



Seasonal variation and sediment–water exchange of antibiotics in a shallower large lake in North China



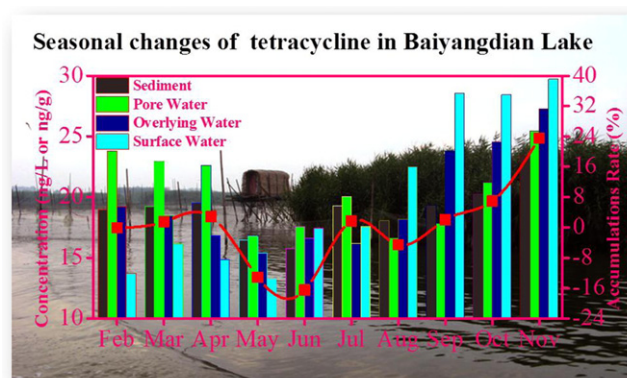
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HIGHLIGHTS

- Antibiotics in different environmental compartments were monitored seasonally.
- Antibiotics showed significant seasonal variations in water and sediment matrix.
- Sediments may act as a second source of antibiotics to aquatic environment.

GRAPHICAL ABSTRACT



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ABSTRACT

The occurrence of four antibiotics, including oxytetracycline (OTC), tetracycline (TC), norfloxacin (NOR) and ofloxacin (OFL), in surface water, overlying water, pore water and sediment samples were studied in the Baiyangdian Lake from February to November in 2009. The total concentrations of these antibiotics ranged among 17.73–281.82, 22.98–258.45, 22.43–198.95 ng L^{-1} and 131.65–750.27 ng g^{-1} in surface water, overlying water, pore water and sediments, respectively. Seasonal variation might be impacted by the frequency of different pattern of antibiotics and the water temperatures of different seasons, where the higher concentrations appeared at different seasons. In addition, the regions with significant sewage discharge or human agricultural activities exhibited high concentrations of antibiotics in water and sediments. The highest accumulation rates of the four antibiotics ranged from 11.27 to 29.71%, which indicated that these compounds exhibited strong adsorption to the sediment. However, higher concentrations of antibiotics in pore water and even overlying water may result in the release of these compounds from the sediment acting as a secondary contaminant source in a certain time period, especially for TC. The pseudo-partitioning values of fluoroquinolones (FQs) ranged from 4493 to 47,093 L kg^{-1} and were much higher than those of tetracyclines (TCs), which ranged from 277 to 1880 L kg^{-1} indicating that the FQs are prone to accumulation in the sediment.

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1. Introduction

Antibiotics as ionic organic contaminants (IOCs) have been widely used for several decades in both humans and livestock (Kümmerer, 2009). The consumption has gradually increased due to global

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economic development. For China, it has been estimated that more than 25,000 tons of antibiotics are used each year (Gao et al., 2012), and except for disease treatment, a large portion of these antibiotics were used by livestock producers (Mellon et al., 2001). Several types of antibiotics have been detected in the aquatic environment, especially fluoroquinolones (FQs) and tetracyclines (TCs) (Leung et al., 2012; Li et al., 2012; Shimizu et al., 2013; Zou et al., 2011). These two compounds are frequently detected in both surface water and sediment and exhibit relatively high pseudo-partitioning coefficients that are calculated from the antibiotic concentrations in sediment divided by the corresponding concentrations in surface water compared to sulfonamides (SAs) and macrolides (MCs) (Gong et al., 2012; Kim and Carlson, 2007; Li et al., 2012; Liang et al., 2013; Zhang et al., 2011).

The environmental effects caused by the widespread use of antibiotics have resulted in much attention due to their consumption and excretion. These antibiotics and their corresponding metabolites are released to the environment after consumption by humans and livestock (McArdell et al., 2003). For antibiotics used to treat humans, previous studies have shown that sewage treatment plants (STPs) are the main component of discharges to the environment, due to the partial elimination during the purification process (Chang et al., 2010; Jia et al., 2012). When the manure is applied to agricultural fields, the soil may be contaminated by livestock treated with antibiotics, and overflow and drain flow can pollute rivers, streams and ditches (Jacobsen et al., 2004; Wei et al., 2011). In addition, the usage of antibiotics in aquaculture is another important source of antibiotics in the environment (Lalumera et al., 2004).

