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From inner-bay preservation to offshore dissipation: Spatiotemporal patterns and tracing of pharmaceuticals in Shenzhen Bay of the greater bay area, South China

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ABSTRACT

Pharmaceutical active compounds (PhACs) are increasingly recognized as contaminants of emerging concern, but their spatiotemporal accumulation patterns in coastal environments remain insufficiently understood. Here, we analyzed 24 PhACs in surface sediments and down cores collected from Shenzhen Bay to trace their residual fate along land-to-sea transport pathways. PhACs exhibited decreasing concentrations from the estuary to offshore and were continuously buried into deeper sediment layers, reaching up to 199.31 ng g^{-1} dry weight. Along the transport pathway, degradation processes affecting multiple PhACs were observed in addition to dilution. We fractionated the surface sediments by grain size for PhAC analysis and found that PhACs were preferentially enriched in the coarse fraction (>20 μm). Based on the observed grain-size patterns and supporting evidence from previous studies, we propose a conceptual framework for the residual accumulation of PhACs in coastal bays. Furthermore, we conducted ^{210}Pb dating of the sediment cores and found that vertical PhAC trends can reflect documented local socio-economic transitions (e.g., population growth and aquaculture). Finally, we established the sedimentary fluxes of the 24 target PhACs and estimated that the cumulative sedimentary inventory of these compounds in the inner bay reached approximately 13.51 ± 4.05 tons over the past 60 years.

1. Introduction

Pharmaceutical active compounds (PhACs) are deliberately designed for high bioactivity widely used in the treatment of human and veterinary diseases. The continuous discharge and widespread distribution have caused them to form pseudo-persistent contamination in the aquatic environment (Bu et al., 2016), classified as emerging organic pollutants (EOPs) (Chaturvedi et al., 2021; Noguera-Oviedo and Aga, 2016). This "pseudo-persistent" arises because the half-life of PhACs in water typically ranges from a few days to months due to a rapid degradation (e.g., hydrolysis or photolysis), and their contamination alleviates once the input decreases (Yu et al., 2024). However, once PhACs are adsorbed and further deposit in sediments, they are not as easily removed as expected, with retention periods even spanning decades (Tamtam et al., 2011; Lara-Martín et al., 2015; Kerrigan et al., 2018). For instance, the half-life of oxytetracycline in well-lit aquatic

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environments is less than five days, whereas it can extend to over two months when covered by just 4 cm of sediment (Samuelsen, 1989). Moreover, as PhACs continuously settle from the water column into sediments and reach deeper, oxygen-depleted layers, their retention time can exceed 50 years (Tamtam et al., 2011). Consequently, PhAC-contaminated sediments will pose a long-term threat to benthic ecosystems, and may serve as a pollution source to overlying waters even after water quality recovery.

The marine sediment is an important sink for pollutants (Leppanen et al., 2024), and PhACs are primarily introduced into the marine environment through water bodies such as groundwater, rivers, and surface runoff (i.e., water flowing over the ground from rain or snow) after being released from land-based sources, including wastewater treatment plants (WWTPs), laboratories, hospitals, pharmaceutical industries, and animal husbandry (Bu et al., 2016; Chaturvedi et al., 2021; Wang et al., 2022a). During this land-to-sea transport, a part of PhACs are adsorbed onto suspended particulates (e.g. via electrostatic attraction, cation exchange, surface complexation, and cation bridging) and subsequently experience repeated cycles of deposition and resuspension under hydrodynamic forcing (Kaeseberg et al., 2018; Luo et al., 2019). The environmental transformation half-life of a chemical is largely determined by its intrinsic chemical properties and by ambient conditions. For active compounds such as pharmaceuticals, transformation rates often depend primarily on environmental factors. Recent studies have focused on the sources, spatial distribution, and ecological risks of pharmaceuticals in marginal seas (Wang et al., 2022b; Chen et al., 2022; Guo et al., 2023a). However, limited attention has been paid to hydrodynamic sorting and long-term accumulation history of pharmaceuticals in sediments during land–sea transport, which constrains our understanding of their environmental fate.

The ongoing sedimentation process enables the vertical preservation of PhACs, thereby recording their temporal variations and long-term accumulation in coastal environments. Lara-Martín et al. (2015) were among the first to investigate PhACs in sediment cores. Using a "matched cores" approach, they demonstrated that the degradation of PhACs within the cores was negligible over at least half a century, allowing reliable reconstruction of historical concentration profiles. Kerrigan et al. (2018) examined the accumulation of antibiotics in sediment cores from Minnesota lakes, successfully reconstructing the depositional fluxes of ten antibiotics and suggesting that some compounds can reflect historical trends in human use. Such long-term preservation has two important implications. On the one hand, buried pharmaceuticals may represent a long-term environmental concern. For example, well-studied antibiotics may maintain selective pressure on sediment microbial communities and promote antibiotic resistance (Huijbers et al., 2015; Liang et al., 2024), while residual psychiatric pharmaceuticals may affect the neurological and behavioral functions of benthic organisms (Sehonova et al., 2018; Fong and Ford, 2014; Ford and Fong, 2016). On the other hand, their preservation provides an opportunity to use sedimentary PhAC records as markers of historical anthropogenic activities, allowing past pharmaceutical inputs and their long-term accumulation to be reconstructed beyond the temporal scope of contemporary water-column monitoring. However, the potential of PhACs as sedimentary markers in coastal environments remains relatively underexplored, particularly where pharmaceuticals are subject to continuous transport, deposition, and burial.

Shenzhen Bay, situated between the Hong Kong Special Administrative Region and Shenzhen City, lies at the heart of the Guangdong-Hong Kong-Macao Greater Bay Area. Over the past 50 years, this region has transformed from a small fishing village into a megacity characterized by high population density, intensive industrial activities, and well-developed (Seto and Fragkias, 2005; Qin et al., 2014a; Peng et al., 2017). During this period of rapid development, substantial amounts of contaminants were introduced into the bay, including heavy metals (Zhang et al., 2021), polycyclic aromatic hydrocarbons (Lin et al., 2023), and *per*- and polyfluoroalkyl substances (PFASs) (Guo et al., 2023b; Li et al., 2024), with PhACs emerging as contaminants of increasing concern (He et al., 2016; Qiu et al., 2019). The shallow depth of Shenzhen Bay (averaging about 5 m) and its semi-enclosed nature limit its water exchange, making it prone to the accumulation of EOPs (Lin et al., 2023; Guo et al., 2023b). In our recent study, PFASs were enriched in the inner bay and preferentially associated with fine-grained sediments (Li et al., 2024). These compounds have been classified as a persistent organic pollutant due to its chemical inertness and longer characteristic travel distances (CTDs) compared to PhACs (Yu et al., 2024). Building on PFASs research in this area, further field studies on the transport and deposition of organic pollutants with varying reactivities can be conducted.

This study aims to investigate the spatiotemporal distribution, hydrodynamic sorting, and burial chronology of 24 PhACs (e.g. antibiotics, psychotropics and cardiovascular drugs) along the land-to-sea transport pathway. First, we measured the concentrations of target compounds at multiple sites along the land-to-sea transport pathway, including surface bulk sediments, size-fractionated sediments, and down cores. Second, we analyzed the potential sources of PhACs and discussed the key factors controlling their residual accumulation in sediments after hydrodynamic sorting. Finally, we reconstructed the sedimentary chronology of PhACs, explored its potential as a historical tracer and estimated their residual accumulation in the region over the past decades. These efforts would provide new insights into the transport-selective retention of PhACs in dynamic coastal systems and inform future environmental management.

