

Article

Occurrence, Spatiotemporal Distribution, Source Apportionment, and Ecological Risk Assessment of PPCPs in the Taipu River, a Strategic Trans-Boundary Water Source in Eastern China

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Abstract

Pharmaceuticals and personal care products (PPCPs) are contaminants of emerging global concern, yet their long-term fate and distribution in strategic trans-boundary water source areas remain underexplored. This study conducted a comprehensive monitoring campaign spanning from 2018 to 2020, with six sampling campaigns carried out in August 2018, December 2018, May 2019, August 2019, December 2019, and May 2020, to investigate the occurrence, spatiotemporal dynamics, source apportionment, and ecological risks of 54 PPCPs in the surface water and sediments of the Taipu River in eastern China. Utilizing non-target screening via liquid chromatography high-resolution mass spectrometry (LC-HRMS), 54 PPCPs across 15 categories were detected, with average total concentrations of 28.60 ng/L in surface water and 13.01 ng/g in sediments. Sulfonamides, quinolones, and non-steroidal anti-inflammatory drugs (NSAIDs) dominated the aqueous phase (with bisphenol A and sulfamethoxazole highly prevalent), while hormones (e.g., estriol) and quinolones (e.g., ciprofloxacin (CIP)) exhibited significant accumulation in the benthic zone. Spatiotemporal analysis revealed a seasonal pattern where PPCP concentrations were higher in the dry season than in the wet season, primarily driven by hydrological dilution and climate-induced degradation. Furthermore, Positive Matrix Factorization (EPA PMF 5.0) extracted five distinct source factors for each matrix. Specifically, surface water pollution was primarily driven by domestic wastewater, municipal effluents, medical discharges, and localized pharmaceutical emissions, whereas sediments acted as a long-term sink predominantly for domestic wastewater, agricultural/veterinary runoff, and municipal/industrial emissions. Ecological risk assessment indicated that while most of the nine high-frequency contaminants posed low risks, specific compounds such as CIP, SM2, and E3 presented medium risks in surface water, whereas ENR, CIP, and SPI posed medium risks in sediments. These findings emphasize that the continuous multi-source input and the resulting pseudo-persistence of PPCPs in sediments warrant prioritized continuous attention and targeted pollution control strategies in the Taipu River Basin.

Keywords: PPCPs; endocrine disrupting chemicals; source apportionment; Taipu River; ecological risk assessment; LC-HRMS; trans-boundary river



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1. Introduction

Over the past few years, pharmaceuticals and personal care products (PPCPs) have become a major focus of scientific research as key contaminants of emerging concern. This growing interest is primarily driven by their pervasive presence in various environmental settings and the substantial risks they pose to aquatic ecosystems at ecologically relevant concentrations [1]. The global markets for PPCPs were valued at approximately USD 1.7 trillion billion and USD 677.19 billion in 2025 [2]. Economic development, population expansion, demographic aging, and advances in healthcare are expected to further increase both the diversity and consumption of PPCPs. However, their removal during treatment processes is highly compound-dependent and is often not complete [3]. Consequently, PPCPs are released into aquatic ecosystems through several sources, including wastewater treatment plant (WWTP) effluents, aquaculture systems, industrial discharges, food and feed processing facilities, and urban stormwater runoff [4,5]. With rapid urbanization and socioeconomic development, PPCP emissions have increased in recent years and become a highly concerned contaminant in the scientific community [6–8].

Despite existing at trace environmental concentrations (typically ng/L to µg/L), PPCPs pose significant ecotoxicological risks to aquatic taxa, potentially inducing developmental malformations, endocrine disruption, alongside neurotoxic and genotoxic damage [9,10]. To better understand their environmental behavior, PPCPs can be broadly categorized into distinct classes based on their therapeutic uses and chemical structures. For instance, antibiotics are highly hydrophilic and persistent; non-steroidal anti-inflammatory drugs (NSAIDs) exhibit high pseudo-persistence and induce severe tissue damage; psychiatric drugs frequently escape WWTP degradation; and personal care products and EDCs possess high lipophilicity, allowing them to bioaccumulate through the food chain [11]. Endocrine disrupting chemicals (EDCs), including bisphenol A and steroid hormones, represent a particularly alarming subset of these contaminants. Unlike bulk organic pollutants, EDCs can severely alter aquatic hormonal systems at mere ng/L concentrations, driving devastating ecological outcomes such as fish feminization and overall loss of biodiversity [12–14]. Therefore, establishing the ecological risks of these bioactive contaminants is critical for environmental safety.

Strategically positioned at the junction of Wujiang (Jiangsu), Jiashan (Zhejiang), and Qingpu (Shanghai), the 57.6 km-long Taipu River is a vital trans-boundary watercourse in the Taihu Lake Basin and a core strategic water body within the Yangtze River Delta (YRD) Integrated Demonstration Zone. As a key ecological corridor and water security shield for the YRD region, it plays an irreplaceable role in regional integrated development and ecological co-governance, supplying drinking water (Jinze reservoir) to over 20 million residents in Shanghai and Jiaxing [15]. However, its multifaceted role also makes it vulnerable to intense anthropogenic disturbances, including industrial and agricultural activities [16]. To date, numerous related studies have been conducted on the seasonal variation characteristics and pollution assessments of conventional pollutants in the surface water of the Taipu River [17]. In recent years, PPCPs have been increasingly detected in river-type drinking water sources in eastern China [18], and extensive monitoring of multiple municipal WWTPs in Jiangsu Province has further revealed the ubiquitous occurrence of diverse PPCPs in final effluents, with maximum concentrations reaching several micrograms per liter [19,20]. However, extensive attention has been paid to conventional pollutants in the Taipu River, few studies have systematically investigated emerging contaminants (ECs) such as PPCPs, and their spatial–temporal distribution patterns and environmental transport mechanisms remain poorly understood. Furthermore, since most existing studies rely heavily on targeted analytical approaches, it remains impossible to comprehensively characterize the holistic fate of all emerging contaminants [21]. This knowledge gap, combined

with the river's unique representative status, ecological sensitivity, and critical societal importance as a drinking water source, motivated our study to systematically comprehend the distribution characteristics, seasonal differences, and risk assessment of PPCPs in the Taipu River Basin.

The specific objectives of this study were as follows: (1) To investigate the occurrence and concentration levels of PPCPs in the surface water of the Taipu River; (2) To analyze their spatial-temporal distribution patterns and seasonal differences; and (3) To assess the ecological risks posed by the detected PPCPs in the study area. The research results are expected to fill the knowledge gap of emerging pollutants in this critical trans-boundary drinking water source and provide scientific data support and a theoretical basis for water quality management, ecological risk mitigation, and pollution control policy-making in the Taipu River Basin and similar regions in the Yangtze River Delta.

