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RESEARCH ARTICLE

Phthalate esters distribution in coastal mariculture of Hong Kong, China

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Abstract

The aim of the study is to evaluate the impact of mariculture on phthalate esters speciation and distribution in sediments and cultured fish in the Hong Kong regions and near mainland China. Concentrations of ∑phthalate esters in mariculture surface sediments (0 to 5 cm) ranged from 0.20 to 54.3 mg/kg dw (mean 10.3 mg/kg dw), with the highest recorded at M2 (20.4 mg/kg dw). Concentrations of phthalate esters were not significantly ($p > 0.05$) enriched in surface and sediment cores at mariculture sites relative to the reference sediments, 1 to 2 km away in areas without mariculture activities. Among different congeners, only butyl benzyl phthalate (BBP) concentrations demonstrated a significant correlation ($R^2 = 0.40$, $p < 0.05$) with TOC values of sediments. The median concentrations of di-2-ethylhexyl phthalate (DEHP) and di-n-butyl phthalate (DBP) in the sediments were 1.57 and 6.96 times higher than the environmental risk levels (ERL), which may pose environmental risks. Results of health risk assessments revealed that the cultured fish (snubnose pompano, orange-spotted grouper, and red snapper) were safe for consumption, in terms of phthalate esters. This is the first study to assess the differences of phthalate esters contamination between mariculture and natural coastal sediments.

Keywords Mariculture · Sediment · Cultured fish · Phthalate esters · Risk assessment

Introduction

Phthalate esters (PAEs) are a class of synthetic compounds mainly utilized in the manufacture of plastics as non-reactive plasticizers to increase the flexibility and durability of high molecular weight polymers, such as polyvinyl chloride (PVC) (Hu et al. 2016; Liu et al. 2014). PAEs have also been widely used in many chemical industries to improve product quality or performance, such as pesticides, personal care products, pharmaceuticals, cleaning materials, lubricants, adhesives, and building materials (Hauser & Calafat 2005; Li et al. 2017b; Mackintosh et al. 2004). Vast consumption and widespread use of PAE-containing products lead to their ubiquitous existence in various environmental matrices, such as in air and air particulate matter (Pei et al. 2013; Wang et al. 2008), water (Li et al. 2017b; Li et al. 2015; Selvaraj et al. 2015), soil (Niu et al. 2014; Skrbic et al. 2016), sediments (Li et al. 2017b; Mohammadian et al. 2016; Wang et al. 2014b), and the tissues and fluids of wildlife and humans (Adeogun et al. 2015; Morgenstern et al. 2017). As the endocrine disrupting chemicals (EDCs) that long-term exposure to PAEs can adversely affect reproduction, impair development,

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and induce genetic aberrations in wildlife, even at low concentrations (Charles et al. 1997; Franco et al. 2007).

Due to the fact that they are not chemically bonded to the polymeric matrix, phthalate esters can enter into the aquatic environment through manufacturing processes and final products, through leaching, migration, or evaporation, from runoffs of storm water, and discharge of domestic and industrial effluent (Liu et al. 2014; Marx 1972; Mayer et al. 1972). After being released into aquatic environment, their distribution is mainly governed by their physical–chemical characteristics and natural degradation (Kickham et al. 2012; Staples et al. 1997). Due to their strong hydrophobicity, phthalate esters are accumulated in sediment and biota (Chen et al. 2012). The organic matter in sediments can regulate their adsorption (Cornelissen et al. 2005). Xu and Li (2008) observed that adsorption capacity of phthalate esters increased according to the increase of sediment organic contents. The inherently biodegradable substances of phthalate esters at high levels of adsorption can possess a longer half-life in the sediment (Kickham et al. 2012). Additionally, their degradation in sediments under anaerobic were 3–10 times higher than those under aerobic conditions (Chang et al. 2005; Yuan et al. 2002). Sediments contained higher levels of phthalate esters would exert toxic effects and enhance accumulation in aquatic organisms (Mackintosh et al. 2004). However, there is less information about phthalate esters in the mariculture systems of coastal area (Mohammadian et al. 2016).