2. Materials and method

2.1. Sample collection

Sampling was conducted in June 2020 during the first phase of a nearshore investigation in Shenzhen Bay using a grab sampler and a stainless-steel box corer, and all samples were subsequently stored at −20 °C until analysis. The hydrological cycle of Shenzhen Bay is influenced by inputs from multiple rivers that traverse urban areas, as well as tidal currents driven by the mixing of the Pearl River Delta Coastal Current (PRDCC) and the South China Sea Coastal Current (SCSCC) outside the bay. To comprehensively investigate the transport and burial of PhACs in the sediments of Shenzhen Bay, 15 surface sediment samples (0–5 cm, S7–S59) and three core sediment samples (C31, 0–125 cm, $n = 15$; C18, 0–110 cm, $n = 14$; C10, 0–96 cm, $n = 15$) were selected, ranging from the inner to outer the bay. The sampling locations are shown in Fig. 1, and detailed sample information is provided in Supporting Information Table S1.

2.2. Fractionation of samples by particle size

The surface sediment samples for particle-size classification were collected from six sites (S7, S10, S17, S23, S42, and S59) spanning the inner to outer bay and representing different hydrodynamic regions of Shenzhen Bay (Fig. 1, black solid circles). The freeze-dried samples were subsequently separated into three grain-size fractions (>63, 20–63, and < 20 μm) using a Sonic Sifter Separator (Model L3D-25, Advantech Manufacturing Inc., USA). The selected size classes represent coarse particles (>63 μm), medium-sized silt (20–63 μm), and fine silt/clay (<20 μm), which differ substantially in settling velocity, specific surface area, resuspension potential, and transport behavior (Li et al., 2024; McCave and Hall, 2006). To prevent electrostatic charging and promote the dispersion of particle aggregates during the separation of fractions smaller than 63 μm, a Horizontal Pulse Accessory (Advantech L3-N8) was employed (Zheng et al., 2025).

2.3. Pharmaceutical extraction and analysis

A total of 24 PhACs were analyzed, including 10 antibiotics, 3

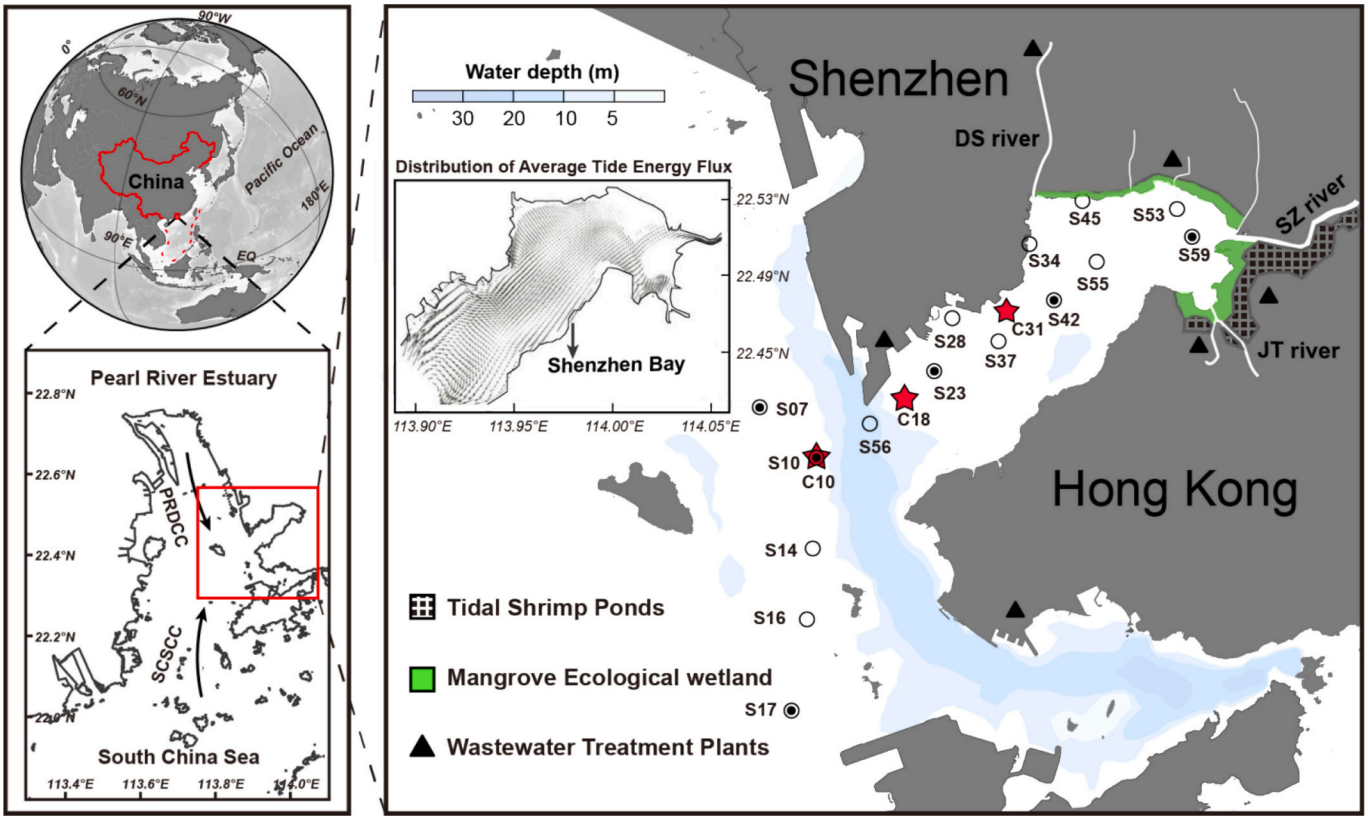


Fig. 1. Sampling Locations in Shenzhen Bay. Black hollow circles are surface sediment samples (0–5 cm), black solid circles are surface sediment samples fractionated by grain size (<20, 20–63, >63 μm), and red pentagons are down core samples. The distribution of average tidal energy flux is derived from Wu et al. (2019) (Wu et al., 2019). Abbreviations: PRDCC = Pearl River Delta Coastal Current, SCSCC = South China Sea Coastal Current, DS river = Dasha river, SZ river = Shenzhen river, JT river = Jintian river. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

cardiovascular drugs, and 11 psychiatric drugs (Table S2). These compounds were selected based on their regional relevance, environmental significance, and previous investigations in the Pearl River Estuary (Wu et al., 2022). Sediment samples were freeze-dried, homogenized, and sieved through a 100-mesh sieve. Approximately 2.0 g of each sample was spiked with isotope-labeled internal standards, followed by overnight extraction in an organic solvent mixture. Samples were ultrasonically extracted three times, and the combined extracts were filtered, diluted with Milli-Q water (<5% organic solvent), and purified using Oasis HLB solid-phase extraction (SPE) cartridges. After washing and elution with methanol containing 0.1% formic acid, the eluates were concentrated under a gentle nitrogen stream at 45 °C, reconstituted to 1.0 mL with methanol/water (6:4, *v*/v, containing 0.1% formic acid), and filtered through a 0.22 μm membrane prior to instrumental analysis.

