



Selection and applicability of quenching agents for the analysis of polar iodinated disinfection byproducts



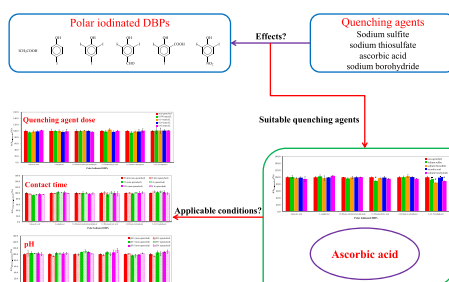
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HIGHLIGHTS

- The quenching agent suitable for the analysis of polar iodinated DBPs was selected.
- The applicable conditions of the suitable quenching agent were investigated.
- The selected quenching agent was widely applicable in various conditions.

GRAPHICAL ABSTRACT



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ABSTRACT

Iodide is widely present in drinking water sources as well as wastewater effluents. Chlorination and chloramination are the most commonly used disinfection methods. During chlorination or chloramination of drinking water/wastewater effluents, iodide may be oxidized to hypiodous acid, which may further react with organic matter to form iodinated disinfection byproducts (DBPs). Recently, several new polar iodinated DBPs have been identified in drinking water as well as chlorinated wastewater effluents, and they have drawn increasing concerns due to their high toxicity. In DBPs studies, the selection of an appropriate quenching agent is critical to prevent further formation or any decomposition of DBPs during the holding time between sample collection and analysis. A previous study reported the applicability of different quenching agents for the analysis of various categories of chlorinated and brominated DBPs. But the applicability of quenching agents for the analysis of polar iodinated DBPs has not been reported. In this study, four different quenching agents (sodium sulfite, sodium thiosulfate, ascorbic acid and sodium borohydride) were tested for their suitability for the analysis of polar iodinated DBPs, and ascorbic acid was selected as the suitable quenching agent. Furthermore, it was found that ascorbic acid was applicable for the analysis of polar iodinated DBPs under the quenching agent doses of 0–0.42 mmol/L (stoichiometric amounts equivalent to 0–30 mg/L Cl₂), contact times within 24 h, and pHs in the range of 6–8. Therefore, ascorbic acid was a widely applicable quenching agent for the analysis of polar iodinated DBPs under various conditions.

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1. Introduction

Iodide is widely present in drinking water sources as well as wastewater effluents (Schwehr and Santschi, 2003; Gilfedder et al., 2008, 2009; Gong and Zhang, 2013). Disinfection is a necessary

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treatment process in drinking water treatment. Nowadays, wastewater effluents are also extensively subjected to disinfection before discharge to inactivate the microorganisms therein. Chlorination and chloramination are the most cost-effective methods for water disinfection. During disinfection of drinking water or wastewater effluents, chlorine or chloramines can oxidize iodide ions to hypiodous acid, which may further react with organic matter to form iodinated disinfection byproducts (DBPs). Previous studies have reported the formation of iodinated DBPs in drinking water and chlorinated wastewater effluents (Plewa et al., 2004; Krasner et al., 2006; Richardson et al., 2008; Pan et al., 2016; Yang and Zhang, 2013, 2014; Gong and Zhang, 2015). Iodinated DBPs may pose adverse effects to humans as well as organisms in the receiving water body of wastewater effluents (Plewa et al., 2004; Cemeli et al., 2006; Richardson et al., 2007, 2008; Yang and Zhang, 2013; Liu and Zhang, 2014). It has been demonstrated that iodinated DBPs are generally several to hundreds of times more toxic than their brominated and chlorinated analogues (Plewa et al., 2008; Cemeli et al., 2006; Richardson et al., 2007, 2008; Yang and Zhang, 2013; Liu and Zhang, 2014), and thus iodinated DBPs have been drawing increasing concerns recently.

Previously, gas chromatography–electron capture detection and gas chromatography–mass spectrometry have been used for detecting and identifying iodinated DBPs in drinking water (Bichsel and von Gunten, 2000; Plewa et al., 2004; Krasner et al., 2006; Hua and Reckhow, 2007; Richardson et al., 2008; Duirk et al., 2011; Jones et al., 2012). However, due to the limitation of these techniques, only a few iodinated DBPs have been detected and identified in drinking water, including mainly iodinated trihalomethanes and iodinated acids (Bichsel and von Gunten, 2000; Plewa et al., 2004; Richardson et al., 2008). Recently, a novel precursor ion scan (PIS) method has been developed for the fast selective detection of polar iodinated DBPs using an electrospray ionization–triple quadrupole mass spectrometer (ESI–tqMS) (Ding and Zhang, 2009). Further, by coupling it with ultra performance liquid chromatography (UPLC) for pre-separation, the ESI–tqMS can be used for identifying unknown polar iodinated DBPs. Previous studies have adopted this method to detect and identify polar iodinated DBPs in simulated drinking water, tap water and chlorinated wastewater effluents (Ding and Zhang, 2009; Pan et al., 2016; Yang and Zhang, 2013; Gong and Zhang, 2015). Up to now, the identified polar iodinated DBPs mainly include iodinated aliphatic acids, iodinated phenols, iodinated nitrophenols, iodinated hydroxybenzaldehydes and iodinated salicylic acids (Plewa et al., 2004; Yang and Zhang, 2013; Pan et al., 2016). Previous studies have demonstrated that these polar iodinated DBPs showed relatively high toxicity among all DBP categories (Plewa et al., 2004; Yang and Zhang, 2013; Liu and Zhang, 2014) and thus the study of these newly identified polar iodinated DBPs is of great significance.

