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Simultaneous formation and biodegradation of HAAs in slow sand filters: effects of residual chlorine in filter influent

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ABSTRACT

Haloacetic acids (HAAs) are one of the major groups of chlorinated disinfection by-products in drinking water. This study explored the formation, speciation, and aerobic biodegradation of HAAs under different influent chlorine doses in batch reactor and simulated slow sand filtration (SSF) columns. The results showed that 90% of HAAs were biodegraded when no residual chlorine was present in SSF influents, and the bromine–HAAs were more biodegradable than chlorine–HAAs. In the batch systems, presence of 0.1 mg/L of free chlorine decreased both the heterotrophic plate counts and HAAs removal rates. The HAAs concentration increased predominantly when the chlorine concentration was higher than 0.5 mg/L. In the simulated labscale SSF columns, the 60 cm depth of SSF column provided the highest HAA percentage removal. When the influent chlorine concentration was higher than 0.5 mg/L, HAA formation was observed and particularly for the tri-haloacetic acids. The results of HAAs analysis in different bed depth showed that the middle and bottom sections of SSF also play an important role for HAA degradation.

Keywords: Aerobic biodegradation; Haloacetic acid; Heterotrophic plate count; Residual free chlorine; Slow sand filtration

1. Introduction

Chlorine is widely used in water purification processes for the inactivation of pathogens and the subsequent control of water-borne diseases. However, chlorine reacts with natural organic matter (NOM) and produces a variety of disinfection by-products (DBPs) in finished water. Among the various categories of chlorinated DBPs, the trihalomethanes (THMs) and haloacetic acids (HAAs) were considered to be the dominant DBPs by weight in chlorinated water. [1,2] Several toxicological and epidemiological studies have suggested that exposure to chlorinated DBPs, such as THMs and HAAs may lead to several adverse health effects. THMs were reported to be related to carcinogenic and reproductive defects and HAAs may be linked to cytotoxicities and genotoxicities in mammalian cells. [3,4] In order to reduce the potential health threats from DBPs in drinking water, many alternative technologies for DBP control have been proposed [5]. For example, THMs can be effectively controlled with boiling and aeration, [6] activated carbon adsorption [7–9], and membrane filtration. [10,11] However, control technologies for THMs may not be feasible for HAAs since HAAs are less volatile.

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It has been shown that HAAs removal in water environment were attributed to microbial degradation [12–14]. Several different bacteria are able to degrade HAA aerobically. TCAA can be degraded slowly by *Pseudomonas* [15]. Other bacteria such as *Xanthobacter autotrophicus* could degrade HAAs under aerobic conditions [16]. Some studies have investigated the biodegradation of HAAs in water environment and suggested that biodegradation is the dominant loss mechanism for HAAs [17,18]. It has been demonstrated that ^{14}C labeled HAAs can be converted to $^{14}CO_2$ in bacterial enrichment cultures [19], and some HAA degraders were isolated successfully [20].

HAA biodegradation can be affected by the concentrations of chlorine in water. For plants practicing prechlorination, a higher prechlorination dosage will be accompanied by a higher HAA concentration in the beginning of treatment train. In addition, higher chlorine dosages also reduce the bioactivities in the treatment systems. For treated water with total organic carbon (TOC) ranged from 1.5–3.9 mg/L, a free chlorine residual of 0.2 mg/L was needed to reduce the biofilm activity to below 50 pg ATP/cm^2. [21] It was also reported that when the chlorine residuals were higher than 0.7 mg/L, no HAAs reduction was observed in the distribution system [22].