2. Materials and Methods

2.1. Reagents and Materials

A total of 54 PPCPs were selected for this study, comprising 12 sulfonamides (SAs), 7 macrolides (MLs), 8 hormones, 4 quinolones (QNs), 4 endocrine disruptors, 4 non-steroidal anti-inflammatory drugs (NSAIDs), 2 anti-epileptics, 2 TCs, 1 penicillin, 2 chloramphenicols, 2 lipid modulators, 2 beta-blockers, 2 antimicrobials, 1 nitrofurantoin, and 1 indole alkaloid. High-performance liquid chromatography (HPLC)-grade methanol and acetonitrile were purchased from Merck (Darmstadt, Germany). Formic acid and ammonium hydroxide (25%) of HPLC grade were obtained from Aladdin (Shanghai, China). Hydrochloric acid (analytical grade) was supplied by Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). High-purity nitrogen ($\geq 99.999\%$) served as the carrier gas, while standard-grade nitrogen was used for solvent evaporation.

2.2. Sample Collection and Preservation

A stock solution of each standard (10,000 mg/L) was prepared in methanol. The individual solutions of 54 standards were mixed and serially diluted in water to appropriate concentrations. All solutions were liquid and were stored in ($< -15\text{ }^{\circ}\text{C}$) until analysis was needed. Water samples (labeled W1–W8) and corresponding surface sediment samples (labeled S1–S8) were collected simultaneously from 8 sampling sites in the Taipu River, China, during 16 August 2018, 22 December 2018, 16 May 2019, 19 August 2019, 17 December 2019, and 23 May 2020. Water samples were grab samples and they were collected in 1 L glass bottles wrapped in aluminum foil and kept at $4\text{ }^{\circ}\text{C}$ until extraction. All of these were extracted within 48 h. The grab sampler was used to collect about 1 kg of riverbed bottom mud, which was placed in a stainless-steel box. After the samples were transported back to the laboratory, they were stored in a $4\text{ }^{\circ}\text{C}$ refrigerator and analyzed within 7 d. The map of the sampling sites (01–08) of the Taipu River is shown in Figure 1. The exact dates for the six monitoring campaigns are detailed in Table S4.

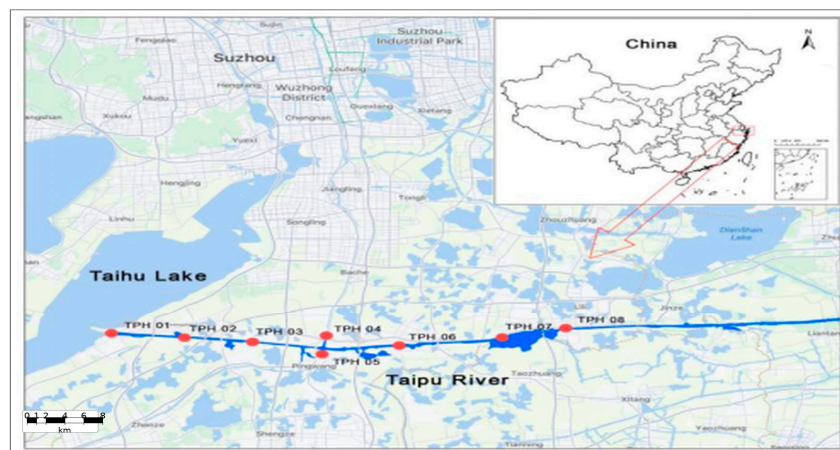


Figure 1. Map of the sampling sites (01–08) of the Taihu River.

2.3. Sample Preparation and Extraction

2.3.1. Pretreatment of Water Samples

A 1000 mL water sample was accurately measured and filtered through a 0.45 μm glass fiber membrane. Solid-phase extraction (SPE) was performed on a 24-port vacuum manifold (CNW Technologies, Shanghai, China) using an Oasis hydrophilic–lipophilic balanced (HLB) cartridge (6 cc; Waters Corporation, Milford, MA, USA), which had been pre-activated with methanol. The sample was loaded onto the cartridge at a flow rate of approximately 5 mL/min. Following sample loading, the cartridge was rinsed with 10 mL of ultrapure water and dried under vacuum for 20 min. The analytes were eluted with 10.0 mL of methanol in two separate aliquots. The resulting eluate was concentrated to near dryness under a gentle stream of nitrogen and then reconstituted to a final volume of 1 mL with methanol. After vortexing for 1 min, the solution was filtered through a 0.22 μm organic microporous membrane prior to analysis using an HPLC system coupled with a Q Exactive mass spectrometer (Thermo Fisher Scientific, Bremen, Germany).

2.3.2. Pretreatment of Sediment Samples

The collected sediment samples were lyophilized using a vacuum freeze-dryer (Scientz, SCIENTZ-10N, Ningbo, China) and subsequently ground into a fine powder. A 5.0 g portion of the powdered sediment was weighed and subjected to ultrasonic extraction for 15 min using 25 mL of an extraction solvent mixture (dichloromethane:n-hexane = 4:1, *v/v*). The mixture was then centrifuged at 5000 r/min for 5 min to facilitate the collection of the organic phase. This extraction process was repeated twice, and the resulting supernatants were pooled. The combined extract was concentrated to near dryness using a rotary evaporator (Buchi, Flawil, Switzerland) and then reconstituted to a final volume of 1 mL with methanol. After vortexing for 1 min, the solution was filtered through a 0.22 μm organic microporous membrane prior to HPLC-Q Exactive analysis.

2.4. Sample Analysis

Chromatography was performed using a Hypersil Gold C18 column (50 mm long, 2.1 mm i.d., 1.9 μm particle diameter; Thermo Fisher Scientific, Waltham, MA, USA). The column was kept at 40 °C. The flow rate was 0.3 mL/min and the injection volume was 5 μL . For the positive ion mode, mobile phase A was 0.1% formic acid in water and mobile phase D was acetonitrile. For the negative ion mode, mobile phase A was 0.05% ammonia solution and mobile phase D was acetonitrile. The gradient elution program was: 0 min, 90% A: 10% D; 0–2 min, 90% A: 10% D; 2–3 min, 70% A: 30% D; 3–6 min, 50% A: 50% D;

6–9 min, 40% A: 60% D; 9–15 min, 0% A: 100% D; 15–23 min, 0% A: 100% D; 23–23.5 min, 90% A: 10% D; 23.5–25 min, 90% A: 10% D.

Ionization was performed using an electrospray ion source with a source temperature of 375 °C and a spray voltage of 3500 V. The sheath gas flow rate was 40 arbitrary units (arb), the auxiliary gas flow rate was 10 arb, and the counter gas flow rate was 0 arb. The S-lens RF value was 50. The auxiliary gas pressure was 10 arb, and nitrogen was used as both spray gas and collision gas. Full MS/ddMS2+Discovery scan mode was used, with samples introduced in both positive and negative ion modes. Full scan resolution was 70,000 for the first level and the data-dependent scan resolution was 17,500 for the second level. The scan range was 100–1500 m/z . The automatic gain control for the first-level scan was 1.0×10^6 and the ion injection time was 100 ms. The automatic gain control for the data-dependent second-level scan was 1.0×10^5 and the maximum ion injection time was 100 ms, the isolation window was 3.0 m/z , the collision energy was 40 eV, the loop count was 3, and the dynamic exclusion time was 10.0 s.

