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RESEARCH ARTICLE

Unlocked disinfection by-product formation potential upon exposure of swimming pool water to additional stimulants

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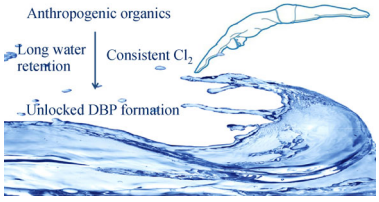
HIGHLIGHTS

- Swimming pool water was studied for DBPs upon exposure to additional stimulants.
- DBP formation could be induced by residual chlorine and extended incubation.
- Urine led to a massive formation of chloroform with additional stimulants.
- Reactions between chlorine and anthropogenic organics were slow and long-lasting.
- Urine control and air ventilation should be on the priority list for pool management.

GRAPHIC ABSTRACT



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ABSTRACT

Anthropogenic organics are known to be responsible for the formation of harmful disinfection by-products (DBPs) in swimming pool water (SPW). The research explored an important scenario of SPW with no additional anthropogenic organic input. With stimulations by residual chlorine or additional chlorine and extended incubation, the formation of DBPs, especially chloroform, was significantly induced. Similar observations were found by investigating synthetic SPW made with sweat and urine. The presence of urine led to a massive formation of chloroform, as noted by an approximate 19-fold increase after 165-day incubation with a shock chlorine dose. The research suggests that consistent residual chlorine and long water retention as two typical features of SPW could unlock the DBP formation potential of anthropogenic organics. Thus, limiting the introduction of anthropogenic organics may not have an immediate effect on reducing DBP levels, because their reactions with chlorine can be slow and long-lasting. Pool management should prioritize on control of urine and improving air ventilation. This work is useful to deepen understandings about DBP formation in SPW and provide implications for pool management and prospective legislation.

1 Introduction

Swimming as an aerobic exercise is good for people of all ages. The venue of this popular activity can be pools of different kinds and sizes in public areas, hotels and spas, or residential homes. Swimming pool water (SPW) must be disinfected to deactivate pathogens; however, the disinfection also introduces non-negligible amounts of undesirable by-products (i.e. disinfection by-products (DBPs)) to the SPW (Wang et al., 2014; Zhang et al., 2015a). For instance, chloroform (one major species of trihalomethanes (THMs)) was found to have concentrations between 8.65 and 243 μg/L, while dichloro- and trichloroacetic acids (DCAA and TCAA, two major species of haloacetic acids (HAAs)) were found at even higher concentrations, up to 6800 (Daiber et al., 2016) and 2600 μg/L, respectively (Carter and Joll, 2017). DBPs are also present in the pool's air, with THM concentrations ranging from 51 to 1225 μg/m^3 (Sá et al., 2011). Thus, pool users may involuntarily inhale, ingest, and absorb these DBPs, leading to potentially serious health consequences (Erdinger et al., 2004; Kogevinas et al., 2010; Cardador and Gallego,

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2011). A blackout case due to narcotic properties of chloroform in a poorly ventilated recreational aquatic center has been reported (Florentin et al., 2011).

To date, SPW is only regulated for free chlorine (Weaver et al. 2009; Dyck et al., 2011), combined chlorine (Bradford 2014; Cimetiere and De Laat, 2014), and THMs (Daiber et al., 2016) in very limited countries or regions. For instance, only Germany and France have limits for total THMs in SPW (Daiber et al., 2016), and for THMs in the pool's air, the European occupational legislation applies, since the chloroform inhalation exposure can not exceed 10000 μg/m^3 (Sá et al., 2011). As DBP regulations for swimming pools are expected to evolve in years to come, research advances in the causes and control of DBPs in SPW are needed.

