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A comparative study on the control of disinfection by-products (DBPs) and toxicity in drinking water

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ABSTRACT

In this work, ozone oxidation combined with ferric chloride and aluminum sulfate (Alum) coagulations was employed to investigate the optimum conditions for the control of the toxicity to *Daphnia magna and Pseudokirchneriella subcapitata* and disinfection by-products (DBPs) formation potential of Buyukcekmece (BC) lake water in Istanbul, Turkey. The efficiency of the treatment was determined by means of UV absorption at 254 nm (UV_{254}) and dissolved organic carbon (DOC) parameters. Four species of trihalomethanes (THMs) and eight species of haloacetic acids (HAAs) were identified and quantified for the evaluation of treatment methods on the formation of DBPs. It was observed that pre-ozonation enhanced DOC removal efficiency of alum and ferric chloride coagulations. 40 mg/l optimal alum and ferric chloride doses respectively exhibited 51% and 56% DOC reduction after 5 min pre-ozonation. As a result, THMs formation potential (THMFP) decreased approximately 43% after ozone enhanced alum and ferric chloride coagulations. Haloacetic acids formation potential (HAAFP) of water was reduced by 61% using pre-ozonation alone but a fluctuating increase and also decrease were reported after coagulations. Chlorinated raw and treated samples displayed acute toxicity in different pattern.

Keywords: Pre-ozonation; Enhanced coagulation; Haloacetic acids (HAAs); Trihalomethanes (THMs); Toxicity; Natural organic matter (NOM)

1. Introduction

Drinking water should be safe from pathogenic organisms and should not contain any harmful chemicals. It should also be aesthetically pleasing having low color and turbidity without the presence of any unpleasant taste or odors. Most of these problems result from natural organic matter (NOM). These organic matters may also react with chlorine and result in adversely harmful chlorinated and brominated organic disinfection by-products (DBPs) such as Trihalomethanes (THMs) and haloacetic acids (HAAs) [1,2]. Due to the importance of NOM, an huge effort has been focused on the nature and removal of NOM by using many advanced treatment technologies such as membrane filtration, enhanced coagulation [3], activated carbon adsorption, catalytic ozonation, ozone enhanced coagulation or ozonation [4,5], photo(electro)catalysis [6,7] to attain safe drinking water quality.

The use of metal-based coagulants is the most common water treatment process for removal of colour, turbidity and organic compounds. Coagulation has

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been shown to be one of efficient process for the overall removal of DBP precursors [8] and is considered by the US Environmental Protection Agency (USEPA) to be the best available technology for the control of DBPs. Dissolved organic matter (DOM) in raw water comprises a wide range of organic compounds, some of which are resistant to removal by coagulation. Otherwise, some protozoas such as *Giardia* and *Cryptosporidium* are very resistant to chlorine thus need high chlorine dose for the inactivation. Recently, pre-ozonation has become a common method to inactivate these resistant microorganisms and also enhances NOM removal in the subsequent coagulation process for the control of DBPs. Ozone does not directly produce chlorinated or brominated organic by-products. Ozone can alter the characteristics of precursors and affect the concentration and speciation of halogenated DBPs when chlorine is subsequently added into water. On the other hand, ozonation can lead to the formation of bromate during ozonation of the bromide-containing waters [9,10]. Thus, pre-ozonation following coagulation processes should be optimized for the control of DBPs especially THMs, HAAs and bromate considering that the United State Environmental Protection Agency [11] set the maximum contaminant levels of 80 µg/l, 60 µg/l and 10 µg/l respectively.

In this study, ozone alone, ferric chloride and aluminum sulfate (Alum) coagulations and pre-ozonation followed by coagulation were studied in detail on the raw water samples from Buyukcekmece (BC) lake to optimize treatment systems in term of NOM removal for the control of the formation of DBPs and their toxicity to *Daphnia magna* and *Pseudokirchneriella subcapitata* as well.

2. Materials and methods

2.1. Water samples

The raw water samples were collected from the influent of BC treatment plant which is one of the important drinking water resources of Istanbul, Turkey. Samples were kept refrigerated at +4°C during experiments. The raw water sample was characterized by measuring UV_{254}, DOC, bromide, chloride concentrations, pH, turbidity and alkalinity. All measurements performed according to Standard Methods [12].