Qualitative and quantitative analyses of the target PhACs were performed using an Agilent 1290 Infinity II ultra-high-performance liquid chromatography system coupled to an Agilent 6465B triple quadrupole tandem mass spectrometer (UHPLC–MS/MS; Agilent Technologies, Santa Clara, CA, USA) equipped with a Zorbax Eclipse Plus C18 column (2.1 × 50 mm, 1.8 μm). Target compounds were identified by their retention times and multiple reaction monitoring (MRM) transitions and quantified using isotope dilution with nine-point matrix-matched calibration curves (0.05–50 ng/mL). Detailed chromatographic conditions, mass spectrometric parameters, and quality assurance/quality control procedures are provided in the Table S3-S6. The reference standards, internal standard substances and detailed laboratory analysis are provided in Text S1-S4.

2.4. Quality assurance and quality control

The quality control of experimental and analytical procedures was conducted in three phases: method blank preparation, blank marking, and sample marking. The process and instrument blank experiments confirmed that no target PhACs were detected, demonstrating effective background control during pre-processing and instrumental analysis. The linear ranges, calibration curves, instrumental limits of detection (LODs) and limits of quantification (LOQs), and recovery rates for each target pharmaceutical are detailed in Tables S6 and S7. Quantification of target PhACs was achieved using internal standard calibration curves with a linear range of 0.05–50 ng/mL and a correlation coefficient (R^2) exceeding 0.99, meeting quantitative standards requirements. The recoveries of the target PhACs ranged from 60.3% to 125.3%, falling within the acceptable range for analytical method validation. LODs and LOQs were determined by serial dilution of standard solutions and defined as the analyte amounts corresponding to signal-to-noise (S/N) ratios of 3 and 10, respectively.

2.5. Dating of sediment cores

The dating of sediment cores (C31, C18, and C10) was based on $^{210}Pb_{ex}$ (i.e., ^{210}Pb – ^{226}Ra) activity and the Constant Initial Concentration (CIC) model (Appleby and Oldfield, 1978; McCall et al., 1984). This approach assumes a constant sedimentation rate over the deposition period and a stable specific activity of initial ^{210}Pb during its initial phase of deposition. Based on radionuclide analysis, the sediment cores C10, C18, and C31 were dated to the periods 1955–2018, 1931–2018, and 1945–2018, respectively, with average sedimentation rates of 1.46 cm/y, 1.22 cm/y, and 1.67 cm/y (Fig. S6 and Table S9). The constant

initial concentration model is represented by Eq. (1):

$$C_m = C_0 e^{-\lambda t} \quad (1)$$

Where C_0 is the initial specific activity of $^{210}Pb_{ex}$ (Bq/kg), C_m is the specific activity of $^{210}Pb_{ex}$ at depth (Bq/kg), and λ is the decay constant (0.03114/y) of ^{210}Pb.

2.6. Ecological risk assessment

In this study, the Risk Quotient (RQ) from the European Union Technical Guidance Document for Environmental Risk Assessment (TGD; EC, 2003) was adopted to quantify the risk of PhACs (European Commission, Joint Research Centre, 2003). To operationalize this, we used the water-sediment partitioning coefficient (K_d) to convert the pharmaceutical $PNEC_{water}$ values into $PNEC_{sediment}$ values, so that the risk of PhACs being released from sediments (both surface sediments and sediment cores) back into the aquatic environment can be assessed. The calculation details for RQ and the toxicological data of PhACs are provided in Text S6 and the Supporting data Excel file.

3. Results and discussion

3.1. Concentrations, composition, and distribution profiles of PhACs

Among the 24 target PhACs, 19 compounds, including 7 antibiotics and 12 psychotropic and cardiovascular pharmaceuticals (PCs), were detected in both surface sediments and sediment cores (Fig. 2a and c). In surface sediments, ∑PhAC concentrations ranged from 1.48 to 85.50 ng g^{-1} dry weight (dw) and significantly decreased with increasing distance from the estuary (Fig. 2b). In the sediment cores, ∑PhAC concentrations followed the order C31 > C18 > C10 and declined with burial depth, with ranges of <LOQ–199.31 ng g^{-1} dw in C31, <LOQ–71.02 ng g^{-1} dw in C18, and < LOQ–13.99 ng g^{-1} dw in C10 (Fig. 2c). Among the detected PhACs, antibiotics dominated the composition, accounting for more than 95% on average. The measured antibiotic concentrations in surface sediments (1.35–82.37 ng g^{-1} dw) were comparable to those reported for estuarine sediments in the Greater Bay Area (6.74–140.00 ng g^{-1} dw) and the South China Sea (1.63–31.70 ng g^{-1} dw) (Wu et al., 2022), but substantially lower than those in riverine sediments in Shenzhen city (28.12–2728.81 ng g^{-1} dw) (Qiu et al., 2019), reflecting dilution and loss during land–sea transport. Meanwhile, in a broad comparison with other bay areas in the world, the PhAC concentrations in the sediments of Shenzhen Bay are at a moderately high level (Table S8).