In coastal area, especially bay and port, receives considerable amounts of chemical substances from the land via surface runoff, sewage discharge, and atmospheric deposition, posing a severe threat to the aquatic environment (Li et al. 2017a; Mohammadian et al. 2016). In addition, plastic debris and marine transportation also constitute a major threat to marine systems (Liang et al. 2011). Chinese mariculture developed rapidly from the 1990s, and China is now the largest fishery country in the world, in terms of overall seafood production volume (Cao et al. 2007). In China, most mariculture activities are practiced in shallow inshore sheltered bays (Li et al. 2017a). However, there has been public concern about the potentially negative impacts on the ambient aquatic environment by these activities (Gao et al. 2005; Yokoyama et al. 2006). The uncontrolled released of nutrients, suspended solids, and organic matters into water and sediment is one of the most negative effects of mariculture commonly encountered in Asian countries (Cornelissen et al. 2005; Yokoyama et al. 2006). Previous studies recorded that copper (Cu), zinc (Zn), lead (Pb), cadmium (Cd), polychlorinated biphenyls (PCBs), organochlorine pesticides (OCPs), and polycyclic aromatic hydrocarbons (PAHs) were enriched in sediments near mariculture sites (Gu et al. 2008, 2013, 2016). Concentrations of PAHs and OCPs in sediments under mariculture rafts were substantially higher than that of sediments without the activities. It has been further indicated that fish feeds are likely to be the major source of the enriched pollutants in the mariculture sediments (Wang et al. 2010, 2014a). Therefore, food safety has raised public health concern regarding marine fish fed with contaminated feeds.

The main objectives of this investigation were to (1) characterize the concentration profile of phthalate esters in surface sediment beneath mariculture rafts of Hong Kong and adjacent areas near mainland China; (2) compare the concentrations of phthalate esters in mariculture sediments and their corresponding non-mariculture sediments; (3) assess their ecological risks in mariculture sediments; and (4) to evaluate the potential human health risks, based on their levels in cultured fish.

Materials and methods

Sampling

Samples of fish and associated sediments were collected from six mariculture sites in the regions of Hong Kong and areas near mainland China including Tsing Yi (H1), Sam Mum Tsai (H2), Sai Kung (H3), Tung Lung Chau (H4), Xi Xiang (M1), and Mirs Bay (M2) (Fig. 1). And the corresponding sites were about 1–2 km away from the mariculture rafts as the non-mariculture zones. Three random sediment samples (0–5 cm) were obtained from each site (beneath cages) with the use of a stainless steel grab sampler, respectively. Three farmed fish species were collected including Red snapper (*Lutjanus campechanus*) ($n = 26$), orange-spotted grouper (*Epinephelus coioides*) ($n = 26$), and snubnose pompano (*Trachinotus blochii*) ($n = 17$) which the main species cultured in in Hong Kong. Each sample was wrapped in aluminum foil, delivered to the laboratory, storing at − 20 °C before further analyses.

Chemical analyses

Preparation and measurements of phthalate esters in sediment and fish were conducted (Cheng et al. 2013). Details about the analysis of Phthalate esters, QA/QC and calculation are described in Supplementary Materials.

Calculation

Risks of exposure to phthalate esters of local citizens through dietary intake of the cultured fish were examined based on the suggested guidelines by the USEPA (2000). For non-carcinogenic risks, comparison was made between