Table 1
Water characteristics of the investigated raw water samples

Parameter	Unit	BC
Alkalinity	(mg $CaCO_3$/l)	133
Turbidity	(NTU)	2.8
UV_{254}	(1/cm)	0.94
DOC	(mg/l)	4.0
Bromide	(µg/l)	75
Chloride	(mg/l)	110
pH	--	7.69

2.2. Treatment methods

Ozonation (O_3): A Corona discharge ozone generator (PCI Model GL–1 type) was used. The ozone gas was transferred into a 5 L cylindrical reactor using a 10 cm ceramic porous tube type of commercial ozone diffuser. The system was operated in a semi-batch mode. Teflon tubing was used for the ozone gas lines. The ozone application rate was kept constant at 4.8 mg/min. In the ozonation experiments the ozone concentrations were monitored using the continuous ozone monitoring system (COMA) that uses the indigo colorimetric procedure [13].

Coagulation: Jar tests were performed using 1 L samples at room temperature for coagulation experiments by applying rapid mixing for 1 min at 100 rpm, then slow mixing without adding polymer for 30 min at 30 rpm, and finally settling for 60 min. Samples were filtered through 0.45 µm membrane (Millipore) to measure absorbance, DOC and DBPs. No pH adjustment was made during treatment of water. Enhanced coagulation experiments were performed according to the target pH value [11].

Chlorination: Raw **(RW)** and treated water **(TW)** samples (250 ml) were chlorinated according to Standard Methods [13].

2.3. Toxicity measurement

D. magna acute toxicity test was conducted on water samples using 24 h newborn daphnids for the end point of immobilization of the animals. The details are explained elsewhere [5,14,15]. Experiments were carried out quadruplicate without diluting the sample. Weekly transplanted unicellular green algae *P: subcapitata* in exponential growth was cultured in a salt solution dissolved in ultrapure water [16]. Cultures were kept in Erlenmeyer flasks at the same conditions of *D. magna*. The toxicity tests were initiated from an algal concentration of 3000 cells/ml and conducted in 3 replicates using 10 ml sample volume. The endpoint consisted of cell growth inhibition, which was measured after 96 h in a Burker cell counting chamber and calculated by dividing the difference of control and sample cells number to control cells number.

2.4. Analytical methodology

UV-vis absorbance values of raw and treated samples were measured by Shimadzu Spectrophotometer with 1.0 cm quartz cells. A Shimadzu 500A Total Carbon Analyzer was used for DOC measurements.

Trihalomethanes formation potential (THMFP) and haloacetic acids formation potential (HAAFP) of raw and treated samples were measured by Gas Chromatography. The THMs, chloroform, dibromochloromethane, dichlorobromomethane, bromoform and the HAAs monochloroacetic acid, monobromoacetic acid, dichloroacetic acid, bromochloroacetic acid, trichloroacetic acid, dibromoacetic acid, dichlorobromoacetic acid, dibromochloroacetic acid, tribromoacetic acid measurements were performed after dosing samples with chlorine (20 mg/I) after a reaction time of 7 d at 35 °C using a Hewlett Packard Gas Chromatograph 5890 Series II with a ^{63}Ni Electron Capture Detector according to standard procedure as described in previous studies [17,18]. Bromate was measured by Dionex ion chromatography with 5 µg/l detection limit.

3. Results and discussion

3.1. Optimization of ozone process

Ozone demand was followed by residual ozone measurements during ozonation of the BCRW sample. Residual ozone was observed in water after 2 min of ozonation period and increased with increasing ozonation time up to 9 min (Fig. 1). Steady state condition occurred after 9 min with residual ozone concentration of 0.9 mg/l which did not change even after prolonged ozonation periods up to 15 min. No bromate formation was reported in 9 min ozonation period in this study due to the low bromide level that changes according to the water level in the lake [5,15,19]. Organic DBPs are formed by the reaction between organic substances, inorganic compounds such as bromide, and oxidizing agents that are added to water during treatment. In water sources, aromatic humic and fulvic materials are the major DBP precursor. UV$_{254}$ is instrumental in identifying aromatic content of water. In treatment process, total organic carbon, DOC and UV$_{254}$ are often used as surrogate parameters for monitoring precursor levels and for determination of precursor removal efficiencies of treatment methods. In this study, NOM removal was followed by nonspecific absorbance and DOC parameters. Absorbance measurements were carried out at 5 different wavelengths (254 nm, 280 nm, 365 nm, 400 nm and 436 nm). The color of water as signified by the absorbance values at 400 nm and 436 nm, disappeared after 2.5 min of ozonation. UV absorbance values at 254 nm, 280 nm, and 365 nm decreased rapidly with increasing ozonation time and reached a steady state condition in 5 min (Fig. 2). High reduction rate of UV$_{254}$ was achieved during the initial stages of ozonation (up to 5 min) where the ozone residual was found to be around 0.25 mg/l (Fig. 1). The absorbance at 254 nm can be directly related to the presence aromatic and unsaturated structures of organic substances and it is considered as an additional quality parameter. Consequently, the reduction in UV absorbance is attributed mainly to the reaction of ozone with the unsaturated bonds and the aromatic rings.

