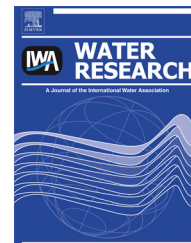


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Occurrence of seven artificial sweeteners in the aquatic environment and precipitation of Tianjin, China

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ABSTRACT

Seventy water samples, including wastewaters, tap waters, fresh surface waters, coastal waters, groundwaters, and precipitation samples, from Tianjin, China, were analyzed for seven commonly used artificial sweeteners (ASs). The concentrations of the investigated ASs were generally in the order of wastewater treatment plant (WWTP) influent > WWTP effluent > surface water > tap water > groundwater ≈ precipitation, while the composition profiles of ASs varied in different waters. Acesulfame, sucralose, cyclamate, and saccharin were consistently detected in surface waters and ranged from 50 ng/L to 0.12 mg/L, while acesulfame was the dominant AS in surface and tap waters. Aspartame was found in all of the surface waters at a concentration up to 0.21 μg/L, but was not found in groundwaters and tap waters. Neotame and neohesperidin dihydrochalcone were less frequently detected and the concentrations were low. The concentrations of the ASs in some of the surface waters were of the same order with those in the WWTP influents, but not with the effluents, indicating there are probably untreated discharges into the surface waters. The ASs were detected in precipitation samples with high frequency, and acesulfame, saccharin, and cyclamate were the predominant ASs, with concentrations ranging from 3.5 ng/L to 1.3 μg/L. A gross estimation revealed that precipitation may act as a source for saccharin and cyclamate in the surface environment of Tianjin city. Moreover, the presence of ASs in the atmosphere was primarily assessed by taking 4 air samples to evaluate their potential source in precipitation.

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1. Introduction

Since saccharin (SAC) was discovered in 1897 (Remsen and Fahlberg, 1879), artificial sweeteners (ASs) have become increasingly popular. Globally, these sugar substitutes are consumed in substantial quantities in food, beverages, pharmaceuticals, personal care products, and even in animal feed (Ferrer and Thurman, 2010; Buerge et al., 2011). However, until

recently, little attention has been paid to these compounds with respect to their occurrence and fate in the environment.

The first large scale screening program on ASs was conducted in Sweden, which documented that sucralose (SUC) was ubiquitous in surface waters with concentrations ranging from 4.0 ng/L to 3.6 μg/L. Waste water treatment plants (WWTPs) were investigated as a possible source for ASs in the surface water; and it was found that SUC levels were

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practically unaffected during wastewater treatment (Brorström-Lundén et al., 2008). Since then, investigations have been conducted in the USA (Mead et al., 2009; Ferrer and Thurman, 2010; Mawhinney et al., 2011; Oppenheimer et al., 2011; Soh et al., 2011), Canada (Van Stempvoort et al., 2011a; Van Stempvoort et al., 2011b), Germany (Scheurer et al., 2009, 2010), Switzerland (Buerge et al., 2009, 2011), and even the whole Europe (Loos et al., 2009). These studies found that ASs were pervasive in surface, underground, marine, and even in drinking waters, with maximum concentrations ranging from 2.9 µg/L to 0.20 mg/L. Additionally, acesulfame (ACE) and SUC have been proven to be the most persistent ASs in WWTPs and drinking water treatment plants.

Although comprehensive toxicological tests have been conducted on ASs and they appear to be nontoxic to humans within regulated concentrations (Kroger et al., 2006; Chattopadhyay et al., 2011), their unintended presence in the environment still cause considerable concern, particularly for SUC, which has been confirmed to have a half-life of up to several years in water (Grice and Goldsmith, 2000). It has been documented that SUC may affect the physiology and locomotion behavior of cladoceran (*Daphnia magna*) and amphipod gammarids (*Gammarus oceanicus* and *Gammarus zaddachi*) (Wiklund et al., 2012).

