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Q6 **Formation and control of nitrogenous DBPs from Western**
 2 **Australian source waters: Investigating the impacts of high**
 3 **nitrogen and bromide concentrations**

Q8 Q7 **Ina Kristiana^{1,*}, Deborah Liew¹, Rita K. Henderson², Cynthia A. Joll¹, Kathryn L. Linge¹**

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ARTICLE INFO

Article history:

11 Received 28 November 2016

12 Revised 20 June 2017

13 Accepted 20 June 2017

14 Available online xxxx

Keywords:

33 Haloacetonitriles

34 Haloacetamides

35 Halonitromethanes

36 N-nitrosamines

37 Chlorination

38 Chloramination

ABSTRACT

We studied the formation of four nitrogenous DBPs (N-DBPs) classes (haloacetonitriles, 16
 halonitromethanes, haloacetamides, and N-nitrosamines), as well as trihalomethanes 17
 and total organic halogen (TOX), after chlorination or chloramination of source waters. We 18
 also evaluated the relative and additive toxicity of N-DBPs and water treatment options 19
 for minimisation of N-DBPs. The formation of halonitromethanes, haloacetamides, and 20
 N-nitrosamines was higher after chloramination and positively correlated with dissolved 21
 organic nitrogen or total nitrogen. N-DBPs were major contributors to the toxicity of both 22
 chlorinated and chloraminated waters. The strong correlation between bromide concentra- 23
 tion and the overall calculated DBP additive toxicity for both chlorinated and chloraminated 24
 source waters demonstrated that formation of brominated haloacetonitriles was the main 25
 contributor to toxicity. Ozone-biological activated carbon treatment was not effective in 26
 removing N-DBP precursors. The occurrence and formation of N-DBPs should be investigated 27
 on a case-by-case basis, especially where advanced water treatment processes are being 28
 considered to minimise their formation in drinking waters, and where chloramination is used 29
 for final disinfection. 30

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Introduction

46 Over the past 15 years, the focus of investigations on
 47 disinfection by-products (DBPs) in drinking water has gradu-
 48 ally shifted from regulated DBPs, such as the trihalomethanes
 49 (THMs) and haloacetic acids (HAAs), to other emerging DBPs
 50 that are suspected to be more relevant from a human health
 51 perspective. Nitrogen-containing DBPs (N-DBPs) are among
 52 these emerging DBPs, since their cytotoxicity and genotoxicity
 53 in mammalian cells have been found to be much higher
 54 than those of THMs and HAAs (Richardson, 2006; Plewa et al.,
 55 2004; Moudgal et al., 2000). To-date, most epidemiological

studies have not included N-DBPs in their assessment 57
 of human health effects (e.g. Botton et al., 2015; Salas et al., 58
 2014; Kogevinas et al., 2010; Nieuwenhuijsen et al., 2009; 59
 Villanueva et al., 2004). One limited study found no associa- 60
 tion between exposure to haloacetonitriles (HANs) during 61
 pregnancy, and small birthweight (Ileka-Priouzeau et al., 62
 2015). While it is not certain that the *in vitro* effects measured 63
 for N-DBPs will translate to human health outcomes, further 64
 investigation of N-DBPs has been identified as a research 65
 priority by numerous researchers and the US EPA (Krasner Q10
 et al., 2006; Woo et al., 2002; Richardson et al., 2007; Bull et al., 67
 2006). 68

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N-DBPs are generally found in drinking waters at significantly lower concentrations than THMs and HAAs. Concentrations of haloacetonitriles (HANs), halonitromethanes (HNMs), and haloacetamides (HAMs) are typically reported up to 10–15 µg/L (Krasner et al., 2006; Goslan et al., 2009; Bond et al., 2015; Liew et al., 2016), with HANs often the most frequently detected class (Krasner et al., 2006; Liew et al., 2016). N-nitrosodimethylamine (NDMA), the most frequently detected N-nitrosamine, is typically detected at concentrations less than 10 ng/L in drinking waters. The concentrations measured are generally lower than published guideline and regulation values (Boyd et al., 2012; Liew et al., 2012a).