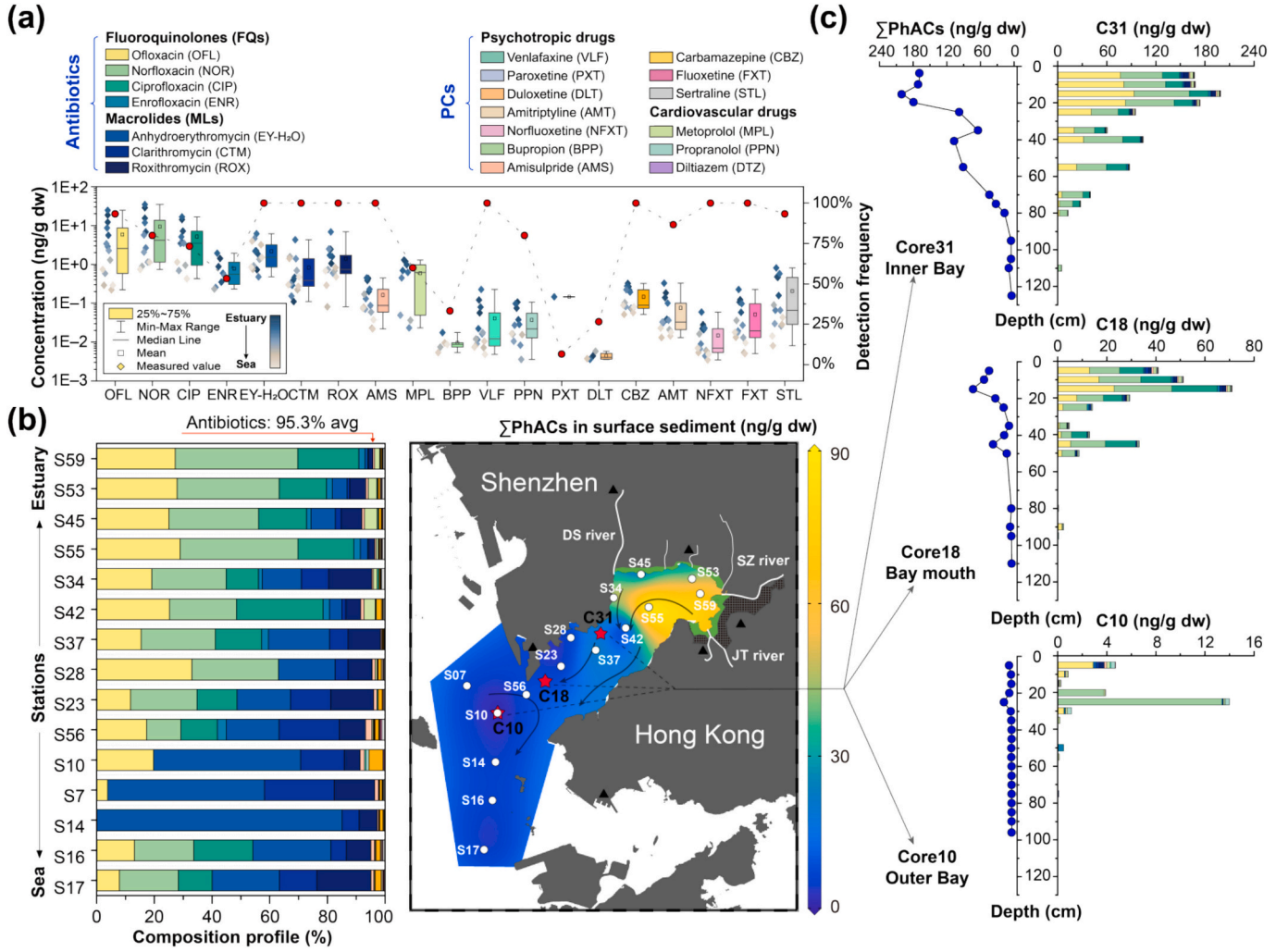


Fig. 2. (a) Concentration levels (left scale) and detection frequencies (right scale) of PhACs detected in surface sediment. (b) Composition profile (left) and spatial concentration distribution (right) of the detected PhACs in surface sediment. (c) Vertical profiles of ∑PhAC concentrations (left; unified x-axis scale) and concentration compositions of individual PhACs (right; variable x-axis scales) in sediment cores C31, C18, and C10.

The 7 antibiotics included 4 fluoroquinolones (FQs) and 3 macrolides (MLs), with FQs (NOR, OFL, CIP and ENR) showing a lower detection rate than MLs (EY-H_2O, CTM and ROX) (Fig. 2a). The FQs exhibited the highest concentrations among all detected compounds and showed a clear spatial clustering in aquaculture-associated areas adjacent to the Shenzhen River estuary (Fig. S1). A gradual shift in pharmaceutical composition of surface sediment was observed from FQs to MLs during transport (Fig. 2b). This pattern is counterintuitive because FQs were detected at higher concentrations near the estuary and exhibit greater aqueous solubility than MLs (Table S2), which would theoretically favour their longer-distance transport. However, FQs have been reported that are rarely detected in the water column of Shenzhen Bay, whereas MLs remain measurable (Ke et al., 2019). Considering the much faster photodegradation of FQs in aquatic environments (Lyu et al., 2022; Kashyap et al., 2023), this transition likely reflects a photochemical filtering effect during water-column transport. Cheng et al. reported that with increasing salinity from estuarine to offshore waters, the formation of reactive halogen species (RHSs) is enhanced, thereby promoting the photodegradation of highly reactive groups, such as FQs (Cheng et al., 2024). The 12 PCs included 9 psychiatric and 3 cardiovascular drugs, all of which were present at low concentrations (<1 ng g^{-1} dw). A similar transport-related transformation may also affect fluoxetine (FXT). Norfluoxetine (NFXT), the active demethylated metabolite of FXT, can serve as a metabolic indicator based on the NFXT/FXT ratio (Kwon and Armbrust, 2006). A gradual increase in the NFXT/FXT ratio from the inner bay to the outer bay was observed (Fig. S1). Because photochemical conversion of FXT to NFXT is minor (<2%) and NFXT photodegrades faster than FXT, this trend likely reflects biological transformation during transport (Kwon and Armbrust, 2006; Tisler et al., 2019).

PhAC concentrations in the sediment cores were generally higher than those in surface sediments, with peak levels occurring at depths of around 15 cm, indicating more severe pharmaceutical contamination in the past (Fig. 2c). The C31 (inner bay) and C18 (bay mouth) showed substantially higher concentrations than C10 (outer bay). This pattern is related to both their closer proximity to riverine inputs and the hydrodynamic setting of Shenzhen Bay. Water circulation within the bay is largely driven by river inflows entering the bay and flowing seaward toward the sea, forming a semi-enclosed system with a general land–sea transport pathway (Zhang and Mao, 2015). In contrast, the outer bay is located within the Pearl River Estuary and is influenced by the Pearl River Delta Coastal Current (PRDCC) and the South China Sea coastal current (SCSCC) (Fig. 1), which may enhance water exchange and reduce pharmaceutical accumulation. Moreover, the deep navigation channel near the bay mouth may further limit sediment exchange between the inner and outer bay.

3.2. Environmental controls on PhAC distribution in sediments

To further examine the marked decrease in PhACs along the land–sea transport pathway, concentrations were normalized to bulk sedimentary TOC. The resulting spatial patterns remained largely consistent with those based on non-normalized concentrations, indicating that bulk TOC alone did not account for the observed spatial variability of PhACs (Fig. S2). We therefore further examined the distribution of PhACs across different grain-size fractions. The results showed that concentrations in the 20–63 μm and > 63 μm fractions were comparable and significantly higher than those in the <20 μm fraction (Fig. 3a). This pattern contrasts with the conventional view that fine-grained sediments, owing to their larger specific surface area, tend to adsorb more organic matter (Ransom et al., 1998; Bao et al., 2019). We argue that for reactive, land-derived pollutants such as PhACs, factors beyond adsorption capacity must be considered, including sedimentation rate, desorption, and degradation during transport. Several lines of evidence support this interpretation. First, the grain-size range investigated in this study (20–63 μm) falls within a range where sedimentation rates