During the early stages of ozonation, fluctuating DOC removal efficiencies ranging from 5% (in 5 min) to 9% (in 1 min) were observed. Highest DOC removal was achieved at 11% and no further enhancement on the removal efficiency was attained even after prolonged ozonation periods. These findings reflect the fact that ozone converts dissolved solids into suspended solids resulting in higher DOC removal but subsequent ozonation breaks large suspended solids into very small molecules which increase DOC concentration in the ozonated water [20].

3.2. Optimization of coagulation processes

Alum was gradually added into raw water until pH reached to the target pH of 7 and alum dose for enhanced coagulation was found as 60 mg/l. Thus, 20–60 mg/l equivalent doses of alum and ferric chloride were used for the optimization and comparison of alum and

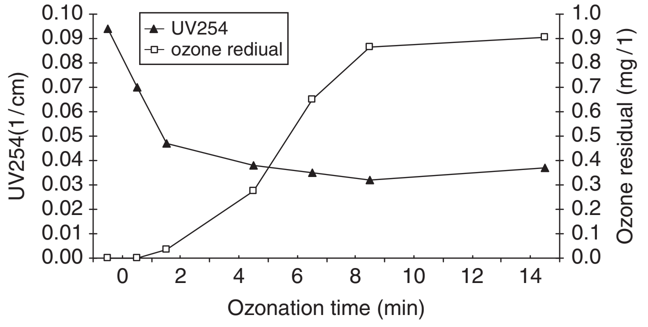

Fig. 1. Ozone demand and UV$_{254}$ removal with respect to ozone contact time.

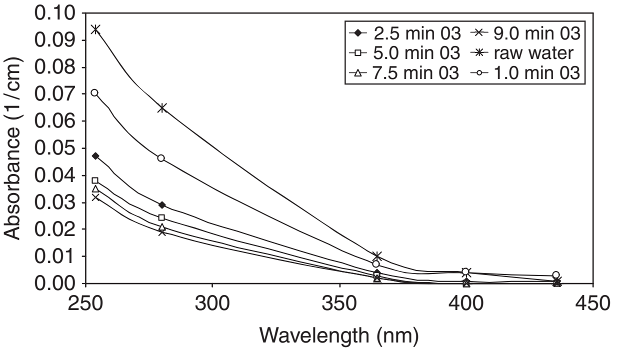

Fig. 2. Effect of ozonation time periods on the absorbance removal at different wavelengths.

ferric chloride coagulation alternatives. As can be followed from Fig. 3, alum doses of 20 mg/l and 40 mg/l reduced the DOC concentration by 15% and 35% respectively. The following alum doses did not change DOC removal significantly. Equivalent ferric chloride doses were also performed for the coagulation of raw water. DOC removal in ferric chloride coagulation was 10% less than that obtained by alum under same experimental conditions. But in contrast to alum coagulation, DOC removal increased using equivalent higher ferric chloride dose and almost same DOC removal (36%) was achieved by using the dose of 60 mg/l found for the enhanced coagulation (Fig. 4). Generally, pre-ozonation has been reported to enhance, hinder or has no effect on DOC removal by coagulation [8,21]. In this work, the effect of pre-ozonation on the following alum and ferric chloride coagulations was investigated by using different pre-ozonation contact time (1 min, 2.5 min, 5.0 min, 7.5 min and 9.0 min). The effect of the gradual addition of alum and ferric chloride coagulants to raw and ozonated samples without pH adjustment are presented in Figs. 3 and 4. Pre-ozonation up to 2.5 min exhibited no significant enhancement or inhabitation effect on the performance of alum coagulation process with respect to the DOC removal and almost same DOC degradation ratio (35%) was achieved after coagulation of ozonated sample. But 5 min pre-ozonation enhanced DOC degradation by ferric chloride coagulation and the performance of alum coagulation increased by 20%. The prolonged pre-ozonation of raw water inhibited DOC removal by alum coagulation however DOC concentration (around 2.0 mg/l) remained almost same due to the higher DOC removal after ozonation. Consequently the ozone enhanced alum coagulation occurred after an optimal ozonation time period of 5 min. This ozonation period was that UV_{254} degradation reached a steady state level (60%) (Fig. 1). The results reflect the fact that there is a correlation between UV_{254} removal and optimal ozone dose for the enhanced alum coagulation. In terms of optimal alum dose, pre-ozonation did not changed the optimal dose of 40 mg/l and under optimum conditions 56% of DOC was removed from the ozonated water.

