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Removal of IBMP using ozonation: role of ozone and hydroxyl radical

Shumin Zhu[a], Bingzhi Dong[a,*], Naiyun Gao[a], Jin Jiang[b]

[a]*State Key Laboratory of Pollution Control and Resource Reuse, College of Environmental Science and Engineering, Tongji University, Shanghai, 200092, China, email: zhushuminwater@sina.com (S. Zhu), Tel. +86 21 65982691; Fax: +86 21 65986313; emails: dbzwater@163.com (B. Dong), gaony2014@163.com (N. Gao)*
[b]*Shanghai Municipal Engineering Design Institute (Group) Co., Ltd, Shanghai, 200092, China, email: 073229@tongji.edu.cn*

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ABSTRACT

Ozonation of a representative taste and odor compound, 2-Isobutyl-3-methoxy pyrazine (IBMP), in drinking water was systematically investigated in this study. Batch experiments were conducted to evaluate the influencing factors including initial IBMP concentration, O_3 addition rate, initial pH, H_2O_2 dosage, and different water qualities on the degradation of IBMP in the ozonation process. Results showed that the degradation of IBMP followed pseudo-first order kinetics. The degradation rate constants increased with the increasing O_3 addition rate and initial pH value, whereas it decreased with the increasing initial IBMP concentration. Degradation of IBMP is mainly dominated by direct ozone oxidation at low pH, while the free radical oxidation may become dominant at high pH levels. Moreover, appropriate H_2O_2 dosage can greatly increase the IBMP degradation rates, but large excess H_2O_2 would act as an $OH^{\cdot}$ scavenger and inhibit the degradation. In addition, lower degradation rate was observed in raw water, compared to that in deionized water. Overall, ozonation can be a promising treatment technology to control IBMP-associated water pollution.

Keywords: Taste and odor compounds; 2-Isobutyl-3-methoxypyrazine; Ozonation; Kinetics; Radical oxidation

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1. Introduction

Cyanobacterial blooming in lakes and reservoirs has increased the environmental concern because of its threat to safe water supplies [1,2]. Recently, more and more attention has been paid to the taste and odor (T&O) issues in drinking water sources as a result of many water consumer complaints. Particularly, the issues frequently occurred subsequent to algal blooming in raw water (e.g. drinking water crisis in Taihu Lake, China). Previous efforts were made to identify a variety of T&O-inducing compounds in water. Effective removal of these T&O compounds from raw water is a great challenge [3,4].

2-Isobutyl-3-methoxypyrazine (IBMP), a metabolite of actinomycetes and other bacteria in soil [5], is a representative of T&O compound in water and plants [6,7]. Early in 1975, IBMP was found in some immature plants, such as pepper, lettuce, and asparagus [8]. Also, it was identified in the grape wine from brewing process [9]. Moreover, IBMP has been widely used in chemical industries because it has a similar odor like coffee, lotus, and green bell pepper. Because of its broad usage and low odor threshold value (0.4–2.0 ng/L), IBMP often enters into our natural environment, and subsequently causes odor problems.

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*Corresponding author.

With a relatively persistent chemical property (Table 1), IBMP can be poorly removed from water by conventional drinking water treatment (i.e. coagulation, sedimentation, filtration, and disinfection). As a result, the T&O compounds are the main cause of consumer complaints in water treatment facilities [4]. Recently, adsorption-based technologies have been evaluated to remove IBMP from water. An et al. concluded that 200 mg/L granular activated carbon could remove approximately 90% of 150 μg/L IBMP [10]. In another study, 20 mg/L powdered activated carbon was reported to achieve 90% removal of 100 ng/L IBMP [11]. To date, a majority of previous studies have been focused on geosmin and 2-methylisoborneol (2-MIB), two most common T&O-induced compounds, so that IBMP has been ignored unfortunately. Therefore, there is an urgent demand to develop new and technically reliable methods for the control of IBMP that caused T&O problems in water.

