and methyl *tert*-butyl ether (MTBE) were used. The chlorine (HOCl) stock solution (2,500 mg/L as Cl_2) was prepared from 4% sodium hypochlorite (NaOCl) solution and was periodically standardized by DPD/FAS titration [20]. The chlorine dioxide stock solution (99% purity) was prepared using the method described by Ruffell et al. [21], and was stored in the dark in amber vials. The disinfectant solutions were stored at 4°C and warmed to room temperature before use. Buffer solutions with pH 5, 6, 7, 8, and 9 were prepared using phosphate (Tianjin Chemical Plant, China); standard samples for THM, HAA, HAN, and HK analyses were obtained from Supelco.

2.2. Sample preparations

Chironomid larvae obtained from adult organisms were bred in our laboratory. The larvae were cultured in aerated 25 L glass aquaria filled with raw water. Subsequently, 500 larvae were put in a 1 L beaker with 500 mL of deionized water and kept at a constant temperature of 20°C. After one day, the water sample with thelarvae metabolites was filtered through a 0.45 μm membrane (to eliminate suspended solids, in order to minimize changes in the constituents), and analyzed within one week. The TOC concentration was measured, and the standards were prepared by diluting reagents to 12.5 mg/L.

2.3. Analytical methods

TOC was analyzed on an analyzer (TOC-VCPH, Shimadzu). The chlorine dioxide concentration was measured by using *N,N*-diethyl-*p*-phenylenediamine (DPD). Available chlorine was determined by DPD/FAS titration [22]. Analysis of selected DBPs was carried out by gas chromatography (GC Agilent 7890) using an electron capture detector (ECD), USEPA methods 551.1 [23] and 552.3 [24]. The THM, HAN, and HK concentrations were measured using a liquid-liquid extraction procedure with MTBE and acidic methanol according to USEPA method 551.1 [23]. An HP-5-fused silica capillary column (30 × 0.25 mm I.D. with 0.25 mm film thickness) was used. The GC-ECD was operated under the following conditions: detector, 290℃; injector, 200℃; injection volume, 1 mL; temperature program, 35℃ for 5 min, ramped to 75℃ at 10℃/min, held for 5 min, then ramped to 100℃ at 10℃/min, and then held for 2 min. For DCAA and TCAA analyses, the samples were pretreated with MTBE and acidic methanol according to the extraction/derivatization procedure uesd (USEPA Method 552.3, US EPA) [24]. An HP-5-fused silica capillary column (30 × 0.25 mm I.D. with 0.25 mm film thickness) was used. The injector, ECD, and GC oven temperature programs for compounds other than HAA_9 were: injector, 200℃; ECD, 290℃; oven, initial temperature of 35℃ for 9 min, ramped to 40℃ at 2℃/min and held for 8 min, ramped to 80℃ at 20℃/min, ramped to 160℃ at 40℃/min and held for 4 min; The HAA temperature program was: injector, 210℃; ECD, 290℃; oven, initial temperature of 30℃ for 20 min, ramped to 40℃ at 1℃/min, ramped to 205℃ at 20℃/min, and held for 4 min.

2.4. Experimental procedures

The stock solution of chironomid larvae metabolites was diluted with deionized water to prepare test solutions of 2 mg/L TOC concentration. Chlorine dioxide dosages of 0, 2, 4, 6, 8, and 10 mg/L and a chlorine dosage of 20 mg/L were used to treat the above-mentioned chironomid larvae metabolite solutions buffered at pH 7.0 and a temperature of 20 ± 2℃. The pre-oxidation time (2, 4, 6, 12, and 24 h), pH values (5, 6, 7, 8, and 9), and temperature (10, 20, and 30℃) were also varied.

