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## Spatio-temporal assessment of perfluorinated compounds in the Brisbane River system, Australia: Impact of a major flood event

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## ABSTRACT

Perfluorinated chemicals including PFOA and PFOS have been widely used in consumer products and have become ubiquitous pollutants widely distributed in the aqueous environment. Following a major flood event in 2011, water samples were collected along a spatial gradient of the Brisbane River system to provide an initial estimate of the release of PFASs from flooded urban areas. PFOA (mean concentrations 0.13–6.1 ng L<sup>-1</sup>) and PFOS (mean concentrations 0.18–15 ng L<sup>-1</sup>) were the most frequently detected and abundant PFASs. Mean total PFASs concentrations increased from 0.83 ng L<sup>-1</sup> at the upstream Wivenhoe Dam to 40 ng L<sup>-1</sup> at Oxley Creek, representing an urban catchment. Total masses of PFOA and PFOS delivered into Moreton Bay from January to March were estimated to be 5.6 kg and 12 kg respectively. From this study, urban floodwaters appear to be a previously overlooked source of PFASs into the surrounding environment.

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### 1. Introduction

Perfluorinated chemicals or perfluoroalkyl substances (herein referred to as PFASs) are ubiquitous pollutants detected globally in a wide range of environmental samples including aquatic and terrestrial biota, humans, air and household dust, with perfluorooctanoate (PFOA) and perfluorooctanoate sulphonate (PFOS) the most commonly detected, well-known and best studied (Giesy and Kannan, 2002; Gerwurtz et al., 2009; Butt et al., 2010; Thompson et al., 2011; Zhang et al., 2011). The strong carbon–fluorine bonds in these compounds make them very resistant to degradation processes (Remde and Debus, 1996; Key et al., 1998; Liou et al., 2010) and as a result, they have the potential to bioaccumulate in the food web (Giesy and Kannan, 2001; Aherns et al., 2010a). The unique physicochemical properties of PFASs (being chemically inert, able to withstand high temperatures and excellent surfactants) have been exploited particularly for use in waterproofing and stain resistant formulations which have been applied to a wide range of consumer products such as carpet, paper, textiles, non-stick cookware, sports and wet weather clothing (Kissa, 2001; Schultz et al., 2003; Carloni, 2009). The sources of PFASs into the

environment are numerous and can be a result of point (wastewater treatment plants (WWTP) or landfill leachate) and non-point (surface runoff) releases or through the degradation of other perfluorinated precursors (Ma and Shih, 2010; Busch et al., 2010; Sun et al., 2011).

Evidence of the global ubiquity, persistence and potential toxicity of these chemicals, prompted the voluntary phase out of PFOS production by the 3 M Company, a major global producer, in the year 2000 and its inclusion onto the Stockholm Convention for Persistent Organic Pollutants (POPs) in 2009 (OECD, 2002; UNEP, 2009). Although the use of fluorochemical products containing PFOA and PFOS has been abandoned in consumer products, they continue to be produced for restricted use in several countries, primarily in industrial processes (such as semiconductors and metal plating) for which suitable replacements have not yet been found (Carloni, 2009; Lim et al., 2011; Xie et al., 2013). Despite the phase out of PFOS from all non-essential uses in consumer products over a decade ago, it is likely that many Australian households still contain older items (such as carpets and furniture) that were once treated with a fluorochemical formulation containing numerous PFASs including PFOS. Whilst the focus is now on developing products that contain perfluorinated alternatives to PFOA and PFOS (such as shorter chain-length PFASs which still retain the properties of PFOS and PFOA but without the bioaccumulation potential),

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understanding the environmental fate and toxicity of these alternative compounds is still ongoing (US EPA, 2012).

Flooding is a common natural phenomena, occurring seasonally particularly in North Queensland, having both benefits and harmful effects on the environment. However, as cities and human activities continue to grow in flood-prone areas, the impact of floods to the natural environment is becoming increasingly negative. Floodwaters are well known to carry loads of nutrients, pesticides and sediment over vast distances (Brodie et al., 2012; Kroon et al., 2012), however the composition of other pollutants carried by urban floodwaters has not been well characterised.

In January 2011, severe flooding occurred in urban areas of the city of Brisbane with an inner city population of approximately 1.1 million (ABS, 2012a). Dam releases combined with heavy rain in the upper Brisbane River catchment resulted in flood water travelling down the Brisbane River into urban areas containing tens of thousands of households and businesses, eventually discharging into the coastal bay area of Moreton Bay (Fig. S1). Low-lying properties began to flood on the 11th of January 2011, with flood waters peaking on the 13th of January, resulting in approximately 23,000 flooded properties (BCC, 2011). Over the month of January, 36% of the volume of Moreton Bay was discharged from the Brisbane River, with the discharge volume 18 times greater than the long-term January mean discharge of 197 GL (data obtained from DERM (2013)). The resulting flood plume covered an area of approximately 400 km<sup>2</sup> in Moreton Bay (Yu et al., 2011) and approximately 1,040,000 tonnes of sediment was deposited (Steven et al., 2013). In addition, nine out of Southeast Queensland's 28 WWTPs were affected by the flooding, resulting in critical failures of treatment systems and the discharge of untreated sewage through overflow relief structures into floodwaters (QFCI, 2012). Floodwaters persisted in some urban areas for up to a week following the peak of the flooding.