The objectives of this study were as follows: (1) to evaluate the treatment feasibility of ozonation for IBMP removal in water; (2) to determine the kinetics parameters of IBMP degradation under different influencing factors, including the initial IBMP concentration, O_3 addition rate, initial pH, H_2O_2 dosage, and different water qualities; and (3) to investigate the role of ozone and hydroxyl radical oxidation in IBMP degradation. The knowledge acquired from this study would provide a scientific basis for ozonation of T&O pollutants in water.

2. Materials and methods

2.1. Chemicals

All the chemicals used were of at least analytical grade, except where noted. IBMP (≥98%), methanol (HPLC grade), chlorooctane, and n-hexane (≥95%) were purchased from Sigma–Adrich (St. Louis, MO). Hydrogen peroxide ($H_2O_2 \geq 30\%$), hydrochloric acid (HCl), sodium hydroxide (NaOH), potassium iodine (KI), and sodium thiosulfate ($Na_2S_2O_3$) were obtained from Sinopharm Chemical Reagent (Shanghai, China). All chemical solutions were prepared using deionized water from a Milli-Q water purification system.

2.2. Experimental procedure

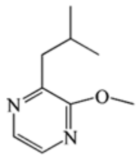
Experimental setup is shown in Fig. 1. All the ozonation tests were conducted in a 1.5 L flat-bottomed flask containing 1.0 L simulated IBMP (0.6 μM) contaminated water. Ozone gas was introduced through a tube into the reactor from an ozone generator (DHX-I multi-function O_3 purifier, Harbin Jiujiu electrochemical technical Co., China). The ozone dosage (0.25–0.75 mg/min) was controlled by adjusting the gas flow rate and aeration time. IBMP concentrations (0.15, 0.3, 0.6, 0.9 μM) were applied with initial concentration of 3.0 mM at 25℃. The pH of 3.1, 4.3, 7.2, 9.1, and 11.0 was adjusted with small value of 0.01, 0.1, or 1 M H_2SO_4 and NaOH. In order to get a better insight into the hydroxyl radical on the oxidation of IBMP, experiments were conducted with applied H_2O_2 dosage of 0.0, 0.2, and 1.0 M, respectively. In addition, raw water from drinking water treatment plant was applied to investigate the effect of different water qualities on IBMP degradation.

During ozonation process, the solution was rapidly mixed (>100 rpm) by a magnetic stirrer to ensure a complete mixing state. Any off-gas ozone was discharged into two sequential 250 mL 2% KI solution for disposal. Oxidation was initiated once ozone entered into the reactor. During the 45-min reaction time, 5 mL solution was collected at each designated sampling time, and immediately added into a bottle prefilled with 100 μL 0.1 M sodium thiosulfate solution that could quench any residual ozone in the sample.

2.3. Analytical method

The concentrations of IBMP were measured using a gas chromatography mass spectrometer (GC-MS, QP2010S, Shimadzu Co., Japan) equipped with a

Table 1
Physical and chemical characteristics and molecular structure of IBMP

Name	Molecular structure	Formula	Molecular weight	Solution (mg/L), 25 (℃)	log k_{ow}	Density (g/mL), 5 (℃)	Boiling point (℃)
IBMP		$C_9H_{14}N_2O$	166.22	1,034	2.72	0.99	83–86

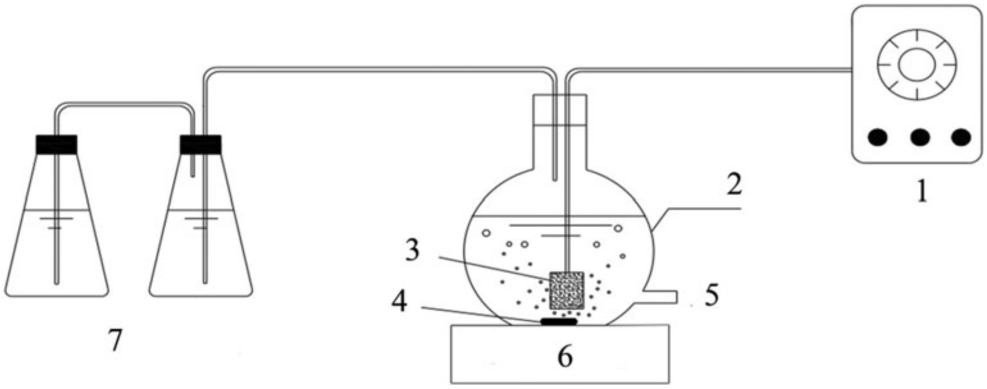


Fig. 1. Schematic diagram of experiment system.
Notes: (1) ozone generator, (2) ozonation contact reactor, (3) the aerator, (4) magnetic rotor, (5) sampling point, (6) magnetic stirrer, and (7) off-gas adsorption bottles.