3. Results and discussion

3.1. Effect of chlorine dioxide pre-oxidation dosage

Fig. 1 shows the formation of DBPs after a two-day disinfection of chironomid larvae metabolite solutions at different chlorine dioxide dosages, with a pre-oxidation time of 30 min and pH 7. The pre-oxidation could reduce the amounts of DCAA, TCAA, DCAN, TCM, 1,1-DCP, and 1,1,1-TCP compared to chlorination alone. The respective concentrations showed a distinct decrease with increasing chlorine dioxide dosage (chlorine dosage fixed at 20 mg/L). For example, the DCAN content decreased dramatically from 13.217 to 8.753 μg/L, when the chlorine dioxide dosage was increased from 4 to 6 mg/L. The amounts of the other DBPs tested decreased significantly when the chlorine dosage was below 2 mg/L, decreasing slowly at higher dosages. The chlorine dioxide can generate DCAN by direct oxidation of the precursor, while a small fraction can be

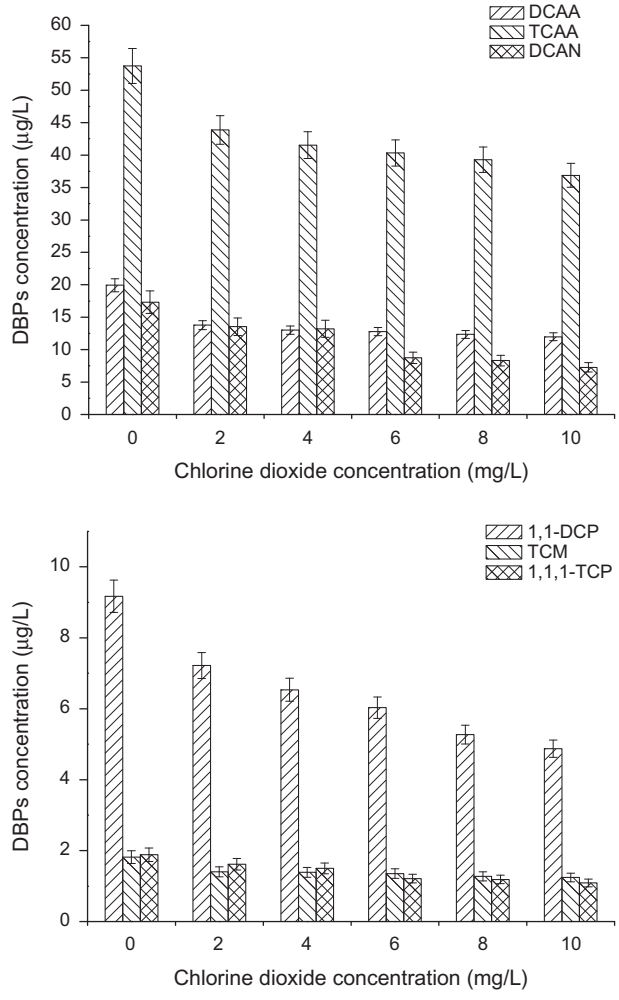

Fig. 1. Impact of chlorine dioxide pre-oxidation dosage on DBPsFP (pre-oxidation time 30 min, TOC 2 mg/L, pH 7, temperature of 20 ± 2℃). The error bars represent the standard deviation of replicate measurements, $n = 2$.

transformed into the precursor of the unstable TCAN. This means that the removal of the DBP precursor occurs when the chlorine dioxide dosage was between 0 and 10 mg/L. DCAN precursors were completely consumed by the disinfectant at 6 mg/L of chlorine dioxide. Aspartic acid (Asp) is an important precursor of DCAN and DCAA, and its removal prior to disinfection may prevent their formation [25]. Besides aspartic acid, some other amino acids may also form DCAN, and further hydrolyze to form DCAcAm and DCAA [26]. In addition, the levels of THMs and HAAs formed by chlorine dioxide oxidation of the organic precursors in raw water are much lower than those for the chlorination process [27], and their concentrations decrease with increasing chlorine dioxide dosage.

