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Evaluation of bromate formation control during drinking water disinfection in Saudi Arabia

Mohammad Saad Algamdi[a], Ibrahim Hotan Alsohaimi[b], Ayoub Abdullah Alqadami[c,*], Ahmed Ali Alshahrani[a], Nasser Fahad Alotaibi[b], Abdullah Mohammed Aldawsari[d]

[a]*King Abdulaziz City for Science and Technology, Riyadh, Saudi Arabia, email: malgamdi@kacst.edu.sa (M.S. Algamdi), ashahrni@kacst.edu.sa (A.A. Alshahrani)*
[b]*Chemistry Department, College of Science, Jouf University, Saudi Arabia, email: ehalshaimi@ju.edu.sa (I.H. Alsohaimi), nfalotaibi@ju.edu.sa (N.F. Alotaibi)*
[c]*Chemistry Department, College of Science, King Saud University, Riyadh 11451, Saudi Arabia, Tel. +966547146717 email: aaaayoub17@gmail.com (A.A. Alqadami)*
[d]*Chemistry Department, College of Arts & Science-Wadi Al-dawaser, Prince Sattam bin Abdulaziz University, Alkharj, Kingdom of Saudi Arabia, email: Abdullah.aldawsari@psau.edu.sa (A.M. Aldawsari)*

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ABSTRACT

Bromate is well known as a carcinogen, thus it is very important to improve the treatment process and reduce or eliminate the bromate formation. This study was performed for synthetic and real samples to achieve the effect of initial Br^-concentration, temperature, and contact time on bromate formation during chlorination and ozonation of water samples. Results presented that the formation of bromate was significantly affected by the initial content of bromide in water. It also displayed that the formation of bromate was maximum when the temperature is closed to 50°C. Less bromate is predicted to be formed during disinfection for lower bromide ion concentrations, shorter contact time, and lower temperature. By increasing of initial bromide ion concentrations and temperature, bromate formation has progressively been increased. The values of bromate formation during chlorination and ozonation at initial concentration of bromide ion solutions containing 100 µg/L were 6.52 and 4.9 µg/L, respectively, and were lower than the WHO guideline value for bromate in drinking water. Because of that product water has been mixed with water wells contain amounts of bromide ion, the drinking water has exceeded the international standards of bromate content. The chlorination process of water presented less formation of bromate than ozonation in the presence of bromide ion.

Keywords: Bromate; Ozonation; Chlorination; UPLC-MS/MS

1. Introduction

The disinfection of drinking water using ozone or sodium hypochlorite (NaOCl) is extensively used because it has been illustrated to be efficient for the oxidation of micro pollutants in drinking water and wastewater [1–5]. Ozone is an effective antibacterial agent and can even be used to inactivate microorganisms such as protozoa which are generally more resistant to conventional disinfectants (chlorine dioxide, chlorine). However, the required ozone exposure is quite high to inactivate these microorganisms. This may lead to excessive amounts formation of undesired disinfection by-products, in particular BrO_3^-, which is considered to be a potential human carcinogen [6–9].

Merit-ens in 1886 use the Ozone as a disinfectant for contaminated water [10]. After a few years, an ozonation pilot plant was created to test ozone's application for the decontamination of drinking water in Germany, and the results displayed that ozone was very active mate-

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*Corresponding author.

rial for the disinfection of bacteria [11]. The treatment of water with ozone gives us many advantages over conventional chlorine. Lin and Yeh revealed that the ozone was much safer to use for disinfection of water than chlorine [12].

Chlorination was massively introduced as a disinfection approach for drinking water supplies in many countries [13,14]. In Saudi Arabia, most of the water supply systems have been using sodium hypochlorite as a disinfectant, especially in desalination plants for pretreatment to provide of safe drinking water. Although hypochlorite (OCl^-) is the most commonly used disinfectant in desalination plants, it produces several decontamination by-products (DBPs) (e.g. haloacetic acids (HAAs), haloacetonitriles (HANs) and trihalomethanes (THMs), and some DBPs are controlled in many countries due to their potential risks to effect on public health. In addition, due to high levels of bromide ion concentrations in seawater, bromate (BrO_3^-) may be formed in water produced by desalination and distribution systems, especially when desalinated water is mixed with other sources of water having higher levels of organic matter. A number of studies on safety assessment of BrO_3^- were performed [15–17]. It was found to be genotoxic, carcinogenic and concerning the toxicological effects of bromate, it is needed to study the factors that affect on bromate formation in order to decrease the concentration of bromate to acceptable levels in drinking water before its intended applications [18,19]. American Water Works Association (AWWA), World Health Organization (WHO) and in Saudi Arabia have been recommended that the 10 µg/L is the a maximum limit of concentration for BrO_3^- in drinking water [20]. Also, there is a federal guideline limitation for bromate concentrations in drinking water in Canada and United States to 10 µg/L [21]. To respect these regulations while still guaranteeing disinfection, bromate control strategies have been proposed for drinking water disinfection using either ozonation or chlorination considering initial concentration of bromide, contact time, and temperature [22,23].