RTX-5 MS capillary column (30 × 0.25 mm, 0.25 μm) and an AOC-20i autosampler. IBMP in the quenched sample was extracted with n-hexane (v(sample):v(n-hexane) = 5:1). Then, 1.0 μL extractant was injected into the GC with a splitless injection mode. The carrier gas was highly purified helium gas (99.999%) at a pressure of 90 kPa with a flow rate of 40 mL/min. The GC injection port temperature was 250℃. Injection port and ion source temperatures of the MS were 200–250℃, respectively. The temperature program was as follows: the initial oven temperature was 40℃ for 1 min, then increased to 210℃ at a rate of 80℃/min, and finally to 250℃ at a rate of 25℃/min. The final 250℃ lasted for 3 min. The characteristic mass to charge ratios of IBMP was 124–151 with a retention time of 13.5 min. The method detection limits (MDL) and relative standard deviations (RSD) of IBMP were 2.0 μg/L and 6.5%, respectively. Ozone concentrations were quantified using indigo trisulfonate colorimetric method [12]. Solution pH was measured with a pH meter (PHS-3C, Shanghai Precision, China).

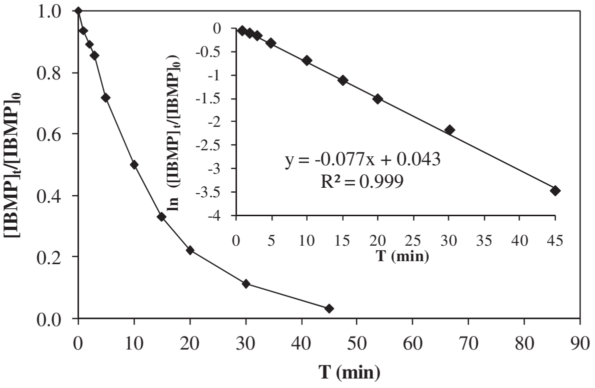


Fig. 2. Degradation of IBMP in ozonation process. Experimental conditions: $[\text{IBMP}]_0 = 0.6$ μM; ozone flow rate = 0.50 mg/min; initial pH 7.2.

3. Results and discussion

3.1. Reaction kinetics of IBMP using ozonation

Fig. 2 shows the decrease in IBMP as a function of reaction time during ozonation. The relationship between the time and $\ln([\text{IBMP}]_t/[\text{IBMP}]_0)$ was linear ($R^2 > 0.99$), where $[\text{IBMP}]_t$ and $[\text{IBMP}]_0$ represent the concentration of IBMP at different reaction time and initial time, respectively. The linear relationship indicated pseudo-first order kinetics with respect to IBMP degradation. In this study, the degradation of IBMP was highly effective. Within 10 min, the residual IBMP was only half of the initial concentration. And almost all the IBMP was degraded during 45 min of ozonation. Generally, direct ozone oxidation and indirect radical oxidation contribute to the organic micropollutant degradation in the presence of oxidants [13]. The direct ozone oxidation is characterized with selective oxidation, as Eq. (1):

$$O_3 + \text{target compound} \rightarrow \text{intermediate}\ldots \rightarrow \text{final oxidation products} \quad (1)$$

The indirect radical oxidation is involved with hydroxyl radicals ($OH^{\bullet}$) generated from ozone, and $OH^{\bullet}$ can drive a non-selective oxidation, as follows:

$$O_3 + OH^- \rightarrow OOH^{\bullet -} + O_2 \quad (2)$$

$$O_3 + OOH^{\bullet -} \rightarrow OH^{\bullet} + O_2^{\bullet -} + O_2 \quad (3)$$

$$OH^{\bullet} + \text{target compound} \rightarrow \text{intermediate}\ldots \rightarrow \text{final oxidation products} \quad (4)$$