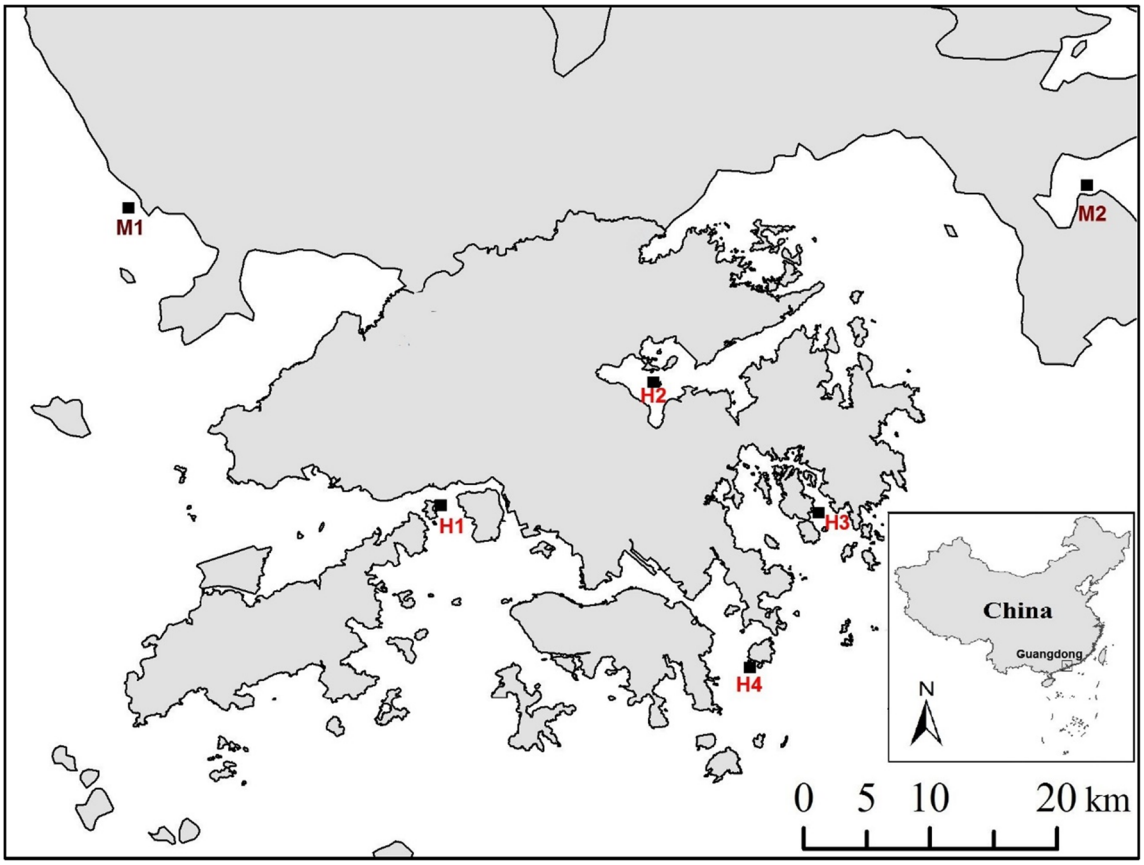

Fig. 1 Map of sampling sites. Mainland China: *M1* Xixiang, *M2* Mirs Bay; Hong Kong: *H1* Tsing yi, *H2* Sam Mun Tsai, *H3* Sai Kung, *H4* Tung Lung Chau

the estimated daily intake and the recommended reference doses (RfD) (BBP 200; DBP 100; and DEHP 20 ng/g bw/day) (USEPA 2014) as stated in Eq. (1):

$$\text{Hazard ratio (HR)} = \text{EDI}/\text{RfD} \quad (1)$$

$$\text{EDI} = \text{C} \times \text{DR}/\text{BW} \quad (2)$$

where EDI is the estimated daily intake, DR daily consumption rate (kg/person/day), *C* the concentration in a given fish species (mg/kg, ww), and BW average body weight (kg). Average body weights used were 58.6 kg (Wang et al. 2005) and 21.8 kg (Leung et al. 2000), and DR 93 and 50 g/day for adults and children, respectively (Leung et al. 2000). When HR is greater than 1, it indicates there is a potential health risk. With regard to carcinogenic risks, the cancer risk (CR) was derived from the oral slope factor (OSF) of phthalate esters (USEPA 2000):

$$\text{CR} = \text{EDI} \times \text{OSF} \quad (3)$$

In our present research, only DEHP has the OSF value (14 μg/kg/day) (USEPA 2014).

Data analyses

Each statistical test was conducted using SPSS 19.0 for Windows. Normality was ractified by the Kolmogorov–Smirnov test, and homogeneity of variances affirmed by the Levene test. Analysis of the data of phthalate esters concentrations was performed by two independent *t* tests, Wilcoxon rank sum test, one-way ANOVA, and Duncan's multiple range test ($p < 0.05$).

