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Optimal methods for quenching H_2O_2 residuals prior to UFC testing

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Abstract

In this paper, the quenching of hydrogen peroxide by catalase, sodium hypochlorite, sodium thiosulfate and sodium sulfite, prior to UFC testing, was investigated. Sodium hypochlorite, sodium thiosulfate and sodium sulfite were found to be unsuitable for quenching H_2O_2 residuals because the procedures are time-consuming and complicated in that they require potentially multiple measurements of the peroxide and chlorine residuals. In contrast, quenching of peroxide with catalase is a simple procedure. Catalase doses of less than 0.2 mg/L were found to have no impact on DBP (TTHM, HAA and aldehyde) formation in the UFC test, and the time that was needed to quench 100 mg/L peroxide (room temperature, pH 8.3) was less than 10 min. In addition, peroxide was found to react with DPD reagents that are used to measure chlorine residuals, a phenomenon that may lead to falsely high chlorine residuals in the UFC test.

Keywords: DBPs; Disinfection byproducts; H_2O_2; UFC test; Scavenger; Catalase

1. Introduction

UV and UV/H_2O_2 oxidation are becoming popular alternative treatment technologies for eliminating trace contaminants from water or wastewater [1–5]. Among all the advanced oxidation processes (AOPs), such as UV, UV/H_2O_2, UV/O_3, UV/H_2O_2/O_3, O_3/H_2O_2, etc. the homogeneous UV/H_2O_2 process has attracted the most attention in recent years due to its relatively low cost and simple operation [6]. However, when applied for drinking water treatment purposes, UV or UV/H_2O_2 processes will likely continue to be followed by secondary disinfection with chlorine or chloramines to maintain disinfection throughout the distribution system. Hence, the impact of UV or UV/H_2O_2 processes on the formation of halogenated disinfection byproducts remains of public concern. Andrews et al. [7,8], Malley et al. [9], Mofidi et al. [10], and Zheng et al. [11] have all investigated the ultimate THM and HAA formation in water that was chlorinated following UV or UV/H_2O_2 irradiation.

In general, distribution system simulation tests such as the Uniform Formation Conditions (UFC) test are used to investigate the effect of UV or UV/H_2O_2 on DBP formation from subsequent chlorine or chloramine disinfection. The UFC test procedure was developed by Summers et al. [12] and has been widely accepted for assessing the DBP formation potential when chlorination is employed. The pH of water samples is adjusted to 8, and a constant reaction temperature of $20 \pm 1°C$ is maintained during the 24 h chlorination period. The ultimate chlorine residuals are required to be within 1.0 ± 0.4 mg/L as Cl_2.

Since H_2O_2 ($E_0 = 1.77$ V) has a higher standard reduction potential than Cl_2 (1.36 V) or OCl^- (0.89 V), it can react with chlorine so that higher chlorine doses are needed to meet desired chlorine residuals, and ultimately have an impact on DBP formation. Studies reported by Albertin et al. [13] and Batterman et al. [14] demonstrated that H_2O_2 followed by chlorination had a

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significant impact on the formation of TTHM and HAAs. Furthermore, as will be reported herein, H_2O_2 was found to interfere with DPD methods that are commonly used to measure chlorine, having a similar color response as chlorine. Hence, prior to UFC testing, residual H_2O_2 in water samples must be quenched accurately.

Theoretically, any reagent that can react with H_2O_2 can be considered as a possible scavenger of H_2O_2 residuals. The ideal reagent will not produce or affect the production of adverse chemical byproducts during H_2O_2 quenching. In this research, four of the most commonly used reagents were selected to assess their quenching efficiencies on H_2O_2 and their effects on DBP formation. These reagents included catalase, sodium hypochlorite, sodium thiosulfate and sodium sulfite.