The overall goal of this work was, to determine the main and interactive effects of temperature, different disinfection type, initial bromide ion concentration of water, and contact time on the extent of bromate formation. The results will decide optimal conditions to meet several water treatment samples while minimizing bromate formation. Applied of (UPLC-MS/MS) method to determine of bromate concentration in water samples [9].

2. Materials and methods

2.1. Materials

All chemicals and solvent used for this work were analytical reagent grade. Sodium hydroxide (NaOH), potassium bromate ($KBrO_3$), hydrochloric acid (HCl) and potassium bro-mide (KBr) were purchased from BDH Chemicals, England. Potassium permanganate ($KMnO_4$) was obtained from Merck (Darmstadt, Germany). Milli-Q water purification system used for purified of water (Millipore Corporation, USA). BrO_3^- stock solutions (10 mg/L) was prepared in Milli-Q water and was used to prepared standard BrO_3^- samples. The standard solution of BrO_3^- concentrations was in the range between 5 and 100 µg/L. The synthetic Br^- samples of concentrations 100, 500, and 1000 µg/L were prepared for the study of converting Br^- to BrO_3^- ions during disinfection. 0.22 mm PVDF syringe filter was used for filtered all samples. All the plastic and glassware were cleaned by using soaking in dilute HNO_3 2% and were rinsed with deionized water before operation.

2.2. Preparation of sodium hypochlorite

Sodium hypochlorite (NaOCl) was prepared according to a previously reported procedure [24]. A typical procedure to prepare NaOCl was as follows: a concentrated of HCl was filled in separatory funnel and slowly added (drop wise) in buchner flask supplied with quick fit containing $KMnO_4$ powder. Then, the chlorine gas (Cl_2) was generated and passed through buchner flask suction plugged with rubber tubing to 1 L of 0.1 M of sodium hydroxide solution. Subsequently, the resulting Cl_2 gas was then reacted with NaOH to produce the hypochlorite solution. A pale yellow solution was obtained which was completed hypochlorite production. The prepared NaOCl was stored for further experiments.

2.3. Ozonation

Ozone was formed from air using Fischer ozone generator 500 M (Germany). 80 L/h is the gas flow rate of the ozone generator and 40 mg/h for ozone mass flow rate. The output gas from the generator was thoroughly mixed with water containing bromide in the venturi injector and continues to flow during a time ranged between 1 and 60 min at varied bromide ions initial concentrations (Fig. 1).

2.4. Analysis

BrO_3^- separation and analysis was achieved on UPLC system, equipped with a quaternary pump system (Waters1, USA). An Acquity1 BEH C_{18} column of dimension, 50 mm × 2.1 mm i.d., 1.7 µm particle size (Waters, USA) was applied. The mobile phase containing 99.99% D.I water with 0.1% of the formic acid was used to achieve the best probable separation of the BrO_3^-. The injection volume of the sample was 5 µL. The detection of BrO_3^- was performed on a triple quadrupole mass spectrometer (MS) equipped with electro spray ionization (ESI) source coupled with the UPLC system. The instrument was run

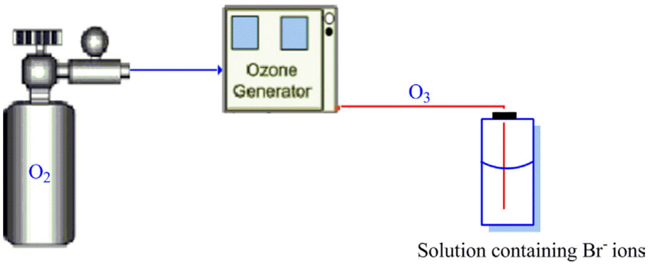

Fig. 1. Schematic system of the ozonation process.

