



Occurrence and distribution of synthetic musks and organic UV filters from riverine and coastal sediments in the Pearl River estuary of China

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ABSTRACT

This study reports the occurrence and distribution of synthetic musks (SMs) and organic UV filters (UVFs) in sediment samples collected in 8 riverine runoffs from the Pearl River and Pearl River estuary (PRE). Here, 6 of the 8 target compounds were detected in all sediments with concentrations ranging from 0.35 ng g⁻¹ to 456 ng g⁻¹. Higher concentrations of SMs and UVFs were evident in the eastern outlets compared to the western suggesting greater input of these contaminants from the Pearl River Delta (PRD) region. All the compounds showed a decreasing trend toward the seaward side which confirming that riverine runoff was the most important source of SMs and UVFs to the coastal environment. Notably, high levels of SMs and UVFs were detected in two fishing harbors in the PRE area. In comparison to UVFs, the SM compounds exhibited a significant correlation with TOC content in the sediments.

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1. Introduction

Synthetic musks (SMs) and organic UV filters (UVFs) are new types of emerging pollutants in the environment that have raised significant concerns in recent years due to their potential threat to the ecological environment and human health (Fromme et al., 2001; Ramos et al., 2015). SMs are widely used by perfume industries as fragrance additives in a variety of consumer products (e.g., perfumes, lotions, sunscreens, shampoos, and laundry detergents) (Reiner and Kannan, 2011; Roosens et al., 2007; Zhang et al., 2008). UVFs are included in various pharmaceutical and personal care products (PPCPs) including body washes, sunscreens, and cosmetic products mainly to protect the skin against ultraviolet radiation (Nakata et al., 2012; Ramos et al., 2015). They are also included in plastics and furniture primarily for photo-protective purposes.

After application, both SMs and UVFs are generally discharged into sewage treatment plants (STPs) or directly into the aquatic environment. However, a number of studies have shown that these compounds are incompletely removed during the treatment at the STPs and do not biodegrade in the environment (Chase et al., 2012; Clara et al., 2011; Tsui et al., 2014a). Consequently, these compounds ultimately enter the aquatic environment and accumulate in various environmental matrices. During the past decade, several studies have documented the presence of SMs and UVFs in rivers, lakes, groundwater, estuaries, and

coastal oceans with concentrations up to µg g⁻¹ levels (Bu et al., 2013; Guo et al., 2013; Kameda et al., 2011; Langford et al., 2015; Nakata et al., 2007; Ramos et al., 2015; Tsui et al., 2014b). In addition, due to their relatively low solubility and high octanol-water partition coefficient (K_{ow}), SMs and UVFs are easily absorbed by particulate matter and eventually accumulate in river sediment (Amine et al., 2012; Ramos et al., 2015; Subedi et al., 2014). These compounds can also be bio-accumulated in organisms at different trophic levels (Fent et al., 2010; Gago-Ferrero et al., 2015; Nakata et al., 2007).

Oceans and coastal zones, act as reservoirs for many contaminants that originate from various sources. Riverine and marine sediments represent the final repository of SMs and UVFs compounds due to their sportive nature and may play a key role as potential sources of these contaminants. The Pearl River Delta (PRD) is one of the most developed and urbanized areas in China, connecting the Pearl River estuary (PRE) to the South China Sea (SCS) via 8 major riverine runoff outlets (Fig. 1). It was estimated that about 1.73 × 10¹⁰ t per year of wastewater are discharged from the PRD into the PRE and the SCS. However, hardly any data are available regarding the occurrence and distribution of SMs and UVFs in sediments of the Pearl River and coastal environment (Chen et al., 2012; Peng et al., 2015; Song et al., 2015; Zeng et al., 2008). In order to understand and assess the impact of increased anthropogenic activities on the coastal environments of the PRD region, the present study investigated the occurrence and spatial distribution of 4 commonly consumed SMs, as well as 3 different UV-F compounds in river and marine sediment samples collected in the Pearl River and the coast of PRE.

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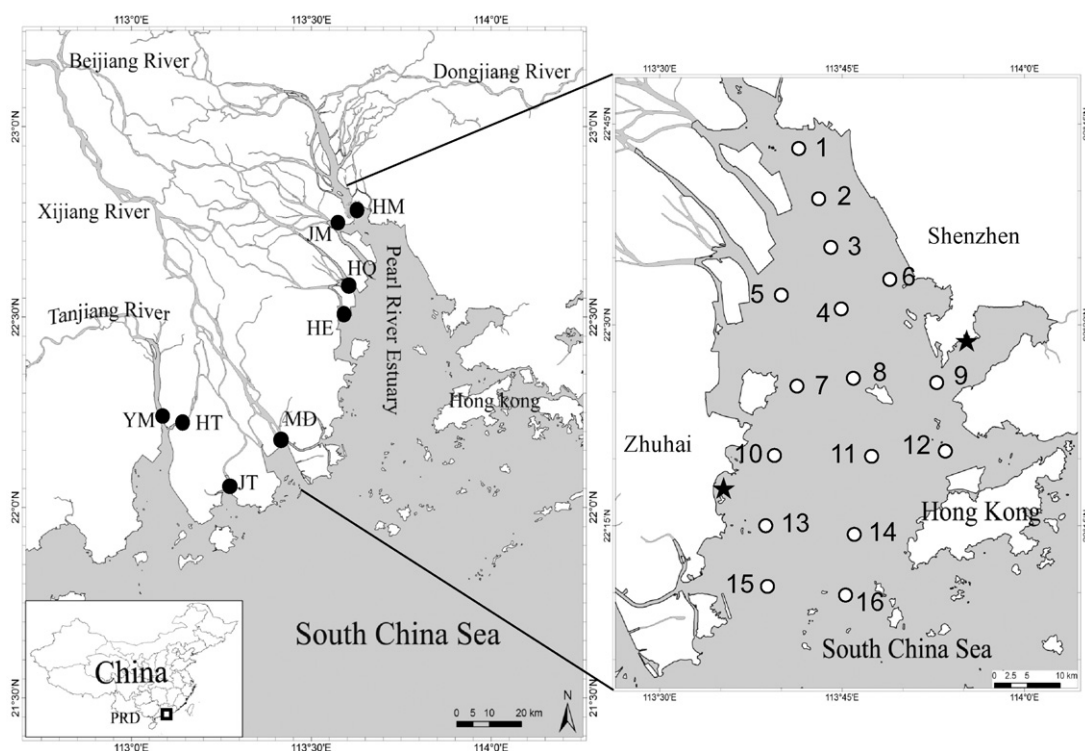