It is well known that catalase quenches H_2O_2 with a high efficiency. Sodium hypochlorite, sodium thiosulfate and sodium sulfite also react with H_2O_2, according to Eqs. (1)–(3). The stoichiometric doses needed for sodium hypochlorite, sodium thiosulfate and sodium sulfite to accurately quench 1 mg/L H_2O_2 are 2.09, 9.29 and 3.7 mg/L, respectively. For experiments in which post-AOP chlorination byproducts are of interest, after completion of AOPs the remaining H_2O_2 residual must first be accurately quenched (preferably without using excess reagent) before the UFC test can begin. This requires very accurate measurement of the peroxide residuals and accurate preparation and measurement of the quenching solution of choice. Overdosing or underdosing any of these reagents will likely result in subsequent chlorine residuals that are outside of the UFC test requirement in the range of 1.0 ± 0.4 mg/L as Cl_2.

$$NaOCl + H_2O_2 \rightarrow NaClO_2 + H_2O \quad (1)$$

$$2Na_2S_2O_3 + H_2O_2 \rightarrow Na_2S_4O_6 + 2NaOH \quad (2)$$

$$Na_2SO_3 + H_2O_2 \rightarrow Na_2SO_4 + H_2O \quad (3)$$

This research was performed a part of the AWWARF project "Innovative UV Technologies to Oxidize Organic and Organoleptic Chemicals". In this paper, quenching of both high (~100 mg/L) and low (~10 mg/L) H_2O_2 concentrations were investigated. The reaction of H_2O_2 with DPD was also investigated in order to verify the colorimetric interference of H_2O_2 residuals on the measurement of chlorine residuals.

2. Experimental design and methods

The experiment was performed in two phases. The first phase, designated as the Pretest, investigated whether commonly used catalase doses, in the range of 2 mg/L, would have any effect on THM, HAA and aldehyde formation. The Pretest was also performed in two steps: one at higher catalase doses (2 and 4 mg/L) and one at lower catalase doses (0.05–0.2 mg/L). The second Pretest in which lower catalase doses were used, was performed to determined the lowest catalase doses that would be enough to scavenge the H_2O_2 residuals, and to determine whether these lower doses would have impacts on DBP formation.

In the second phase, designated as the Confirmation Test, experiments were performed to confirm the Pretest results, and also to investigate the quenching efficiency and effects on DBP formation by selected inorganic reagents (sodium hypochlorite, sodium thiosulfate and sodium sulfite). These tests were also performed under conditions of both high initial H_2O_2 concentration (~100 mg/L) and low initial H_2O_2 concentration (~10 mg/L).

All tests were performed with freshly prepared synthetic water whose constituents are shown in Table 1. The synthetic water was intended to simulate typical drinking water quality with a total organic carbon (TOC) level of approximately 3 mg/L by combining of these constituents into Millipore-UV® water. All of the inorganic chemicals used in the preparation of the synthetic water were analytical or USP grade. Humic acid was IHSS Suwannee River NOM and alginic acid was purchased from Aldrich Chemical Company Inc. The pH of the synthetic water was approximately 8.3. The quenching agents used in this research included bovine catalase (Sigma), sodium hypochlorite (VWR), sodium thiosulfate (BDH) and sodium sulfite (BDH).

The H_2O_2 quenching tests and UFC tests were performed in 250 mL amber bottles with Teflon liners inside the caps. All chemical reagents (catalase, sodium hypochlorite, sodium thiosulfate and sodium sulfite) used in the tests were prepared in appropriate concentrations so that the added reagent volume would not exceed one percent of the water sample volume (250 mL). The chemical concentrations of the stock solutions used in the tests are listed as follows: 0.1 mg/mL catalase, 31 mg/mL as Cl_2 sodium hypochlorite,

Table 1
Synthetic water constituents

	Ca^{2+}	Mg^{2+}	Na^{+}	$(CO_3)_{TOT}$	NO_3^{-}	Cl^{-}	SO_4^{2-}	Humic acid	Alginic acid
mg/L	29.1	9.99	36.2	90	3.00	40.0	55.0	2.56	5.32

100 mg/mL sodium thiosulfate, 50 mg/mL sodium sulfite. Three different doses for each inorganic chemical scavenger were applied according to the scavenger: H_2O_2 stoichiometric ratio of 0.9:1, 1:1 and 1.1:1. All quench tests were performed at room temperature. All of the samples that were quenched with catalase and those samples quenched by inorganic scavengers stoichiometrically were subjected to the UFC test.