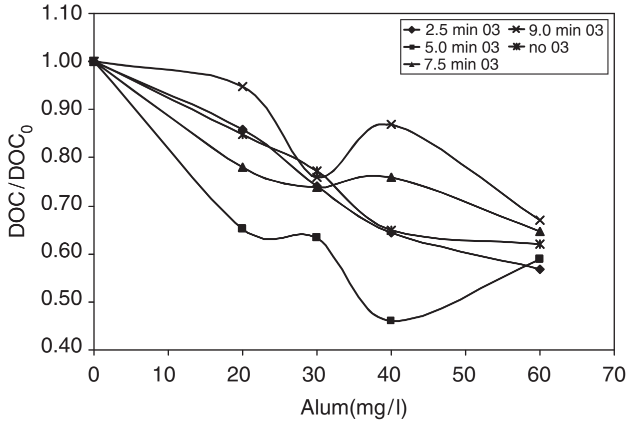

Fig. 3. The effect of pre-ozonation on DOC removal by alum coagulation.

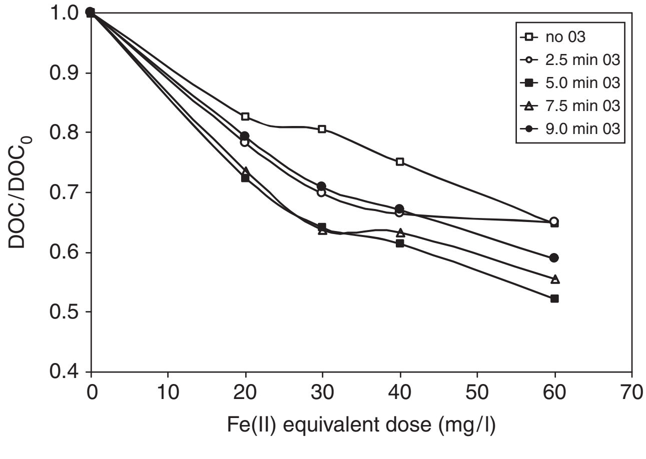

Fig. 4. The effect of pre-ozonation on DOC removal by ferric chloride.

The pre-ozonation showed similar enhancement effect on the performance of ferric chloride coagulation. The efficiency of ferric chloride coagulation increased with increasing pre-ozonation contact time up to 5 min. The following pre-ozonation periods slightly changed DOC removal by ferric coagulation. 40 mg/l ferric chloride concentration was found to be optimal for the ozonated sample while it was 60 mg/l without pre-ozonation. After pre-ozonation, DOC removal (56%) by alum coagulation was slightly higher than that achieved by ferric chloride coagulation (51%).

3.3. THMs formation

Enhanced ferric and alum coagulations were applied for the treatment of raw and ozonated water without pH adjustment to find efficient treatment process for the control of halogenated DBPs of THMs and HAAs. 60 mg/l of alum and equivalent ferric chloride doses ware used for the comparison of all treatment methods. Among the THMs, chloroform was found to be the dominant species in all of the raw, ozonated, alum and ferric chloride treated waters (Table 2). The change in the distribution of THM species was insignificant (5%) after all treatment methods. Pre-ozonation alone caused 35% decrease in THMFP of water even though DOC removal after ozone was around 10%. This result was attributed to its effect on the THM precursors that was monitored as UV_{254} (Fig. 1). Alum and ferric chloride coagulations with and without

Table 2
The effect of treatment methods on the THMFP of BCRW

Treatment method	UV_{254} (1/cm)	DOC (mg/l)	pH	THMFP (µg/l)	THM species (µg/l)			
					$CHCl_3$	$CHCl_2Br$	$CHClBr_2$	$CHBr_3$
Ozone + ferric chloride	0.017	2.10	6.80	100	51	31	16	2
Ozone + alum	0.018	2.06	7.10	102	51	32	17	2
Alum	0.044	2.48	6.70	140	64	45	28	4
Ferric chloride	0.048	2.59	7.00	142	69	44	26	4
Ozone alone	0.032	3.50	7.67	116	74	31	11	1
Raw water	0.940	4.00	7.69	178	103	51	21	3