3.2. Effect of pre-oxidation time

Fig. 2 shows the time-dependent results of chlorine dioxide pre-oxidation. Among the tested DBPs, the DCAA and TCAA concentrations were the highest, while those of TCM and 1,1,1-TCP were the lowest. The yields of DCAA, TCAA, DCAN, TCM, and 1,1-DCP decreased with increasing pre-oxidation time, whereas that of 1,1,1-TCP changed in an opposite way, leading to the suggestion that, the precursor of 1,1-DCP could be easily converted to that of 1,1,1-TCP by chlorine dioxide. When the pre-oxidation times are extended, chlorine dioxide was persistently consumed and we could not confirm whether its final amount is sufficient to oxidize the precursor. Besides, the high precursor concentration could lead to a condition when the concentration of TCM is stable. The concentrations of DCAA, TCAA, DCAN, TCM, and 1,1-DCP reached their maximum after 2 h of reaction, and decreased continuously thereafter at extended pre-oxidation times. The above could be explained by hydrolysis and oxidation by chlorine after a certain time, since the presence of chlorine is known to increase the hydrolysis rate of DBPs [2]. The HAA precursor is highly reactive and can be easily oxidized by chlorine dioxide. The data indicate that the amount of DCAA formed dropped by 52%, and that of TCAA by 46%, showing that the amount of HAAs generated by chlorination can be effectively controlled. THMs are stable in the presence of chlorine, being a terminal product [7]; therefore, the concentration change in TCMs with increased pre-oxidation time is not obvious. In addition, it has been reported that 1,1-DCP can be oxidized to 1,1,1-TCP, in agreement with the increasing amount of 1,1,1-TCP with extended pre-oxidation time.

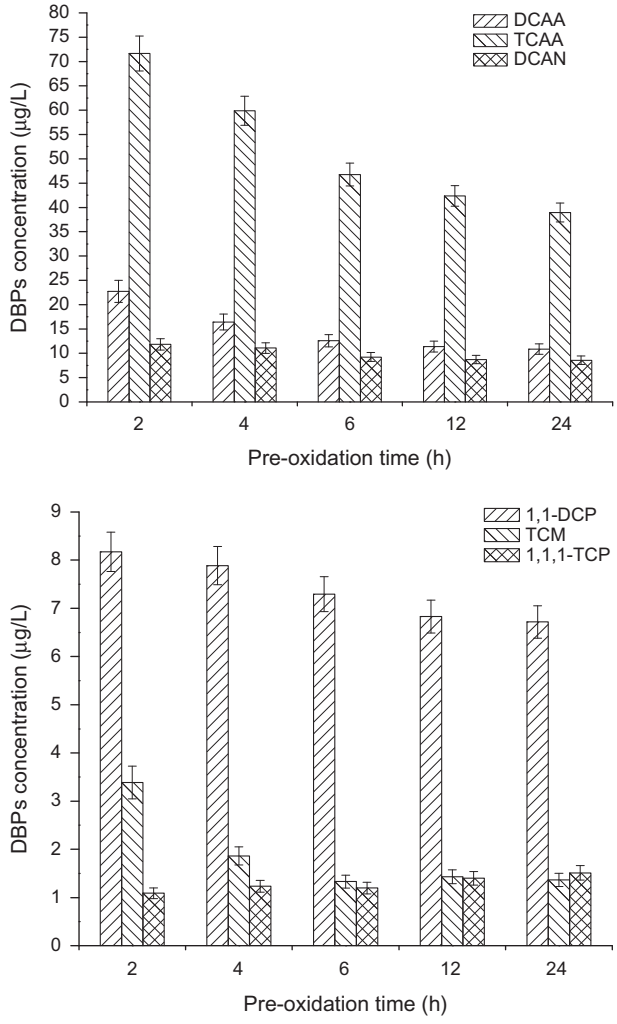

Fig. 2. Impact of pre-oxidation time on DBPsFP (chlorine dioxide dosage 2 mg/L, chlorine dosage 20 mg/L, TOC 2 mg/L, pH 7, temperature of 20 ± 2°C). The error bars represent the standard deviation of replicate measurements, $n = 2$.

3.3. Effect of pH

Fig. 3 shows the amounts of DBPs formed after two days of disinfection, i.e. chlorine dioxide pre-treatment (pre-oxidation time of 30 min) in combination with chlorination at various pH values. The content of DCAA and TCAA increased continuously when the pH was increased from 5 to 8, and then decreased as the pH was increased further from 8 to 9. The yields of 1,1,1-TCP increased quickly as the pH rose from 5 to 7, and then remained almost unchanged with increasing pH, with the maximal yield detected at close to neutral conditions. The amount of 1,1-DCP, and DCAN increased continuously with the pH increasing from 5 to 7, and then decreased quickly as the pH was further