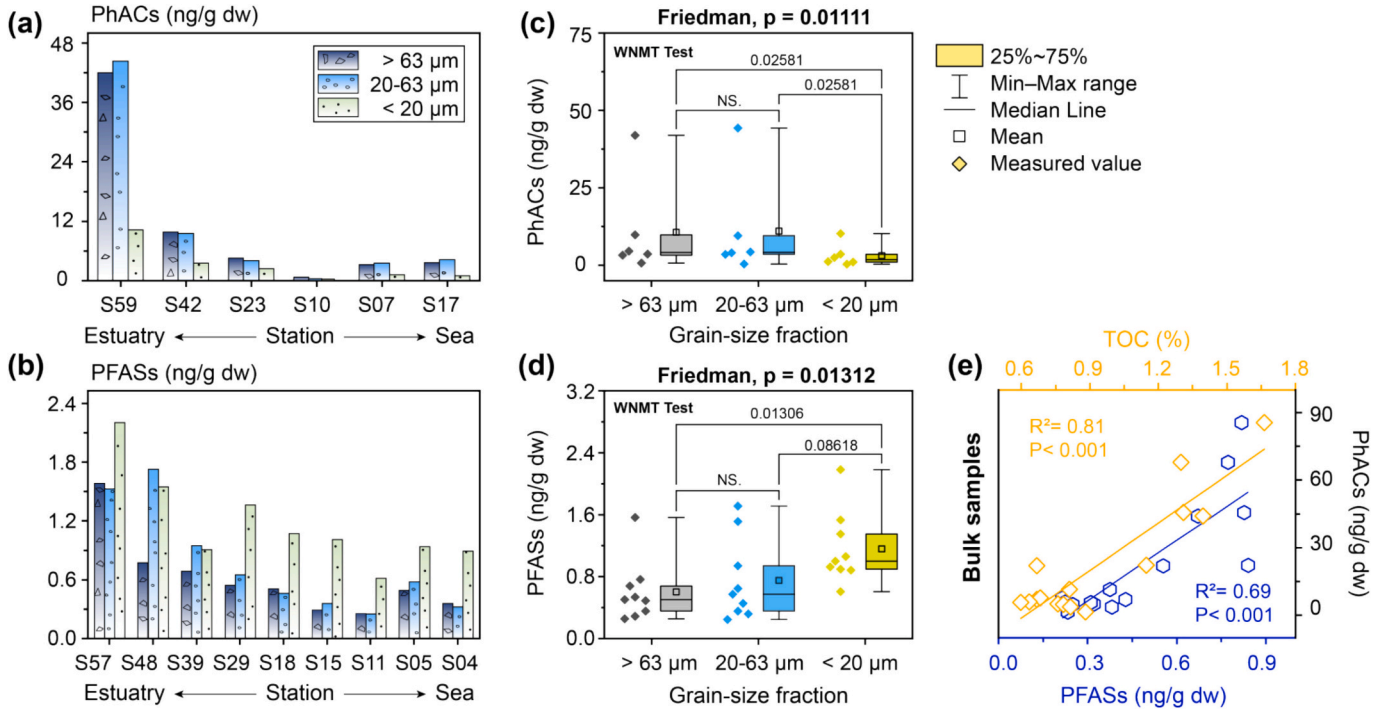


Fig. 3. (a) Concentration levels of PhACs in sediments of different grain-size fractions (<20, 20–63, >63 μm). (b) Concentration levels of PFASs in corresponding grain-size fractions. (c) Boxplots showing ∑PhACs in each grain-size fraction. (d) Boxplots of ∑PFASs in each grain-size fraction. Significant differences were assessed using the Friedman test with Wilcoxon–Nemenyi–McDonald–Thompson post hoc comparisons ($p < 0.05$ indicates significant differences). (e) Linear relationships among TOC and PhACs (yellow line), PFASs and PhACs (blue line). PFASs data are adapted from Haoshuai Li et al., (2024) (Li et al., 2024). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

increase with increasing grain size (Fig. S4) (Lamb et al., 2020). In Shenzhen Bay, suspended particles are typically fine (8–15 μm), whereas surface sediments contain relatively coarser particles (<32 μm) (Zhang and Mao, 2015), indicating that coarser particles are more readily removed from the water column by settling. Second, dissolved oxygen concentrations and water temperature decrease, whereas salinity increases, with increasing water depth in Shenzhen Bay (Xu et al., 2010). These conditions may reduce the transformation of particle-associated PhACs during transport through deeper waters. Third, coarse-grained particles contain less organic matter and are transported laterally at greater depths, where weaker light penetration may limit the formation of reactive intermediates such as RHSs and ^{3}DOM* involved in pharmaceutical phototransformation (Cheng et al., 2024). In contrast, fine-grained particles are more susceptible to resuspension and prolonged suspension, potentially increasing their exposure to sunlight and oxygen and facilitating PhAC transformation (Ou et al., 2025). Together, these processes may favour the preferential preservation of PhACs in coarse-grained sediments. Nevertheless, this pattern should not be interpreted as a simple monotonic grain-size effect. Previous experiments on norfloxacin and tetracycline showed that pharmaceutical adsorption varied among particle-size fractions, with the 63–150 μm fraction showing greater adsorption than both finer and much coarser fractions, and this variation was related to particle properties such as specific surface area, cation exchange capacity, and organic matter content (Luo et al., 2019). In addition, the intrinsic physicochemical properties of PhACs also influence their transport and partitioning among sediment size fractions, alongside environmental conditions. For example, venlafaxine (VLF) was predominantly associated with the coarse fraction in the estuarine region but became enriched in the fine fraction at offshore sites (Fig. S3), indicating that the transport and redistribution of individual compounds may differ during estuarine–marine transport.

We compared the grain-size distribution of PhACs with previously published data for PFASs (Li et al., 2024), a group of relatively inert and environmentally persistent contaminants, obtained from sediments collected during the same sampling campaign. Despite significant variability ($p < 0.05$) within this fraction (<20 μm), PFASs and PhACs display contrasting patterns, with PFASs enriched in fine particles and PhACs markedly depleted (Fig. 3c, d). Several factors may contribute to this difference, with differences in chemical structure and environmental reactivity being among the most important. As persistent organic pollutants, PFASs contain multiple carbon-fluorine (C—F) bonds with exceptionally high dissociation energies (460 kJ/mol) (Higgins and Luthy, 2006), granting them remarkable thermal and chemical stability. Previous studies summarized by Yu et al. (2024) indicate that PhACs are more susceptible to photodegradation than PFASs in aquatic environments. Therefore, prolonged suspension of fine particles in the water column is unlikely to result in substantial degradation of PFASs. Besides differences in environmental reactivity, differences in sorption and partitioning properties may also contribute to the contrasting distributions. PhACs have diverse molecular structures, ionization states, and functional groups, whereas PFAS partitioning is strongly influenced by fluorinated chain length and functional head groups (Yu et al., 2024). These differences may lead to distinct interactions with organic matter and mineral surfaces and consequently affect their particle association. Consistent with this possibility, the stronger relationship between bulk TOC and PhACs than between bulk TOC and PFASs (Fig. 3e) suggests that organic matter may play a more important role in the sedimentary behavior of PhACs. Overall, these observations suggest that the rapid deposition of coarse-grained sediments may be more conducive to the preservation of PhACs.