Kinmen is a small island off the southeast shore of China. Due to the lack of water resources and severe pollution by domestic sewage and sea water intrusion, high total organic carbon (TOC, 8–12 mg/L) and bromide (0.1–0.5 mg/L) concentrations are commonly observed in the raw water. Tai-Lake Water Treatment Plant (WTP) is one of the main WTPs on Kinmen Island. The overall treatment processes are: prechlorination, coagulation, floatation, slow sand filtration (SSF), and postchlorination. Due to the high TOC in raw water, high prechlorination dosage was applied in raw water commonly to achieve desirable oxidation. Therefore, a substantial amount of DBPs was formed before entering the treatment process. It has been reported that HAA concentrations will be greatly reduced after SSF and the reduction of HAAs can be attributed to biodegradation in the SSF units [23]. In this study, the HAAs formation and speciation in the SSF units under different prechlorination dosages were investigated to reveal the mechanisms of HAA biodegradation in SSF.

2. Materials and methods

2.1. DBPs stock solution

The concentrated DBPs stock solution was prepared by mixing 40 mg/L of free chlorine, 20 mg C/L of humic acids (Aldrich, USA), and 10 mg/L of bromide (Br^-) and the solution was allowed to react in dark for 5 days. A fairly high concentration of HAAs (1973 μg/L) was formed in this solution, and this stock solution was used to prepare the influents for the following batch and column experiments.

2.2. Batch experiments

HAA biodegradation was confirmed by batch experiments. Two amber glass reactors (2 L) were used for batch biodegradation tests, one was for experiment (denoted as bioreactor) and the other was the control (denoted as control reactor). Both reactors were inoculated with biofilms detached from SSF. To prepare the biofilm suspension, 10 g of SSF sand (wet weight) was placed in 15 mL sterilized 0.01 M Tris buffer, and treated with 1 min ultrasound followed by 3 min vortex. After a brief settling, the biomass-rich supernatant was used as inoculants for batch experiments. To facilitate microbial growth, the reactors were amended with the following inorganic nutrients: 0.1% (w/v) NH_4NO_3, 0.1% (w/v) K_2HPO_4, 0.1% (w/v) KH_2PO_4, 0.01% (w/v) $MgSO_4{\cdot}7H_2O$, 0.01% (w/v) $FeCl_3{\cdot}6H_2O$ and 0.001% (w/v) $CaCl_2{\cdot}H_2O$. For control reactor, 3 mM of NaN_3 was added to prevent bacterial respiration. The initial dissolved oxygen in batch reactors was 8.8 mg/L, and both the reactors were supplied with filtered air (2 mL/min) continuously to assist aerobic microbial degradation. Since the biomass used for batch experiments was detached from the SSF column, the results obtained in batch reactor can then be used to describe the HAAs degradation in the column experiments.

2.3. Laboratory column experiments

The filter sands collected from the SSF unit in Tai-Lake WTP were sterilized (autoclaved at 121 ℃ for 20 min) and packed into the labscale SSF columns (60 cm bed depth and 3 cm of internal diameter). Four sampling ports were installed at different depths along the laboratory SSF columns. The labscale SSF columns (denoted as the biofilter column after) was inoculated with the SSF influent from Tai-Lake WTP continuously and operated with an empty bed contact time of 173 min (equivalent to a filtration rate of 5 m/day) at room temperature (25 ± 2 ℃) for over 30 days to promote the development of biofilms. The Tai-Lake SSF influent water contained 7.6 ± 2.3 mg/L of NPDOC, 0.03 ± 0.01 mg/L of NH_4^+–N, and 0.42 ± 0.03 mg/L of NO_3^-–N. The mean pH of the SSF influent solution was 6.44, and it was 6.73 for the effluent water. Water samples (40 mL) were collected

periodically from the influent, effluent, and sampling ports at different bed depths and HAA concentrations were determined.