Thus, the degradation rate of IBMP can be expressed as Eq. (5):

$$-\mathrm{d}[\mathrm{IBMP}]/\mathrm{d}t = k_{\mathrm{o}_3}[\mathrm{IBMP}][\mathrm{O}_3] + k_{\mathrm{OH}^{\cdot}}[\mathrm{IBMP}][\mathrm{OH}^{\cdot}] \tag{5}$$

where k_{o_3} and $k_{\mathrm{OH}^{\cdot}}$ represent the second-order reaction rate constants of IBMP with ozone and hydroxyl radicals, respectively; and [IBMP], $[\mathrm{O}_3]$, and $[\mathrm{OH}^{\cdot}]$ are the molar concentrations of IBMP, O_3, and $\mathrm{OH}^{\cdot}$, respectively.

Of note, the ratio of $[\mathrm{OH}^{\cdot}]$ to $[\mathrm{O}_3]$, defined as R_{C}, is constant at any specific pH.

$$R_{\mathrm{C}} = [\mathrm{OH}^{\cdot}]/[\mathrm{O}_3] \tag{6}$$

Therefore, Eq. (5) can be rewritten as Eq. (7):

$$-\mathrm{d}[\mathrm{IBMP}]/\mathrm{d}t = (k_{\mathrm{o}_3} + k_{\mathrm{OH}^{\cdot}}R_{\mathrm{C}})[\mathrm{IBMP}][\mathrm{O}_3] \tag{7}$$

Considering that k_{o_3}, $k_{\mathrm{OH}^{\cdot}}$, and R_{C} are constant, Eq. (7) can be simplified to Eq. (8) when O_3 is in large excess to IBMP.

$$-\mathrm{d}[\mathrm{IBMP}]/\mathrm{d}t = k_{\mathrm{obs}}[\mathrm{IBMP}] \tag{8}$$

Here $k_{\mathrm{obs}} = (k_{\mathrm{o}_3} + k_{\mathrm{OH}^{\cdot}}\ R_{\mathrm{C}})\ [\mathrm{O}_3]$, is the observed pseudo-first order reaction rate constant. In this study, the observed rate constant was 0.077 min^{-1}. As explained before, the reaction rate constant might vary with solution pH and the ozone dosage.

3.2. Effect of initial IBMP concentration

Fig. 3 shows the graph of $[\mathrm{IBMP}]_t/[\mathrm{IBMP}]_0$ with different IBMP concentrations as a function of reaction time. A sharp reduction in IBMP occurred within the first 10–15 min, followed by a much slow IBMP degradation. The degradation that significantly slowed down in the latter phase could be attributed to two reasons. First, ozone was greatly reduced as a result of self-decay and consumption by the target compound (i.e. IBMP). Second, the intermediate oxidation products were accumulated as the IBMP degradation proceeded, and their potential competition with IBMP for oxidizing agents in the system was gradually pronounced. For any particular initial IBMP concentration, the IBMP degradation still followed pseudo-first order kinetics well ($R^2 > 0.99$). The observed rate constants are summarized in Table 2. As the initial IBMP concentrations increased from 0.15 to 0.9 μM, the observed rate constants (k_{obs}) of IBMP decreased from 0.221 to 0.069 min^{-1}. The result can be attributed to that a higher fraction of IBMP could be removed at a low initial IBMP concentration when the amount of oxidizing agent available was constant (i.e. at a fixed ozone dosage). This finding is consistent with Dai et al. [14], who reported that the pseudo-first order rates decreased from 0.315 to 0.016 min^{-1} during ozonation as the concentration of phenoxyacetic acid rose from 100 to 2,000 mg/L.