The occurrence of DBPs in SPW is ascribed to chemical reactions between chlorine and organic matter, which is the same as the formation of DBPs in drinking water. However, DBPs in drinking water occur at much lower concentrations within the Unites States Environmental Protection Agency (USEPA)'s Stage 2 regulatory limits of 80 and 60 μg/L for THMs and HAAs, respectively. Conventional wisdoms have attributed this phenomenon to the pool's unique settings: continuous loading of anthropogenic substances (such as sweat, urine, saliva, lotion, hair, and skin particles) from pool users (Keuten et al., 2012; 2014), continuous dosing of chlorine, and a high level of water age (Zwiener et al., 2007; Kanan and Karanfil, 2011). Additionally, the less-volatile nature of HAAs compared to THMs results in the accumulation of HAAs in SPW, leading to high HAAs/THMs ratios (Chowdhury et al., 2014; Teo et al., 2015). The theory appears to be able to explain the DBP profile in SPW well. For instance, it was employed to interpret a progressive increase of HAAs (from 87 to 1600 μg/L) and a relatively low level of THMs (62±35 μg/L) in SPW as the water age increased from 0 to 1.5 years (Tang et al., 2015). Since the continuously introduced anthropogenic organics from pool users are considered as the main DBP precursors in SPW other than the natural organic matter (NOM) in the filling water, management should consider limiting the introduction of anthropogenic organics to SPW. However, research is inadequate regarding what will happen in SPW if no more anthropogenic organics is added, that is, a pool stops accepting users or the hygiene rules (e.g., pre-swim showering, no urination in pools) have been strictly followed and the pool operates under the influence of pre-existing anthropogenic organics only. This is an important scenario because a pool's consistent residual chlorine, long water retention, and DBP transfer at the water-air interface as additional stimulants may confound the DBP formation mechanism and it is unclear whether the conventional wisdoms regarding DBP control by limiting anthropogenic organic input are still sound.

In this study, we exposed real and synthetic SPW with pre-existing anthropogenic organics to additional stimulants in the laboratory with a purpose of quantifying the DBP formation potential of such waters. The research tries to answer the question: Will the SPW be safer after limiting new anthropogenic organics to swimming pools? The results are useful to deepen our understandings about DBP formation in SPW and provide implications for pool management and prospective legislation.

2 Materials and methods

2.1 Chemicals

THM Calibration Mix containing chloroform, bromodichloromethane (BDCM), chlorodibromomethane (CDBM) and bromoform (100 μg/mL each component in methanol), and HAA stock standard (EPA 552 Halogenated Acetic Acid Mix) containing monochloroacetic acid (MCAA), monobromoacetic acid (MBAA), DCAA, TCAA, bromochloroacetic acid (BCAA), and dibromoacetic acid (DBAA) (2000 μg/mL each component in methyl tert-butyl ether (MtBE)) were obtained from Supelco (Bellefonte, Pennsylvania, USA). A 1000 mg/L chlorine stock solution was made by diluting a commercially available 6% (v/v) sodium hypochlorite solution and was standardized periodically.

2.2 Real and synthetic SPW samples

Real SPW was obtained from an indoor university swimming pool open to students, employees, and local residents. Approximately 1400 users were admitted to the pool every month. At the time of sampling, the SPW had a water age of 5 months, total organic carbon (TOC) of 5.9 mg/L, and total THMs of 37 μg/L, while its total HAAs had reached 1220 μg/L. The residual chlorine was 1.9 mg/L. Two synthetic SPW samples were made with anthropogenic organics (e.g. sweat and urine) obtained from a 24-year-old college male. One synthetic SPW was made by diluting a sweat sample with distilled water, resulting in a TOC of 7.3 mg/L, and the other one was made based on the first synthetic SPW with urine spiked at a ratio of 0.01%. The second synthetic SPW with both sweat and urine resembled real SPW without urine control and had a TOC of 8.3 mg/L. Note that direct chlorination of urine for determination of DBP formation potential would be difficult due to the massive chlorine demand, and it was challenging to maintain the free chlorine residual in an acceptable range after an extremely long incubation period. Table 1 provides a list of water quality parameters including TOC, UV absorbance at 254 nm (UV_{254}), specific UV absorbance (SUVA), ammonia-nitrogen (NH_3-N), nitrate-nitrogen (NO_3-N), chlorine residual, and DBP concentrations for the three SPW samples.

Table 1 Water quality parameters of real and synthetic SPW samples

Parameter	Unit	Real SPW	Synthetic SPW with sweat	Synthetic SPW with sweat and urine
TOC	mg/L	5.9	7.3	8.3
UV_{254}	cm^{-1}	0.026	0.037	0.075
SUVA	L/mg-m	0.44	0.51	0.90
NH_3-N	mg/L	0	0.6	1.2
NO_3-N	mg/L	4.0	3.8	3.8
Cl_2 residual	mg/L	1.9	0	0
Total THMs	μg/L	37	0	0
Total HAAs	μg/L	1220	0	0