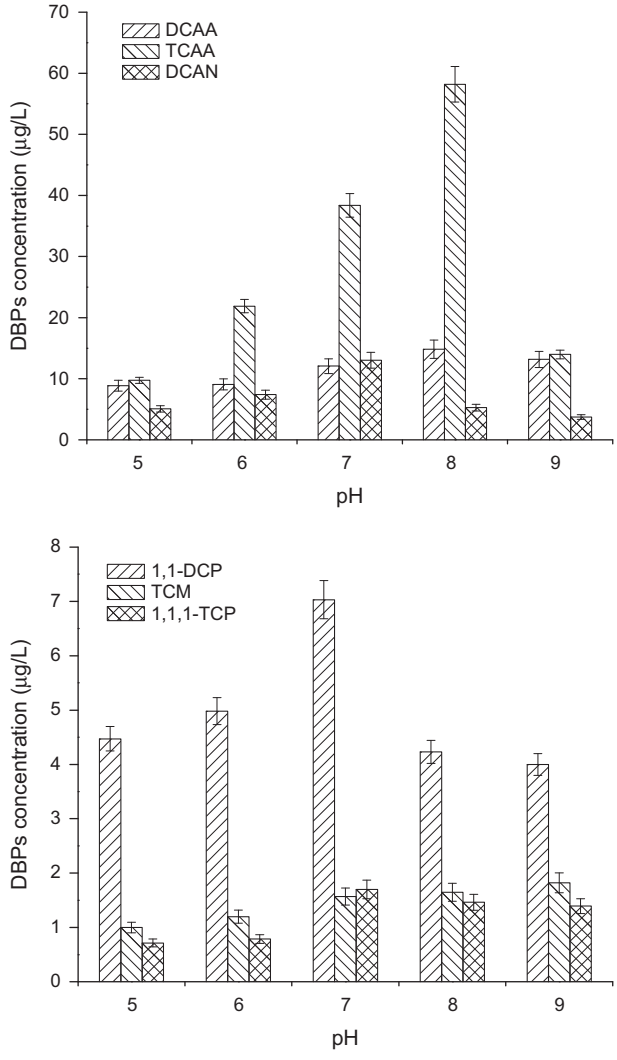

Fig. 3. Impact of pH on DBPsFP after 2 d of combined chlorine dioxide pre-oxidation and chlorination of the chironomid larvae metabolite (pre-oxidation time 30 min, chlorine dioxide dosage 2 mg/L, chlorine dosage 20 mg/L, TOC 2 mg/L, temperature of 20 ± 2℃). The error bars represent the standard deviation of replicate measurements, $n = 2$.

raised from 7 to 9. The 1,1-DCP hydrolysis constant increased with increasing pH values, from 0.21 (pH 5) to 1.55 (pH 7) causing a rising concentration trend that is easily decomposed under alkaline conditions, with the decomposition rate being lower than the oxidation rate. Therefore, 1,1-DCP is mainly oxidized to 1,1,1-TCP to give TCM as opposed to being decomposed. Since the hydrolysis rate constant is quite low under acidic conditions, with the formation rate being significantly higher, DCAN reaches its minimum concentration at pH 9. The influence of pH on HAA formation is more complicated, owing to the coexistence of two opposing processes of formation and hydrolysis for each individual species. The response might be related to the change of the organic precursors themselves [28]. A change of the chlorine species might also retard DBP formation. The TCM concentration increases as the pH is increased to 9. However, the pH also affects unstable DBPs with increasing pH, since DCAN, 1,1-DCP, and 1,1,1-TCP can undergo hydrolytic decomposition under alkaline conditions [2], with increase in their hydrolysis rates [29]. In addition, TCM is a common hydrolysis product of both 1,1-DCP and 1,1,1-TCP. Therefore, the increase in concentration with increasing pH was observed only in TCM.