2.4. Analytical procedures

The HAA concentrations were measured by liquid–liquid extraction, derivatization, and analyzed by a GC/ECD following the USEPA Method 552.3. In general, the water samples (40 ml) were spiked with surrogate standard (bromobutanoic acid, 20 μg/mL) then acidified to pH < 0.5, and extracted by 4 mL of methyl-tert-butyl-ether (MTBE) with 1,2,3-trichloropropane as the internal standard (1 μg/mL). The extract (3 mL) was then derivatized with 10% sulfuric methanol at 50°C for 2 h. After derivatization, the solution was back-extracted and neutralized with sodium sulfate solution (150 g/L) and saturated sodium bicarbonate solution. HAA concentrations were determined by a GC/ECD (Agilent 6890GC) with capillary column (DB1701), and the overall recovery efficiencies for HAAs were 80–120%. The detection limits for MCAA, MBAA, DCAA, TCAA, BCAA, BDCAA, DBAA, CDBAA, and TBAA were 0.17, 0.10, 0.12, 0.08, 0.09, 0.06, 0.07, 0.05, and 0.10 μg/L, respectively. The nonpurgeable dissolved organic carbon (NPDOC) was measured with an ASI 5000 TOC Analyzer (Shimadzu). The heterotrophic plate counts (HPCs) were determined by spreading of the batch water samples (0.1 mL) on R2A agar media (Difco), and the plates were incubated at 28°C for 7 days before counting of the total bacteria.

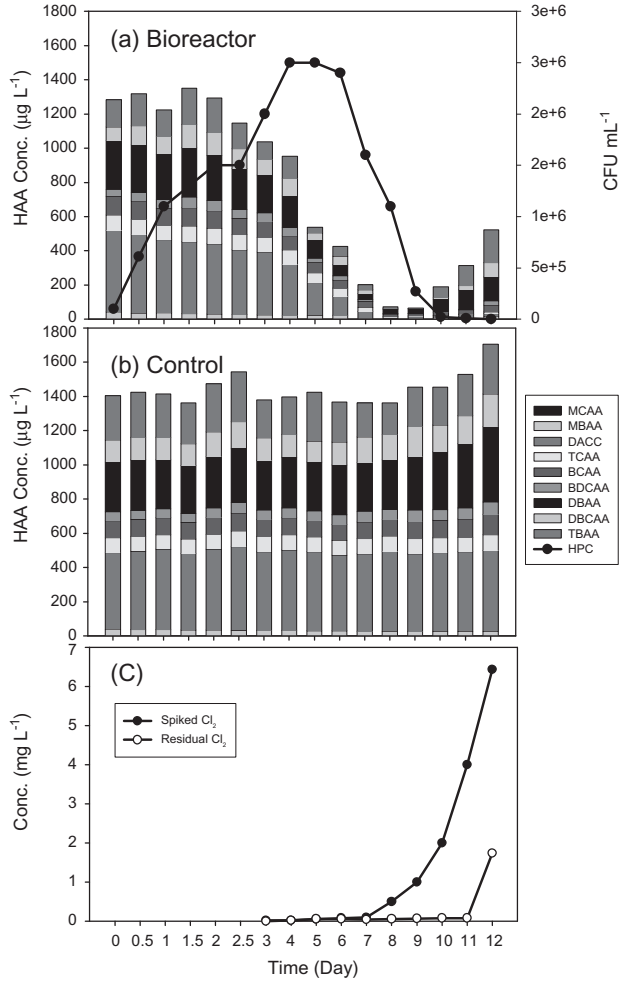

Fig. 1. HAA concentrations in the batch degradation experiments: (a) bioreactor, (b) control reactor, (c) spiked and residual chlorine in the bioreactor.

3. Results and discussion

3.1. Degradation of HAAs in batch reactors

In batch experiments, inorganic salts and biofilms detached from inoculated sands were added to the bioreactors to evaluate the HAAs degradation. For control reactor, the inoculants were autoclaved from sands. Fig. 1 shows the results of HAAs degradation profiles in the batch reactors. The HAA concentration in bioreactor started to decrease at the second day of incubation.

