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Distribution, mass inventories, and ecological risk assessment of legacy and emerging contaminants in sediments from the Pearl River Estuary in China

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HIGHLIGHTS

- Surfactants are the most predominant contaminants in PRE sediments followed by PAHs.
- Highest concentrations were found near Guangzhou city and Macao.
- Wastewater discharges are a major source of contamination, except for UV filters.
- Triisobutylphosphate and 2-ethylxheyldiphenylphospate are detected for the first time.
- Legacy contaminants still pose a significant risk for the benthic communities at PRE.

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ABSTRACT

This study focused on comparing the occurrences and environmental toxic risks for diverse priority and emerging contaminants (>100 chemicals) in the sediments from the Pearl River Estuary (PRE, China). The most predominant compounds were cationic surfactants, organophosphate flame retardants (e.g., triisobutylphosphate), and polycyclic aromatic hydrocarbons (PAHs), accounting for >75% of the total mass inventory (~330 metric tons). Wastewater discharges seem to be one of the main sources of pollution in the area, as the highest concentrations (>1000 ng g^{-1} for some chemicals) were reported in the upper part of the PRE (near Guangzhou city) and Macau. Highest levels of ultraviolet (UV) filters, however, were observed in recreational areas, revealing the importance of direct sources (e.g., outdoor activities). An environmental risk assessment showed that PAHs and dichlorodiphenyl dichloroethylene had the highest hazard quotient (HQ) values (up to 233). Nonylphenol, a metabolite from nonionic surfactant, and two UV filters (2-ethyl-hexyl-4-trimethoxycinnamate and 4-methylbenzylidene camphor) also posed a significant threat to benthic species (HQ > 1). Further research through the realization of monitoring campaigns and toxicity tests is encouraged, as the exposure of the resident aquatic organisms and human population to these and other emerging chemicals is expected to increase over the years.

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1. Introduction

Since the second half of the 20th Century, the synthesis of new chemicals has experienced an exponential growth. Negative effects of some synthetic chemicals on the environment and human health became apparent soon after their widespread release into the environment during the 1960–70s. This resulted in contaminant phase-outs or restrictive legal actions in the case of some compounds such as polychlorinated biphenyls (PCBs) or many organochlorinated pesticides (e.g., DDT), now known as legacy contaminants that are still detected in the environment because of their high persistence. Some of these anthropogenic chemicals may adversely affect the biota even at very low concentrations, e. g, endocrine disrupting compounds (EDCs) such as nonylphenol (NP) and polybrominated diphenyl ethers (PBDEs) that can act through the receptor mediated responses. In recent years, there

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has been a significant concern focused on new contaminants called emerging pollutants [1]; however, unfortunately, the same analytical advances that have allowed for their low-level detection, neither offer knowledge about whether a newly detected contaminant is of eco/toxicological interest nor their environmental behavior and fate. Therefore, assessing the occurrence, distribution, reactivity, and effects of these chemicals in the environment remains major time- and resource-intensive challenges.

Ecosystems of urbanized estuaries are directly threatened by organic contaminants due to direct and in-direct inputs derived from enhanced human activity. More specifically, Pearl River Estuary (PRE) located in southern China (Fig. 1), one of the most urbanized estuaries heavily affected by the rapid economic (GDP > 400 million USD) and population growth (>80 million inhabitants). The concentrations of organic contaminants in this aquatic system have been extensively monitored over the last decades through a series of sampling campaigns, and most of them aimed to the screening of persistent organic pollutants (POPs). Thus, the presence of PCBs and PBDEs has been confirmed in the air [2], sediment [3,4], soils [5], and biota [6]. The annual release of PBDEs in wastewater was estimated at 2280 kg/year by measuring their concentrations in the sewage effluents discharged to the PRE [7]. Previous results showed that the levels of PAHs, organochlorine pesticides (OCPs), and halogenated flame retardants and their composition patterns were well preserved in the sediments, and their chronological records could reflect the evolution of the pollution inputs, because of the rapid growth of population and the development of manufacturing industries in the PRE [4,8–10]. Additionally, the use of geochemical markers such as linear alkylbenzenes (LABs) [11] and quaternary ammonium compounds (QACs) [12] has confirmed the effect of municipal sewage pollution in the sediments of the PRE and South China Sea (SCS). High correlation coefficients were found among these markers and the POPs in the Pearl River, indicating that they had similar sources, and both were associated with sewage-derived particles. The decrease in the concentrations of contaminants with increasing distance from the PRE towards the ocean also suggests advective transport. Chen and coworkers [13] estimated that the riverine input influence reached approximately between 124 and 276 km from the end of the estuary. To better evaluate the role of sediments as a sink for a significant fraction of organic contaminants and their potential risk as secondary sources, mass inventories in the PRE have been also calculated for PAHs (126 tons) and OCPs (0.4 tons) in the PRE sediments [14], as well as PBDEs in the PRE sediments [9] and soils [5].