2.3 Exposure to stimulants

For the real SPW, three scenarios of stimulation were conducted with no additional anthropogenic organic input. In Scenario 1, extended incubations of 3, 7, and 14 days were employed on the pre-existing 2 mg/L residual chlorine. In Scenario 2, a shock chlorine dose of 20 mg/L was applied to the SPW and the samples were incubated for 3, 7, and 14 days. In Scenario 3, a step chlorine dose of 4 mg/L at a dosing interval of 3 days was applied to the SPW 5 times within 14 days, and the incubation conditions were the same. The three scenarios were designed to explore the trends of DBP formation in SPW on residual chlorine, a shock chlorine dose, and step chlorine doses, respectively, which resembled either real pool operations or DBP formation potential tests environment. To minimize photodecay of residual chlorine and DBPs (Weng et al., 2012), all incubations were performed in amber borosilicate bottles (250 mL) in the dark at room temperature (22°C). It is important to note that these 3-, 7-, and 14-day incubations had separate bottles for reaction, and no head space was allowed in each bottle during the incubation period to prevent loss of DBPs due to volatilization. For each synthetic SPW sample, the reactions occurred in two bottles for 3-day and 165-day incubations, respectively, and a shock chlorine dose of 20 mg/L was applied to 10-fold diluted solutions to ensure residual chlorine was not depleted at the end of incubation.

2.4 Analysis

TOC was measured with an O.I. Analytical 1010 TOC analyzer (O.I. Analytical, Maryland, USA) following Standard Methods 5310 D. UV_{254} was measured with an Agilent 8453 UV-Visible Spectrophotometer (Agilent technologies, California, USA) following EPA Method 415.3. SUVA (L/mg-m) was calculated by dividing a sample's UV_{254} (cm^{-1}) by its TOC in mg/L and then multiplying by 100. NH_3-N and NO_3-N were measured following the Standard Methods 4500-NH_3 D and 4500 NO_3^- B, respectively (APHA, 2005). Free chlorine residuals were analyzed using the *N,N*-diethyl-*p*-phenylenediamine (DPD) method with a Hach DR/890 colorimeter (Hach Co., Colorado, USA). THMs and HAAs were analyzed by gas chromatographs (GC) following USEPA Methods 551.1 and 552.3, respectively. Details on DBP extraction procedures and GC program settings can be found elsewhere (Tang et al., 2015). DBP concentrations were validated against a comprehensive set of procedural blanks, matrix spikes, and replicate samples, and the results presented in the study were averages of duplicate analyses.

3 Results and discussion

3.1 DBP formation in SPW induced by additional stimulants

Figure 1 shows the concentrations of major DBP species in real SPW under three scenarios of additional stimulation. The "0-day" legend indicates the starting conditions of the SPW, which included 37 μg/L chloroform, 847 μg/L DCAA, and 341 μg/L TCAA. It was typical to have DCAA and TCAA as the most abundant HAAs in SPW (Simard et al., 2013), and due to their conservative nature (Zhang et al., 2015b), they tended to accumulate in SPW while much of the chloroform was volatilized. Thus, it appeared that the THMs with less than the USEPA Stage 2 regulatory level were not accumulated in the 5-month old SPW. The low concentration of THMs in the pool could be benefited from the constant water splashing by swimmers and significant air ventilation which facilitated water-air transfer, and water recirculation pumps which promoted the breakup of stratification (Weng and Blatchley 3rd, 2011).

Exposure of such SPW to extended chlorination on 2 mg/L residual chlorine in Scenario 1, however, led to accumulation of THMs significantly. After 14-day incubation, the chloroform concentration increased to 142 μg/L, which was an approximate 4-fold increase. It was deduced that the pre-existing organics in SPW continued to react with the residual chlorine to form chloroform. DCAA and TCAA also increased. However, their percentages of