UFC tests were performed according to the procedure reported by Summers et al. [12]. A stock solution of 0.9 mg/mL (as Cl_2) sodium hypochlorite was used in the UFC test. Chlorine residuals were determined by reacting with DPD for three minutes followed by reading the concentration on a Hach® DR/2010 Portable Datalogging Spectrophotometer (4500-Cl G). Four drops of 8 g/L sodium thiosulfate were added to quench the chlorine residual when the UFC test was finished. Then the samples were prepared for DBP analysis. Methods for the analysis of TTHM (6232 B), HAA_9 (6251 B) and aldehydes (6252 B) were from Standard Methods [15] with minor modifications. The minimum detection limits in this laboratory for THMs were: $CHCl_3$: 0.08 μg/L, $CHCl_2Br$: 0.05 μg/L, $CHClBr_2$: 0.05 μg/L, $CHBr_3$: 0.01 μg/L; for HAA_9: ClAA: 0.3 μg/L, BrAA: 0.4 μg/L, Cl_2AA: 0.3 μg/L, Cl_3AA: 0.5 μg/L, BrClAA: 0.5 μg/L, $BrCl_2AA$: 1.1 μg/L, Br_2AA: 0.4 μg/L, Br_2ClAA: 0.5 μg/L, Br_3AA: 0.4 μg/L; for aldehydes: formaldehyde: 1.6 μg/L, acetaldehyde: 2.0 μg/L, glyoxal: 3.5 μg/L, methylglyoxal: 2.5 μg/L, respectively. Laboratory blanks and standards were measured simultaneously with each batch of samples to monitor measurement accuracy. Relative standard deviations for THMs were within $100 \pm 5\%$, for HAAs within $100 \pm 7\%$, and for aldehydes within $100 \pm 5\%$. Hydrogen peroxide concentrations were measured by reaction with iodide catalyzed by molybdate [16]. An HP UV-VIS spectrophotometer was used to determine the concentrations of H_2O_2. The minimum detection limit for H_2O_2 measurement was 50 μg/L.

3. Results and discussion

3.1. Peroxide scavenging efficiency

Higher catalase doses (2 or 4 mg/L) resulted in the rapid decay of high concentrations of H_2O_2 (Table 2). It took less than 30 min to quench 142 mg/L H_2O_2 with 2 mg/L catalase (Pretest 1). While longer times were found to quench almost the same concentration of H_2O_2 with lower catalase doses (0.05–0.2 mg/L, Pretest 2), it took only 60 min for 0.2 mg/L catalase to quench 141 mg/L H_2O_2 and 180 min for 0.05 mg/L catalase in pretest.

In the Confirmation Test (Table 3), even less time was found necessary for the same catalase dose used in the Pretest to scavenge high concentrations of H_2O_2. 0.2 mg/L catalase quenched 105 mg/L H_2O_2 within 10 min, and 11.5 mg/L H_2O_2 was quenched within 5 min. This discrepancy between the Pretest and Confirmation Test might be due to the preparation of the catalase stock solution. In the Pretest, the catalase stock solution was prepared the day before the quench test, whereas the catalase stock solution was prepared fresh for the Confirmation Test. Hence, daily fresh prepared catalase is recommended to ensure its activity and high efficiency to eliminate H_2O_2 residual. Regardless, 10 min was demonstrated to be enough time for 0.2 mg/L catalase to quench H_2O_2 at concentrations of as high as approximately 100 mg/L.