Results and discussion

Phthalate esters concentrations in sediments

Figure 2 shows phthalate ester concentrations in both types of sediments (mariculture and non-mariculture) of each site. Total concentrations in mariculture sediments ranged from 0.20 to 54.3 mg/kg dw (mean 10.3 mg/kg dw) while non-mariculture sediments ranged from 0.39 to 98.8 mg/kg dw (mean 14.8 mg/kg dw), without significant ($p > 0.05$) difference between mariculture sediments and non-mariculture sediments. The concentrations of this study matched those from

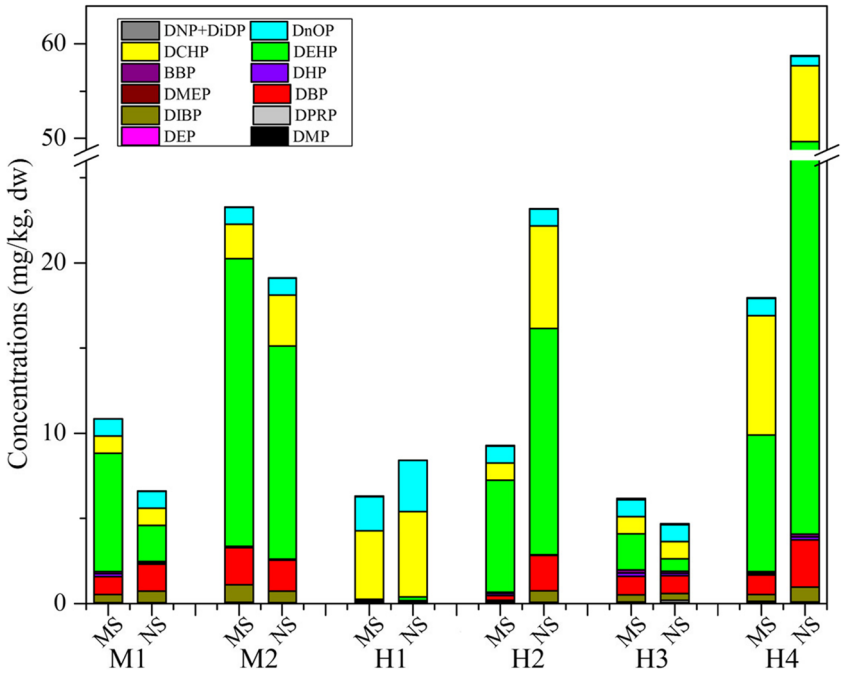

Fig. 2 Concentrations of phthalate esters of surface sediments in mariculture and non-mariculture. MS: mariculture sediments; NS: non-mariculture sediments; M1; Xixiang; M2: Mirs Bay; H1: Tsing yi; H2: Sam Mun Tsai; H3: Sai Kung; H4: Tung Lung Chau

Kaohsiung Harbor (0.40 to 34.8 mg/kg dw), Taiwan (Chen et al. 2013) and Pearl River (2.76 to 47.3 mg/kg dw), and the mainland China (Liu et al. 2014), but higher than that from False Creek Harbor (0.004 to 2.10 mg/kg dw), Canada (Mackintosh et al. 2006). The non-mariculture sediment of H4, with the highest concentration (50.0 mg/kg dw), was located at Tung Lung Chau, a largely uninhabited island. However, the traffic around the area was busy, and the main sources of phthalate esters could be derived from cargo vessels and cruise ships (Wang et al. 2010). The mariculture sediment from M2 (20.4 mg/kg dw) possessed higher concentrations compared with those from other sites. M2 site located at Mirs Bay, a major mariculture area of in Guangdong province, China, and next to the economic district of the Pearl River Delta. Due to the semi-enclosed situation of the bay, the water exchange in Mirs Bay is poor, and anthropogenic activities and development of mariculture fishery further accelerated deterioration of the environment (Yu et al. 2007). In general, the results indicated that the high concentrations of phthalate esters are ubiquitous in sediments of the regions but mariculture does not seem to significantly contribute to the concentrations.