Table 3
The effect of treatment methods on the HAAFP of BCRW

Treatment method	HAAFP (µg/l)	HAA species (µg/l)		
		Monochloroacetic acid	Dichloroacetic acid	Trichloroacetic acid
Ozone + ferric chloride	81	5	47	29
Ozone + alum	43	5	34	4
Alum	141	14	30	98
Ferric chloride	62	5	8	49
Ozone alone	28	10	12	6
Raw water	71	27	40	5

pre-ozonation showed almost same effect on the THMFP. The enhanced alum and ferric chloride coagulation did not reduce the THMFP much and THMFP of alum and ferric chloride treated water was just 20% less than that of raw water whereas the pH of both coagulated waters (around 7.0) were less than pH value of raw water (7.69). Pre-ozonation did not enhance the performance of alum and ferric coagulation for the reduction of THMFP however the highest THMFP reduction (approximately 43%) was observed after both ozone enhanced alum and ferric chloride coagulations.

3.4. HAAs formation

The distribution of trichloroacetic acid increased especially after enhanced alum and ferric chloride coagulations (63% and 73% respectively) probably due to the lower pH values.The increase in the portion of trichloroacetic acid was much less (35% and 9% respectively) after ozone enhanced alum and ferric chloride coagulations. The percent of monochloroacetic acid to HAAs decreased significantly after both enhanced and ozone enhanced alum and ferric chloride coagulations while it was almost same after pre-ozonation (Table 3).

Because of lower pH values, alum and ferric chloride coagulated water had a higher HAAs/THMs ratio compared with that of the raw water. On the other hand, at the same pH value pre-ozonation alone decreased HAAs/THMs value two fold. It indicates that ozone removes more HAAs precursors than THMs precursors. Without pH adjustment, HAAFP of water was reduced by 61% using pre-ozonation alone while it was decreased by 13% and 39% by enhanced ferric chloride and ozone enhanced alum coagulations respectively (Table 3). An increase in HAAFP after enhanced alum and ozone enhanced ferric chloride coagulation was attributed to the lower pH values after coagulation. Bekbolet et al. (2005) investigated the formation of brominated HAAs in bromide containing BCRW and they reported significant brominated HAAs concentrations after pre-ozonation. In contrast, in this study some brominated haloacetic acids (Monobromoacetic acid, bromochloroacetic acid, dibromoacetic acid, dichlorobromoacetic acid, dibromochloroacetic acid, tribromoacetic) concentrations were found to be under detection limits in all treated samples probably due to the low bromide level of the raw water sample [22].

3.5. Toxicity

As it is seen in Fig. 5 that the *P. subcapitata* cell growth ws stimulated when this specie was exposed to

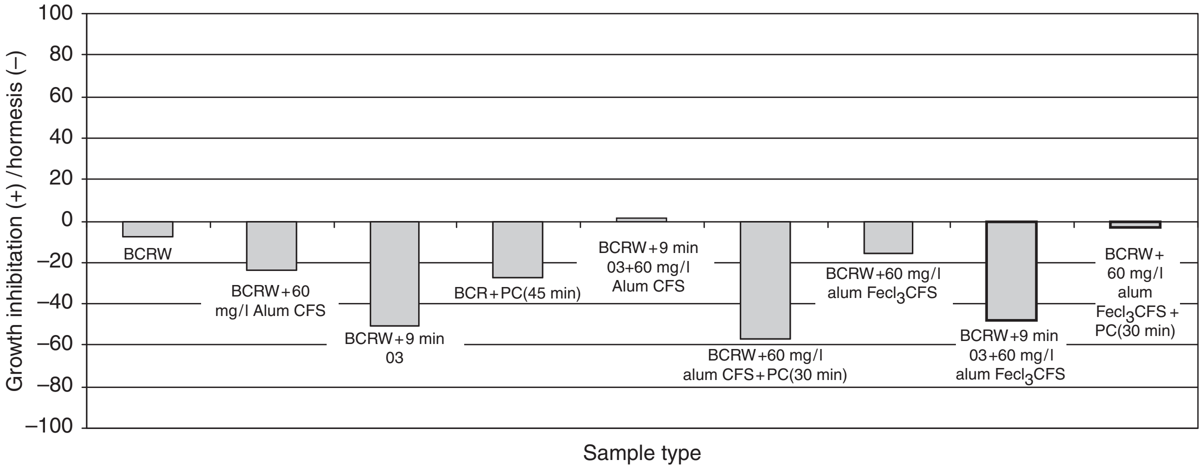

Fig. 5. Changes in toxicity of *P. Subcapitata* exposed to raw and treated samples before chlorination.