The reaction between inorganic scavengers and H_2O_2 occurred rapidly both under high and low H_2O_2 concentrations when appropriate doses were used (Tables 4 and 5). This demonstrated that quenching time should not be a factor if inorganic scavengers were to be used. However, the scavenger dose should at least be the stoichiometric equivalent or be added in excess. If peroxide quenching is to be followed by a UFC test, only stoichiometric doses can be used. Overdosing will result in the requirement for additional chlorine to be

Table 2
H_2O_2 decay and catalase dose—Pretest

Reaction time (min)	Pretest 1			Pretest 2			
	Catalase dose (mg/L)			Catalase dose (mg/L)			
	0	2	4	0	0.05	0.1	0.2
	H_2O_2 (mg/L)			H_2O_2 (mg/L)			
0	142	142	142	141	141	141	141
5	NA	NA	NA	NA	54.3	58.9	48.0
30	140	LDL	LDL	NA	45.1	12.7	0.8
60	NA	NA	NA	NA	20.9	1.7	LDL
120	NA	NA	NA	NA	2.6	LDL	NA
180	NA	NA	NA	NA	LDL	NA	NA

Note: NA—not applicable; LDL—less than the minimum detection limit.

Table 3
H_2O_2 decay and catalase dose—Confirmation Test

Reaction time (min)	Catalase dose (mg/L)							
	0.05	0.1	0.2	0.3	0.05	0.1	0.2	0.3
	H_2O_2 (mg/L)				H_2O_2 (mg/L)			
0	105	105	105	105	11.5	11.5	11.5	11.5
5	5.8	2.3	0.2	LDL	3.8	0.9	LDL	LDL
10	0.7	LDL	LDL		0.2	LDL		
30	LDL				LDL			

Note: LDL—less than the minimum detection limit.

Table 4
Quenching of high concentrations of H_2O_2 with inorganic reagents

Reaction time (min)	NaOCl (mg/L)			$Na_2S_2O_3$ (mg/L)			Na_2SO_3 (mg/L)		
	192	213	234	853	948	1042	340	377	415
	H_2O_2 (mg/L)			H_2O_2 (mg/L)			H_2O_2 (mg/L)		
0	102	102	102	102	102	102	102	102	102
5	13.6	0.6	LDL	11.6	0.2	LDL	12.5	0.3	LDL
10	12.9	0.8	LDL	11.8	0.3	LDL	12.9	0.2	LDL
30	13.2	0.6	LDL	11.6	0.1	LDL	12.8	0.2	LDL

Note: LDL—less than the minimum detection limit.

Table 5
Quenching of low concentrations of H_2O_2 with inorganic reagents

Reaction time (min)	NaOCl (mg/L)			$Na_2S_2O_3$ (mg/L)			Na_2SO_3 (mg/L)		
	19.8	21.9	24.1	87.8	97.5	107	35.0	38.9	42.7
	H_2O_2 (mg/L)			H_2O_2 (mg/L)			H_2O_2 (mg/L)		
0	10.5	10.5	10.5	10.5	10.5	10.5	10.5	10.5	10.5
5	1.2	0.2	LDL	1.1	LDL	LDL	1.4	0.1	LDL
10	1.2	0.3	LDL	1.3	LDL	LDL	1.4	0.2	LDL
30	1.2	0.2	LDL	1.3	LDL	LDL	1.4	0.1	LDL

Note: LDL—less than the minimum detection limit.

added to quench the residual scavenger in the water samples prior to UFC testing.

The data in Tables 4 and 5 also confirm that the stoichiometric amounts were calculated correctly. For example, approximately 10% of the H_2O_2 remained when a 90% stoichiometric dose was applied.

3.2. Effect of scavenger doses on DBP formation

The DBPs formed in samples in which different catalase doses were used to quench H_2O_2 residuals are shown in Tables 6 and 7. The most commonly used dose of 2 mg/L or more resulted in much greater concentrations of TTHM, HAA_9 and aldehydes than were observed in control samples (without catalase), whereas 0.2 mg/L or less catalase contributed nothing to the DBP formation. These results were first observed in the Pretest and were further confirmed by the Confirmation Test, both with high and low initial H_2O_2 concentrations. Since a concentration of 0.2 mg/L catalase quenched 105 mg/L H_2O_2 within 10 min and had no effect to DBP formation, this dose is recommended for practical use.