The chemical composition of PAEs in sediments

The detection frequency was noticeably different among the different sampling sites and phthalates. Thirteen congeners of phthalate esters were identified (Fig. 2), with DEHP being the dominating compounds in all the samples, accounting for 28.3 to 90.2%, with a median of 78.2%. The predominance of DEHP was also observed in other marine (Chen et al. 2013; Hassanzadeh et al. 2014) and river sediments (Liu et al. 2014). DEHP is mainly applied as plasticizers and added in a broad types of products (e.g., polyvinyl chloride) to enhance flexibility and practicability (Gómez-Hens and Aguilar-Caballos 2003). As DEHP has a relatively low vapor pressure (Staples et al. 1997), it could be commonly found in the environment (Kong et al. 2012; Liu et al. 2014; Peijnenburg and Struijs 2006).

In view of coastal sediment being the habitat for aquatic organisms as well as the source of pollutants, the environmental risk levels (ERL) could be used as a useful tool to evaluate the environmental safety of surface mariculture (and reference sediments) in posing potential risks to the neighboring aquatic environment and corresponding biota and the ERLs determined for DBP and DEHP were 0.7 and 1 mg/kg, respectively, according to the data of ecotoxicology and environmental chemistry of the ecosystem (van Wezel et al. 2000). The median concentrations of DEHP (6.96 mg/kg) and DBP (1.10 mg/kg) of the present study in all mariculture and reference surface sediments exceeded the ERL (except H1 site). In general, the median concentrations of DEHP and DBP in sediments were 1.57 and 6.96 times higher than the specified ERL. These findings indicated that DEHP and DBP would cause potential harm to the marine environment.

Relationships between PAEs and sediment geochemical parameters

Table S1 showed the correlations between the phthalate ester concentrations and the geochemical parameters in surface sediments. No significant correlations ($p > 0.05$) were found

among $\sum_{13}$PAEs concentrations with total organic carbon (TOC), nitrogen, and sulfur element contents as well as particle size and pH values. All the congeners were also no correlations with TOC, nitrogen, particle size, and pH values, expect the concentrations of BBP demonstrated a significant correlation ($R^2 = 0.40$, $p < 0.05$) with TOC values of sediments. Previous study showed the content of organic matter is crucial in controlling the adsorption of chemical contaminants onto sediments (Cornelissen et al. 2005). The TOC of sediments have been also found an important factor in governing the adsorption of the contaminants onto sediments (Huang et al. 2008; Zeng et al. 2008). The present results showed a different phenomenon might be caused by the influence of mariculture activities. Because of the attachment of carboxylic groups, aromatic domains non-hydrolyzable carbon in sediment could inhibit sorption of phthalate esters, while facilitated elevated sorption of PAHs (Jin et al. 2015). The uneaten feed and feces from fish cage caused the accumulation of organic matter in mariculture sediments, and previous study observed the PAHs concentrations in mariculture sediments were higher than that in the sediments from the reference sites (Wang et al. 2010). Moreover, no significant correlations were noted between phthalate ester concentrations and the geochemical parameters, indicating that their concentrations in mariculture sediments may also depend on other factors, such as characteristics of the water body, transport, mixing, and mechanism of sedimentation, source compositions, input flows, and the physical and social characteristics of the coastline region (Chen et al. 2013; Liu et al. 2014; Zeng et al. 2008).

Phthalate esters concentrations in cultured fish

Table 1 lists mean values of phthalate esters in the muscle of three fish species. The concentrations detected in fish samples ranged from 0.25 to 3.93 mg/kg ww (average 1.38 mg/kg ww), with snubnose pompano (2.01 ± 0.68 mg/kg ww) > orange-spotted grouper (1.38 ± 0.44) > red snapper (0.89 ± 0.49). The concentrations measured in fish muscle of the present study were lower than those collected from the market in Hong Kong (3.33 to 23.7 mg/kg dw) (Cheng et al. 2013), farmed ponds in the mainland China (1.87–52.4 mg/kg mg/kg dw) (Chen et al. 2012), and the estuary in Vancouver, Canada (mean 27.0 mg/kg dw) (Mackintosh et al. 2004).