in the negative mode. The data was carried out in multiple reactions monitoring (MRM) mode via the protonated molecular ion of each compound as a precursor ion. The optimized working conditions were as follows: capillary voltage, 3.0 kV; cone voltage, 30 V; desolvation temperature, 350°C; source temperature, 120°C; cone gas flow rate, 60 L/h; desolvation gas flow rate, 600 L/h. Nitrogen (99.99% purity, model NM30LA, UK) and Argon (99.99% purity, Saudi Arabia) were used as cone and collision gases, respectively. The primary vacuum for the MS with rotary pump was provided, Oerlikon model SOGEVAC SV40 BI (Paris, France). The MRM conditions with the MS system for the analysis of BrO_3^- have been presented in Table 1. Data acquisition was performed by Mass Lynx V 4.1 software.

2.5. Experimental procedure

In the case of ozonation disinfection, the experimental work had been applied on the synthetic water using D.I water with initial Br^- concentrations (100, 500, and 1000 µg/L). The batch experiments was then switched on for the required time (1, 5, 10, 15, 20, 30, 45 and 60 min) while keeping the water flow rate of ozone constant. Whole samples were collected and then filtered through 0.22 µm PVDF filter prior to injection. On the other hand, the experiment of the disinfection with hypochlorite, the same conditions were applied with the injection of 500 µg/L of chlorine to keep sufficient amount and control the chlorine in drinking water lower than the WHO guideline value. The different parameters were well studied such as initial Br^- concentrations (100, 500, and 1000 µg/L), temperature ranged between 303 and 323 K, and contact time ranged between 1 and 60 min. The samples also were collected and filtered through 0.22 µm PVDF filter prior to injection. Experiments were also achieved in the real water samples (metropolitan and bottled water) spiked with 100 µg/L Br^- to test the significance of BrO_3^- formation through disinfection under a more realistic condition.

3. Results and discussion

3.1. Effect of disinfection type and initial bromide concentration

Chlorination and ozonation of water samples were applied under a given set conditions to assess the effectiveness of disinfection species to form the lowest level of BrO_3^-. The BrO_3^- formation were conducted either by ozone or sodium hypochlorite. The results confirmed that the disinfection of water using ozonation process was enhanced the formation of BrO_3^- more than hypochlorite. As can be seen, the BrO_3^- formation favors high concentration of initial Br^- whereas the reduction of BrO_3^- formation was achieved during chlorination or ozonation with reducing the initial bromide concentration (Fig. 2).

The content of bromide ion levels in the solution and natural waters is one of the most important factors affecting BrO_3^- formation. When the initial Br^- concentration in the solution is higher than 100 µg/L, the concentration of BrO_3^- will be formed during ozonation and chlorination and may exceed maximum pollutant level (10 µg/L) [25]. At constant disinfection time (60 min), the experiments were carried out under conditions of gas flow rate of ozone mass flow rate is fixed at 40 mg/h, initial Br^- concentration ranged from 100 to 1000 µg/L, and hypochlorite concentration was fixed at 0.5 mg/L for the chlorination process. The results are presented in Fig. 2. Generally, BrO_3^- concentration was increased with increase in initial Br^- concentration through disinfection by both chlorination and ozonation process. For chlorination process, the concentration of BrO_3^- increased from 6.51 to 20.12 µg/L when the initial Br^- concentration varied from 100 to 1000 µg/L whereas during ozonation process, the concentration of bromate increased from 4.9 to 19.40 µg/L when the initial concentration of Br^- various from 100 to 1000 µg/L.

3.2. Effect of contact time on BrO_3^- formation

The influence of contact time on BrO_3^- formation was examined at the time intervals of 0, 5, 10, 20, 30, 45, 60 and 120 min with an initial concentration of Br^- (100, 500 and 1000 µg/L) at 25 ± 0.5°C. Also, the effect of contact time for