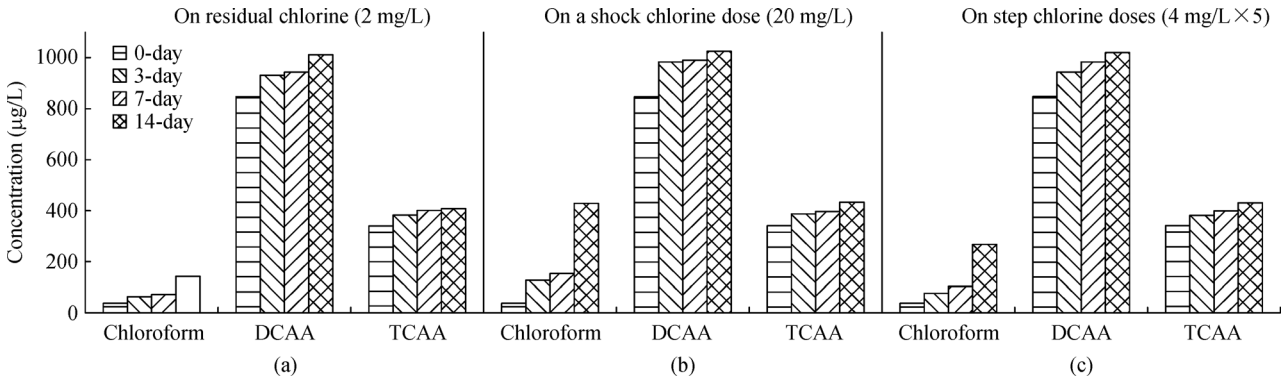

Fig. 1 Concentrations of major DBP species in real SPW samples induced by extended incubation and additional chlorine dosing: (a) on residual chlorine (2mg/L); (b) on a shock chlorine dose (20 mg/L); (c) on step chlorine doses (4 mg/L×5).

increase (19% and 18%) were not as great as that of chloroform. This was due to the high base level of HAAs as a result of previous accumulation. Scenario 1 showed that with simple stimulants of residual chlorine and extended incubation, additional formation of DBPs could be induced. It suggested that the reaction between anthropogenic organics and chlorine could be long-lasting, and those organics had a high DBP formation potential.

In Scenario 2, we applied a shock chlorine dose of 20 mg/L to the SPW, which resembled a DBP formation potential test for drinking water (Xie, 2004) and wastewater (Tang et al., 2012). The shock chlorine dose promoted the formation of chloroform further. With a concentration of 429 μg/L after a 14-day incubation period, it is striking to note this was an approximate 12-fold increase on chloroform. The increase percentages of DCAA and TCAA were 21% and 27%, respectively, which were comparable to the results on the 2 mg/L residual chlorine. The results indicated the shock chlorine dosing unlocked the chloroform formation, and it appeared the formation potential of HAAs was depleted. The unlocked chloroform formation could be ascribed to the increased chlorine/carbon ratio, which favored THM formation. The HAAs, especially the DCAA, appeared unresponsive to the shock chlorine dose. Since the HAA precursors were primarily associated with easily oxidizable organics with smaller molecular weight (Hua and Reckhow, 2007), the pre-existing anthropogenic organics in the SPW could be in shortage of such precursors as a result of consistent reactions with residual chlorine before the experiments.

The step chlorine dosing of 4 mg/L for 5 times in Scenario 3 resembled the situation of pool operation without air-water transfer and water recirculation. Although the results revealed an approximate 7-fold increase on chloroform after 14 days compared to Day 0, with a reduced chlorine/carbon ratio during the incubation, the induced chloroform formation was much less compared to that of Scenario 2. Still, the chlorine/carbon ratio did not appear to influence HAAs that much, attributable to the depletion of HAA precursors in the anthropogenic organics.

It can be deduced that the 5-month old SPW contained chlorine-reactive organics in forming DBPs. Even if there was no continuous input of anthropogenic organics, the DBP formation potential of existing substances in SPW could be unlocked upon additional stimulation during the pool's operation. The increase in DBP formation was due to a great excess of free chlorine residual in these scenarios. Considering sweat and urine are two typical anthropogenic inputs to swimming pools, in the section to follow, we made synthetic SPW with the two components, with a purpose of quantifying their DBP formation potential over an extended period of incubation on a 20 mg/L shock chlorine dose.

3.2 DBP formation of sweat and urine induced by additional stimulants

Figure 2 shows the concentrations of major DBP species in synthetic SPW after conventional 3-day incubation for DBP formation potential quantification and 165-day incubation to mimic the water age of the real SPW. With 3-day incubation, both synthetic SPW samples formed chloroform (100 and 110 μg/L), DCAA (245 and 264 μg/L), and TCAA (58 and 63 μg/L) at similar levels. However, the 165-day incubation unlocked the formation potential of all these species, and it was especially true for the sample with urine. At a chloroform concentration of 1930 μg/L, it was an approximate 18-fold increase for the sample with urine, while the sample with only sweat resulted in an approximate 5-fold increase. Urine was previously believed to suppress the formation of THMs (Judd and Jeffrey, 1995; Kim et al., 2002). It was not until recently that researchers found urea, a component of urine and skin (De Laat et al., 2011), reacts with chlorine quite slowly in SPW (Zare Afifi and Blatchley 3rd, 2016) and a mechanism between urea and chlorine was suggested (Blatchley 3rd and Cheng, 2010). In a SPW setting with long water retention and consistent residual chlorine, the