Similarly, the same 13 congeners of phthalate esters were found in fish samples (Table 1), with both DBP and DEHP the predominance congeners, accounting for 9.09 to 57.4% and 11.4 to 52.1%, respectively. The predominant DEHP and DBP in freshwater and marine fish was also observed in various studies (Adeniyi et al. 2011; Cheng et al. 2013; Mackintosh et al. 2004). DBP is commonly added into personal care goods (perfumes, lotions, cosmetics etc.), while DEHP largely applied as plasticizers in manufacturing of polymer. Therefore,

Table 1 Phthalate ester concentrations (mg/kg, ww) in different fish tissues

Location	Name	*n*	DMP	DEP	DPRP	DIBP	DBP	DMEP	DHP	BBP	DEHP	DCHP	DnOP	DNP + DiDP	Total	Lipid (%)
M1	Snubnose pompano	6	< 0.01	< 0.01	< 0.01	0.23	0.47	< 0.01	< 0.01	< 0.01	0.53	0.04	nd	0.02	1.30	39.8
	Orange-spotted grouper	6	nd	0.01	< 0.01	0.50	1.01	< 0.01	0.01	0.01	0.20	0.02	< 0.01	< 0.01	1.76	18.2
	Red snapper	6	< 0.01	< 0.01	nd	0.01	0.04	0.01	nd	0.04	0.11	0.10	0.11	0.01	0.44	23.3
M2	Snubnose pompano	5	< 0.01	0.02	0.02	0.38	0.70	0.01	< 0.01	0.04	0.76	0.08	< 0.01	0.05	2.06	23.6
	Orange-spotted grouper	6	< 0.01	0.01	0.01	0.29	0.73	0.01	< 0.01	0.02	0.23	0.04	< 0.01	< 0.01	1.34	23.7
	Red snapper	6	< 0.01	0.01	nd	0.26	0.61	< 0.01	< 0.01	0.02	0.15	0.03	0.05	< 0.01	1.15	21.1
H1	Orange-spotted grouper	6	< 0.01	0.01	0.02	0.37	0.79	< 0.01	< 0.01	0.05	0.30	0.08	< 0.01	0.06	1.69	9.54
	Red snapper	4	< 0.01	< 0.01	0.02	0.01	0.03	< 0.01	< 0.01	0.06	0.17	0.15	0.03	0.01	0.50	18.0
H2	Orange-spotted grouper	6	nd	0.01	< 0.01	0.21	0.60	0.01	< 0.01	0.01	0.15	0.02	0.01	0.01	1.04	14.6
H3	Orange-spotted grouper	4	< 0.01	0.01	0.01	0.12	0.28	0.026	nd	0.03	0.11	0.06	nd	0.02	0.65	5.40
	Red snapper	4	< 0.01	< 0.01	0.02	0.04	0.06	0.02	< 0.01	0.04	0.38	0.10	0.08	0.01	0.73	9.74
H4	Snubnose pompano	6	< 0.01	0.01	< 0.01	0.47	0.90	< 0.01	0.03	0.07	1.05	0.11	nd	0.02	2.66	23.3
	Orange-spotted grouper	4	nd	0.01	< 0.01	0.37	0.80	< 0.01	< 0.01	0.02	0.23	0.02	< 0.01	< 0.01	1.47	13.2
	Red snapper	6	< 0.01	0.01	nd	0.46	0.91	< 0.01	0.01	< 0.01	0.19	0.02	0.01	0.01	1.61	9.68

Note: nd: not detected; M1: Xixiang; M2: Mirs Bay; H1: Tsing yi; H2: Sam Mun Tsai; H3: Sai Kung; H4: Tung Lung Chau