Even when sodium hypochlorite, sodium thiosulfate or sodium sulfite were added stoichiometrically into the samples to quench H_2O_2, small H_2O_2 residuals were still found in some samples (Tables 4 and 5). Slight overdosing or underdosing of inorganic scavengers

Table 6
Effect of catalase dose on the formation of DBPs—Pretest

DBPs	Pretest 1 ($[H_2O_2]_0 = 142$ mg/L)			Pretest 2 ($[H_2O_2]_0 = 141$ mg/L)			
	Catalase dose (mg/L)						
	0	2	4	0	0.05	0.1	0.2
TTHM (μg/L)	88.4	103	133	71.4	69.9	66.2	65.7
HAA_9 (μg/L)	70.0	103	118	49.8	48.9	49.4	48.2
Aldehydes (μg/L)	4.6	9.6	18.0	LDL	LDL	LDL	LDL

Note: LDL—less than the minimum detection limit.

Table 7
Effect of catalase dose on the formation of DBPs—Confirmation Test

DBPs	$[H_2O_2]_0 = 105$ mg/L				$[H_2O_2]_0 = 11.5$ mg/L			
	Catalase dose (mg/L)							
	0.05	0.1	0.2	0.3	0.05	0.1	0.2	0.3
TTHM (μg/L)	87.0	89.7	85.3	86.6	85.2	83.9	83.4	81.7
HAA_9 (μg/L	54.6	53.8	56.5	56.2	52.5	55.1	53.1	56.1
Aldehydes (μg/L)	LDL	LDL	LDL	LDL	LDL	LDL	LDL	LDL

Note: LDL—less than the minimum detection limit. DBPs in the control sample in the UFC test (without catalase added) were: 87.0 μg/L TTHM, 55.4 μg/L HAA_9.

Table 8
DBPs from the inorganic quenching of 102 mg/L H_2O_2 followed by UFC testing

DBPs	$[H_2O_2]_0 = 102$ mg/L		
	NaOCl (mg/L)	$Na_2S_2O_3$ (mg/L)	Na_2SO_3 (mg/L)
	213	948	377
TTHM (μg/L)	89.6	93.2	91.6
HAA_9 (μg/L)	59.3	59.9	60.0
Aldehydes (μg/L)	LDL	LDL	LDL

Note: LDL—less than the minimum detection limit. DBPs in the control sample in the UFC test (0 mg/L scavengers) were: 87.0 μg/L TTHM, 55.4 μg/L HAA_9.

appears inevitable because of the error associated with the measurement of H_2O_2, variations in the preparation of the scavenger solution, and uncontrolled reactions between the components of the synthetic water (especially NOM) and the added inorganic compounds. When high peroxide concentrations are present (approximately 100 mg/L), even a small error (2–5%) may result in significant scavenger overdosing or underdosing. The extra sodium hypochlorite doses calculated stoichiometrically were used to scavenge the quenching reagent residuals in the UFC test so that the appropriate chlorine residuals were achieved.

Inorganic scavengers that are added stoichiometrically to samples to test DBP formation with subsequent UFC testing did not appear to affect DBP test results (Tables 8 and 9). However, since 0.2 mg/L catalase could quench a very broad range of H_2O_2 concentration (as high as 140 mg/L) and contributed nothing to DBP formation, inorganic scavengers are not as highly recommended for quenching high concentrations of H_2O_2. Nevertheless, if no UFC test is to be performed or if chlorine/chloramine disinfection follows the quenching process, inorganic scavengers may be used effectively.