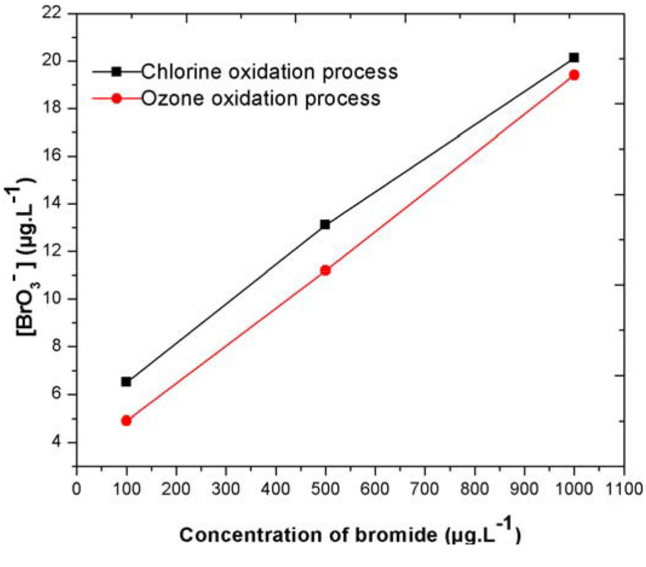

Fig. 2. Effect of initial Br- concentration on bromate formation during chlorination and ozonation process.

Table 1
MRM conditions used with the triple quadrupole MS instrument[a]

Analyte	Precursor ion (m/z)	Quantitation		Confirmation	
		Product ion (m/z)	Collision energy (eV)	Product ion (m/z)	Collision energy (eV)
BrO_3^-	128.85	112.85	24	116	28

real sample was studied after the addition of 100 μg/L bromide for metropolitan and bottled water in order to assess the effect of co-existing matrix on BrO_3^- formation. Fig. 3 displays the formation of BrO_3^- during chlorination increased with increasing contact time at a varied initial concentrations of bromide ion (100, 500 and 1000 μg/L) while the formation of BrO_3^- remained quite constant after 20 min and 45 min for real samples and the solution containing 100 μg/L of Br^-, respectively. The concentration of BrO_3^- was 6.52 μg/L in a solution containing 100 μg/L of Br^-. This value is lower than the WHO guideline value for bromate in drinking water, while the values of BrO_3^- formation at initial concentration of Br^- solutions more than 100 μg/L were more than the WHO guideline value for BrO_3^- in drinking water.

Fig. 4 demonstrates the influence of contact time on BrO_3^- formation through ozonation. It can be observed that the BrO_3^- formation increased with increasing contact time at a varied initial concentrations of bromide ion (100, 500 and 1000 μg/L) while the formation of BrO_3^- remained quite constant after 10 min for the synthetic samples containing a varied initial concentrations of bromide ion. In comparison between ozonation and chlorination on bromate formation, the results revealed that ozonation oxidized bromide ion more than chlorination and converted to bromate. While the chlorination of water samples containing bromide ion was needed long time to convert bromide ion to bromate. For the real samples, the bromate concentrations were detected in both metropolitan and bottled water samples before disinfectant addition and found to be 12 and 15 μg/L, respectively. The BrO_3^- formation increased with increasing contact time when water disinfected using chlorine and reached a maximum of 20 and 25 μg/L at 20 min for bottled water and metropolitan water, respectively (Fig. 3). While the formation of BrO_3^- increased with increasing contact time when the real water samples disinfected using ozone and reached a maximum of 110 and 130 μg/L at 30 min for metropolitan and bottled water, respectively. Then, the formation of BrO_3^- in metropolitan sample again increased until it reached 160 μg/L at 60 min (Fig. 4). The real samples reach the maximum conversion of bromide to bromate due to the real samples have a lot of matrixes that effect on this reaction. Minimum bromate formation occurred with chlorination and the maximum bromate formation occurred with ozonation because ozone is one of the most effective oxidants it is capable of oxidizing many organic and inorganic compounds better than conventional disinfectants (chlorine). These values were higher than the WHO guideline value for BrO_3^- in drinking water samples.

3.3. Effect of temperature on bromate formation

The influence of temperature on BrO_3^- formation was achieved for initial Br^- concentration ranged between 100 and 1000 μg/L at a varied temperatures (30, 40 50°C) with hypochlorite dosage at 500 μg/L. Fig. 5 displays the influence of temperature on BrO_3^- formation during chlorination process. The results revealed that the formation of BrO_3^- increased with the increase of temperature at different concentrations of bromide ion. The concentration of BrO_3^- formation was increased from 6.52 to 11 μg/L when the temperature increased from 30 to 40°C in a solution containing 100 μg/L of Br^-. The increase of BrO_3^- formation value at initial concentration of Br^- solution 100 μg/L was more than the WHO guideline value for BrO_3^- in drinking water. These results showed an increase of BrO_3^- formation as temperature increase. The increase of BrO_3^- formation was ascribed to the increase in OBr^- that played an important role in BrO_3^- formation. This is agreed with results found in the literature reported [26].