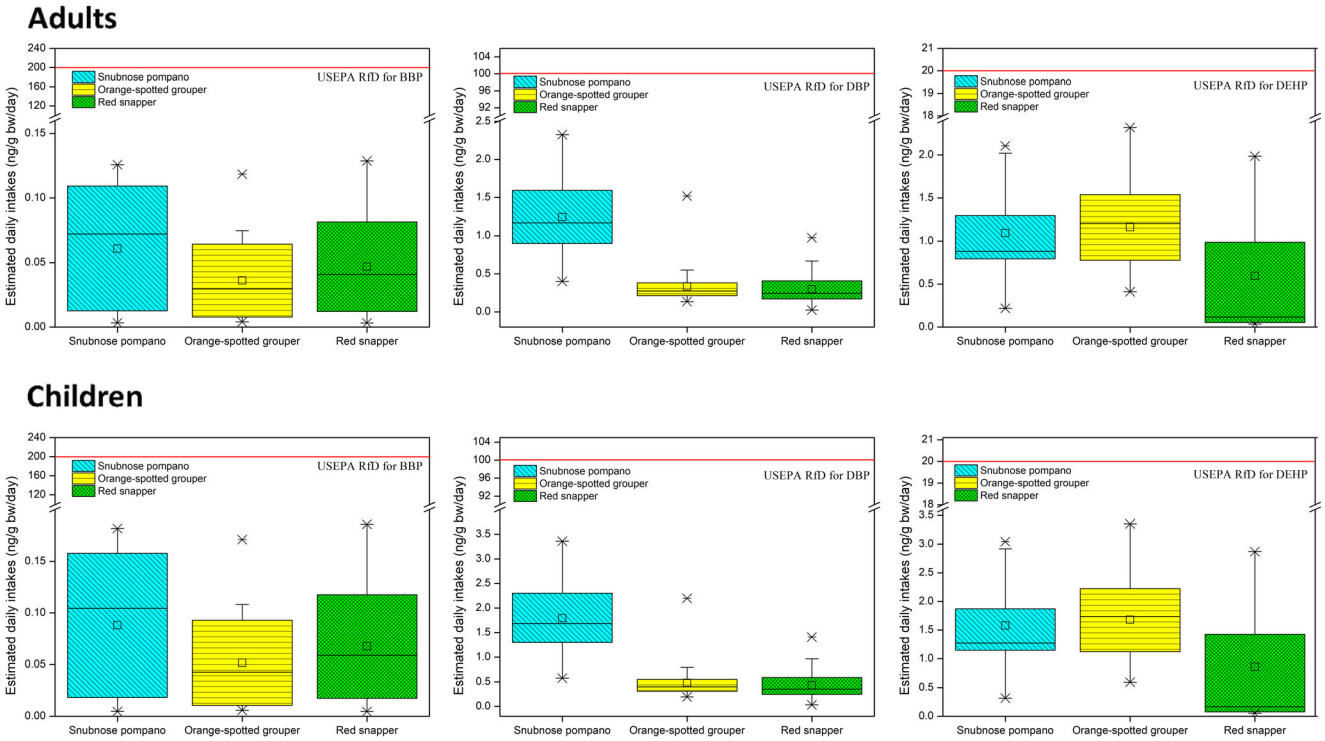

Fig. 3 Estimated daily intakes of DBP, BBP, and DEHP through mariculture fish consumption by adults and children in Hong Kong. RfD: reference dose (BBP: 200 ng/g bw/day; DBP: 100 ng/g bw/day; and DEHP: 20 ng/g bw/day (USEPA 2014)). Each box represents interquartile range (25th and 75th percentile) of estimated daily intakes

both DBP and DEHP have been extensively recorded in aquatic environments (Hassanzadeh et al. 2014; Sun et al. 2013). The entry of DBP and DEHP to fish is from water through gill and skin and consumption of contaminated food and sediment. DEHP concentrations in the three studied fish species ranged from 0.01 to 1.46 mg/kg ww, (median 0.19 mg/kg ww), which were slightly lower than those collected from Netherlands (Peijnenburg and Struijs 2006), but about 10–30 times below the values collected from Taiwan (13.6 to 70.0 μg/g dw) (Huang et al. 2008). DBP