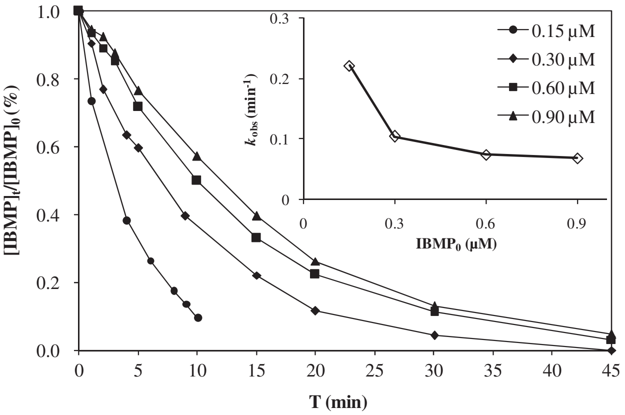


Fig. 3. IBMP degradation using ozonation under different initial IBMP concentrations. Experimental conditions: ozone flow rate = 0.50 mg/min; initial pH 7.2.

3.3. Effect of ozone dosage

Semi-logarithmic graph of $[\mathrm{IBMP}]_t/[\mathrm{IBMP}]_0$ with different ozone addition rates as a function of reaction time was constructed (Fig. 4). As presented, pseudo-first order kinetics was a good fit for the degradation of IBMP under different ozone addition rates ($R^2 > 0.99$). Furthermore, as shown in the insert image of Fig. 4, the degradation-observed rate constants exhibit a linear trend as a function of ozone dosage in this study ($k_{\mathrm{obs}} = 0.376 \times [\mathrm{O}_3] + 0.053$, $R^2 = 0.967$). A rapid degradation ($k_{\mathrm{obs}} = 0.345\ \mathrm{min}^{-1}$) was achieved at the higher ozone addition dosage (0.75 mg/min), in comparison to a slow decomposition ($k_{\mathrm{obs}} = 0.157\ \mathrm{min}^{-1}$) accomplished by 0.25 mg/min O_3 (Table 2). Similar finding was reported by Dai et al. [14], who reported that the pseudo-first order rates of ozonation of phenoxyacetic acid increased obviously with the variation in the O_3 dosage. The improvement in the reaction rate constant was because higher ozone addition rate could ensure higher $[\mathrm{O}_3]$ during the treatment, and thus increase the value of k_{obs} in Eq. (8). The results indicated that high ozone addition rate could increase the amount of oxidizing agents (ozone or hydroxyl radicals produced

Table 2
Reaction rate constants of IBMP degradation under different experimental conditions

Factor	Level	k_{obs} (min^{-1})	Half-life (h^{-1})	R^2
$[IBMP]_0$ (μM)	0.15	0.221	3.12	0.995
	0.30	0.104	6.66	0.998
	0.60	0.074	9.36	0.998
	0.90	0.069	10.02	0.998
O_3 dosage (mg/min)	0.25	0.157	4.44	0.995
	0.50	0.221	3.12	0.995
	0.75	0.345	1.98	0.990
pH	3.1	0.173	18.24	0.997
	4.3	0.293	2.34	0.993
	7.2	0.345	1.98	0.990
	9.1	0.376	1.74	0.997
	11.0	0.518	1.14	0.998
H_2O_2 (M)	0.0	0.176	3.96	0.996
	0.2	0.364	1.92	0.993
	1.0	0.054	12.84	0.993
Water qualities	Raw water	0.127	5.46	0.993
	Deionized water	0.176	3.96	0.996

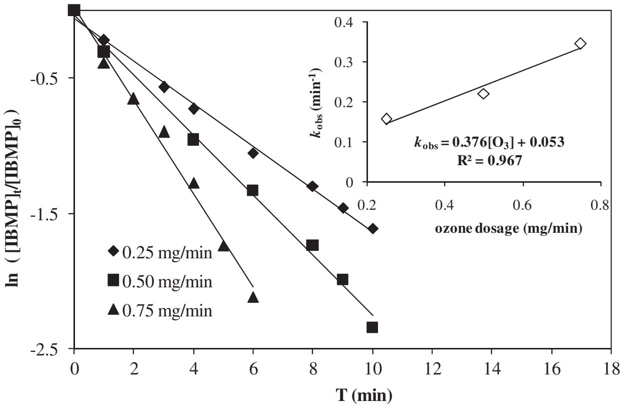


Fig. 4. IBMP degradation using ozonation under different ozone dosage. Experimental conditions: $[IBMP]_0 = 0.15$ μM; initial pH 7.2.

from ozone) available for IBMP degradation, thereby increasing the overall reaction rate.