Regarding emerging contaminants, most of the research in China has been focused on pharmaceuticals and related compounds (PhACs). Recently, Bu et al. [15] reviewed PhACs and identified six compounds (three antibiotics and three anti-inflammatories) as priority contaminants in waters [15]. However, there remains a paucity of information on the relative importance of these and other emerging sewage-derived organic contaminants (e.g., personal care products, PCPs) in sewage-affected sediments. There are only a few studies on the distribution of polycyclic fragrances [16], triclosan [17], and other EDCs [18,19], and most of them focused in the upper part of the PRE (and adjacent rivers) and their removal efficiencies in local wastewater treatment plants (WWTPs). Two notable exceptions are very recent data on perfluorinated surfactants [20] and endocrine disrupting compounds (EDCs) [21] from the two surveys in marine areas. The rapid evolution of production and usage for newly introduced organic chemicals and the lack of data on their occurrence in the PRE contrast with the extensively studied distribution of legacy contaminants in this area. Monitoring both classes together has not been performed until now and it would allow for a direct comparison between their levels and their relative risk to aquatic biota trough environmental risk assessments for those chemicals for which ecotoxicological data is available

Therefore, the main goal of the study was to establish the first comprehensive and comparative study on the current status of legacy and emerging chemicals in the PRE region. More specifically, we aimed to: a) evaluate the occurrence and distribution of diverse particle-reactive organic contaminants in surface sediments, b) calculate and compare their mass inventories to establish their relative abundance, and c) perform a preliminary environmental risk assessment for selected chemicals with preexisting ecotoxicological information in PRE sediments. To achieve these objectives, we have covered a large area (3295 km^2, 31 sampling stations) and screened over one hundred relatively highly hydrophobic compounds ($\log K_{ow}$ >4) including hydrocarbons (16 PAHs), flame retardants (five PCB congeners and nine recently introduced organophosphate compounds), pesticides (18 organochlorines, five organophosphorus, eight triazines, and seven pyrethroids), surfactants (NP and the homologous series of two major QAC groups), and PCPs (13 fragrances, 10 UV filters, and two antimicrobials).

2. Experimental

2.1. Study area and sampling

The PRE is one of the most urbanized areas in China, because of the rapid economic and population growth in the region. It receives 14,000 kilotons of wastewater per day from the major cities such as Guangzhou, Foshan, and Dongguan (Environmental Status Bulletins of Guangdong Province, China; http://www.gdepb.gov.cn/). The organic contaminants are generated by the industries, agriculture, and electronic waste (E-waste) recycling areas [22]. In this study, 31 surface sediment samples were collected from the PRE region and the adjacent northern SCS during two cruises in July and December 2012 (Fig. 1). Top 5 cm sediment samples were collected using a Van Veen stainless steel grab sampler. Each sediment sample was stored in a glass bottle pre-baked at 450 °C for 8 h and stored at −20 °C until further treatment. All the samples were then freeze-dried in the lab and homogenized prior to the analysis of different organic contaminants. Specific details on each sampling station (location, total organic carbon content, and concentration of target compounds) are listed in Table 1S in the Supporting Information.

2.2. Chemicals, sample extraction, purification, and analysis

The extraction and determination of QACs were carried out using a previous methodology [12] based on ultra-performance liquid chromatography coupled to tandem mass spectrometry (UPLC–MS/MS). The rest of target compounds are susceptible for gas chromatography–tandem mass spectrometry (GC–MS/MS) analysis and were determined following another methodology developed by our group [23]. More information on this can be found in these references and in the Supporting Information.

2.3. Data analysis

Ordinary Kriging interpolation analysis based on the spherical model was used to predict and visualize the spatial distribution of the data in the PRE region by using ArcGIS 10 software. Further information on this approach has been reported by Chen et al. [24], where they plotted the distribution maps of PBDEs in the sediments from the same sampling area. We also used this interpolation analysis to calculate the mass inventories of all the contaminants detected in the sampling area using a modification of the approach followed by Chen et al. [14]. Briefly, for the Kriging analysis, the PRE region area plotted in Fig. 1S (Supporting Information) was

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