2.5. Quality Assurance and Quality Control

Quality control (QC) was performed throughout the entire analytical process: one procedural blank, one sample duplicate, and one matrix spike recovery sample were included for every 10 environmental samples. All glassware used in the experiment (including conical flasks and heart-shaped flasks) was soaked in acetone and oven-dried before use to prevent dust and cross-contamination. Prior to sample injection, the instrument performance was calibrated to ensure that the limit of detection (LOD), sensitivity, and limit of quantification (LOQ) met the methodological requirements. A standard curve consisting of at least five concentration points was established, with a correlation coefficient R^2 exceeding 0.99. To evaluate instrumental precision, the midpoint of the calibration curve was analyzed for six consecutive injections, with the intra-day relative standard deviation (RSD) maintained below 10%. Inter-day precision was further assessed by repeating the six-injection analysis every 24 h. The recoveries of the 54 target PPCPs in surface water and sediment ranged from 72.1% to 115.3% and 60.5% to 102.6%, respectively. Quantification of target PPCPs was performed using an external standard calibration curve. No internal standard was used in this study.

2.6. Source Apportionment Analysis

This study employed the Positive Matrix Factorization (PMF) 5.0 model for source apportionment. PMF is one such model and data analysis tool that decomposes a matrix of speciated sample data into two matrices—factor contributions and factor profiles—in order to understand the factors or sources impacting the speciated sample data [22]. A total of 8 water samples and 8 sediment samples were included in the PMF model for the water and sediment datasets, respectively. The optimal number of factors for both water and sediment datasets was determined based on a combination of quantitative and qualitative criteria: (1) the ratio of Q (robust) to Q (true) was close to 1 with more than 80% of scaled residuals ranging from -3 to 3 ; (2) the F_{peak} value was maintained within a range from -1 to 1 to avoid excessive rotation; and (3) bootstrap (BS) analysis confirmed the stability and reproducibility of factor profiles (BS uncertainty $< 20\%$ for most species).

For the concentration data, the assigned value and its uncertainty depended on what the measurement result was relative to the method detection limit (MDL). Zeroes and negatives are not permitted for either the detection limit or the percent uncertainty. If the concentration in the sample was less than or equal to the MDL, the sample was given a concentration of half of the MDL and the uncertainty was calculated as five-sixths of the MDL (Equation (3)). If the concentration in the sample is greater than the MDL, the

assigned sample concentration is the measured value and the uncertainty is calculated as Equation (4). The functions are shown in Equations (2)–(5) [23].

$$x_{ij} = \sum_{k=1}^p g_{ik}f_{kj} + e_{ij} \quad (1)$$

$$S_{ij} = \begin{cases} \frac{5}{6} \times MDL & x_{ij} \leq MDL \\ \sqrt{(RSD \times x_{ij})^2 + (0.5 \times MDL)^2} & x_{ij} > MDL \end{cases} \quad (2)$$

$$Q = \sum_{i=1}^m \sum_{j=1}^n \left[\frac{x_{ij} - \sum_{k=1}^p g_{ik}f_{kj}}{S_{ij}} \right]^2 \quad (3)$$

$$Q = \sum_{i=1}^m \sum_{j=1}^n \left[\frac{x_{ij} - \sum_{k=1}^p g_{ik}f_{kj}}{S_{ij}} \right]^2 \quad (4)$$

2.7. Ecological Risk Assessment

To evaluate the ecological risks posed by EDCs, we applied the RQ (risk quotients) method [24]. The RQ for each aquatic compound was determined using the following formula:

$$RQ = \frac{MEC}{PNEC} = \frac{MEC}{E(L)C_{50}} \times AF \quad (5)$$

where MEC is the measured concentration of EDCs in the environment, PNEC is the predicted non-effect concentration, and LC50 or EC50 is the median lethal or effective concentration for crustaceans, algae, and fish. The resulting RQ values represent the severity of the potential ecological risk associated with contaminant exposure. $RQ > 1.0$ indicates high risk, $0.1 < RQ < 1$ indicates medium risk, and $0.01 < RQ < 0.1$ indicates low potential risk.

3. Result and Discussion

3.1. Occurrence and Spatial Distribution of PPCPs in Water

Non-target screening was performed using the following parameters: accurate mass error < 5 ppm, fragment ions mass error < 5 ppm, matches fragment number ≥ 1 , isotopic distribution $< 30\%$, and peak intensity in the sample above five times of that in the blank. As shown in the figure (Figure 2), through non-targeted screening across three consecutive years, a total of 54 PPCPs of concern were detected in the Taipu River Basin. Through the screening and categorization of PPCPs, PPCP suspects are classified into 15 categories based on therapeutic use, including 12 SAs, 7 MLs, 8 hormonal drugs, 4 QNs, 4 environmental hormones, 4 NSAIDs, 2 anti-epileptic drugs (AEDs), 2 tetracyclines (TCs), 1 penicillin (P), 2 chloramphenicols (CAPs), 2 lipid-regulating drugs (LRs), 2 blockers (BLs), 2 antimicrobials (ABs), 1 nitrofurantoin, and 1 indole-type alkaloid. These contaminants are distributed at different concentrations in the Taipu River Basin and are affected by the complex ecological environment and human activities in the surrounding areas of the Taipu River. Their concentrations vary at different times and periods. Six sampling campaigns were conducted across the basin between August 2018 and May 2020 to comprehensively evaluate their quantitative occurrence (Figures 3 and 4). Overall, the mean total concentration of the 54 PPCPs ($\sum 54\text{PPCPs}$) across all individual surface water samples was 28.60 ng/L, ranging from 0.1 to 178.00 ng/L. Among the screened contaminants, bisphenol A (BPA) was universally detected (100% DF) in all surface water samples, while a subset of high-frequency contaminants (DF $> 50\%$) dominated the aqueous profile, including spiramycin (SPI), ciprofloxacin (CIP), NP, indomethacin (IMC), fenoprofen (FP), CAP, and nimodipine (NM).



Figure 2. Sunburst diagram demonstrates classification of PPCPs in the Taipu River by off-target screening.