3.4 Effect of initial pH

As aforementioned, pH is a key factor controlling the dominant oxidation mechanism. The effect of initial solution pH (3.1, 4.3, 7.2, 9.1, and 11.0) on the IBMP degradation was investigated. Semi-logarithmic graph of $[IBMP]_t/[IBMP]_0$ with different initial pH as a function of reaction time was constructed (Fig. 5). As seen, IBMP degradation at all pH values followed pseudo-first order kinetics. The IBMP degradation is pH-dependent and the degradation rates increased as the initial solution pH increased. The most appropriate pH for IBMP degradation in this study was 11.0, and the corresponding degradation rate constant was 0.518 min^{-1}. The reaction rate constants are summarized in Table 2. The rate constant significantly increased from 0.173 to 0.293 min^{-1} as the pH increased from 3.1 to 4.3, but slightly went up to 0.376 min^{-1} as the pH further increased to 9.1. When the pH continued to rise to 11.0, the rate constant again sharply went up to 0.518 min^{-1}. As explained in Section 3.1, ozone oxidation pathways include direct oxidation by ozone or radical oxidation by $OH^{\cdot}$ radical. Direct oxidation by ozone is more selective and predominates under acidic conditions, whereas radical

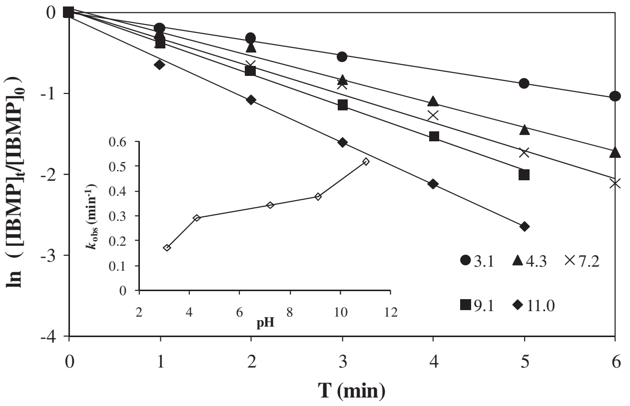


Fig. 5. IBMP degradation using ozonation under different initial solution pH. Experimental conditions: $[IBMP]_0 = 0.15$ μM; ozone flow rate = 0.75 mg/min.

oxidation by $OH^{\bullet}$ radical is less selective and predominates under alkaline conditions.

As shown in Eqs. (2)–(4), indirect radical oxidation contributes to the IBMP degradation. By applying the pseudo-steady state conditions to the free radicals, the concentration of $OH^{\bullet}$ radicals can by estimated by Eq. (9) [15]:

$$[OH^{\bullet}] = 3\ k_{OOH}[O_3][OH^-]/(k_{OH^{\bullet}}[IBMP]) \tag{9}$$

where k_{OOH} is the kinetic rate constant for the formation of free radical $OOH^{\bullet -}$ from ozone decomposition at high pH values. By substituting Eq. (9) for Eq. (5), the oxidation rate of IBMP degradation can be rewritten as Eq. (10):

$$-d[IBMP]/dt = k_{o_3}[IBMP][O_3] + \ 3\ k_{OOH}[O_3][OH^-] \tag{10}$$

Based on aforementioned observation, the ozonation of IBMP is dominated by the pseudo-first order reaction, so Eq. (10) can be restated as:

$$\begin{aligned} -d[IBMP]/dt &= k_{obs}[IBMP] \\ &= k_{o_3}[IBMP][O_3] + \ 3k_{OOH}[O_3][OH^-] \\ &= a_1[IBMP] + a_2[OH^-] \end{aligned} \tag{11}$$