3.4. Determination of BrO_3^- formation in water samples

The analysis of BrO_3^- in synthetic and real water samples was carried out using UPLC-MS/MS method [9,27,28].

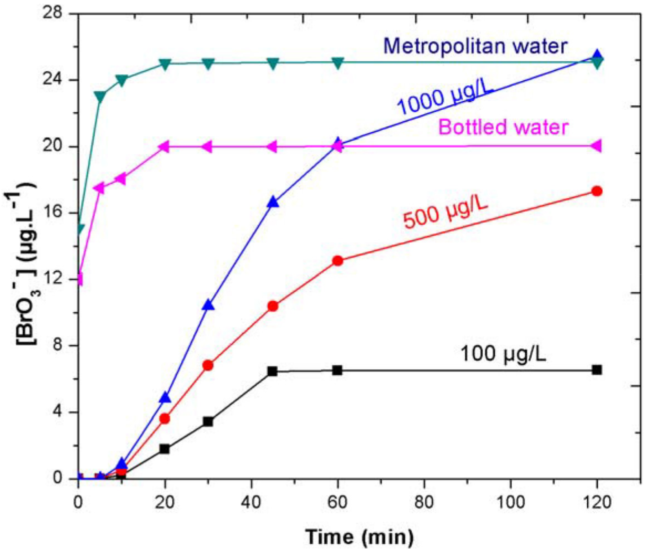

Fig. 3. The formation of bromate with time during chlorination for samples containing 100, 500, and 1000 μg·l^{-1} of Br^-, and metropolitan and bottled water.

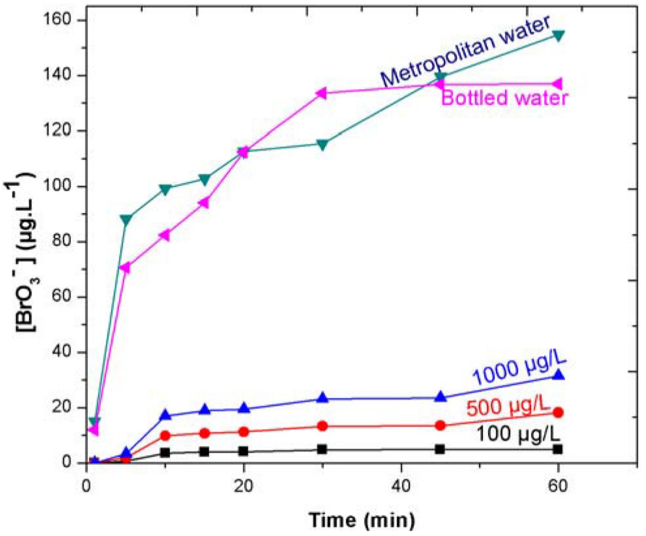

Fig. 4. The formation of bromate with time during ozonation for samples containing 100, 500, and 1000 μg·l^{-1} of Br^-, and metropolitan and bottled water.

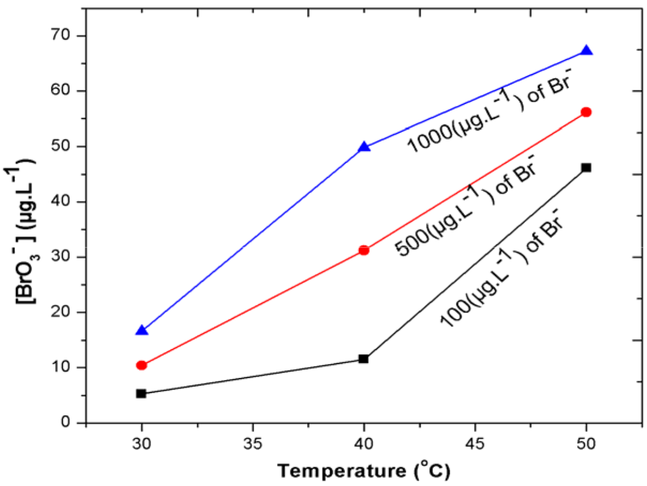

Fig. 5. The formation of bromate during chlorination at various temperatures. (Co: Br^- 100, 500 and 1000 µg/L).