According to the HPC in the bioreactor, the microbial population in the bioreactor was following the common bacterial growth curve (Fig. 1(a)). From the 4th to the 6th day, the microbial population was at the stationary phase and the microbial population declined after the 6th day of incubation. For the control reactor (Fig. 1(b)), no culturable colony was observed during the course of study. In order to simulate the effects of residual chlorine on the microbial growth in the reactor, sodium hypochlorite was spiked into both the reactors from the 3rd day of incubation, and the concentrations of the residual chlorine was slowly increased (Fig. 1(c)).

As shown in Fig. 1(a), when the free residual chlorine was under 0.02 mg/L in the bioreactor, the HAA concentration kept decreasing in the reactor. This indicated that the microbial activity was not affected by the residual chlorine added in the system. At the 7th day, the HPC decreased largely when the spiked chlorine increased to 0.1 mg/L. However, the HAA concentration kept decreasing until the 9th day. This may indicate that micro-organisms were still able to degrade HAAs under this chlorine concentration. Ndiongue et al. [24] also reported that, when no measurable residual chlorine was present, a reduction on HPC numbers can still be observed. On the 9th day, the spiked chlorine in the bioreactor was around

1.0 mg/L, and the HPCs decreased from 1.1×10^6 to 2.7×10^5 CFU/100 mL. As shown in Fig. 1(a), the HAAs degradation was terminated and HAA formation was observed which may be due to the reaction of chlorine and organic matter in the bioreactor. The concentrations of HAAs in control reactor (Fig. 1(b)) remain similar during the first 9 days of study and increased slightly when chlorine residual was increased from the 10th to 12th day.

Fig. 2 compares the concentrations of Cl–HAA and Br–HAA in the bioreactor. When preparing the HAA stock solution, a high dosage of sodium hypochlorite was added to react with humic acid, no MCAA was produced due to high chlorine concentration and the major Cl–HAAs were DCAA and TCAA. During the degradation test, DCAA was more biodegradable compare to TCAA and can be degraded without acclimatization (Fig. 2). The DCAA degradation was not affected by the chlorine addition from the 3rd to the 9th day of incubation. TCAA is more difficult to be degraded by micro-organisms and required an acclimation period, so the degradation rate of TCAA was slower than DCAA [12]. For TCAA, it required 4–5 days of acclimation before its concentration started to decline. Between the 4th and the 6th day of batch experiment, HPC number was stable, and the decrease of HAAs obeyed the pseudo-first-order kinetics:

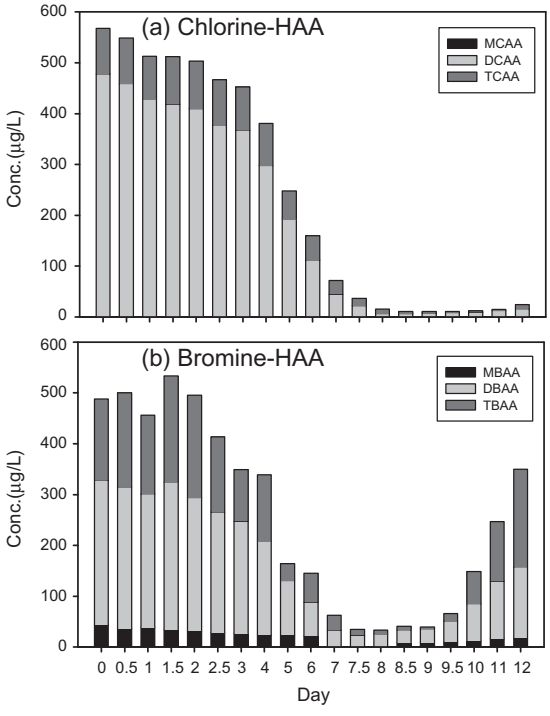

Fig. 2. Comparisons on the concentrations of chloro-only HAAs and bromo-only HAAs in the batch degradation experiments.

$$C_t/C_o = \exp(-kt)$$

where C_t is the concentration of HAA at the t th day, C_o is the initial HAA concentration, k is the rate constant. Based on the three days data between the 4th to the 6th day, the degradation rate constants were $0.285\,day^{-1}$ for DCAA and $0.240\,day^{-1}$ for TCAA, respectively.