Proper sample pretreatment procedures are necessary to obtain accurate and reliable concentration data of these polar iodinated DBPs. In particular, the use of an appropriate quenching agent is critical to prevent further formation or any decomposition of DBPs during the holding time between sample collection and analysis. An ideal quenching agent should have the following properties: ① rapid and complete reaction with the residual disinfectants; ② chemical inertness towards the analyzed DBPs; ③ non-detectable signal for the quenching agent itself and its reaction products during sample analysis. The commonly used quenching agents include sodium sulfite, sodium thiosulfate, sodium arsenite, ascorbic acid, sodium borohydride, and ammonium chloride. The quenching agents undergo redox reactions with disinfectants except for ammonium chloride. Ammonium chloride is a quenching agent specific for free chlorine since it converts free chlorine to monochloramine, which generally does not generate chlorinated

and brominated trihalomethanes and haloacetic acids, which are the regulated DBPs (Malliarou et al., 2005; Munch and Hautman, 1995). A previous study investigated the applicability of five quenching agents, namely sodium sulfite, sodium arsenite, sodium borohydride, ascorbic acid and ammonium chloride, for the analysis of seven different categories of chlorinated and brominated DBPs in drinking water, and the results indicated that ascorbic acid could be used for the analysis of most organic chlorinated and brominated DBPs while sodium sulfite was suitable for the analysis of inorganic DBPs (Kristiana et al., 2014). However, the applicability of these quenching agents for the analysis of polar iodinated DBPs has not been studied.

The purposes of this study were thus to select the suitable quenching agents for the analysis of polar iodinated DBPs, and further to investigate the applicable conditions (quenching agent doses, contact times, and pHs) of the specific suitable quenching agents.

2. Materials and methods

2.1. Chemicals and reagents

Iodoacetic acid ($\geq 98\%$), 4-iodophenol (99%), 2,4,6-triiodophenol (97%), sodium sulfite ($\geq 98\%$), sodium thiosulfate (1 mol/L), L-ascorbic acid (reagent grade), and sodium borohydride ($\geq 99\%$) were purchased from Sigma Aldrich. 3,5-Diiodosalicylic acid (97%) and 3,5-diiodo-4-hydroxybenzaldehyde ($\geq 98\%$) were purchased from Alfa Aesar. 2,6-Diiodo-4-nitrophenol ($\geq 97\%$) was purchased from CHEMPARTNER (Chengdu, China). Methanol (HPLC grade) and acetonitrile (HPLC grade) were purchased from LiChrosolv. Methyl tert-butyl ether (99.9%) was purchased from Tedia. Ultrapure water (18.2 M Ω cm) was supplied by a Simplicity UV ultrapure water system (Merck Millipore).

2.2. Preparation of solutions

The stock solutions (100 mg/L) of iodoacetic acid, 4-iodophenol, 3,5-diiodo-4-hydroxybenzaldehyde, 3,5-diiodosalicylic acid, 2,6-diiodo-4-nitrophenol, and 2,4,6-triiodophenol were prepared by dissolving each organic compound in methanol. They were stored in amber glass bottles at 4 °C and newly prepared every week. The working solutions of these organic compounds with lower concentrations were freshly prepared daily by diluting the stock solutions with ultrapure water.

A sodium sulfite solution (0.1 mol/L), a sodium thiosulfate solution (0.1 mol/L), an ascorbic acid solution (0.1 mol/L), and a sodium borohydride solution (0.1 mol/L) were prepared and stored in amber glass bottles at 4 °C. They were newly prepared every week.

2.3. Sample preparation

Four series (Series A, B, C and D) of samples were prepared. In Series A, the effects of different quenching agents on the polar iodinated DBPs were studied. In Series B, the effects of ascorbic acid on the polar iodinated DBPs under different quenching agent doses were investigated. In Series C, the effects of ascorbic acid on the polar iodinated DBPs under different contact times were tested. In Series D, the effects of ascorbic acid on the polar iodinated DBPs under different pHs were studied.

For Series A, five aliquots (100 mL) of a DBP mixture solution containing 20 $\mu\text{g/L}$ of iodoacetic acid, 5 $\mu\text{g/L}$ of 4-iodophenol, 3,5-diiodo-4-hydroxybenzaldehyde, 3,5-diiodosalicylic acid, 2,6-diiodo-4-nitrophenol, and 2,4,6-triiodophenol were prepared. For aliquot 1, no quenching agent was added. For aliquots 2–5, sodium sulfite, sodium thiosulfate, ascorbic acid, and sodium borohydride

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