where k_{obs} is the overall (pseudo-first order) rate constant, and a_1 and a_2 are coefficients equivalent to $k_{o_3}[O_3]$ and $3k_{OOH}[O_3]$. The first term in Eq. (11) stands for the pathway of direct ozone oxidation, and the second term represents the indirect free radical oxidation. As the $[O_3]$ concentration is constant, the value of both a_1 and a_2 become constants. Multiple linear least-square regression analysis of the experimental data-set of k_{obs} measured at different pHs was used to calculate the coefficients. Accordingly, the coefficients of a_1 and a_2 were calculated as 1.46×10^{-3}–$6.08 \times 10^{-4}\ s^{-1}$, respectively. From the ratio of a_1/a_2, we can also calculate the term $3k_{OOH}/k_{o_3}$ which indicates the contribution of two parallel pathways (i.e. through direct or indirect pathways in Eq. (11)) in the overall reaction. In general, the degradation of IBMP at low pH is mainly dominated by the direct ozone oxidation because the ratio of $3k_{OOH}/k_{o_3} = 0.42$ is less than 1.0. However, at high pH levels, the free radical oxidation may become dominant if the product of a_2 by $[OH^-]$ is higher than that of a_1 by [IBMP].

3.5. Effect of H_2O_2 dosage

In order to further verify the involvement of free radical reactions in the degradation of IBMP, the effect of H_2O_2 on the IBMP degradation has been investigated during the ozonation process. Semi-logarithmic graph of $[IBMP]_t/[IBMP]_0$ with different H_2O_2 dosage as a function of reaction time is shown in Fig. 6. The reaction rate constant significantly increased from 0.176 to 0.364 min^{-1} when the H_2O_2 dosage was increased from 0 to 0.2 M (Table 2). However, when the H_2O_2 dosage was further increased to 1.0 M, the rate constant greatly dropped to 0.054 min^{-1}. As known, H_2O_2 can react with O_3 to directly produce hydroxyl radicals as Eqs. (12) and (3).

$$H_2O_2 + H_2O \rightarrow OOH^{\bullet -} + H_3O^+ \tag{12}$$

$$OH^{\bullet} + \ OOH^{\bullet -} \rightarrow H_2O + O_2^{\bullet -} \tag{13}$$

The O_3/H_2O_2 system is completely different from ozone at an alkaline condition. In the latter condition, OH^- reacts with ozone to produce $OH^{\bullet}$. However, the O_3/H_2O_2 system is much less affected by solution pH. The O_3/H_2O_2 system is more efficient in generating hydroxyl radicals than ozonation under an alkalinecondition. Therefore, the IBMP degradation rate was substantially increased when H_2O_2 dosage was increased from 0 to 0.2 M. Moreover, H_2O_2 is an $OH^{\bullet}$ scavenger itself (Eq. (13)). Once the H_2O_2 concentration was in large excess, H_2O_2 could compete with IBMP for $OH^{\bullet}$ and indirectly reduce the IBMP degradation rate. When the H_2O_2 was 1.0 M, the degradation rate dropped below the rate without H_2O_2. Therefore, to achieve an optimal removal, the most favorable ratio of H_2O_2 to O_3 exists for a specific O_3/H_2O_2 treatment system [16].

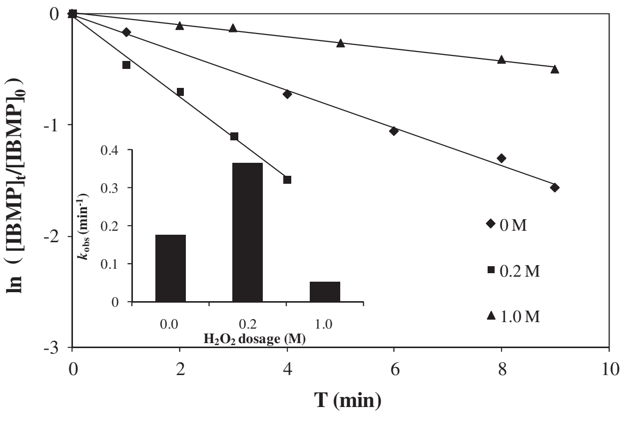


Fig. 6. IBMP degradation using ozonation under different H_2O_2 dosage. Experimental conditions: $[IBMP]_0 = 0.3\ \mu M$; ozone flow rate = 0.75 mg/min; initial pH 7.2.