The HAA stock solution was prepared with 10 mg/L of Br^- to enhance the bromine–HAAs (Br–HAAs) formation, which resulted in substantial amounts of bromine–HAA species. Bayless and Andrews [25] reported that the order of biodegradability of HAAs by biofilm was MBAA > MCAA > BCAA > DCAA > DBAA > TCAA. This order of biodegradability showed that Br–HAAs were easier to be biodegraded than chlorine–HAAs (Cl–HAAs). The results in Fig. 2 show that TBAA can be degraded up to 81.6% on the 7th day, which is higher than the 71.8% of TCAA degradation at the same reaction time. When the chlorine dosage was increased to 0.5 mg/L (the 8th day), the rates of HAAs formation were higher than the rates of HAAs degradation due to the inhibition from high chlorine concentration in the system, and the HAAs formation tends to generate much more Br–HAAs than Cl–HAAs due to the presence of bromide ion in the system. Since the rates of Br–HAAs formation was much faster, the accumulations of MBAA, DBAA, and TBAA increased considerably since the 8th day (Fig. 2).

3.2. Simulated SSF column studies

The percentage removals of monohalo-AAs (MXAA: MBAA), dihalo-AAs (DXAA: DCAA + DBAA + BCAA), and trihalo-AAs (TXAA: TCAA + BDCAA + DBCAA + TBAA) degradations after flowing through the labscale SSFs are shown in Fig. 3. The overall HAAs percentage removals increased with increasing bed depth in SSF columns. The MXAA were not completely degraded even though the MXAA was considered to be more biodegradable than DXAA and TXAA. Although it has been shown in literatures that the primary degradation for organic pollutants was obtained in the top portion of the filter units, better degradation efficiencies can be obtained for the less biodegradable TXAA at a deeper filter bed depth (e.g. 30 and 60 cm filter depth). As shown in Fig. 3, a 25.3% removal was obtained for TXAA at 10 cm depth and 61.5% removal was observed after flowed throughout the 60 cm bed depth. Fig. 4 shows the results of percentage removals for each TXAA species at different bed depths. The overall removal efficiency was TBAA (90.9%) ⩾ DBCAA (90.0%) ⩾ BDCAA (89.9%) > TCAA (77.3%). The results in Fig. 4 support the observation

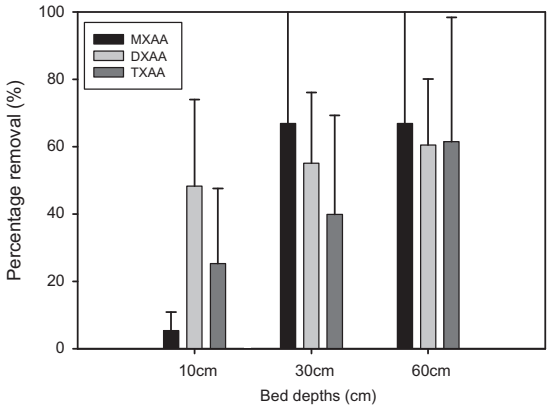

Fig. 3. Removal efficiencies of mono-HAAs, di-HAAs and tri-HAAs in column degradation systems with different bed depths. (MXAA = MBAA + MCAA, DXAA = DCAA + DBAA + BCAA, TXAA = TCAA + TBAA + BDCAA + DBCAA) The error bars indicate the standard deviations of duplicate experiments.