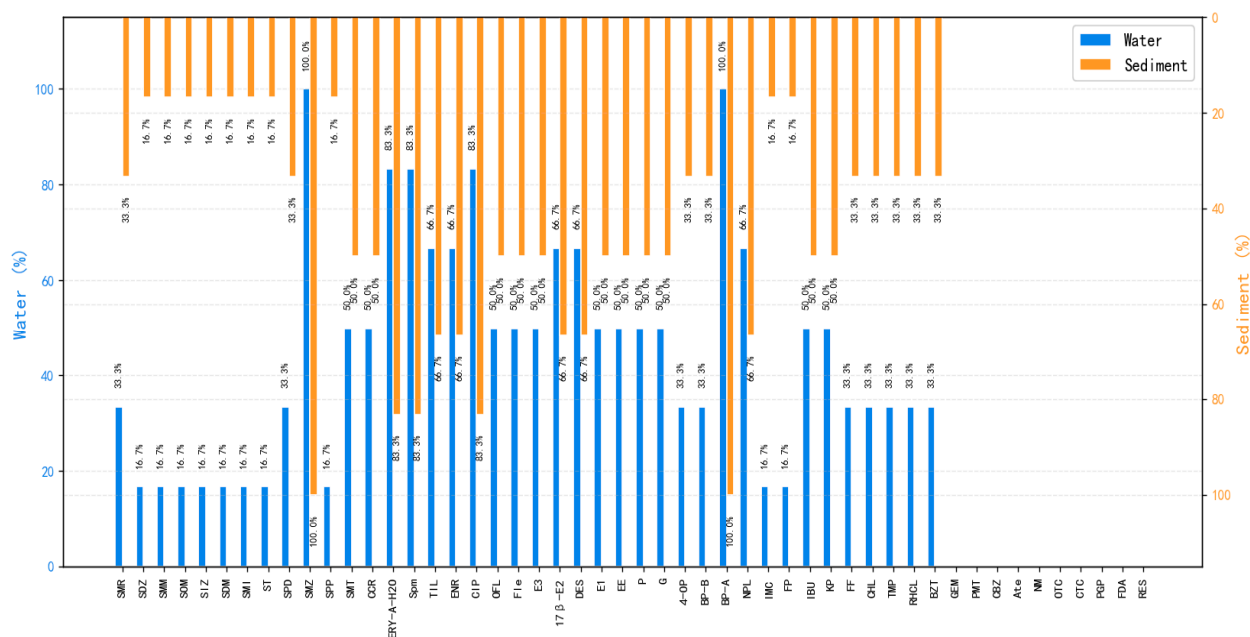


Figure 3. Frequency of detection of contaminants in river water and sediment of the Taipu River.

Antibiotics constituted a major fraction of the targeted PPCPs in the Taipu River, exhibiting distinct spatiotemporal distribution patterns in the aqueous phase. Quinolones (QNs) were predominant, exhibiting an average aqueous concentration of 44.88 ng/L (range: 7.52–144.88 ng/L). The total QN concentrations in surface water peaked in May 2018 (mean: 92.39 ng/L) but subsequently stabilized around 42.00 ng/L from December 2019 to May 2020. Within this class, enrofloxacin (ENR) and ciprofloxacin (CIP) consistently presented detection frequencies (DFs) exceeding 60% in surface water. Regarding macrolides (MLs), the mean concentration was 9.61 ng/L in the aqueous phase (range: 0.59–20.97 ng/L). Conventional MLs like erythromycin and roxithromycin were absent in water, primarily attributed to their high susceptibility to hydrolysis and biodegradation

in the aquatic environment. Spiramycin (SPI) and azithromycin (AZM) emerged as dominant contaminants (DF > 50%) in both water and sediment. Notably, AZM was stable in surface water at a mean of 18.54 ng/L. Among sulfonamides (SAs), sulfadimethazine and sulfamethoxazole (SM2) were the primary contaminants [25]. SM2, in particular, exhibited isolated concentration spikes reaching 109.54 ng/L in December 2019 and 174.88 ng/L in May 2020, which were much higher than other contaminants. As the source of the Taipu River, the changes in Taihu Lake have deeply affected the ecological balance of the Taipu River Basin [26]. Compared to the aforementioned classes, tetracyclines (TCs) were detected at relatively lower frequencies (DF < 40%) primarily during late-stage monitoring, with chlortetracycline and hygromycin hydrochloride presenting comparable aqueous concentrations of 19.93 ng/L and 21.67 ng/L, respectively.

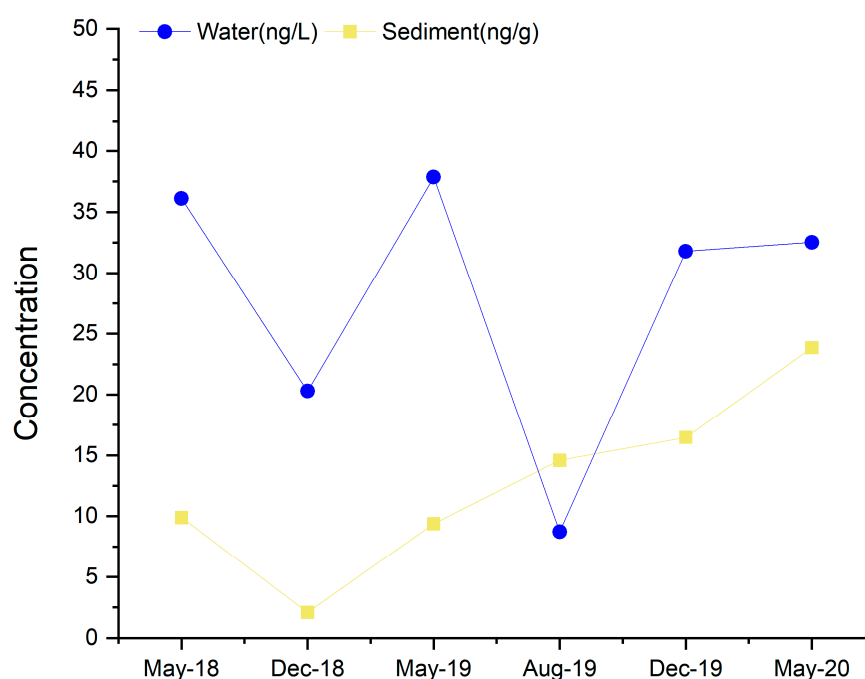


Figure 4. Trends of PPCPs in water samples and sediments in the Taipu River Basin over a three-year period.

The occurrence of endocrine-disrupting chemicals (EDCs), hormones, and NSAIDs also contributed to the aqueous contamination profile. The majority of general hormones were E3, a class of hormones found mainly in women's bodies, but rarely in men's bodies. Estrogens, discharged into rivers with domestic wastewater, can have many effects on aquatic ecosystems [27]. General hormones exhibited an average aqueous concentration of 18.75 ng/L (range: 0.16–65.46 ng/L), with 17 β -E2 predominantly detected in surface water. The average aqueous concentration of the four targeted EDCs was 18.50 ng/L, peaking during the May 2019 sampling campaign. Bisphenol A (BPA) was widely distributed, detected in over 80% of the water samples. Liu et al. investigated BPA in the water and sediments of Taihu Lake water bodies with concentrations of 13.5 ng/L and 1.9 ng/g dw, respectively [28]. The concentration levels of BPA in the water and in the sediment of Beibu Gulf in the South China Sea were 8.38 ng/L (mean) and a range of 0.56–5.22 ng/g, respectively [29]. The concentration of BPA in the water samples from the Taipu River was 7.49 ng/L, which was lower than the levels reported in Taihu Lake (13.5 ng/L) and Beibu Gulf (8.38 ng/L). The presence of BPB was also detected in the first two assays, but due to the structural similarity between BPB and BPA, BPB became a class of contaminants replacing BPA [30]. Furthermore, NSAIDs exhibited an average concentration of 31.14 ng/L in the aqueous phase (range: 0.12–148.80 ng/L); however, these contaminants were entirely

absent during the latter monitoring campaigns in December 2019 and May 2020. Other trace pharmaceuticals, including chloramphenicol (CHL), beta-blockers, and lipid-regulating agents, generally exhibited low DF and trace concentrations, contributing marginally to the overall profile.

