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Assessment of bisphenol-A in the urban water cycle

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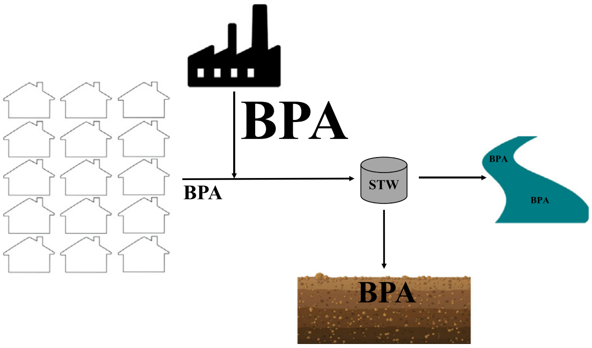
HIGHLIGHTS

- Comprehensive study of BPA in the water cycle reported.
- Industrial discharges of BPA in municipal wastewater identified through wastewater analysis.
- Considerable temporal and spatial variability of BPA loads in wastewater.
- Effluent discharges had little impact to river quality and levels below current PNEC.
- High BPA concentrations (4.6 and 38.7 μg g^{-1}) in digested sludge for land application.

GRAPHICAL ABSTRACT



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ABSTRACT

The plasticizer bisphenol-A (BPA) is common to municipal wastewaters and can exert toxicity to exposed organisms in the environment. Here BPA concentration at 5 sewage treatment works (STW) and distribution throughout a river catchment in South West UK were investigated. Sampling sites included influent and effluent wastewater ($n = 5$), river water ($n = 7$) and digested sludge ($n = 2$) which were monitored for 7 consecutive days. Findings revealed average BPA loads in influent wastewater at two STWs were 10–37 times greater than the other wastewaters monitored. Concentrations up to ~100 μg L^{-1} were measured considerably higher than previously reported for municipal wastewaters. Temporal variability throughout the week (i.e., highest concentrations during weekdays) suggests these high concentrations are linked with industrial activity. Despite ≥90% removal during wastewater treatment, notable concentrations remained in tested effluent (62–892 ng L^{-1}). However, minimal impact on BPA concentrations in river water was observed for any of the effluents. The maximum BPA concentration found in river water was 117 ng L^{-1} which is considerably lower than the current predicted no effect concentration of 1.6 μg L^{-1}. Nevertheless, analysis of digested sludge from sites which received these elevated BPA levels revealed average concentrations of 4.6 ± 0.3 and 38.7 ± 5.4 μg g^{-1}. These sludge BPA concentrations are considerably greater than previously reported and are attributed to the high BPA loading in influent wastewater. A typical sludge application regime to agricultural land would result in a predicted BPA concentration of 297 ng g^{-1} in soil. Further studies are needed on the toxicological thresholds of exposed terrestrial organisms in amended soils to better assess the environmental risk here.

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1. Introduction

It is estimated that 2.2 million tonnes of bisphenol-A (BPA) is manufactured globally each year (Kelland, 2010), and demand is expected to rise by 6–7% per year (Wright-Walters et al., 2011). BPA is used in the production of epoxy resins and polycarbonate plastics as well as phenol resins, polyacrylates and polyesters (Staples et al., 1998). Due to its high production and widespread usage in numerous products, BPA is commonly found in the environment, including surface waters (Belfroid et al., 2002; Santhi et al., 2012), despite a relatively short environmental half-life of 2.5 to 5 days (Babić et al., 2016). Concentrations previously reported in river water are generally in the ng L^{-1} range (Belfroid et al., 2002; Santhi et al., 2012; Petrie et al., 2015). However, concentrations exceeding 1 μg L^{-1} have been reported including river waters in the United States, Japan, Spain, China and the Netherlands (Matsumoto, 1982; Staples et al., 2000; Belfroid et al., 2002; Vethaak et al., 2005; Céspedes et al., 2005). The presence of BPA in the aquatic environment is concerning due to its possible toxic effects on exposed biota (Brian et al., 2005). Evidence of endocrine disruption caused by BPA has been observed in numerous aquatic organisms including fish, amphibians and invertebrate (Kang et al., 2008). These toxicity studies conducted under laboratory conditions typically use BPA at μg L^{-1} concentration levels. Risk assessment of BPA has established a predicted no effect concentration (PNEC) of 1.6 μg L^{-1} established (EU, 2008). However, this PNEC has been questioned by the scientific community with the suggestion that such levels may not be adequate to protect the most sensitive organisms to BPA exposure (Oehlmann et al., 2008).

The main source of BPA in rivers is from the discharge of wastewater effluents. There are other possible sources such as the leaching of BPA from submerged or discarded plastic in the river itself. Nevertheless, BPA is present in municipal wastewaters at high concentration due to the continual leaching from plastics and resins (Rubin, 2011; Flint et al., 2012), including pipes and drinking cups and bottles. Typical concentrations of BPA in municipal wastewater influent are <5 μg L^{-1} (Rudel et al., 1998; Lee and Peart, 2000; Zafra et al., 2003; Hernando et al., 2004; Drewes et al., 2005; Petrie et al., 2015). However it can also be present in municipal wastewater from the discharge of industrial wastewater. Manufacturing and processing industries can be permitted to discharge their waste into municipal wastewater systems for treatment. Industrial wastewater from a paper production facility has previously been found to contain BPA concentrations ranging from 28 to 72 μg L^{-1} (Fürhaker et al., 2000).