Fig. 1. Maps of sampling sites in the eight major runoff outlets (●), Pearl River estuary (○) and fishing harbors (★).

2. Methods and materials

2.1. Reagents and materials

In this study, 5 SMS [including tonalide (AHTN), galaxolide (HHCB), celestolide (ADBI), phantolide (AHMI), musk ketone (MK)], and 3 UVFs [including 4-methylbenzylidene camphor (4-MBC), octocrylene (OC) and octyl-*p*-methoxycinnamate (OMC)] were selected. Standards ($\geq 98\%$) were purchased from Sigma-Aldrich, Germany. All organic solvents used were of analytical grade and redistilled using glass system. Laboratory glassware was baked at 250 °C for 12 h and then rinsed with acetone and *n*-hexane. Neutral silica gel (80–100 mesh) and anhydrous sodium sulfate were cleaned with dichloromethane (DCM) using MX-Soxhlet extractor for 24 h and then baked at 450 °C for 12 h. Silica gel was deactivated with 10% (w:w) of precleaned deionized water (extracted with DCM).

2.2. Samples collection

The Pearl River is the largest and most complex water system in the South China. It has a catchment area of $>450,000 \text{ km}^2$, with an annual mean runoff volume of 326 billion m^3 . The river water finally enters the SCS via 8 major outlets that include Humen (HM), Jiaomen (JM), Hongqilimen (HQ), Hengmen (HE) on the eastern side, and Modaomen (MD), Jitimen (JT), Hutiaomen (HT), and Yamen (YM) on the western side (Fig. 1). Detailed information of the 8 outlets has been previously described (Xu et al., 2013). Surface sediment samples ($\leq 10 \text{ cm}$) from the 8 outlets were collected in 2010 using a stainless steel grab. A total of 16 sediment samples in the PRE and 6 sediment samples from 2 fishing harbors (Shenzhen and Zhuhai) were also collected during the same period. Detailed information on sampling locations is shown in Fig. 1. All the samples were placed into polytetrafluoroethylene bags and immediately stored at $-20 \text{ }^\circ\text{C}$.

2.3. Samples preparation and extraction

The collected sediment samples were freeze-dried at $-20 \text{ }^\circ\text{C}$ and ground into powder using an agate pestle and mortar. Briefly, the sediment samples (10.0 g) were extracted with DCM for 16 h at a flow rate of 5 mL min^{-1} using MX-Soxhlet extractor. Before extraction, $10 \mu\text{L}$ of 500 $\text{pg } \mu\text{L}^{-1}$ surrogate standards (d_3 -AHTN) was added to monitor the recovery and approximately 1.0 g of activated copper granules was added to remove elemental sulfur. Extracts were evaporated down to 1–2 mL using hexane as keeper and further cleaned on a silica column (2.5 g, 10% water deactivated) topped with 3 g anhydrous granulated sodium sulfate. The extract was then purified by eluting with 15 mL hexane followed by 20 mL DCM/acetone (1:1, V/V). The final solution was concentrated down to 200 μL by roto-evaporation and nitrogen blower.

2.4. Compounds analysis

The samples were analyzed with an Agilent 6890N gas chromatography coupled to an Agilent 5973 mass spectrometer (GC-MS) (Agilent Technologies, Avondale, PA, USA), operating in electron impact and selective ion monitoring modes (SIM). An HP-5MS capillary column (J&W Scientific, 30 m \times 250 μm i.d., 0.25 μm film thickness) was employed. The transfer line and the ion source temperature were maintained at 280 °C and 230 °C, respectively. The temperature of the oven was initiated at 60 °C for 1.0 min, increased to 180 °C at a rate of $30 \text{ }^\circ\text{C min}^{-1}$, and ramped at $2 \text{ }^\circ\text{C min}^{-1}$ to 300 °C and held for 10 min. The flow rate of the carrier gas helium was kept constant at 1.3 mL min^{-1} . The extracts (1.0 μL) were injected onto GC-MS in pulsed splitless mode with an inlet temperature program as following: 60 °C for 0.1 min, increased to 280 °C at a rate of $300 \text{ }^\circ\text{C min}^{-1}$ and held for 10 min. Quantitation was performed using the internal calibration method based on a 9-point calibration curve.

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