Table 3
Water characteristics of raw water and deionized water used in the experiments

	pH	Turbidity (NTU)	DOC (mg/L)	UV_{254} (cm^{-1})	Ammonia (mg/L)	Conductivity (μs/cm)
Raw water	7.71	16.50	20.66	0.172	5.43	681.0
Deionized water	7.20	0.15	0.55	0.002	0.07	2.0

3.6. Effect of different water qualities

The ozonation studies were also conducted in deionized water and raw water sampled from drinking water treatment plant. The water quality parameters of deionized water and raw water are listed in Table 3. The effect of water qualities on IBMP degradation is shown in Fig. 7, and the kinetics data are summarized in Table 2. It indicated that the higher complexity of the sample interferes with IBMP degradation. A lower degradation efficiency of IBMP was observed in raw water, achieving only 72.7% degradation in 10 min, compared with 84.1% degradation in deionized water.

The coexisting anions in aqueous samples can interfere in different manner with the degradation of target compounds. It was reported that bicarbonate and carbonate ions can strongly decrease the degradation efficiency by quenching hydroxyl radicals generated from ozone [17], according to Eqs. (14) and (15):

$$OH^{\bullet} + HCO_3^- \rightarrow H_2O + CO_3^{\bullet -} \quad (14)$$

$$OH^{\bullet} + CO_3^{2-} \rightarrow OH^- + CO_3^{\bullet -} \quad (15)$$

The presence of dissolved organic carbon (DOC), like humic acids, can also inhibit the degradation through competition for hydroxyl radicals [18]. The high DOC concentration of raw water may contribute to more interference with the degradation of IBMP when compared to the results obtained in deionized water. Furthermore, the concentration of ammonia may be another factor because ozone can react with NH_3 [19], leading to an attenuation of ozone and hydroxyl radicals, thus hindering the degradation.

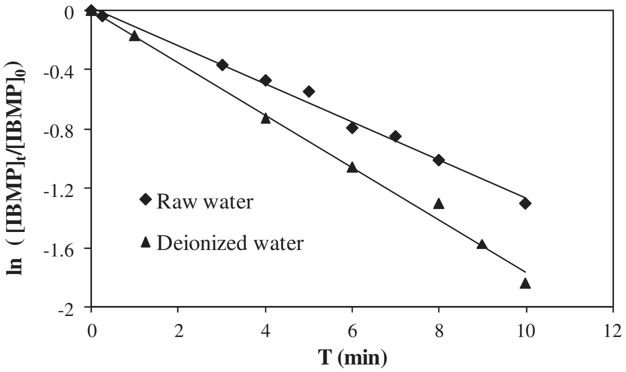


Fig. 7. IBMP degradation using ozonation in raw water and deionized water. Experimental conditions: $[IBMP]_0$ = 0.3 μM; ozone flow rate = 0.75 mg/min; pH 7.2.

4. Conclusions

Bench-scale kinetics tests were conducted to study the ozonation of IBMP at different experimental conditions. Ozonation was highly effective for IBMP degradation in water because of direct ozone oxidation and/or indirect hydroxyl radical oxidation. Affecting factors such as initial IBMP concentration, O_3 addition rate, initial pH, H_2O_2 dosage, and different water qualities were also evaluated. The degradation of IBMP fitted pseudo-first order kinetics well in the ozonation process and the observed rate constants increased as the initial IBMP concentration decreased and O_3 addition rate increased. Moreover, the IBMP degradation is pH-dependent and the degradation rates increased with the increase in the pH value. In addition, appropriate H_2O_2 dosage can significantly increase the IBMP degradation rates, whereas large excess H_2O_2 can act as an $OH^{\bullet}$ scavenger and reduce the degradation rate. This study demonstrated that ozonation is a promising treatment technology to control IBMP, as well as other similar T&O compounds in water.

Acknowledgments

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