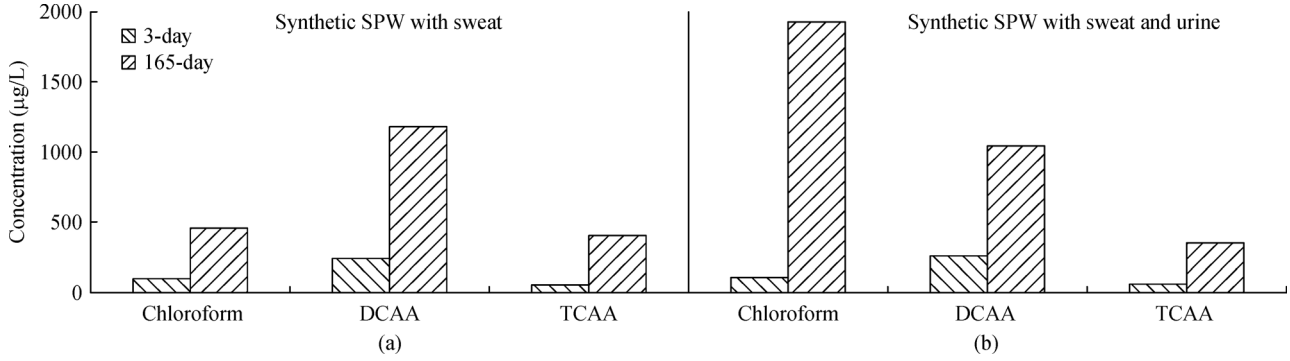

Fig. 2 Concentrations of major DBP species in synthetic SPW samples induced by a shock chlorine dose of 20 mg/L and extended incubation: (a) synthetic SPW with sweat; (b) synthetic SPW with sweat and urine.

formation potential of urine could be massive. For DCAA and TCAA, without high base levels as a result of previous accumulation in the synthetic SPW, it was clear that their formation was also greatly induced by extended incubation. However, a noteworthy massive increase similar to the chloroform did not appear for the sample with urine. This implied the urine did not have fundamentally different HAA precursors compared to the sweat. Several researchers (Judd and Jeffrey, 1995; Yang et al., 2016; Carter and Joll, 2017) included citric acid in their recipes of body fluid analogs for SPW research, and it is possible that citric acid – a typical DCAA precursor that reacts fast with chlorine, is produced as an intermediate product of degradation metabolism and exists in the extended incubation period. On the other hand, the THM precursors in SPW are believed to be associated with larger carbon chain organic molecules (Lee et al., 2009) and their reactions with chlorine are slow (Weng and Blatchley 3rd, 2011). More studies on model components (e.g., uric acid (Lian et al., 2014)) would be helpful to elucidate the DBP precursors in SPW. Details on the concentration of individual DBP species in different scenarios and the corresponding chlorine residuals can be found in Table 2.

3.3 Implications

The study broadens existing understandings regarding anthropogenic substance as the primary cause of DBP formation in SPW. Consistent residual chlorine and long water retention are two typical features of SPW, and these stimulants could significantly affect DBP formation in SPW as well. Given the same amount of anthropogenic organics, if one SPW is exposed to additional stimulation that resembles the pool's environment, it could have more DBP formation. The results of this study also suggest that limiting new anthropogenic organic input might not have an immediate effect on reducing DBP formation in SPW. The pre-existing anthropogenic organics would continue to slowly react with the residual chlorine and consistently release more DBPs, especially chloroform. Because chloroform formation is induced at a high chlorine/carbon ratio, pool management should run the pool with an acceptably-low residual chlorine concentration. Considering that a complete stop on anthropogenic organics introduction is nearly impossible, the priority should be given to the control of urine, which has a massive chloroform formation potential if unlocked by additional stimulation in pool's settings. As a result of water-air partitioning, the additionally formed chloroform is more likely to end up in the pool's air. Thus, improving air ventilation is crucial to reduce its negative health impacts on pool users and attendants. Since the source of DBP precursors in SPW is mainly from the human and the control of anthropogenic inputs would only alleviate, not eliminate the DBP problems, the ultimate solution would need to target on the removal of anthropogenic organics and the formed DBPs. Since water replacement is costly and not energy-efficient, advances in SPW treatment technologies are needed. For instance, biologically active carbon filtration has been found capable of removing HAAs from SPW (Tang and Xie, 2016). Other technologies such as ozonation (Hansen et al., 2016), UV irradiation (Spiliotopoulou et al., 2015), ultrafiltration-adsorption (Barbot and Moulin, 2008) have been explored, but certainly warrant further investigations. Studies that focus on characterization of DBP precursors in SPW and their removal technologies would be desirable.