BPA is considered moderately biodegradable during biological wastewater treatment with removal transformation >80% commonly reported (Clara et al., 2005; Petrie et al., 2015). Nevertheless BPA is commonly reported in treated effluents discharged into the environment (Nakada et al., 2006; Gardner et al., 2012; Petrie et al., 2015).

Other than being biodegradable BPA is considered moderately hydrophobic and has a Log K_{OW} of 3.4 (Staples et al., 1998). Therefore it can adsorb to particulate organic matter including sludge during biological wastewater treatment. BPA has been reported from ng g^{-1} levels to >300 μg g^{-1} in activated sludge (Meesters and Schröder, 2002; Nie et al., 2009). Excess or waste activated sludge produced is normally digested anaerobically. Organic contaminants such as BPA have been found to persist during anaerobic digestion with concentrations up to 1 μg g^{-1} reported (Tan et al., 2007; Pothitou and Voutsa, 2008; Stasinakis et al., 2008; Nie et al., 2009; Langdon et al., 2011; Guerra et al., 2015). Persisting BPA can be a significant issue as digested sludge (or biosolids) are recycled to land as fertilizer. Staples et al. (2010) investigated the possible risks of BPA exposure to organisms at the base of terrestrial food webs (2 species of invertebrates and 6 plant species). A soil PNEC of 3.7 μg g^{-1} was proposed indicating the risk to these organisms is low based on current BPA concentrations reported in sludge for land application. However, a lowest observed effect concentration of 10 μg g^{-1} has been reported for female juvenile woodlouse (Lemos et al., 2010). Applying an assessment factor of 10 would result in a soil PNEC of 1 μg g^{-1}. There are comparatively fewer studies which investigate BPA toxicity to exposed organisms in the terrestrial environment which make the derivation of a PNEC challenging.

Due to the continuing increase in BPA production and use, it is essential to understand its current distribution in the water cycle. Such information is vital for assessing environmental risk and developing environmental policy. Therefore the aim of this study was to provide a holistic understanding of the source, fate and behaviour of BPA throughout a river catchment. To achieve this understanding the following objectives were identified: (i) to investigate the presence and source of BPA in influent wastewaters, (ii) to determine the fate and behaviour of BPA during wastewater treatment, (iii) to assess the impact of effluent discharges to river water quality with respect to BPA, and (iv) to evaluate the significance of BPA in digested sludge destined for land application. This was achieved by investigating BPA at 5 strategic sampling sites throughout a river catchment in South West UK. At each site, influent and effluent wastewater, and river water from upstream and downstream of the effluent discharge point (where possible) were sampled for 7 consecutive days. Digested sludge was also collected from sites with sludge treatment facilities.

2. Materials and methods

2.1. Materials

The analytical reference standards BPA and BPA-d16 were purchased from Sigma-Aldrich (Gillingham, UK). Methanol (MeOH) was HPLC grade and obtained from Sigma-Aldrich. Water used throughout the study was 18.2 MΩ quality (Elga, Marlow, UK). Ammonium fluoride (NH_4F) for the preparation of mobile phases was obtained from Fisher Scientific (Loughborough, UK). Oasis® HLB and MCX (60 mg, 3 mL) SPE cartridges were purchased from Waters (Manchester, UK).

2.2. Extraction methods

Aqueous matrices (influent wastewater – 50 mL, effluent wastewater – 50 mL and river waters – 100 mL) were filtered using GF/F filters (0.7 μm) and adjusted to pH 7.5 ± 0.1. Samples were then spiked with 50 ng (50 μL of a 1 μg mL^{-1} methanolic solution) of BPA-d16 and loaded onto Oasis® HLB cartridges conditioned using 2 mL MeOH followed by 2 mL H_2O at 1 mL min^{-1}. Samples were loaded at 5 mL min^{-1} and dried under vacuum. Elution was performed using 4 mL MeOH at a rate of 1 mL min^{-1}. Methanolic extracts were subsequently dried under nitrogen using a TurboVap evaporator (Caliper, UK, 40 °C, N_2, <5 psi). Dried extracts were reconstituted in 500 μL 80:20 H_2O:MeOH ready for analysis.

Suspended particulate matter from influent wastewaters and digested sludge were frozen, freeze-dried and 0.25 g samples spiked with 50 ng of BPA-d16. Samples were then left for a minimum of 1 h. Extraction was achieved using 25 mL of 50:50 MeOH:H_2O (pH 2) at 110 °C using a 800 W MARS 6 microwave (CEM, UK). Samples extracts were adjusted to <5% MeOH using H_2O (pH 2). SPE was performed using Oasis® MCX cartridges conditioned with 2 mL MeOH followed by 2 mL H_2O (pH 2). Samples were loaded and dried as described previously. Elution was performed using 2 mL 0.6% HCOOH in MeOH. Once dried extracts were reconstituted in 500 μL 80:20 H_2O:MeOH and filtered using pre-LCMS 0.2 μm PTFE filters (Whatman, Puradisc). The full methodology is described in Petrie et al. (2016).

2.3. UPLC-MS/MS analysis

Chromatography was performed on a Waters Acquity UPLC system (Waters, Manchester, UK). A reversed-phase BEH C18 column (150 × 1.0 mm, 1.7 μm particle size) (Waters, Manchester, UK) was utilised with a 0.2 μm, 2.1 mm in-line column filter maintained at 25

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