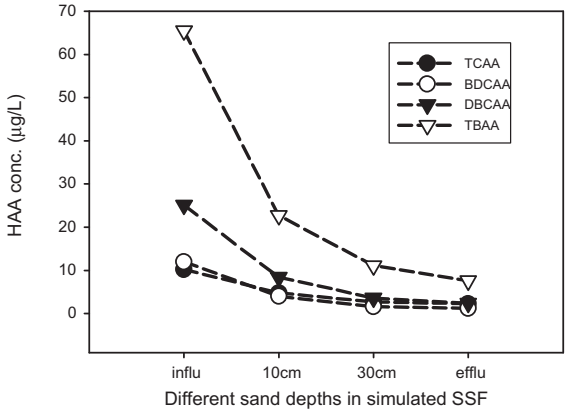

Fig. 4. Concentrations of tri-halo HAAs in the column degradation tests with different bed depths.

that the Br–HAAs are more biodegradable than the Cl–HAAs.

3.3. Degradations of Cl–HAAs and Br–HAAs in the lab-scale SSF columns

Fig. 5 compares the degradation of Cl–HAAs and Br–HAAs in the labscale SSF columns (denoted as biofilter). The influent was prepared with DBPs stock solutions as described in the previous sections. When no or low residual chlorine was present in the influent, approximately 90% of the HAAs can be removed after flowing through the biofilter (HAA9 was decreased from 314 to 33 μg/L, see data in Fig. 5 with 0 mg/L of influent chlorine). The primary degradation was observed in the top portion (10 cm) of the filter, where more than 60% removal was obtained (from 314 to 124 μg/L). The results showed that the main degradation of HAAs was from the surface portion of the filter; however, the middle and bottom sections of the filter can also provide some HAA removals.

The microbial community structure of the labscale SSF column sand was analyzed by PCR amplification and 16S rDNA genes, it revealed that alpha proteobacteria and beta proteobacteria were the dominant species of the biofilm. After RFLP analysis and nucleotide sequencing, *Sphingomona*, *Pseudomonas*, and *Burkholderiales* were identified as the dominated bacterial population of the biofilm. [26] These bacteria have been shown to be able to degrade HAAs [19,27–28].

Br–HAAs were easily removed by the SSF than Cl–HAAs. The overall percentage removals for Cl–HAAs (DCAA + TCAA) and for Br–HAAs (BCAA + TBAA) were 78.3 and 86.5%, respectively (Fig. 5(a) and 5(b)). When the dosages of sodium hypochlorite in the influent were gradually increased, at the beginning, the chlorine residuals at the 10 cm filter depth effluent remained 0.01–0.02 mg/L. Until the influent chlorine concentration reached 2.2 mg/L, the chlorine residual in the effluent collected at 10 cm filter depth went up thereafter. This showed that the chlorine may be consumed by organic matters and biofilms on the sand surface, and the chlorine applied in the influent water was consumed rapidly at the top portion of the SSF columns. The chlorine consumption at the top layer could generate more HAAs due to the reactions between chlorine and the organics on the sand surface. Higher chlorine in the influent can also affect microbial activities. When the concentration of influent chlorine reached 0.25 mg/L or more, the concentration of TCAA increased considerably (as shown in Fig. 5(a)). This may be due to the reason that the rate of TCAA formation was higher than the rate of TCAA degradation when the influent chlorine concentration was high. Because most chlorine was consumed by the top 10 cm section of filter sands, bioactivity could still be active at the middle and bottom sections of the SSF. The TBAA, BDCAA, and DBCAA have similar removal profiles (Fig. 5(b) and 5(c)) in the labscale SSF. The concentrations of brominated tri-haloacetic acids were increased at first 10 cm of SSF bed depth, and were removed at deeper bed depth. As a comparison, no apparent increases were observed for DCAA, DBAA, and BCAA at the top 10 cm bed depth even with increasing chlorine concentration in the filter influent. Based on the results observed in both the batch and column experiments, the profiles of HAAs degradation and regeneration were consistent: the presence of chlorine can inhibit the bacterial activities, reduce the rates of HAAs biodegradations, and lead