3.3. Source apportionment

The 16 PhACs with high detection frequencies were selected from the 19 detected compounds for correlation heatmap analysis and principal component analysis (PCA). Two distinct correlation patterns were observed in surface sediments: one linking FQs and most psychiatric drugs and the other involving MLs (Fig. 4a, up). Factor 1 was characterized by high positive loadings of FQs (NOR, OFL, CIP and ENR), metoprolol (MPL), carbamazepine (CBZ), and several psychiatric pharmaceuticals (Fig. 4b, left), indicating that these compounds shared a common transport pathway. Spatially, these pharmaceuticals generally exhibited their highest concentrations in sediments adjacent to the Shenzhen River estuary (Figure S1and S5), suggesting that Factor 1 primarily represents contaminant inputs delivered by the Shenzhen River. Because the Shenzhen River drains densely urbanized districts as well as aquaculture regions before discharging into Shenzhen Bay, the pharmaceutical assemblage associated with Factor 1 most likely reflects mixed anthropogenic inputs, including municipal wastewater and aquaculture activities. Among these compounds, CBZ and MPL are widely recognized as indicators of municipal wastewater because of their extensive human consumption, frequent occurrence in wastewater effluents, and incomplete removal during wastewater treatment (Sörengård et al., 2019; Hai et al., 2018), whereas FQs are extensively used in aquaculture and are commonly detected in aquaculture-impacted environments (Chen et al., 2022; Liu et al., 2017). The co-occurrence of these compounds on Factor 1 therefore indicates a mixed anthropogenic source, integrating inputs from municipal wastewater and aquaculture. Factor 2 was dominated by MLs (EY-H_2O, CTM and ROX), indicating a distinct distribution pattern from that represented by Factor 1. Spatially, these compounds exhibited their highest concentrations in sediments near the Dasha River estuary (Fig. S1), suggesting that Factor 2 is primarily associated with contaminant inputs transported by the Dasha River. The Dasha River drains Shenzhen's High-tech Industrial Park, where pharmaceutical industries (including macrolide producers) and medical facilities are concentrated (Fig. 4c), and a previous study have reported relatively high concentrations of MLs in wastewater treatment plant effluents within this watershed (Pan and Yau, 2021). Therefore, the predominance of MLs on Factor 2 likely reflects a mixed anthropogenic source associated with the Dasha River catchment, including wastewater discharge and pharmaceutical industry-related inputs.

Unlike the surface sediments, almost all target PhACs exhibited high positive loadings on Factor 1 in the sediment cores, which explained 79.8% of the total variance (Fig. 4b, right). This pattern is consistent with the strong positive correlations observed in the heatmap (Fig. 4a, down), indicating that the temporal variations of most compounds were governed by a common controlling factor. Given the gradual decrease in pharmaceutical concentrations from the inner bay to offshore and the continuous burial of contaminants in the sediment column, Factor 1 is interpreted as representing the long-term accumulation of river-derived anthropogenic inputs combined with sedimentary burial processes rather than individual emission sources. The progressively smaller score distributions from the C31 to the C18 and C10 further suggest that the variability of pharmaceutical composition decreases seaward. This trend likely reflects the attenuation and homogenization of river-derived pharmaceutical inputs during offshore transport, resulting in increasingly uniform contaminant assemblages in the outer bay.

3.4. Sedimentary chronology and anthropogenic tracing

To better understand the temporal sedimentation patterns of PhACs along the land–sea transport continuum, we reconstructed the sedimentary fluxes of pharmaceuticals in Shenzhen Bay. The sedimentary fluxes of pharmaceuticals in the three sediment cores exhibit similar vertical trends, with magnitudes decreasing toward the outer bay (Figue S7). This pattern suggests that pharmaceutical inputs were propagated through the bay as a coherent environmental signal. Although the magnitude of this signal gradually attenuated during seaward transport, its temporal pattern was largely preserved, reflecting a relatively stable land–sea transport pathway. This phenomenon is consistent with the concept of environmental signal propagation in sediment-routing

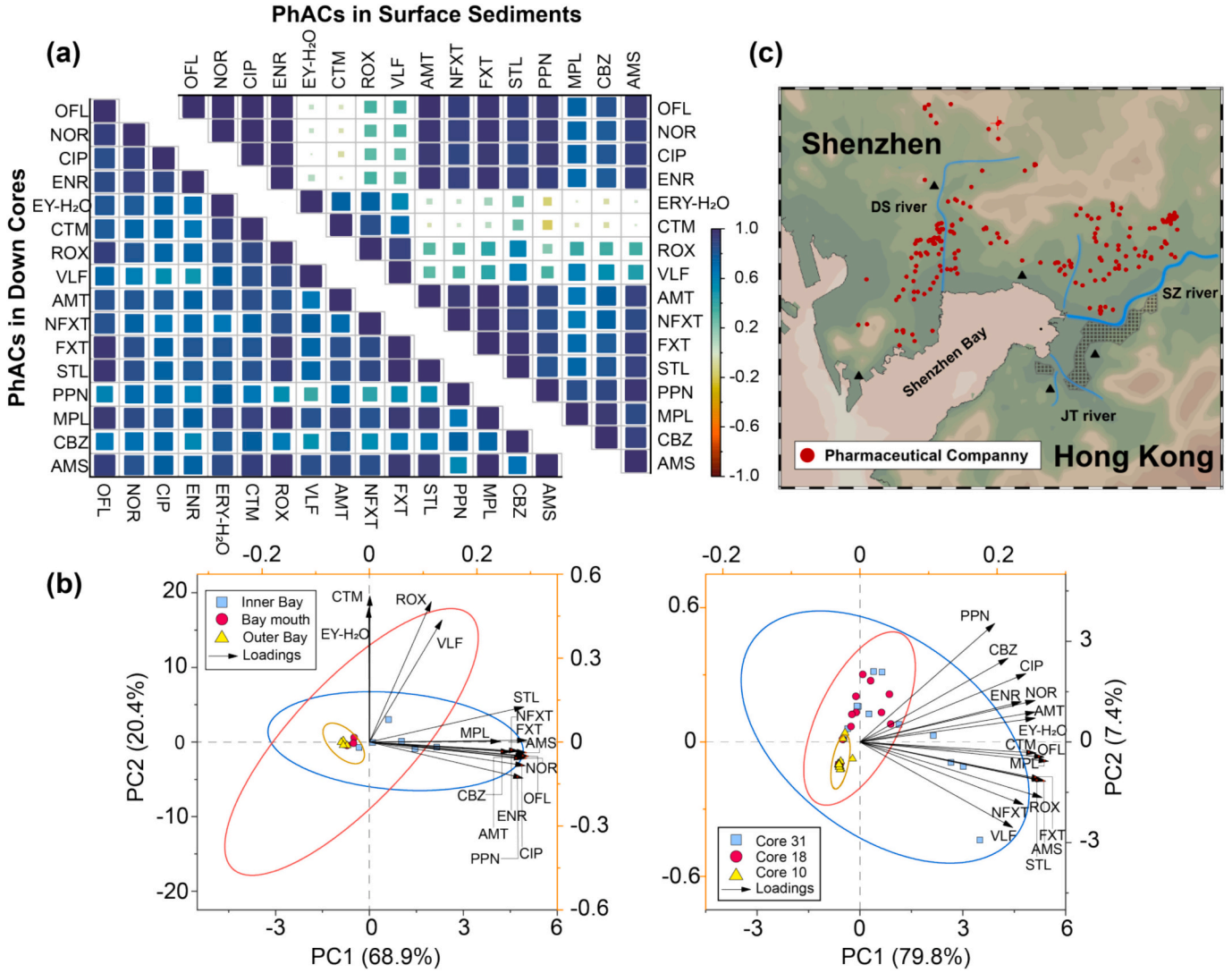


Fig. 4. (a) Heatmap of correlations for PhACs in surface sediments (top) and down cores (bottom). (b) Principal Component Analysis (PCA) of PhACs in surface sediments (left) and sediment cores (right). (c) Distribution of pharmaceutical companies in Nanshan District ($n = 121$) and Futian District ($n = 115$), Shenzhen city. The data were obtained from the website (https://api.map.baidu.com/lbsapi/getpoint/index.html).

systems, in which source signals are transmitted downstream while progressively dampened by transport and temporary sediment storage (Romans et al., 2016; Pizzuto et al., 2017).

