



# Mass flows and removal of eight bisphenol analogs, bisphenol A diglycidyl ether and its derivatives in two wastewater treatment plants in New York State, USA

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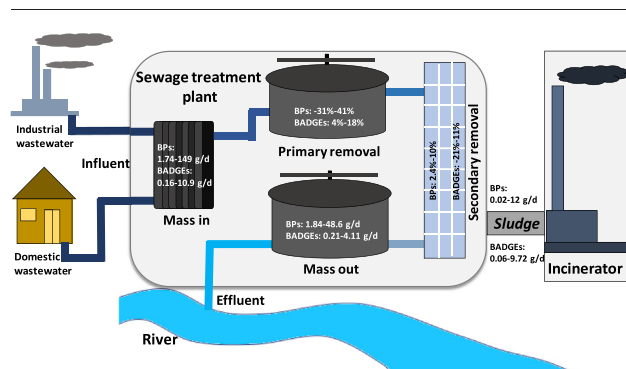
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## HIGHLIGHTS

- Mass flows of BPA, BPS, BPF and BADGEs were studied in WWTPs.
- Bisphenols were found in influents at concentrations of hundreds to thousands of ng/L.
- Removal rates of bisphenols were <52%.
- Mass loadings of bisphenols were at several tens to hundreds of mg/d/1000 people.
- Formation of bisphenols from the degradation of microplastics in WWTPs is suggested.

## GRAPHICAL ABSTRACT



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## ABSTRACT

Despite high production and usage of bisphenols including bisphenol A (BPA) as well as bisphenol A diglycidyl ether and its derivatives (BADGEs), little is known about the occurrence and fate of these substances in wastewater treatment plants (WWTPs) in the U.S. In this study, we investigated the occurrence, removal, mass flows, and fate of eight bisphenol analogues and six BADGEs based on the concentrations measured in influent, primary effluent, final effluent, and sludge from two WWTPs (WWTP<sub>A</sub> and WWTP<sub>B</sub>) in the Albany area of New York State, USA. BPA, bisphenol F, bisphenol S, and BADGE·2H<sub>2</sub>O were the predominant compounds found in influents of both WWTPs, at respective geometric mean (GM) concentrations of 90.0, 90.2, 31.2, and 6.48 ng/L in WWTP<sub>A</sub>, and 53.3, <MLOQ, 27.6, 2.25 ng/L in WWTP<sub>B</sub>. Incomplete removal of these compounds was observed in both WWTPs with the highest removal rate (52%) was found for BPA after the secondary treatment in WWTP<sub>A</sub>. The fraction of BPA sorbed to suspended particulate matter (SPM) was 6.83%. Mean daily mass loadings of total bisphenols and BADGEs ranged from 9.2 [∑(BADGEs) in WWTP<sub>B</sub>] to 226 mg/d/1000 inhabitants [∑(BPs) in WWTP<sub>A</sub>]. The environmental emission rates of total bisphenols and BADGEs through effluent discharges from WWTPs ranged from 13.7 [∑(BADGEs) in WWTP<sub>B</sub>] to 246 mg/d/1000 inhabitants [∑(BPs) in WWTP<sub>A</sub>]. Overall, bisphenols and BADGEs were not removed completely by activated sludge treatment. Furthermore, formation of bisphenols from the degradation of microplastics or other precursors such as alkylated bisphenols, in WWTPs is suggested.

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## 1. Introduction

Bisphenol analogs (hereafter referred as 'bisphenols') and bisphenol A diglycidyl ether (BADGE) and its derivatives (hereafter referred as 'BADGEs') are high production volume phenolic chemicals with widespread industrial and commercial applications, including inner coating of food cans, dental sealants, paints, electronic equipment, thermal papers, and polycarbonate plastics (Eddo and Allan, 2011; Lee and Neville, 1982; Liao et al., 2012a; Xue et al., 2018). In the past few years, bisphenols and BADGEs have emerged as contaminants of concern owing to their widespread human exposures and toxicity including estrogenicity, cytotoxicity, genotoxicity, and carcinogenicity (Chen et al., 2002; Kitamura et al., 2005; Nakazawa et al., 2002; Ramilo et al., 2006; Rivas et al., 2002; Satoh et al., 2004; Suarez et al., 2000; Sueiro et al., 2001; Sueiro et al., 2006).

Studies have reported ubiquitous occurrence of bisphenols and BADGEs in humans and the environment (Cobellis et al., 2009; Xue et al., 2015a; Wang et al., 2015). Bisphenols and BADGEs were reported to occur in environmental samples, including air and indoor dust, biosolids, and sediment, at concentrations as high as several parts per million (Xue et al., 2015b; Xue et al., 2016; Yu et al., 2015; Liao et al., 2012b; Fu and Kawamura, 2010; Wang et al., 2012). This raised concern about the fate of these substances in the environment. Similar to that reported for many chemicals that are used in consumer products, down-the-drain route is an important pathway for the environmental release of bisphenols and BADGEs. Bisphenols and BADGEs can reach wastewater treatment plants (WWTPs) through the sewer system; thus, WWTPs can act as conduits for the environmental release of these compounds. Nevertheless, information about the occurrence and fate of these compounds in WWTPs in the USA are limited. Two studies have examined the occurrence of BPA in WWTPs in the U.S. (Guerra et al., 2015; Hunter, 2015). However, the fate of other bisphenol analogues and BADGEs in WWTPs has not been studied yet. In this study, 24-h composite samples of influent, primary effluent, final effluent, and sludge, were collected from two WWTPs (hereafter referred as WWTP<sub>A</sub> and WWTP<sub>B</sub>) in the Albany area of New York State. The aim of this study was to determine mass flows and fate of bisphenols and BADGEs in WWTPs based on the concentrations measured in wastewaters and sludge collected at various stages of treatment. This is the first study to investigate the occurrence, removal and fate of bisphenols and BADGEs in WWTPs in the USA.