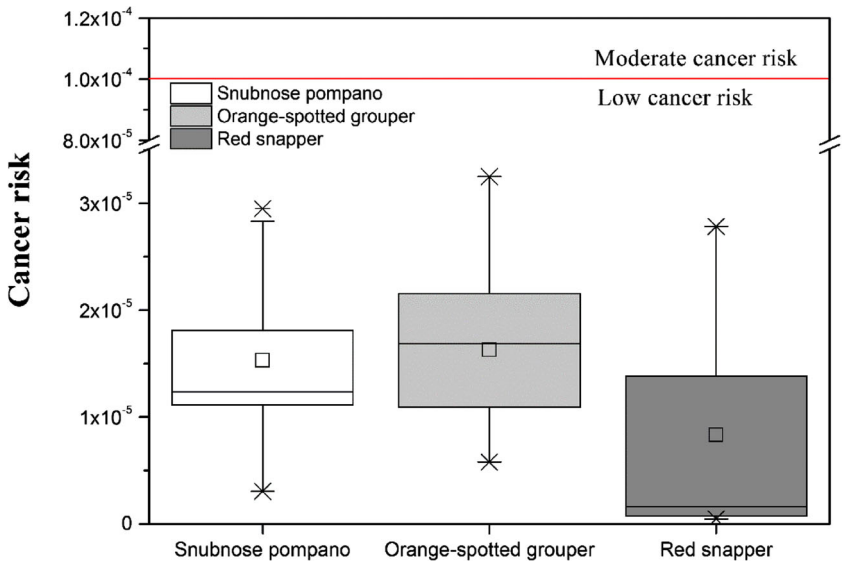

Fig. 4 Cancer risks of DEHP through mariculture fish consumption by adults and children in Hong Kong. Each box represents interquartile range (25th and 75th percentile) of estimated daily intakes. USEPA (2014)

concentration (from 0.02 to 1.46 mg/kg ww, median 0.55 mg/kg ww) detected in our present study was comparable to the concentration found in Nigeria, and Taiwan (Adeniyi et al. 2011; Huang et al. 2008).

Health risk assessment

Organic pollutants like OCPs, and phthalate esters in human body usually show a positive correlation with the intake of fish, which accounted for the majority of total dietary intakes (Chen et al. 2014; Wang et al. 2011, 2013). Dietary protein source for the majority of Hong Kong residents is mainly relied on aquatic products. Figures 3 and 4 show the EDI and CR of phthalate esters through intake of the three fish species by Hong Kong adults and children. An HR greater than 1 indicates that the EDI surpassed the contaminant RfD and potential occurrence of systemic effect. No HR values are found to be over 1 via consumption of all three fish species, indicating non-cancer risk due to intake of phthalate esters through intake of fish is unlikely for both adults and children. According to the USEPA (1989), a risk value surpassing 10^{-6} is an acceptable risk for cancer when assessing the lifetime excess CR of DEHP. The CR values of the three fish species were above 10^{-6}, which were lower than the upper limit of the acceptable risk levels (10^{-4}) (USEPA 2014). Therefore, consumption of the three cultured fish (snubnose pompano, orange-spotted grouper, and red snapper) collected from Hong Kong and adjacent areas were safe, in terms of phthalate esters.

Conclusion

The present study evaluated the spatial distribution and profile of mariculture phthalate esters in coastal sediments and fish in Hong Kong regions and near mainland China. No significant ($p > 0.05$) difference was observed between mariculture and non-mariculture sediments. Results reflected that mariculture does not significantly affect the concentrations of phthalates in sediments of this region. Based on the ecological risk assessment, there was a possible hazard for DEHP and DBP enriched in the sediments to contaminate the neighboring marine environment. Phthalate ester concentrations and the geochemical parameters have no significant correlations, which were not in consistent with previous study. Further studies are needed to explore the role of specific factors within the sediments, water body, and others. Concentrations of phthalate esters detected in fish muscle were lower or close to those of fish from other countries and areas reported by previous studies. The outcome of health risk assessment suggested that the three cultured fish species collected from Hong Kong and adjacent areas were safe for human consumption, in the light of phthalate esters.

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