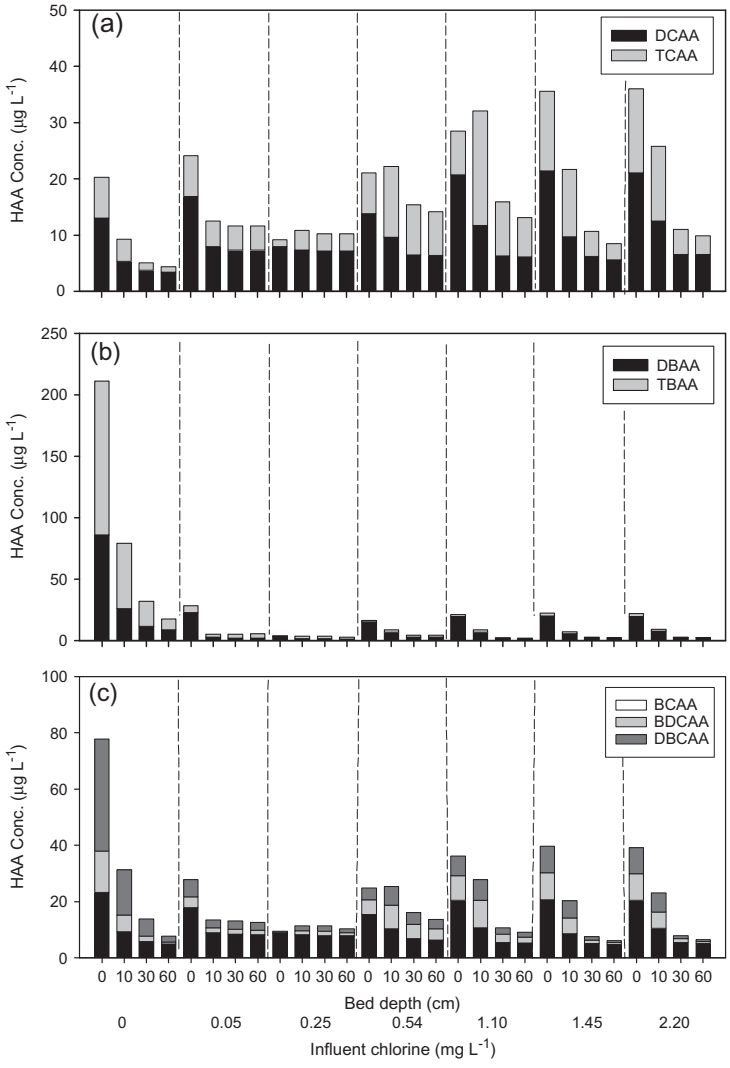

Fig. 5. Effects of influent chlorine concentrations on HAAs formation and degradation within SSF columns (a: Cl-HAAs, b: Br-HAAs, and c: bromo-chloro-HAAs).

to the reformation of HAAs when the chlorine concentration was increased.

Fig. 6 compares the degradation profiles of DCAA and TCAA spiked in the influent water in the labscale SSF column study. When chlorine was not present (residual chlorine was negligible in the influent), 93% of DCAA had been removed after flowed through 10 cm of the bed depth (Fig. 6(a)) even though the initial concentration of DCAA was high, and the remaining DCAA can be completely removed (99.7%) after flowing through the entire column. TCAA was less biodegradable than DCAA, and only 15.8% of TCAA was removed after 10 cm of contact in SSF; however, the overall percentage removal for TCAA was 50% after flowing through the entire column (Fig. 6(b)). After spiked with more chlorine into the influent solutions, the percentage removal of TCAA was reduced to 45.1% after flowed through the 60 cm bed depth; this observation indicated that the TCAA removal was affected by chlorine. When the chlorine dosages in the influent water reached 0.6 mg/L, the DCAA can still be removed effectively; however, the concentration of TCAA was increased in the sample collected from effluent at 10 cm bed depth. As a comparison, the concentration of DCAA at 10 cm bed depth was increased when the influent chlorine was increased to 2.1 mg/L (see circles in Fig. 6). This may be due to the reaction of chlorine and NOM in the filter and produced more HAAs.