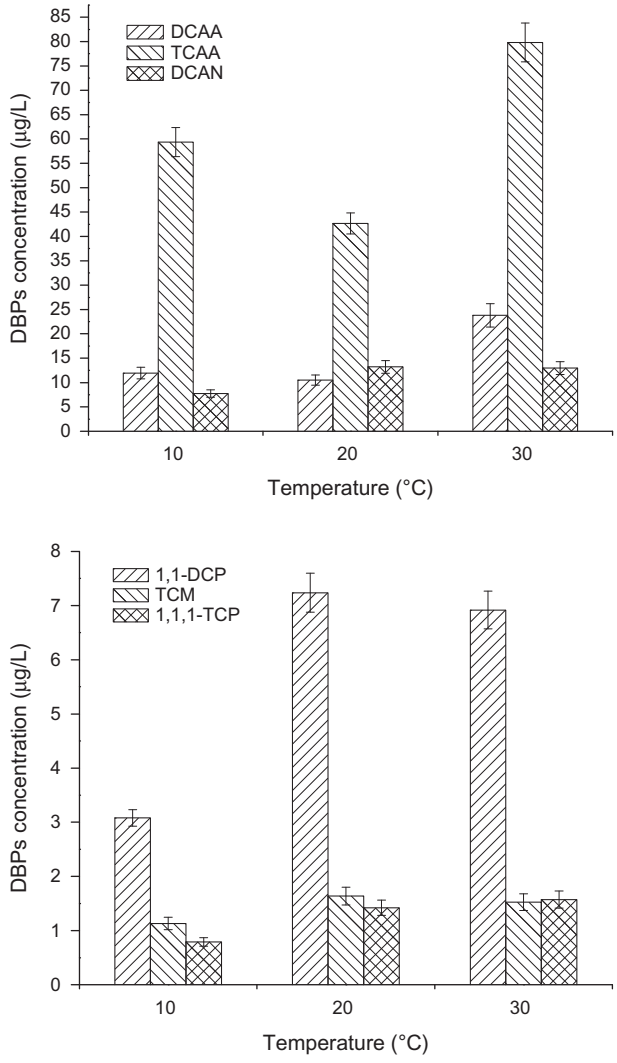

Fig. 4. Impact of temperature on DBPsFP after 2 d of combined chlorine dioxide pre-oxidation and chlorination of the chironomid larvae metabolite (pre-oxidation time 1 h, chlorine dioxide dosage 2 mg/L, chlorine dosage 20 mg/L, TOC 2 mg/L, pH 7). The error bars represent the standard deviation of replicate measurements, $n = 2$.

3.4. Effect of temperature

Fig. 4 shows the amounts of DBPs formed after two days of disinfection, i.e. chlorine dioxide pre-treatment (pre-oxidation time of 30 min) in combination with chlorination at three designated temperatures: 10, 20, and 30˚C. The formation of 1,1,1-TCP was significantly enhanced when the temperature is increased from 10 to 30˚C, while the concentrations of TCAA and DCAA first decreased and then increased again with the increase in temperature. The maximum amounts of DCAN, 1,1-DCP, and TCM were formed at 20˚C. The concentrations of DBPs at different temperatures reflect the balance of their formation and decomposition rates. Therefore, increasing temperature is expected to accelerate the decomposition reactions (e.g. hydrolysis), since they are always endothermic [30]. Indeed, there is a strong relationship between the formation of DCAA and TCAA. Since DCAA is the precursor of TCAA, increasing the concentration of the former decreases the concentration of the latter; therefore, the TCAA concentration exhibits an initial decrease and then an increase with increasing temperature. In summary, the temperature can fundamentally influence the decomposition of DBPsFP by affecting the interplay between the formation and decomposition rates.

4. Conclusions

A distinct decrease in the amount of DBPs formed was observed as the chlorine dioxide dosage increased at fixed chlorine dosages. Compared to chlorination alone, the chlorine dioxide pre-oxidation step could reduce the DBP quantity significantly, in terms of THMs, HAAs, HANs, and HKs produced. The yields of DCAA, TCAA, DCAN, TCM, and 1,1-DCP decreased with increasing pre-oxidation time, while the content of 1,1,1-TCP increased continuously with increasing pre-oxidation time. TCM was the only species showing a steady concentration increase with increasing pH, while the concentrations of other species initially increased and then decreased at basic pH values. Higher temperature enhanced 1,1,1-TCP formation, whereas the DCAN, 1,1-DCP, and TCM concentrations increased between 10 and 20˚C, followed by a decrease between 20 and 30˚C. The content of other DBPs decreased between 10 and 20˚C and then increased between 20 and 30˚C.

Acknowledgment

The project was supported by Foundation item: the National Natural Science Foundation of China (No. 503780262); Supporting Certificate of China Postdoctoral Science Foundation (No. 20070420882); and the National Natural Science Foundation of Heilongjiang Province of China (No. E200812).

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