3.2. Occurrence and Spatial Distribution of PPCPs in Sediments

Regarding the solid phase, database-based identification revealed multiple characteristic contaminants across the eight sediment monitoring sites (TPH01–TPH08) with varying numbers of signatures (Figure 5). In sediments, the average $\Sigma 54$ PPCPs concentration was 13.01 ng/g, with a range of 0.01 to 259.59 ng/g. Notably, CIP and E3 exhibited a 100% detection frequency in sediment samples throughout the three-year study period. This widespread and stable distribution underscores continuous regional partitioning and point-source deposition into the benthic catchment.

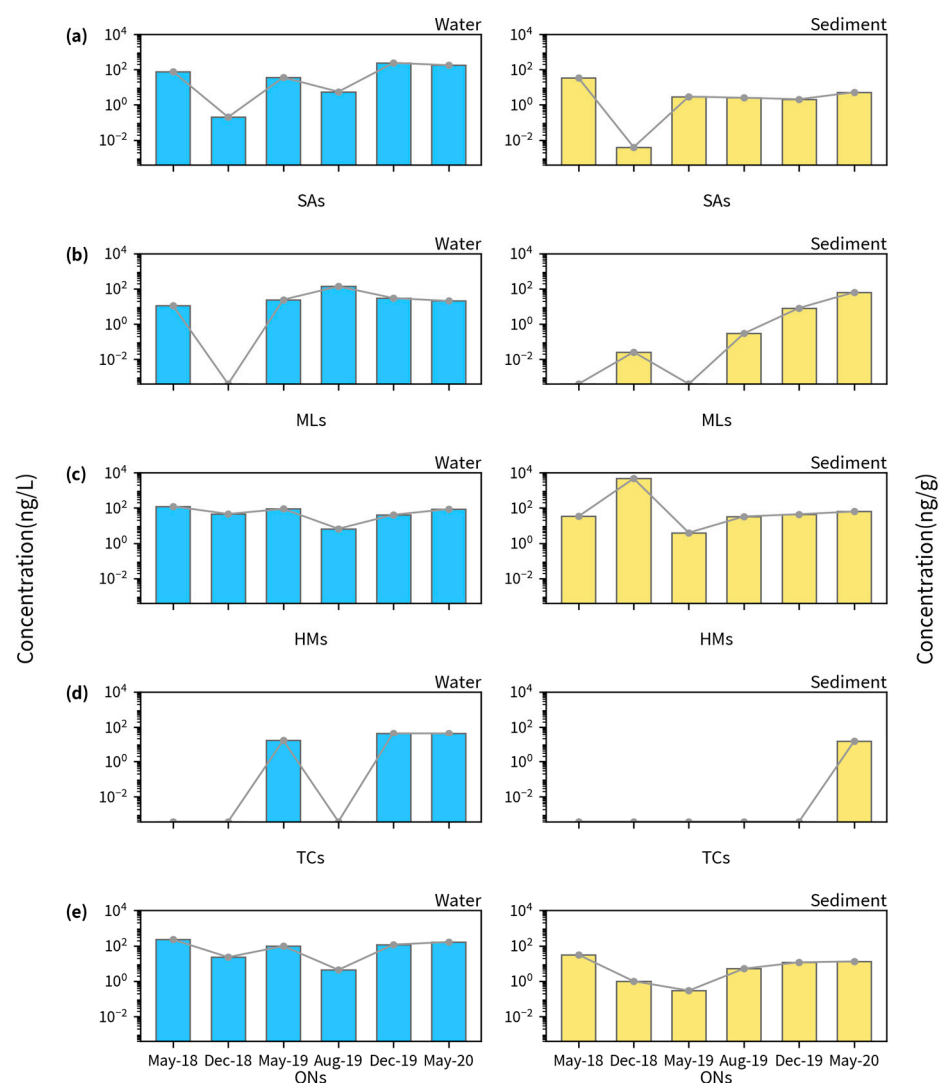


Figure 5. Trends in water samples (ng/L) and sediments (ng/g) in the Taipu River over a 3-year period (May 2018 to May 2020) for various contaminants: (a) sulfonamides (SAs); (b) macrolides (MLs); (c) heavy metals (HMs); (d) tetracyclines (TCs); and (e) quinolones (QNs).

The solid phase of the Taipu River demonstrated varied accumulation capabilities for different PPCP categories. In sediments, the average QNs concentration was 4.59 ng/g, with ENR and CIP presenting detection frequencies exceeding 50%. For MLs, the mean concentration was 0.00455 ng/g (range: 0.000001–0.05167 ng/g). Although conventional

MLs such as erythromycin and roxithromycin (ROX) were rarely detected (DF < 20%) due to their high susceptibility to degradation, both SPI and AZM demonstrated a continuous accumulation trend in sediments over the three-year monitoring period. Sulfadiazine (SDZ) and SM2 were the primary SAs contaminants detected in the sediment. Compared to the aforementioned classes, TCs were detected at relatively lower frequencies (DF < 40%) and were primarily observed during the late-stage monitoring campaigns in December 2019 and May 2020. Oxytetracycline hydrochloride (OTC) and chlortetracycline hydrochloride (CTC) detected in sediments remained relatively low, at 6.36 ng/g and 7.91 ng/g, indicating limited benthic accumulation for this specific group within the Taipu River.

Significant benthic accumulation was particularly evident for hormones and EDCs. General hormones exhibited a sediment concentration of 7.49 ng/g (range: 0.01–55.19 ng/g). Distinct phase partitioning was observed, as estriol (E3) and diethylstilbestrol (DES) demonstrated a strong tendency to accumulate in the solid phase. EDCs represented a significant contamination burden, reaching a mean sediment concentration of 47.61 ng/g (range: 0.10–259.58 ng/g). BPA was the most pervasive contaminant, being detected in 100% of the sediment samples. The concentration of BPA in the sediments was 5.40 ng/g, which exceeded the levels in Taihu Lake sediments (1.9 ng/g) and was comparable to the upper end of the concentration range reported in Beibu Gulf. Given the increasing use of bisphenol B (BPB) as a substitute for BPA, emerging bisphenol analogs should be included in future monitoring priorities. While bisphenol B (BPB) was detected in the first two assays, it was not detected in sediments in the latter four assays. Since BPA is the most typical contaminant of BPs, and there are well-documented studies confirming that BPA has adverse health effects on humans and various wildlife, such as infertility, cancer, cardiovascular diseases, etc. [31], it has been estimated that there is more than 7.7 million tons of BPA consumption worldwide [32]. In China, the annual production of BPA is about 1.67×10^5 tons, which is a relatively large chemical production level in China [33]. Due to its adverse effects on the ecological environment, the use of BPA has been strictly controlled and regulated in many regions, such as the European Union, North America, China, and Japan [30,34]. Lastly, NSAIDs exhibited a low average concentration of 0.83 ng/g in the solid phase (range: 0.01–2.19 ng/g), and similar to the aqueous phase, they were entirely absent during the latter monitoring campaigns.