The use of chloramine as a disinfectant has been associated with elevated concentrations of N-DBPs relative to chlorination (e.g. Kristiana et al., 2014; Bond et al., 2011; Lee et al., 2007), with chloramine itself reported to be an inorganic precursor to N-DBPs (Yang et al., 2010). Nitrogen-enriched fractions of organic matter have also been found to have a higher propensity to form N-DBPs (Bond et al., 2012; Dotson et al., 2009). Algal organic matter is a known major source of dissolved organic nitrogen (DON) in the natural environment, and waters containing higher concentrations of algal organic matter have been reported to form higher concentrations of N-DBPs (Bond et al., 2012; Shah and Mitch, 2012). Roccaro et al. (2011) have further specified that the formation of N-DBPs is associated with the chlorination of nitrogen-containing activated aromatic groups in NOM, such as amino acids and N-containing heterocyclic aromatic rings.

Thus far, there is no indication that a single treatment method exists for the management of all N-DBPs, with different treatments reported to be effective for removal of precursors of the different N-DBP classes (Liew et al., 2012a). In contrast to THM precursors, N-DBP precursors tend to be of low molecular weight and low electrostatic charge (Bond et al., 2012), and include free amino acids, as well as the colloidal and hydrophilic fractions of NOM (Mitch et al., 2009). While conventional water treatment has been reported to be moderately effective in removing N-DBP precursors (Bond et al., 2011), treatments that remove lower molecular weight NOM more efficiently, such as activated carbon and riverbank filtration, can sometimes remove higher percentages of HAN and HNM precursors (Liew et al., 2012a).

In this study we investigated the formation four N-DBP classes (HANs, HNMs, HAMs, and N-nitrosamines) after chlorination or chloramination of source waters that are rich in N-DBP precursors. DBP formation potential was studied with respect to water quality and organic matter characteristics, providing some insights into the reactivity of the complex mixture of organic matter contained in natural waters and the resulting N-DBP formation. In order to quantify the contribution of N-DBPs to the overall formation of DBPs, the formation of THMs and total organic halogen (TOX) were also measured. Since most source waters in Western Australia contain high concentrations of bromide, we also evaluated the relative and additive toxicity of N-DBPs, in particular brominated N-DBPs. Finally, the effect of conventional water treatment (coagulation–flocculation–clarification–filtration) and ozone–biological activated carbon (O₃ + BAC) treatment on N-DBP formation was investigated using a groundwater source known to contain high concentrations of dissolved organic carbon (DOC), bromide, and

ammonia, and which had previously shown high concentrations of HANs in the treated (disinfected) water (Liew et al., 2016).

1. Materials and methods

1.1. Chemicals

All chemicals and standards used in this study were of analytical grade purity, while organic solvents were of HPLC grade purity. Specific details on these chemicals are provided in the Supporting Information S11 (Table S1).

1.2. Study design and sample collection

Four surface waters (HD — reservoir, RV — reservoir, GR — lake, and HE — reservoir) and one groundwater (JD) from Western Australia (WA) were selected for this study. The surface waters were from different climatic regions (HD: North West of WA, RV: South East of WA, GR: South East of WA, HE: East of Perth Metropolitan Area), and all have anecdotally experienced periodic blue-green algal blooms, in particular HD surface water (Antenucci et al., 2016), and thus represent source waters that are likely to be rich in N-DBP precursors. HD, RV, and GR surface waters were each sampled once during the winter season, while HE surface water was sampled in the spring. Sample collection times were determined by availability of operators and accessibility to each site at the commencement of the study. Grab samples were collected from the inlet to the respective treatment plants at these locations. All samples were collected in 4 L amber glass bottles, kept cool (in an ice box) and transported back to the laboratory, where they were refrigerated at 4°C until analysis of water quality parameters, which was typically within 24 hr. Samples were used for formation potential experiments within 1 week of collection.

The groundwater JD (south of Perth metropolitan area) was an ideal source water for meeting two objectives of our study. As well as containing high concentrations of DOC and total N (mostly due to high ammonia concentrations), it has a very high concentration of bromide, allowing for evaluation of the potential toxicity of brominated N-DBPs. This groundwater was treated at a treatment plant where a pilot plant was in operation, which provided an opportunity to evaluate treatment options for minimising the formation of N-DBPs, thus meeting another study objective. At the treatment plant, groundwater JD undergoes pre-chlorination, coagulation, flocculation, clarification and dual media gravity filtration before final disinfection and distribution to customers. Initially, the pilot plant was assembled to evaluate whether the addition of O₃ + BAC treatment improved treated water quality, particularly through improved removal of organic matter, reduced chlorine demand, increased chlorine residual stability, and reduced formation of THMs. For this study, the pilot plant provided an opportunity to evaluate the impact of O₃ + BAC treatment on the removal of N-DBP precursors. At the pilot plant, three treatment trains were operational with three different types of biologically activated carbon (JD-O1:

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