We utilized FQs, the dominant pharmaceutical components in this region, to trace historical changes in anthropogenic activities. The FQs historical deposition flux (based on C31, which showed the highest R^2 for the ^{210}Pb age mode, Fig. S6) can be categorized into two growth phases and two decline phases (Figue 5a), which closely align with the development history of this area aquaculture industry (Yuan et al., 2024; Jia et al., 2016). The first growth phase occurred from 1945 to 1997, exhibiting a sustained increase with a marked acceleration after 1980. As noted in historical reviews, after 1949, fishponds near the Shenzhen River estuary became major suppliers of aquatic products due to their proximity to Hong Kong. In the early Special Economic Zone period (after 1978s), Shenzhen city remained primarily agricultural, with thousands of acres of fishponds concentrated in Futian, Luohu districts and along both banks of the Shenzhen River (Jia et al., 2016). Following the introduction of FQs in the 1980s, their widespread use in aquaculture likely increased FQ inputs into the bay (Andersson and MacGowan, 2003). FQs subsequent decline occurred from 1997 to 2000. This decline was likely driven by the rapid urbanization of Shenzhen, which significantly increased land demand and led to the widespread infilling of traditional fishponds. Studies showed a significant decline in Shenzhen Bay's aquaculture area by 1996, with only the fishponds in Mai Po Nature Reserve, Hong Kong, partially preserved under ecological protection policies (Yuan et al., 2024; Jia et al., 2016). In addition, the decline in FQs may also have been influenced by improvements in wastewater management. Since the 1990s, a series of pollution-control and wastewater infrastructure measures have been implemented in the Shenzhen River catchment, including the construction and expansion of wastewater treatment and transfer systems, which contributed to improving river water quality after the mid-1990s (Qin et al., 2014b). The second growth phase occurred from 2000 to 2012. After 2000, China's antibiotic production has increased rapidly and, by 2009, the country had become the world's largest producer (Liu et al., 2017; Shao et al., 2021). This led to widespread application and even misuse in aquaculture, further contributed to the increasing deposition flux of FQs (Liu et al., 2017). In response to these challenges, the Chinese government implemented the "Antibiotic Restriction Order" in 2012 to regulate antibiotic production and usage (Shao et al., 2021). From 2012 to 2020, additional policies were enacted to further control antibiotic use, including a 2015 decision to ban the use of four veterinary drugs (i.e., lomefloxacin, pefloxacin, ofloxacin, and norfloxacin) in food animals (Shao et al., 2021). In this study, the sediment fluxes of FQs (OFL, NOR, CIP, and ENR) in 2015

(175.9, 110.4, 45.06, and 2.90 ng/cm^2/y, respectively) were lower than those in 2012 (202.8, 146.7, 50.36, and 5.50 ng/cm^2/y, respectively) (Fig. S7). This reflects the positive impact of China's regulatory measures on antibiotics use and highlights the role of sedimentary PhACs as real-time indicators of anthropogenic activities (Thiebault et al., 2017).

For compounds present at relatively low concentrations, we also reconstructed their temporal distribution. It should be noted that the historical deposition flux of ML antibiotics showed a continuous increase from 1945 to 2017 (Fig. S7), in contrast to the event-driven fluctuations observed for FQs. A possible explanation is that MLs, due to their lower resistance rates and better safety profiles compared to FQs, have been more commonly used in the general population, with usage steadily increasing alongside Shenzhen's population growth. Psychotropic and Cardiovascular drugs (PCs) have shown a consistent upward trend in flux since 1945 (Fig. 5b), with a significant increase after 2000. This sharp rise can reflect a growing prevalence of mental health conditions, such as depression and anxiety, in the Shenzhen Bay area over the past 25 years. Contributing factors may include rapid social changes, economic pressures, environmental stressors, lifestyle shifts, the rise of social media, and greater awareness of mental health. A comparison of Shenzhen's population growth and the deposition flux of PCs revealed a significant positive correlation (Fig. S8) (Yang et al., 2019), indicating that population growth may be a key driver of rising these pharmaceutical consumption.

3.5. Accumulation mechanism, quantification, and risk assessment

The accumulation of PhACs within the sediments of coastal bays predominantly initiates within estuarine zones. These transitionary regions are characterized by a marked decline in hydrodynamic energy as riverine currents encounter the resistance of the receiving basin waters (Wu et al., 2022). Concurrently, the mixing of freshwater and seawater leads to a progressive increase in salinity, triggering significant physicochemical changes (Wu et al., 2022; Cheng et al., 2024). Crucially, this combination of reduced flow velocity and changing ionic strength profoundly influences the behavior of suspended particulate matter (SPM) (Luo et al., 2019; Zhang and Mao, 2015). Coarser fractions of SPM, possessing higher settling velocities due to their larger size and density, experience a rapid loss of transport capacity (Fig. 5c). Consequently, they tend to settle out preferentially and accumulate within the estuarine and proximal bay sediments (Wu et al., 2022). In contrast, finer colloidal particles and clays, remaining buoyant for longer periods under lower energy conditions, largely bypass this initial depositional trap. These finer particles stay suspended within the water column and are advected further seaward, often undergoing subsequent depositional cycles in more distal, lower-energy environments of the inner shelf.

This hydrodynamic sorting and preferential settling of coarse particles play a disproportionately significant role in the fate of highly reactive organic contaminants like PhACs (Wu et al., 2022). The rapid incorporation of PhACs into the sediment layer effectively sequesters them from the water column. More importantly, this burial process rapidly removes them from exposure to oxidative processes occurring in the aerobic water column and upper sediment layers, significantly decreasing their oxygen exposure time (OET) and protecting them from photolytic degradation by sunlight near the surface (Luo et al., 2019; Hartnett et al., 1998; Zhao et al., 2024). A recent study in a stratified reservoir found that the transformation rate of sulfamethoxazole at 40 m decreased to only 4.8% of the surface value, alongside pronounced vertical changes in solar irradiance, dissolved oxygen, and reactive iron species (Wang et al., 2026). This suggests that deeper transport may reduce exposure to reactive conditions and potentially favour the preservation of particle-associated PhACs.

The limited mobility of these deposited coarse sediments further compounds the environmental impact. Once settled within the relatively confined estuarine and inner bay depositional zones, these sediment-bound PhACs exhibit minimal potential for significant further lateral transport under typical hydrodynamic regimes (Wu et al., 2022). This results in a pronounced spatial focusing or concentration of PhAC contamination within these specific nearshore environments. Consequently, the localized sediment reservoirs become significant, long-term sources of these micropollutants. This persistent reservoir effect not only exacerbates the immediate severity of pollution within these sensitive