3.3. Seasonal Variations and Driving Forces of PPCPs in Taipu River

The three-year monitoring program (2018–2020) revealed distinct seasonal fluctuations in the occurrence of PPCPs within the Taipu River. Generally, the total concentrations in surface water followed a discernible pattern: dry season (December and May) > wet season (August). This spatiotemporal distribution is driven by a complex interplay of hydrological conditions, physicochemical degradation, and anthropogenic activities.

Similar seasonal variations have been reported in previous studies. Regarding EDCs, our three-year monitoring (2018–2020) revealed a complex temporal evolution between the aqueous and solid phases. In the aqueous phase, typical EDCs such as BPA and E3 exhibited significant inter-annual fluctuations; for instance, BPA concentrations at certain sites showed a marked peak during 2019 (reaching up to 62.3 ng/L) before undergoing a relative decline in 2020. In stark contrast, the concentration of EDCs in sediments, particularly NP and 4-OP, demonstrated a steady and continuous upward trend over the three-year period, with NP levels in sediments escalating significantly from 2018 to 2020. This divergence suggests that while water-phase concentrations are subject to immediate hydrological dilution and seasonal degradation, the sediments act as a cumulative long-term sink for these hydrophobic contaminants.

The total concentration of PPCPs in the dry season was higher than that in the wet season. The total concentration of PPCPs in surface water showed a clear seasonal trend: dry season (December and May) > wet season (August). This distribution is primarily driven by hydrological conditions and climate-induced degradation. During the dry season, reduced river flow leads to a lower dilution capacity, resulting in the accumulation of pharmaceutical residues. In contrast, during the wet season (summer), higher temperatures and intense solar radiation enhance biodegradation and photochemical degradation processes. For instance, antibiotics such as SM2 and CIP reached their peak concentrations in the winter and spring months, likely due to increased clinical consumption and increased environmental persistence in colder temperatures. Among the various categories, QNs and SAs were the dominant antibiotic groups. CIP was detected at nearly 100% frequency in both water and sediment phases, highlighting its widespread use and environmental stability. Notably, while some NSAIDs such as ibuprofen and naproxen were intermittently detected, they were absent in several monitoring cycles (e.g., December 2019 and May 2020), suggesting a higher removal efficiency in local wastewater treatment plants or faster natural attenuation for these specific contaminants. In the sediment phase, the concentrations of several persistent PPCPs showed a progressive accumulation trend from 2018 to 2020. This indicates that the sediment acts as a long-term “sink” for hydrophobic pharmaceuticals. The continuous input from upstream trans-boundary sources and local domestic discharge exceeds the natural self-purification capacity of the river, leading to a “pseudo-persistence” phenomenon. Pseudo-persistence arises when continuous or recurrent inputs (e.g., municipal wastewater discharges) replenish compounds at timescales comparable to, or faster than, their removal by dilution, transformation, and sorption processes. Seasonality can modulate this behavior through changes in river discharge, temperature, and runoff. Similar pseudo-persistent behavior of PPCPs has been widely discussed in previous studies [35]. Our results therefore suggest that the observed seasonal patterns reflect not only environmental attenuation but also the balance between ongoing inputs and removal processes.

A previous study suggested that WWTP efficiency was affected by effluents in the wet season. From urban areas, CECs enter open water bodies mainly through WWTPs and during rain events through combined sewer overflows or stormwater outlets. Seasonal differences in PPCP use may be the main driver of seasonal variations in PPCP concentrations in the environment. This is consistent with an earlier study [36], where the overall amounts of ABs found at sampling sites were lower during summer than during any other season. PPCP degradation processes may play an important role in river water during the summer months when sunlight exposure might induce photochemical degradation processes and higher temperatures can enhance the biodegrading activities of bacterial communities. Rainfall and agricultural irrigation can cause substantial quantities of ABs entering rivers via runoff during the wet season [1].

3.4. PMF Analysis

3.4.1. Water

To identify the pollution sources of PPCPs in the Taipu River surface water, a PMF [37,38] model was applied, extracting five distinct factors (Figure 6). The percentage contributions of Factor 1 to Factor 5 were 25.4%, 24.0%, 15.0%, 21.9%, and 13.7%, respectively. Factor 1 and Factor 2 are dominated by 17 β -E2, SM2, BPA, NP, CIP, and E3, reflecting the discharge of domestic wastewater and municipal WWTP effluents. This aligns with broader regional assessments in China [39], which similarly reported these endocrine-disrupting chemicals and antibiotics as ubiquitous municipal tracers and WWTP effluents in surface waters. Factor 3 and Factor 4 are distinguished by the QNs fleroxacin

(representing 40% of the profile) and ENR, pointing strongly to medical facility wastewater and clinical pharmacology discharges, while previous source apportionment studies often attribute elevated ENR levels primarily to veterinary use and intensive aquaculture [36]. Finally, Factor 5 is primarily composed of the MLs (SPI, reaching 60%), suggesting a mixed non-point source such as localized sanitary sewer overflows or specific pharmaceutical emissions. This mixed-source profile is supported by the findings of Phillips et al. [40], who demonstrated that the distributions of pharmaceutical compounds in surface waters are highly susceptible to episodic non-point source inputs, such as combined sewer overflows during wet weather conditions. Factor 4 is the dominant contributor at W01 and W02, Factor 1 is the dominant contributor at W03 and W06, and Factor 2 is the dominant contributor at W04, W07, and W08. W05 shows a mixed pattern, with Factors 3 and 5 contributing at similarly high levels. Overall, the occurrence of PPCPs in the surface water is driven by a highly complex interplay of domestic, agricultural, medical, and industrial activities.