It is important to note that the THMs and HAAs levels would eventually decrease if there is no input of DBP precursors and the physicochemical loss of DBPs prevails as the dominant mechanism. This has been discussed by the authors (Tang et al., 2015) by presenting a mass balance model to study the DBP profiles of an indoor pool over a 1.5-year period. The computational algorithm of the model was based on the contribution from daily anthropogenic input and a first-order kinetic DBP loss due to evaporation, degradation, dilution and adsorption. Despite the existing knowledge on the importance of anthropogenic organics control, this research prompts pool designers, engineers, maintenance staff, and legislators to rethink how to improve the quality of their SPW further. Three comparisons in this research provide the unequal

Table 2 Concentrations of DBP species in real and synthetic SPW samples under stimulations by extended incubation and additional chlorine dosing

Scenario	Days of incubation	Chloroform	BDCM	DBCM	DCAA	TCAA	BCAA	Total THMs	Total HAAs	Total DBPs	Cl_2 residual
Real SPW on residual chlorine of 2 mg/L	0	37	0	0	847	341	30	37	1218	1255	1.92
	3	64	1	0	931	382	32	65	1345	1410	1.88
	7	72	0	0	944	402	34	72	1380	1452	1.86
	14	142	1	0	1010	408	34	143	1452	1595	1.86
Real SPW on a shock chlorine dose of 20 mg/L	0	37	0	0	847	341	30	37	1218	1255	21.9
	3	127	1	0	983	386	33	128	1402	1530	19.4
	7	153	0	0	990	396	36	153	1422	1575	18.5
	14	429	2	0	1024	432	36	431	1492	1923	17.9
Real SPW on step chlorine doses of 4 mg/L× 5	0	37	0	0	847	341	30	37	1218	1255	6.9
	3	76	1	0	944	382	32	77	1358	1435	8.1
	7	103	0	0	983	400	36	103	1419	1522	11.2
	14	267	1	0	1020	431	36	268	1487	1755	18.3
Synthetic SPW with sweat on a shock chlorine dose of 20 mg/L	3	100	0	0	245	58	12	100	315	415	15.8
	165	460	160	0	1184	408	42	620	1634	2254	7.1
Synthetic SPW with sweat and urine on a shock chlorine dose of 20 mg/L	3	110	0	0	263	63	11	110	337	447	13.4
	165	1930	90	10	1044	355	39	2030	1438	3468	5.6

situations that can represent certain aspects of SPW under different conditions. The higher formation potential of DBPs is caused by two external stimulations, sweat and urine, from the pool users, with two internal stimulations, chlorine doses and chlorination time. All internal and external stimulations in the swimming pool lead to the formation of DBPs, which is the main product in swimming pools that needs to be reduced. The three clear ways to minimize DBPs in SPW are 1) to prevent significant amounts of harmful DBP formation in the first place by reducing DBP precursors and chlorine residual, 2) to quickly remove from the air and the water enough DBPs that do form by having acceptable air ventilation and water DBP removal technologies, and 3) to increase the amount of fresh water added to the pool and limit the extended use of recycled, old water. The third way is perhaps the least worthwhile because it could be expensive and wasteful to continually use more fresh water than necessary.

4 Conclusions

The research explored an important scenario of SPW with no additional anthropogenic organic input. With pre-existing anthropogenic organics and stimulations by residual chlorine or additional chlorine and extended incubation, the formation of DBPs, especially chloroform, was significantly induced. Similar observations were found by investigating synthetic SPW made with sweat and urine. The presence of urine led to a massive formation of chloroform, as noted by an approximate 19-fold increase after 165-day incubation with a shock chlorine dose. The research suggests that consistent residual chlorine and long water retention as two typical features of SPW could unlock the DBP formation potential of anthropogenic organics. Thus, limiting the introduction of anthropogenic organics may not have an immediate effect on reducing DBP levels, because their reactions with chlorine can be slow and long-lasting. Pool management should prioritize on control of urine and improving air ventilation. This work is useful to deepen understandings about DBP formation in SPW and provide implications for pool management and prospective legislation.

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