Analytical testing of the BrO_3^- in the different samples in a laboratory shown that the concentration of BrO_3^- was in the range of 0–160 µg/L. Fig. 6 presented the chromatograms of bottled water sample before and after ozonation process. As can be observed from the figure, it is clearly that the formation of BrO_3^- increased after treatment with ozone. The chromatograms did not show any interference peak at the retention time of the target analyte. This showed an influence of the disinfection type of water samples on BrO_3^- formation under the experimental conditions.

2.5. Bromate control

Previous results have shown that there is a relationship between the initial concentration of Br^- and BrO_3^- formation after disinfection process. It was observed that the low initial concentrations of Br^- needed long time to be converted to BrO_3^-. The resulting BrO_3^- was a slight increase progressively and well under the allowed limits. While the samples containing high initial Br^- concentrations have been exceeded the permissible limits of the converted BrO_3^- in water. For bottled water and metropolitan water samples, it has been estimated BrO_3^- before the sterilization process and was a slightly above the allowed limits. Despite the addition of the amount of Br^- and exposing all of the substances themselves, it's noted that the BrO_3^- formation during chlorination was increased 10 µg/L to the content concentration levels whereas the formation of bromate strongly increased during ozonation more than 100 µg/L to the content concentration levels. From the experimental results, it is better that the Br^- concentration in the produced water no more than 0.1 mg·L^{-1} before disinfection under low temperature to reduce BrO_3^- formation in drinking water and to ensure that the BrO_3^- is contained within the permissible limits. Also, chlorination disinfection presented less oxidized of bromide ion to bromate than ozonation. Thus, a safe disinfectant is chlorination that will keep water content level of bromate in the permissible limits.

4. Conclusion

In summary, BrO_3^- formation through disinfection of synthetic and real water samples was performed. The disinfection of water using hypochlorite (OCl^-) presented less formation of BrO_3^- than ozonation in the presence of Br^- ion. Results presented that the formation of BrO_3^- was gradually increased with increasing temperature. It also presented that BrO_3^- formation is maximum when ozonated water containing high initial bromide ion concentration. The results revealed that BrO_3^- formation during disinfection was affected by initial Br^- concentration, disinfection type, temperature and contact time. BrO_3^- concentration increased as the operating conditions moved towards the higher level. BrO_3^- formation increased more than 100% (6–20 µg/L) with the initial Br^- concentration increased (100, 500, 1000 µg/L) at constant disinfection time (25 min). The influence of the spiked Br^- to the bottled and metropolitan water followed by ozonation or chlorination was confirmed that the Br^- converted to BrO_3^-. The disinfection process can be controlled on basis of the parameters water temperature, type of disinfection, initial bromide ion concentration and contact time. The formation of bromate then can be adjusted to the acceptable limits. The conditions and parameters were applied to the optimise the use of disinfectant either ozonation or chlorination of drinking water restricted by a bromate target value of 10 µg/L and recommended to apply in all desalination plants and bottled water factories in Saudi Arabia.

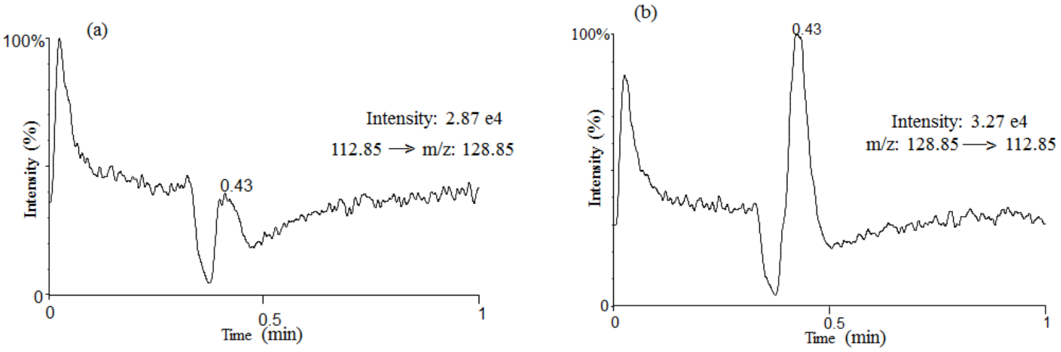

Fig. 6. UPLC–MS/MS chromatograms of BrO_3^- concentration in commercial bottled water before (a), and after ozonation (b).

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