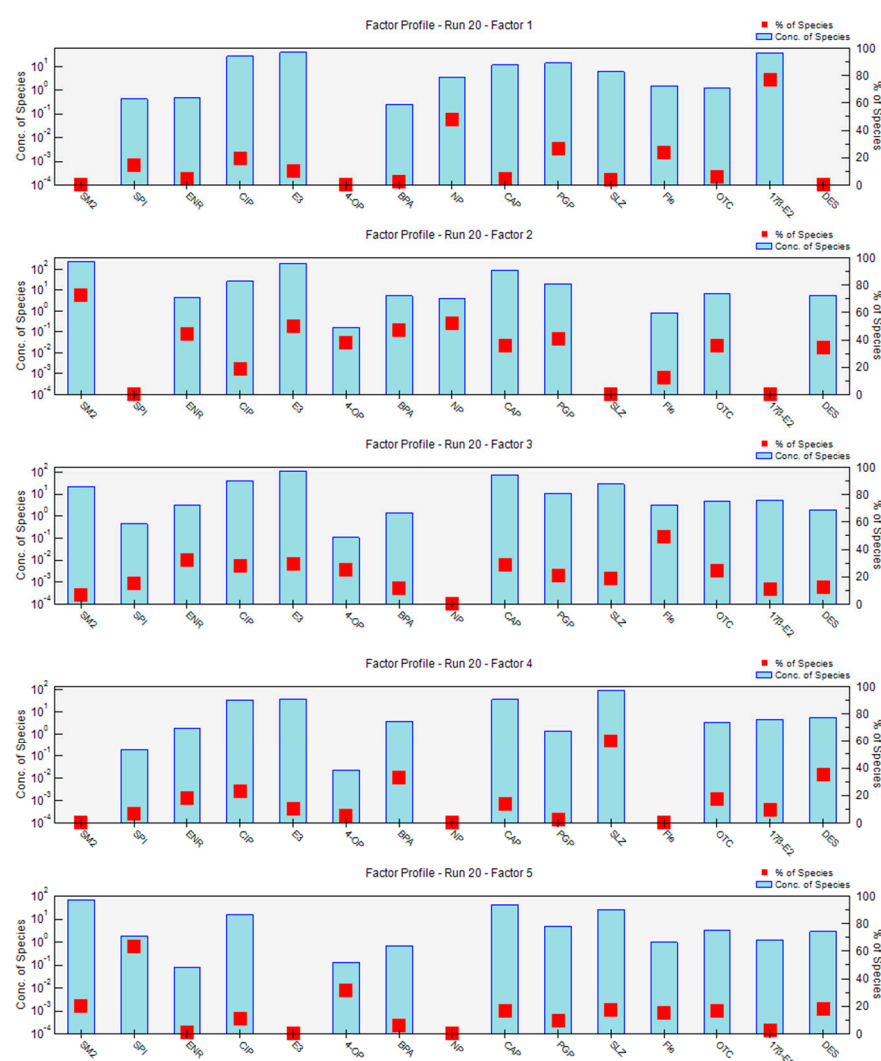


Figure 6. Source profiles of PPCPs extracted by the PMF model in the surface water of the Taipu River.

3.4.2. Sediment

To further trace the accumulation of these contaminants in the solid phase, a PMF model was applied to the Taipu River sediment samples, extracting five distinct source factors (Figure 7). The percentage contributions of Factors 1 to 5 were 8.7%, 13.3%, 26.8%, 10.2%, and 41.0%, respectively. Factor 1 and Factor 3 are overwhelmingly dominated by

the general hormone E3 (accounting for nearly 95%), identifying it as domestic wastewater discharge, as sediments act as a long-term sink for these hydrophobic contaminants. Factor 2 is characterized by the high loading of the tetracycline antibiotic OTC (reaching 80%) alongside 4-OP and fleroxacin (Fle), since TCs are broadly applied in veterinary contexts. The representative PPCPs of Factor 4 were the SA SM2 (contributing up to 50%) and the QNs OFL (45%) and ENR (40%). Factor 2 and Factor 4 suggest a combination of agricultural runoff and industrial or medical effluents. Finally, Factor 5 is primarily composed of the MLs AZM (reaching 80%) and SPI (60%). This indicates municipal wastewater or specific pharmaceutical emissions settling into the benthic zone, where macrolides often exhibit prolonged persistence under anoxic sediment conditions [41]. Factor 2 is the dominant contributor at S01 and S07, Factor 3 is the dominant contributor at S02 and S08, and Factor 5 is the dominant contributor at S03, S05, and S06. Among these sites, S05 and S06 show particularly strong dominance of Factor 5, whereas S03 exhibits a more mixed contribution pattern. Overall, the sediment PMF highlights that benthic layers serve as a cumulative sink for persistent hormones, veterinary antibiotics, and MLs originating from surrounding agricultural and domestic activities, underscoring the ecological risks posed by the potential secondary release of these contaminants during sediment resuspension.

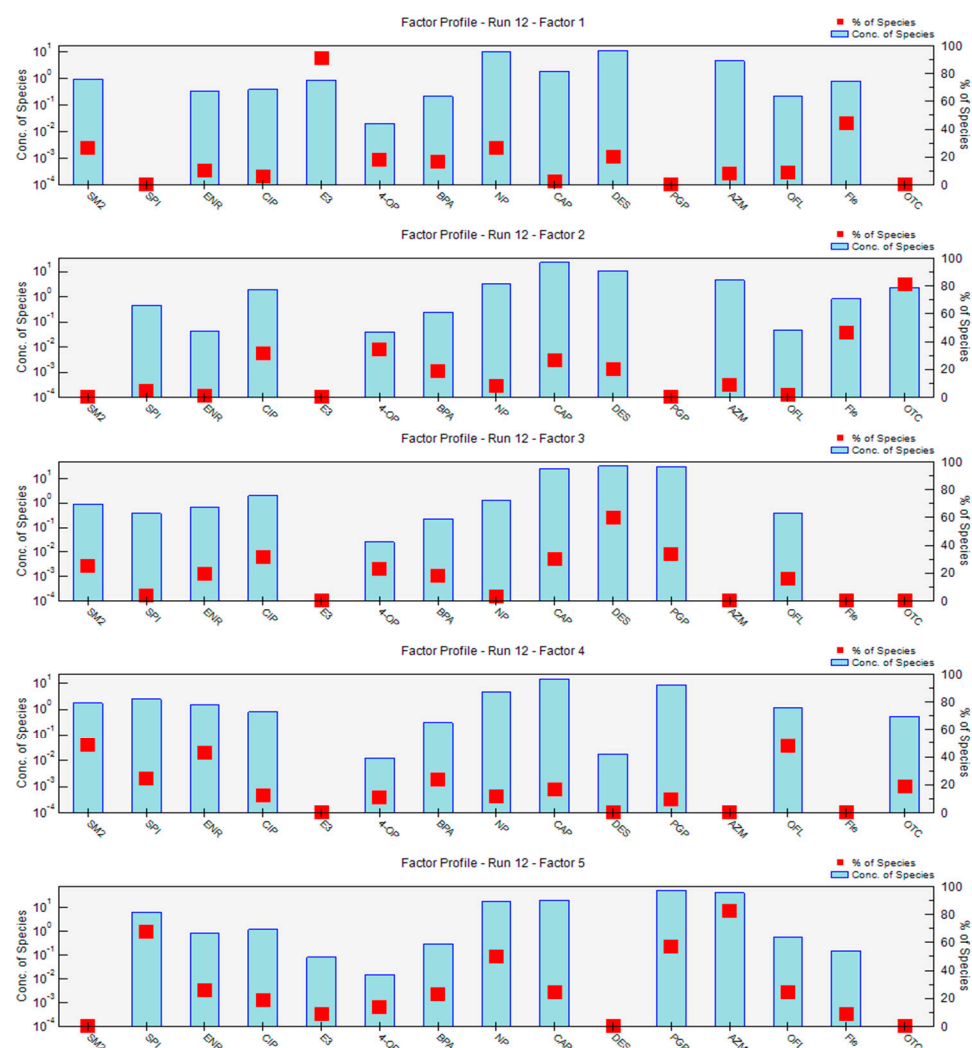


Figure 7. Source profiles of PPCPs extracted by the PMF model in the sediments of the Taipu River.