3.3. Interference of H_2O_2 residuals on the measurement of chlorine

Since DPD (*N*,*N*-diethyl-*p*-phenylenediamine) is a reducing agent, it can react with H_2O_2 as it does with

Table 9
DBPs from the inorganic quenching of 10.5 mg/L H_2O_2 followed by UFC testing

DBPs	$[H_2O_2]_0 = 10.5$ mg/L		
	NaOCl (mg/L)	$Na_2S_2O_3$ (mg/L)	Na_2SO_3 (mg/L)
	21.9	97.5	38.9
TTHM (μg/L)	85.6	90.5	88.6
HAA_9 (μg/L	58.7	59.1	56.4
Aldehydes (μg/L)	LDL	LDL	LDL

Note: LDL—less than the minimum detection limit. DBPs in the control sample in the UFC test (0 mg/L scavengers) were: 87.0 μg/L TTHM, 55.4 μg/L HAA_9.

Table 10
Reaction of H_2O_2 with DPD reagents

	Sample1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6
H_2O_2 conc. (mg/L)	0	5	10	20	30	50
The color response as mg/L Cl_2	0	0.13	0.20	0.63	0.86	1.00

chlorine and erroneously be measured as a chlorine residual when, in fact, an H_2O_2 residual exists. Table 10 shows the effect of the H_2O_2 reaction with DPD reagents that are supplied in Hach kit powder pillows for Total/Free Cl_2 tests. It implies that if inorganic scavengers used to quench H_2O_2 were underdosed, an error would result in that it would seem that the chlorine residual met the requirement of the UFC test but actually it did not.

4. Conclusions

Although UV or UV/H_2O_2 processes are becoming widely used technologies, when used for water disinfection they would likely be followed by secondary disinfection with chlorine or chloramines to maintain disinfection throughout the distribution system. In general, UFC tests would be used to investigate the effect of UV or UV/H_2O_2 on DBP formation from subsequent chlorine or chloramine disinfection. Prior to UFC testing, residual H_2O_2 in water samples must be quenched.

In this paper, the quenching of hydrogen peroxide by catalase, sodium hypochlorite, sodium thiosulfate and sodium sulfite was investigated. Quenching of peroxide with catalase is a simple procedure. Low catalase doses of less than 0.2 mg/L were found to have no impact on DBP (THM, HAA and aldehyde) formation in the UFC test, and the time that was needed to quench 100 mg/L peroxide (room temperature, pH 8.3) was less than 10 min. However, doses of 2 mg/L or more were found to produce elevated TTHM, HAA and aldehyde concentrations in the UFC test. The HAAs increased from 70.0 μg/L in the sample without catalase to 103 μg/L at 2 mg/L and 118 μg/L at 4 mg/L catalase, TTHM from 88.4 to 103 and 133 μg/L, respectively. Aldehydes were also found to increase from 4.6 to 9.6 and 18.0 μg/L.

Sodium hypochlorite, sodium thiosulfate and sodium sulfite were found to be unsuitable for quenching H_2O_2 residuals if subsequent UFC testing was to be performed or chlorine/chloramine disinfection process would be applied. Even when the inorganic scavengers were added stoichiometrically, there were still measurable H_2O_2 residuals in the sample by the end of the quench test. It was required that extra chlorine be accurately added to quench the residuals prior to the UFC test. The procedure is time-consuming in that it requires multiple measurements of the peroxide and chlorine residuals. In addition, overdosing or underdosing of chlorine may be problematic because of the error associated with the measurement of H_2O_2. When high peroxide concentrations are present (approximately 100 mg/L), even a small error (2–5%) may result in significant chlorine overdosing or underdosing because chlorine doses employed in UFC tests are typically in the range of 3–10 mg/L.

In this study, peroxide was also found to react with DPD reagents that are used to measure chlorine residuals. This phenomenon may yield false chlorine residuals in the UFC test.

In conclusion, based on a comparison of the quenching of H_2O_2 by catalase and inorganic scavengers, use of a catalase dose of less than 0.2 mg/L was found to be an optimal option. At the dose of 0.2 mg/L, catalase can effectively quench H_2O_2 concentrations of as high as

145 mg/L in less than 10 min with no impact on DBP formation in the UFC test.

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