Since the influent chlorine was kept in contact with the micro-organisms and other organic matters on the sand surface, the chlorine will be gradually consumed

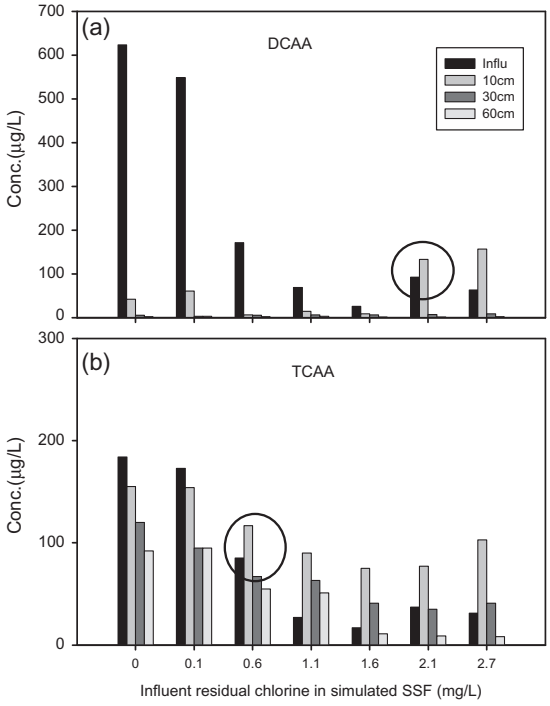

Fig. 6. Effects of residual chlorine on DCAA and TCAA concentrations in the SSF column studies.

and the chlorine concentration might be lower in the middle and bottom sections of the SSF. Therefore, the DCAA and TCAA concentrations were removed at deeper bed depth. Compared to the results obtained from 10 cm bed depth, the TCAA removal at 30 and 60 cm depth could reach 30% and 92%, respectively. Although the chlorine dosage in influent was increased, its concentration will soon be reduced after reactions with the organic matters on the sand surface. For example, as the influent chlorine was increased to 2.7 mg/L, less than 0.01 mg/L of chlorine was detected at 10 cm bed depth. The residual chlorine at the deeper bed depths were essentially not detectable, indicating that the primary inhibition to HAA removal in presence of chlorine only occurred in the top section of the SSF, and the deeper sand filter may still exert the biodegradation ability.

4. Conclusions

HAAs degradation and formation in the simulated SSF column under different influent chlorine dosages were assessed in this study. The results showed that SSF units can effectively remove the HAAs, but the chlorine concentration in the influent can affect the HAA removal efficiencies. The influent chlorine not only inhibit the HAAs biodegradation, but also react with NOM accumulated on sand surface and generate more HAAs when the concentrations of influent chlorine were high. In batch experiment, spike 0.1 mg/L of free chlorine can decrease HPC numbers and reduce HAA removal rates. When no residual chlorine is presented in the system, 89% of total HAAs could be degraded and the Br–HAAs were more biodegradable than the Cl–HAAs. TBAA can be degraded up to 81.6%, which was higher than the 71.8% of TCAA degradation at the same contact time. In the labscale SSF column study, when influent chlorine residual reached up to 0.6 mg/L and, TCAA concentration increased at the first 10 cm bed depth but later being removed by 53% (compared with the results at 10 cm depth) after the water flowed through the 60 cm bed depth. For DCAA, similar situation occurred when the residual chlorine reached 2.1 mg/L. The profile of residual chlorine at different bed depth showed that no residual chlorine was detectable at 10 cm depth. It appeared that the top section of SSF would consume a large amount of sodium hypochlorite and only minimal residual chlorine could be detected in the middle and bottom portions of SSF which create an ideal condition for HAA biodegradation.

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