3.5. Ecological Risk of PPCPs in Taipu River

To characterize the potential ecological impacts of nine high-frequency contaminants in the Taipu River, the ecological risk assessment was performed for both surface water and sediment compartments. The mixture toxicity was considered under an additive assumption (concentration addition) as the default/conservative approach, while synergistic effects were not explicitly assessed due to limited interaction data [42]. The assessment focused on nine contaminants with high DF in this study: SM2, SPI, ENR, CIP, E3, 4-OP, BPA, NP, and CAP. The ecological risk distribution of these contaminants at each monitoring site in the Taipu River is shown in Figure 8. Based on the analysis of RQ distribution maps from six monitoring campaigns, the RQ values for these nine high-frequency contaminants in both surface water (W01–W08) and sediments (S01–S08, excluding S04) of the Taipu River were all below 1.0. Overall, the target contaminants exhibited low-to-medium ecological risk levels throughout the study period. From the perspective of specific monitoring data, CIP and E3 exhibited prominent medium-risk characteristics in surface water (W01–W08). For instance, the RQ value of CIP at site W04 once approached 0.97, while the RQ of E3 at site W07 exceeded 0.74, both falling within the medium-risk range (0.1–1.0). In comparison, the RQ values for SM2 in the water phase were predominantly distributed between 0.1 and 0.55. In sediments (S01–S08, excluding S04), the risk levels of ENR and CIP were generally higher than those in the aqueous phase. Monitoring data revealed that the RQ values of ENR at multiple sites (e.g., S02 and S06) remained consistently around 0.76 to 0.79, and the maximum RQ of CIP in sediments reached 0.64 (site S07), both indicating a stable medium-risk status. Meanwhile, the RQ of SPI in sediments showed greater fluctuations, with a maximum value of approximately 0.27. The integrated analysis of the six distribution maps indicates that although the Taipu River is currently not threatened by high ecological risk ($RQ < 1.0$) from any single high-frequency contaminant, most antibiotics and hormones widely maintain medium-risk levels in both water and sediment matrices. Given the extremely high DF and the co-occurrence of these contaminants, their long-term combined pressure on the ecosystem of the Taipu River—a strategic water source—warrants continuous and prioritized attention.

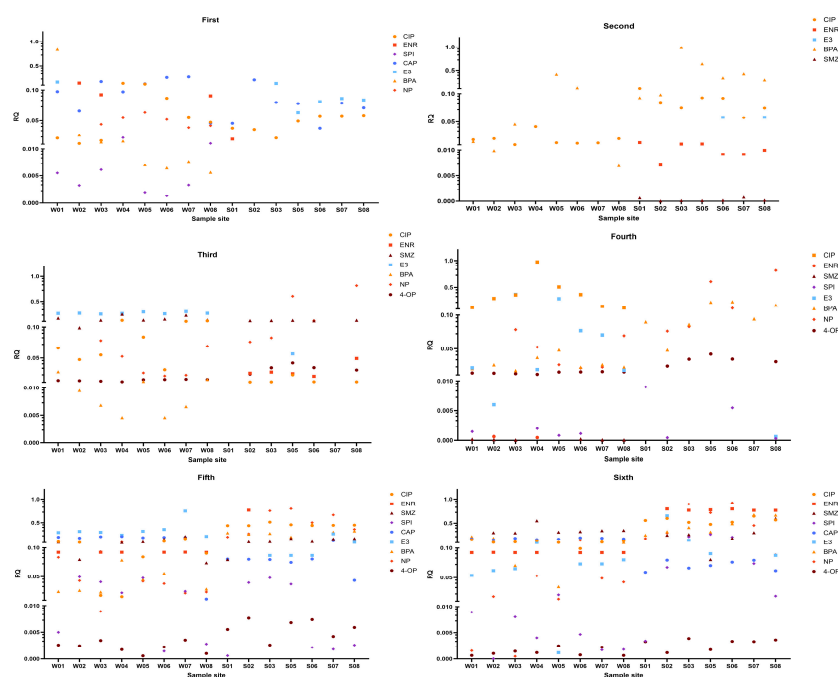


Figure 8. Distribution of risk quotients (RQs) for nine high-frequency contaminants in surface water and sediments across six monitoring campaigns in the Taipu River.

4. Conclusions

This comprehensive three-year study (2018–2020) elucidates the environmental fate, source dynamics, and ecological risks of 54 targeted PPCPs within the Taipu River. The results revealed spatiotemporal heterogeneity in PPCP distribution, with fluoroquinolones and sulfonamides dominating in the aqueous phase, whereas macrolides and fluoroquinolones showed relatively greater contributions in sediment. While surface water concentrations were highly susceptible to hydrological variations and climate-induced degradation—typically peaking during the dry season—sediments consistently acted as a cumulative, long-term sink for hydrophobic contaminants such as specific endocrine-disrupting chemicals and quinolones. Furthermore, source apportionment via the PMF model revealed that the PPCP burden is not derived from a single point source, but rather represents a complex interplay of domestic wastewater, agricultural and aquaculture runoff, and medical facility discharges. Ecological risk assessment indicated that the risk quotients (RQs) for nine high-frequency contaminants were low-to-medium risk. Future monitoring could consider focusing on potential indicator PPCPs (e.g., compounds with consistently high detection frequencies, elevated concentrations, and/or notable risk contributions), while the final indicator list should be refined as more temporal data become available. Specifically, management efforts should prioritize upgrading wastewater treatment plants to enhance PPCP removal, strengthening controls on agricultural and aquaculture runoff, and improving pretreatment and discharge management for medical facility effluents, in order to reduce multi-source PPCP inputs more effectively.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/w18141694/s1>, Table S1: Occurrence of PPCPs in waters and sediments of the Taipu River Basin; Table S2. Comprehensive list of 54 PPCPs with CAS numbers, molecular formulas, exact masses, and identification metadata; Table S3. (a) PMF input matrix for water samples (concentrations and uncertainties). (b) PMF input matrix for sediment samples (concentrations and uncertainties); Table S4. Exact sampling dates for the six monitoring campaigns in the Taipu River Basin (2018–2020); Table S5. Locations of Water and Sediment Samples; Table S6. Predicted no-effect concentrations (PNECs) of target PPCPs in water and sediment; Table S7. Minimum, maximum, and mean Risk Quotient (RQ) values for the nine high-frequency contaminants in water and sediment matrices; Table S8. The meteorological data for the sampling periods; Table S9 The PMF diagnostics of water samples; Table S10 The PMF diagnostics of sediment samples; Table S11. (1) The compound loading of water samples. (2) The compound loading of sediment samples. (3) The the percent variance of water and sediment samples; Figure S1 the TIC of water samples; Figure S2 the TIC of sediment samples; Figure S3 the concentration of PPCPs in water and sediment samples. References [43–46] are cited in the supplementary materials.

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