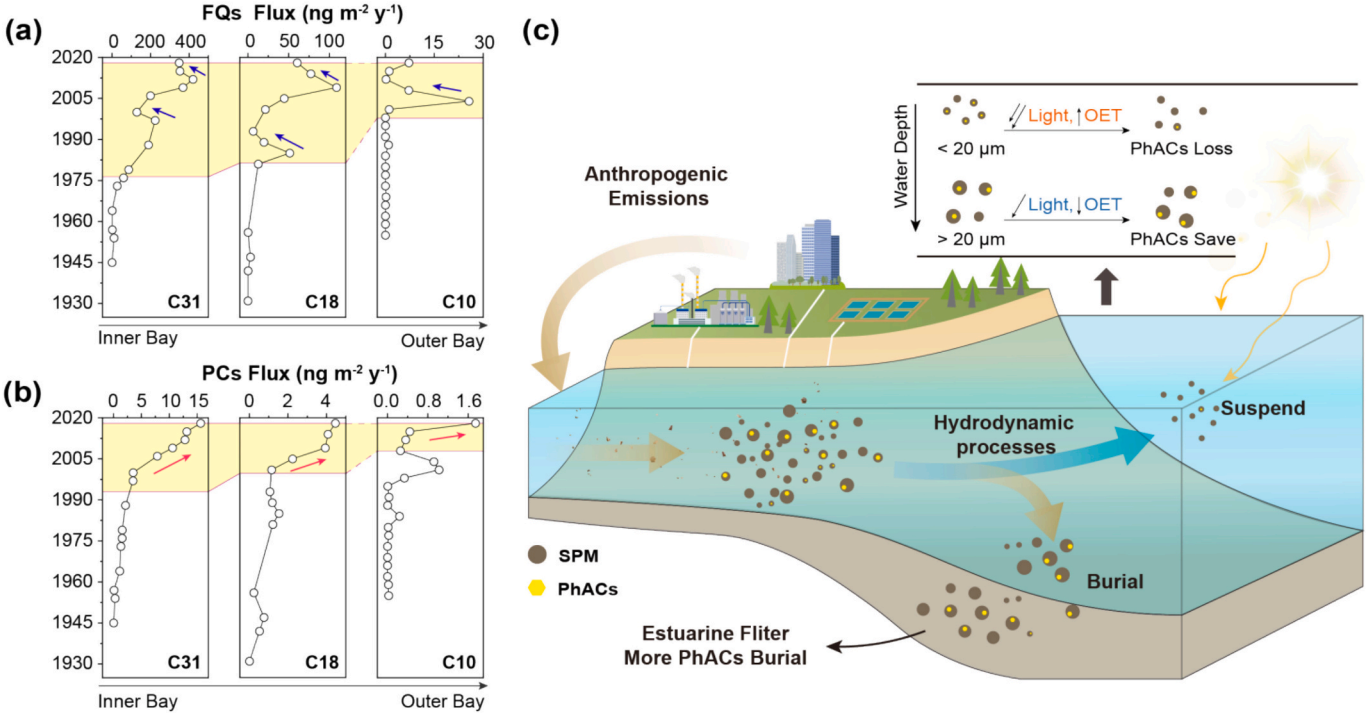


Fig. 5. (a) Chronological flux profiles of fluoroquinolone (FQs) in down cores. (b) Chronological flux profiles of Psychotropic and Cardiovascular drugs (PCs) in down cores. (c) Accumulation mechanisms of PhACs in coastal bay sediments. SPM = suspended partical matter; OET = oxygen exposure time.

coastal habitats but also underpins its extended persistence, posing ongoing ecological risks through potential remobilization or bioaccumulation within benthic food webs long after the initial input event (Lara-Martín et al., 2015; Kerrigan et al., 2018; Guo et al., 2023a).

We estimated the total mass of detected PhACs buried within the top 1 m (approximately 60 years) of sediment in the inner bay, bay mouth, and outer bay regions of Shenzhen Bay (details in Text S5 and Fig. S9). The results show these regions contain approximately 13.51 ± 4.05, 1.42 ± 0.43, and 0.62 ± 0.19 tons of PhACs, respectively. Although the inner bay represents less than one-third of the total evaluated sediment volume, it harbors more than 90% of the total buried PhACs, indicating that the inner bay functions as an important sink and bears a high pollution burden. We calculated risk quotients (RQs) using the maximum detected concentrations of each PhAC, with detailed calculations and risk assessment provided in Text S6. The results showed OFL, CTM, and CBZ as high-risk compounds, six PhACs (i.e., NOR, ROX, VLF, NFXT, FXT, and PPN) as medium-risk, and the remaining as low-risk or negligible (Fig. S10). High-risk compounds are predominantly FQs, indicating that risks stemming from historical aquaculture activities continue to threaten benthic ecosystems. Liang et al. (2024) pointed out that Shenzhen Bay exhibits a higher antimicrobial resistance risk compared to other marine environments. Fortunately, strengthened environmental policies have led to a noticeable decline in major antibiotic concentrations, reflecting effective pollution control and the need for continued monitoring to mitigate residual risks.

4. Conclusion

This study systematically characterized the spatial and temporal distribution of PhACs along the land–sea transport continuum in Shenzhen Bay. PhACs exhibited progressive attenuation from the estuary toward offshore sediments and distinct grain-size partitioning, with preferential enrichment in coarse sediment fractions (>20 μm). Based on these observations, we propose a conceptual hydrodynamic sorting framework suggesting that selective removal of fine particles during transport may contribute to the residual enrichment of reactive PhACs in coarse sediments. Historical reconstruction further demonstrated that sedimentary PhAC records preserve clear anthropogenic signals over decadal timescales, highlighting their potential as chronological tracers of socio-economic development, aquaculture expansion, and environmental regulation. In addition, the long-term accumulation of PhACs in Shenzhen Bay sediments and their associated ecological risks highlight the need for continuous monitoring and effective management to prevent further environmental deterioration.

More broadly, the conceptual framework proposed in this study provides a new perspective for understanding the long-term environmental fate of reactive organic contaminants in dynamic coastal systems. By integrating spatial distributions, sedimentary archives, and particle-size characteristics, this approach may be applicable to other estuarine and coastal environments worldwide for investigating contaminant transport pathways, sedimentary preservation, and historical anthropogenic impacts. Nevertheless, several limitations should be acknowledged. The proposed hydrodynamic sorting framework remains conceptual and requires further validation through experimental and hydrodynamic modeling studies. Furthermore, the ecological risk assessment was based on current sediment concentrations and did not account for the cumulative risks associated with the long-term burial and continuous accumulation of PhACs. Future studies should incorporate temporal accumulation processes and potential remobilization to better evaluate their long-term ecological impacts.

CRediT authorship contribution statement

Guangyang Cheng: Writing – review & editing, Writing – original draft, Visualization, Data curation. **Haoshuai Li:** Writing – review & editing, Supervision, Investigation. **Lei Xing:** Writing – review & editing. **Yang Zhou:** Resources, Formal analysis, Data curation. **Danxu Guo:** Writing – original draft, Visualization, Validation, Formal analysis, Data curation. **Zimin Wang:** Formal analysis. **Siqi Zhao:** Writing – review & editing. **Zongshan Zhao:** Writing – review & editing. **Shuhong Fang:** Writing – review & editing, Methodology, Formal analysis. **Fang Chen:** Resources, Formal analysis. **Rui Bao:** Writing – review & editing, Supervision, Project administration, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Detailed information on standard preparation, sample pretreatment, analytical procedures, qualitative and quantitative anlysis, sediment sample parameters, ^{210}Pb-based sediment dating data, accumulation estimation, and graphical representations of individual PhACs was provided. Supplementary data to this article can be found online at https://doi.org/10.1016/j.marpolbul.2026.120299.

Data availability

Data will be made available on request.

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