It is necessary to grasp the level of ASs in the environment before assessing their fate and their possible risk. Compared to the wide investigations in the western countries, there are only few relevant reports in China and other Asian countries (Ma et al., 2012), where food habits are quite different from those in western countries. The objective of this study was to determine the prevalence of seven commonly used ASs in the aquatic environment of Tianjin, China. Tianjin is the third largest city in China and it currently has the most rapid economic development in China. The city is a seashore city, and rivers across the city all belong to downstream. Hence, contamination in surface water is severe. The samples investigated including inland fresh water, marine water, groundwater, tap water, samples from WWTPs as well as precipitation samples. To investigate the potential source of ASs in precipitation, we primarily analyzed 4 air samples. To our knowledge, this is the first study to systematically evaluate the occurrence of ASs in Chinese water samples, and the first report on seven commonly used ASs in precipitation and the atmosphere, except for SUC, which was found in rainfall samples from the USA by Oppenheimer et al. (2012). The results expand knowledge concerning the presence and distribution of ASs in the environment, particularly for the occurrence of ASs in precipitation and the atmosphere.

2. Materials and methods

2.1. Chemicals and reagents

The standards for ACE, SAC, SUC, cyclamate (CYC), and neohesperidin dihydrochalcone (NHDC) were procured from Sigma–Aldrich (St. Louis, MO, USA); neotame (NEO) was acquired from USP Reference Standards (Rockville, MD, USA); and aspartame (ASP) was purchased from Supelco (Bellefonte, PA, USA). Sucralose-d6 (SUC-d6), aspartame-d5 (ASP-d5), and

acesulfame-d4 (ACE-d4), used as internal standards, were obtained from TRC (North York, ON, Canada). The structures of the investigated ASs are shown in Table S1 (Supporting Information). The ion pair reagent tris(hydroxymethyl)aminomethane (TRIS) was obtained from Sigma–Aldrich (St. Louis, MO, USA). All other solvents and reagents were of HPLC or analytical grade.

Poly-Sery PWAX (3 mL/60 mg) cartridges from CNW (CNW Technologies GmbH, Düsseldorf, Germany) were used for concentrating the ASs in water samples by solid phase extraction (SPE). Polyurethane foam (PUFs) slices (3 × 2.5 inch) and quartz fiber filters (QFFs) (10 × 8 inch), used in air sampling were procured from Tisch Environmental Incorporation (Clevs, OH, USA). The PUFs were cleaned in Soxhlet apparatus consecutively by methanol and ethyl acetate for 24 h, respectively, followed by drying with high purity nitrogen. The QFFs were baked at 600 °C for 5 h and wrapped in aluminum foil. Both of the cleaned materials were sealed inside polyethylene bags prior to use.

2.2. Sampling campaign

Seventy water samples (including wastewater, tap water, surface water, groundwater, and precipitation) were collected in triplicate from Tianjin in 2011 (Fig. 1). Wastewaters samples including the influents and effluents were collected from two WWTPs (WWTP-1 and WWTP-2) in Tianjin. WWTP-1 is located in the central city, using a conventional activated sludge process with hydraulic retention time (HRT) of 12 h, while WWTP-2 is located in a special economic development zone along the coast, employing a sequencing batch reactor activated sludge process with the HRT of 15 h. The WWTPs treat both sewage water and industrial water. Precipitation samples (rainfall) were collected directly using a pre-cleaned stainless steel bucket, and the precipitation within the first half hour was discarded. All samples were collected as grab samples with chain-of-custody documentation. Raw water samples were preserved in 500 mL PP plastic bottles and stored in the dark at 4 °C until analysis. No preservation agents were added. During sample collection, travel blanks were prepared using 50 mL Milli-Q water in PP plastic bottles to check for any contamination. The travel blank was extracted in the same manner as the samples; and none of the seven ASs were found in the blanks (Fig. S1). Four air samples were collected consecutively using high volume samplers (TH-1000, Wuhan, China) containing cleaned PUF and QFF on March 17, 18, and 27, 2012 at sampling sites of P3 and P4 for 10 h at 1 m³/min. After sampling, the PUF and QFF were stored at –20 °C in polyethylene bags and extracted within 24 h. The details and geographic information of the sampling sites are described in Section S1, Table S2, and Table S3 (Supporting Information).

2.3. Sample extraction and analysis

2.3.1. Water samples

The extraction and analysis methodologies for water samples have well been developed in our previous study (Gan et al., 2013). Water samples were filtered using 0.45 µm cellulose nitrate membrane filters (0.45 µm, 47 mm, Whatman, UK),

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