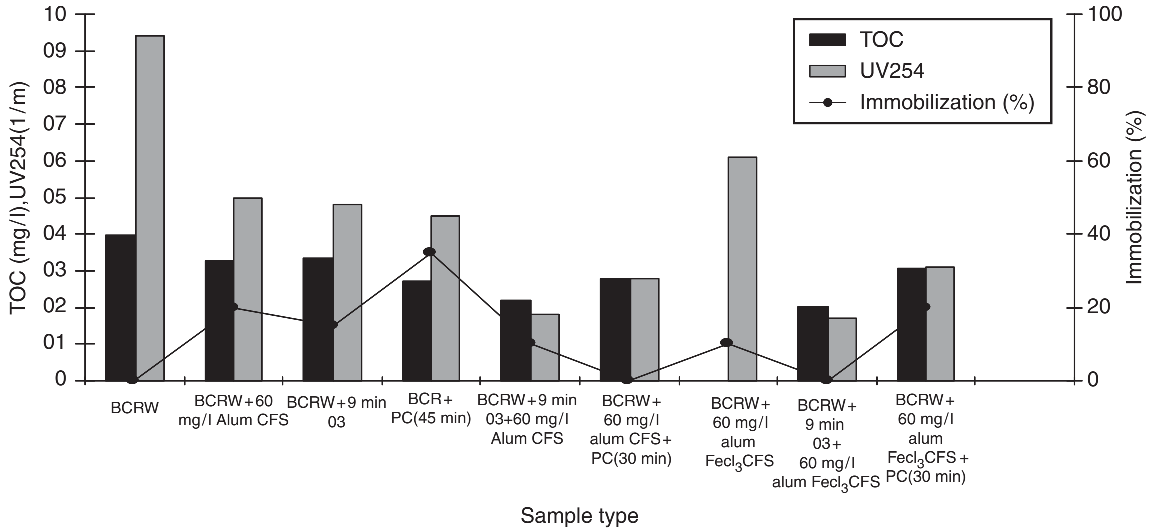

Fig. 6. Changes in toxicity of *D. magna* exposed to raw and treated samples after chlorination.

unchlorinated samples whereas except one sample whole chlorinated samples displayed 100% toxicity to *P. subcapitata*.

None of raw water samples before introduced to the treatment and sequentially chlorination displayed toxicity to *D. magna* (data not shown) (Fig. 6).

4. Conclusions

The effects of pre-ozonation on the following alum and ferric chloride coagulations were investigated in terms of DOC removal, acute toxicity and DBP formation potentials and the following conclusions have been drawn.

Almost same DOC reduction (35%) was achieved by both alum and ferric chloride coagulations however optimal alum and ferric doses were found as 40 mg/l and 60 mg/l respectively. Pre-ozonation enhanced both alum and ferric chloride coagulation around 15–20% after an optimal ozone contact time of 5 min. 20 mg/l doses of alum and ferric chloride resulted in 35% DOC removal while 40 mg/l optimal alum and ferric chloride doses respectively exhibited 51% and 56% DOC reduction after 5 min pre-ozonation. Prolonged pre-ozonation time periods inhibited the performance of alum coagulation while it had insignificant effect on the ferric chloride coagulation.

THMFP of BCRW was reduced by 35% using pre-ozonation alone. The enhanced and ozone enhanced alum and ferric chloride coagulations exhibited almost same effect on the THMFP. The decrease in THMFP in both coagulation processes were found to be 20% by coagulation alone and 43% by pre-ozonation enhanced coagulation. The distribution of trichloroacetic acid increased after enhanced alum and ferric chloride coagulations by 63% and 73% respectively. Pre-ozonation alone decreased HAAs/THMs value, achieved in raw water by two fold. It reflects the fact that ozone removes more HAAs precursors than THMs precursors. Without pH adjustment, HAAFP of water was reduced by 61% using pre-ozonation alone but a fluctuating increase and also decrease were reported after both enhanced coagulations because of the lower pH values of treated sample. Raw and treated samples displayed acute toxicity on *Daphnia magna* in different pattern and practically "no dose-response behavior" was observed.

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