## 2. Materials and methods

### 2.1. Standards and reagents

Information regarding analytical standards and reagents used in this study is provided in the Supporting Information.

### 2.2. Sample collection

Detailed information on sample collection has been described elsewhere (Subedi and Kannan, 2014; Subedi and Kannan, 2015). Briefly, 24-h composite wastewater samples, including raw wastewater (influent), primary-treated wastewater (primary effluent), secondary-treated wastewater (final effluent), and sludge were collected every morning from WWTP<sub>A</sub> and WWTP<sub>B</sub>, in the Albany area of New York State. Characteristics of the two WWTPs and composite sample collection are shown in Table S1.

The sampling period consisted of daily collection from April 27th to May 1st, and from June 27th to 28th in 2015 in WWTP<sub>A</sub> and from July 7th to 13th in 2015 in WWTP<sub>B</sub>. These samples represented daily samples from Monday to Sunday in a week, and were used to investigate the diel variations in bisphenols and BADGEs concentrations in WWTPs. Furthermore, samples were also collected monthly from August 2013 to April 2014 in both WWTPs to investigate seasonal

differences in mass loadings. Detailed information regarding influent, primary effluent and final effluent samples is provided in the Supporting Information.

The activated sludge samples were combined sludge obtained after primary and secondary treatments, and were collected for 7 consecutive days from WWTP<sub>B</sub> and four consecutive days (April 28th to May 1st, 2015) within the sampling week from WWTP<sub>A</sub>. In WWTP<sub>A</sub>, the combined sludge was further thickened and dewatered to produce sludge cake, which was then incinerated in a hearth incinerator yielding sludge ash. Sludge cake was produced when the combined sludge was generated, however, incineration of sludge cake was not performed routinely. All samples were collected in certified pre-cleaned amber glass jars (Thermo Scientific; cleaned in the laboratory with acetone, hexane and methanol) with Teflon-faced caps, shipped to the laboratory, and stored in a refrigerator at 4 °C until further analysis.

### 2.3. Sample preparation

Wastewater samples were mixed thoroughly prior to analysis, and 100 mL of the sample was filtered through a glass fiber filter (37 mm, pore size 1 mm; GVS North America, Sanford, ME, USA) to separate suspended particulate matter (SPM) from the aqueous fraction. All the apparatus used in the filtration and separation were rinsed with Milli-Q water and methanol in sequence to remove any potential contamination.

### 2.4. Wastewater

Formic acid (0.2%) was added to wastewater samples to adjust the pH of the samples to 2.5. The aqueous phase was allowed to equilibrate for ~30 min at room temperature, then purified by passage through a Strata™ - X 33 µm Polymeric Reversed Phase (200 mg/3 mL) cartridge. Fifty nanograms of internal standard mixture (ISs: *d*<sub>6</sub>-BADGE, <sup>13</sup>C<sub>12</sub>-BPA, <sup>13</sup>C<sub>12</sub>-BPF, and <sup>13</sup>C<sub>12</sub>-BPS) were spiked into samples, procedural blanks, and quality control (QC) samples prior to extraction. Prior to use, the cartridge was conditioned with 6 mL of methanol and 6 mL of acidified Milli-Q water (0.2% formic acid) in sequence. After loading the sample (~1 drop per second), the cartridge was washed with 10 mL of acidified Milli-Q water (0.2% formic acid), and then eluted with 9 mL of methanol after drying for ~30 min under vacuum. The eluate was concentrated to ~100 µL under a gentle stream of nitrogen using a TurboVap evaporator (Zymark, Inc., Hopkinton, MA, USA). The final volume of the extract was adjusted to 1 mL with methanol in an amber glass vial, and further diluted when needed before injection into high performance-liquid chromatography-tandem mass spectrometer (HPLC-MS/MS) for the analysis of bisphenols and BADGEs.

### 2.5. Suspended particulate matter (SPM)

Bisphenols and BADGEs in SPM samples were sequentially extracted with 8 mL of ethyl acetate and then with 6 mL of methanol. The combined extracts were concentrated to near dryness under a gentle nitrogen stream. The final volume of the extract was adjusted to 1 mL and then filtered through a 0.2 µm nylon filter prior to instrumental analysis. The glass fiber filters were pre-weighed after baking at 60 °C in an oven for 3 h. After filtration of wastewater, the entire content on the glass fiber filter (i.e., SPM) was freeze-dried and weighed to calculate the mass of SPM present in wastewater.

### 2.6. Sludge

Subsamples of sludge were freeze-dried, and approximately 0.1–0.2 g of sludge/sludge cake and 0.3–0.4 g for ash was transferred into a 15-mL polypropylene (PP) tube. Various extraction methods were employed for the analysis of bisphenols and BADGEs in sludge, cake and ash samples. Sample extraction for bisphenols analysis in

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