The contamination of river and coastal waters in association with urban areas with PFASs has been studied worldwide (Loos et al., 2008; Nguyen et al., 2011; Thompson et al., 2011; Sun et al., 2011; Sakurai et al., 2010) however, to our knowledge, no data is available on the role of major floods on PFASs input into the environment. Flooding of urban areas has likely been overlooked as a significant source of PFASs released into the environment.

With PFASs being associated with many consumer products commonly used in homes and businesses, the aim of this study was to investigate the impact of urban floodwaters on the concentrations of PFASs in the Brisbane River and coastal bay system. Water samples were collected along a spatial gradient from the upper Brisbane River catchment to the marine environment to provide an initial estimate of the release of PFASs from urban areas during the flood. Samples were collected from (a) the origin of Brisbane River (i.e. Wivenhoe and Somerset Dams), (b) the Brisbane River, (c) Oxley Creek (a tributary of the Brisbane River and (d) Moreton Bay (which receives the water discharged from the Brisbane River). By monitoring upstream and downstream of intensive urban areas, the contribution of these areas to the amounts of PFASs entering the environment may be estimated for the first time.

## 2. Materials and methods

### 2.1. Sampling sites

Sampling sites were located from the two water reservoirs along a transect from the fresh water to estuarine area of the Brisbane River and extending up to a further 15 km from the River mouth out into Moreton Bay (Table S1). The locations of the ten grab water sampling sites are detailed in Fig. 1.

The Brisbane River is the longest in Southeast Queensland, stretching approximately 344 km in length. Approximately 150 km upstream from the Brisbane River mouth, the river is dammed by Wivenhoe Dam (Site D2), with a total storage capacity of 2.6 million ML and catchment area of 7020 km<sup>2</sup> (SEQWater, 2013a). Wivenhoe Dam was designed primarily to provide a reliable water supply for the city of Brisbane as well as flood mitigation. Somerset Dam (Site D1) is located on the nearby Stanley River, with a total storage capacity of 380,000 ML and catchment area of 1340 km<sup>2</sup> (SEQWater, 2013b). Somerset Dam is released into Lake Wivenhoe and also supplements Brisbane drinking water supplies.

Three sites (R1, R2, R3; Jindalee, Orleigh Park, Bulimba) are located on the Brisbane River between 50 and 14 km upstream of the river mouth respectively. Population densities of the three suburbs in 2011 were 2053, 4259 and 3050 persons per km<sup>2</sup> respectively with West End (Orleigh Park) one of the most densely populated areas in Brisbane city (ABS, 2012b). One site was located in Oxley Creek (Site C), a major tributary of the Brisbane River located approximately 38 km upstream of the river mouth. A more intensive sample collection program (samples collected at both high and low tide) was undertaken at Oxley Creek for four months following the flood, as prolonged sampling would give an indication of the recovery of a seriously flood-affected catchment over time. Oxley Creek is approximately 70 km long and the Oxley Creek catchment is one of Brisbane City's largest, covering an area of over 260 km<sup>2</sup>. This low-lying catchment (which contains a WWTP, general industry and areas of low to medium density housing) experienced some of the most devastating flooding, with the Oxley flood plain covering approximately 15 km<sup>2</sup> (Fig. S2). Four sites (B1–B4) were located in Moreton Bay, extending between 6 and 15 km from the river mouth.

### 2.2. Grab sample collection

1L water samples were collected using a solvent-rinsed stainless steel bucket at an approximate depth of 1–2 m. High density polyethylene (HDPE) bottles were rinsed with analytical grade acetone and again with water collected at the site prior to collection. The steel bucket was rinsed with site water at each location prior to sample collection. Collected water samples were stored frozen in HDPE bottles prior to extraction. A total of 42 grab samples were collected from the 10 sampling locations between the 22nd of January and the 21st April 2011. Dangerous river conditions prevented samples being collected at any earlier dates following the peak of flooding. Nine time points also had replicate grab samples collected.

### 2.3. Grab sample extraction

PFASs investigated in this work were perfluorohexanoate (PFHxA), perfluoroheptanoate (PFHpA), perfluorooctanoate (PFOA), perfluorononanoate (PFNA) and perfluorodecanoate (PFDA), perfluorobutanesulfonate (PFBS), perfluorohexanesulfonate (PFHxS), perfluorooctanesulfonate (PFOS) and 6:2 fluorotelomer sulfonate (6:2 FTSA).

Methanol (HPLC grade) and ammonia solution (HPLC grade; 32% w/w) were purchased from Merck (Darmstadt, Germany). Ultrapure water (Millipore, 0.22 µm filtered, 18.2 mΩ cm<sup>-1</sup>) was used in sample extraction and chemical analysis. Mobile phases were filtered using Phenex 0.20 µm 47 mm nylon filter membranes (Lane Cove, Australia). All laboratory glassware and equipment was rinsed with analytical grade acetone followed by methanol (HPLC grade) prior to use. Internal standards were added to all water samples prior to extraction (50 µL; 0.08 ng µL<sup>-1</sup>), consisting of mass labelled perfluoro[1,2,3,4-<sup>13</sup>C<sub>4</sub>]butanoic acid, perfluoro[1,2-<sup>13</sup>C<sub>2</sub>]hexanoic acid, perfluoro[1,2,3,4-<sup>13</sup>C<sub>4</sub>]octanoic acid, perfluoro[1,2,3,4,5-<sup>13</sup>C<sub>5</sub>]nonanoic acid, perfluoro[1,2-<sup>13</sup>C<sub>2</sub>]decanoic

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