Currently, antibiotic resistance in bacteria has become a new challenge for infection control worldwide (Wellington et al., 2013). Antibiotics were initially designed to produce biological effects and interact with specific biological systems. However, the continual exposure of the bacterial community, even at small concentrations of antibiotics or active metabolites, could lead to the emergence or persistence of antibiotic-resistant bacteria in the natural aquatic environment, which poses a potential threat to human health (Schwartz et al., 2003). In the long term, humans will be threatened by infections caused by exposure to bacteria (Wellington et al., 2013). In addition, a variety of antibiotics have been detected in the sediment matrix which is an important habitat for bacteria and sequesters more pollutants (Kim and Carlson, 2007; Li et al., 2012). These results also indicate that sediment can potentially act as a significant secondary source of antibiotics that can be released into the water if the aquatic environment changes. Many studies have indicated that changes in the aquatic environment (e.g., water volume, flow and physicochemical properties such as water temperature and pH) substantially contribute to the ad/desorption behaviour of pharmaceuticals and other contaminants in sediment (Gong et al., 2012; Kümmerer, 2009; Rosen et al., 2010; Westerhoff et al., 2005). Therefore, it is important to explore the antibiotic partitioning behaviour between the water phase and sediment to predict and control the risk of aquatic organisms and humans which have been directly or indirectly exposed to a contaminated aquatic environment (Chau, 2005).

It has been confirmed that hydrophobic organic contaminants (HOCs), such as PCDD/Fs, PCBs and OCPs, participates in sediment–water exchange in natural aquatic environments (Dai et al., 2013; Dalla Valle et al., 2003). For HOCs, it is generally believed that sediment–water exchange includes two main processes: (i) diffusion of dissolved HOCs between sediment and water phases and (ii) particle-phase HOC deposition and resuspension (Lun et al., 1998). However, the sediment–water exchange of IOCs has been rarely studied and is urgently needed for a better understanding of the environmental behaviour of IOCs.

In recent years, there has been an abundance of research focused on determining the presence of antibiotics in environmental waters (Yan et al., 2013; Zhang et al., 2013). These studies typically focus on the lateral distribution of antibiotics via short-term monitoring. However, antibiotics will continue to move within a water column (i.e., a river or

lake) until they are removed by attenuation processes, such as sorption or degradation. Less is understood about the vertical distribution and exchange between the sediment and water phase of antibiotics in a natural water system. Long-term continuous monitoring combined with multiple media vertical sampling is necessary to better understand the behaviour of antibiotics in a natural aquatic environment.

Motivated by our previous study (Dai et al., 2013), our hypothesis in this paper is that distributions of antibiotics will vary depending on sampling locations and time due to different human factors and environmental conditions (e.g., water temperature, volume, and physical disturbance) in the Baiyangdian Lake. Therefore, the major objectives of this study were (1) to investigate the seasonal and spatial behaviours of antibiotics between sediments and the water phase and (2) to understand the exchange and partitioning behaviours of different antibiotics between water and sediment in aquatic environment. Two classes of antibiotics were included in the study, including ofloxacin and norfloxacin (FQs) and oxytetracycline and tetracycline (TCs) (Table 1).

2. Material and methods

2.1. Site description and sample collection

As the largest shallow freshwater lake in North China, Baiyangdian Lake (Fig. 1) is located approximately 200 km southwest of Beijing and covers more than 366 km² with an average depth of approximately 2–4 m. This lake consists of more than 100 small and shallow lakes linked by thousands of ditches and large areas of reed marshes. Currently, there are more than 243,000 people living in 39 villages scattered in and around it.

Ten different sampling events were conducted from February to November 2009 (February in frozen period) at six sampling sites (Sites 1–6, given in Fig. 1 and Supporting information Table S3) representing slightly polluted, urban, and agriculturally influenced areas in the Baiyangdian Lake in North China. Water and sediment samples were collected according to our previously published protocols (Dai et al., 2013). Surface water samples were collected approximately 0.5 m below the water surface using a stainless-steel submersible pump. Overlying water samples were collected approximately 0.2 m off the bottom using a peristaltic pump to ensure that the surface sediments would not be disturbed. In situ pore water samples were collected using a Rhizon in situ sampler (RISS, Rhizosphere Research Products, Wageningen). All of the water samples were stored in pre-cleaned amber glass bottles. Sediments (0–5 cm deep) were collected at the corresponding water sampling sites using a stainless steel static gravity corer. The top surface layer (approximately 1 cm) was carefully removed with a stainless steel spoon, and stored in aluminium containers. All of the samples were stored in a cooler during sampling events and were immediately transported to the laboratory. Sediment samples were freeze-dried using a vacuum freeze-drier (FD-1A, China), and water samples were stored at –20 °C. Freeze-dried sediments were ground by an agate mortar and sieved through a 100 mesh sieve, wrapped in pre-cleaned aluminium foil, sealed in plastic bags and stored at –20 °C prior to extraction.

2.2. Chemicals and materials

Four antibiotic compounds (Table 1) (oxytetracycline (OTC), tetracycline (TC), norfloxacin (NOR) and ofloxacin (OFL)) were purchased from Dr. Ehrenstorfer (Augsburg, Germany). HPLC grade methanol, acetonitrile and formic acid were purchased from Fisher Science Co. The internal standard (i.e., diuron-d6) was acquired from Cambridge Isotope Laboratories, USA.

Unless otherwise indicated, the other chemicals used in this study were of analytical grade and used without further purification. Separate stock solutions (1000 mg L⁻¹) of individual compounds and internal standards were prepared by dissolving an appropriate amount of each

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