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白洋淀典型抗生素的源解析及其特定源风险评估

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摘要: 目前我国湖泊中抗生素污染形势严峻, 研究多集中于抗生素的时空分布与风险评价等, 而有关源解析的研究则较少. 鉴于此, 选取白洋淀为研究区, 探究典型抗生素的污染来源及其特定源风险. 运用高效液相色谱串联质谱法 (HPLC-MS) 测定样品中的四环素类 (TCs)、磺胺类 (SAs) 和喹诺酮类 (QNs) 抗生素, 并运用正定矩阵因子分解 (PMF) 模型和风险商值法 (RQ) 相结合的方法对典型抗生素进行源解析和特定源风险评估. 结果表明: ①水体和沉积物中抗生素含量范围分别为 ND ~ 2 635 ng·L⁻¹ 和 ND ~ 259.8 ng·g⁻¹; ②就水体中抗生素浓度的空间分布而言, QNs 呈“西高东低”, SAs 呈“中部高、南北低”, TCs 呈“中部低、南北高”的分布特征; 就沉积物中抗生素含量的空间分布而言, QNs 呈“中部高, 东西低”, 而 SAs 和 TCs 均呈“西高东低”的分布特征; ③就抗生素的来源而言, 水产养殖 (33.2%) 占比最高, 其次为污水处理厂 (29.2%)、畜禽养殖 (18.9%) 和生活污水 (18.7%); ④就生态风险而言, 恩诺沙星 (ENR) 和氟甲喹 (FLU) 处于中高风险水平; ⑤就特定源风险的空间分布而言, 除 S1 处水产养殖处于高风险水平, 其余样点各源均处于中低风险水平; 就源的种类而言, 水产养殖处于中高风险水平, 其余各源均处于中低风险水平. 因此, 针对白洋淀抗生素的主要来源及其特定源风险等级, 需采取更为精准科学的抗生素风险管控.

关键词: 白洋淀; 抗生素; 空间分异; 源解析; 特定源风险; PMF 模型

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Source Apportionment and Source-specific Risk of Typical Antibiotics in Baiyangdian Lake

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Abstract: The current situation of antibiotic pollution in lakes is critical. At present, most of the previous studies on antibiotics in lakes have focused on the spatiotemporal distribution and risk assessment, while less attention has been paid to the source apportionment. Ultra-high performance liquid chromatography-mass spectrometry was used to determine the concentration of tetracyclines (TCs), sulfonamides (SAs), and quinolones (QNs) in the samples. The source apportionment and source-specific risk of typical antibiotics in the study area were analyzed using the combination of a PMF model and risk quotients (RQ). The results showed that ① the total concentrations of target antibiotics (Σ antibiotics) ranged from ND to 2 635 ng·L⁻¹ for surface water and from ND to 259.8 ng·g⁻¹ for sediments. ② The spatial distribution of QNs in surface water decreased from west to east, SAs decreased from middle to north and south, and TCs increased from middle to north and south. In the sediment, QNs decreased from middle to east and west, whereas SAs and TCs increased from east to west. ③ Aquaculture was the major antibiotic source, accounting for the highest proportion (33.2%), followed by sewage treatment plants (29.2%), livestock activities (18.9%), and domestic sewage (18.7%). ④ The ecological risk assessment results showed that enrofloxacin and flumequine were at a medium-high risk level. ⑤ For the spatial distribution of source-specific risk, the results showed that the aquaculture at S1 was at a high risk level, whereas the source-specific risks for other sites were at a medium-low risk level. In terms of source types, aquaculture was at a medium-high risk level, whereas the other sources were at a medium-low risk level. Therefore, considering the major sources and source-specific risk level of antibiotics, more precise and scientific antibiotic risk control should be adopted in Baiyangdian Lake.

Key words: Baiyangdian Lake; antibiotics; spatial distribution; source apportionment; source-specific risk; PMF model

自 1928 年青霉素发现以来, 抗生素广泛用于疾病预防和治疗, 以及动物生长促进剂^[1,2]. 根据其化学结构, 抗生素可以分为喹诺酮类 (quinolones, QNs)、磺胺类 (sulfonamides, SAs)、大环内酯类 (macrolides, MLs)、四环素类 (tetracyclines, TCs) 和 β -内酰胺类 (β -lactam) 等^[3]. 目前, 我国已成为抗生素最大的消费国和生产国, 如 2013 年我国抗生素的生产量已达 24.8 万 t, 使用量高达 16.2 万 t, 其中 SAs、TCs 和 QNs 的使用量分别占 5%、7% 和 17%^[4]. 然而, 抗生素不能完全被人体和动物吸收, 约有 30%~90% 会以母体化合物或代谢产物的形式随

尿液或者粪便排出体外^[5-7]. 随后, 抗生素通过各种途径源源不断进入水环境中^[8], 例如: 城市污水处理厂尾水排放、畜禽养殖废水、医疗废水和农业排水等^[9-13]. 据统计我国每年约有 2.47 万 t 抗生素进入水环境中^[4]. 在水环境中抗生素可通过食物网在生物体中进行生物累积和营养放大, 进而对水生生物和生态系统造成危害^[14]. 此外, 抗生素还会诱导产生耐药

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菌(antibiotics resistance bacterias, ARBs)和耐药基因(antibiotics resistance genes, ARGs),对人体健康和生态安全构成严重威胁^[15].因此,抗生素耐药性问题已成为21世纪人类面临的重大挑战之一^[16].

目前,已在河流^[17]、河口^[18]、海湾^[19]和湖泊^[20]等多种水环境中检出70余种抗生素.其中,湖泊作为抗生素的重要储库,在2019年排放到我国湖泊中抗生素总量高达5711 t^[21].整体而言,我国湖泊中抗生素污染形势较为严峻,如:洞庭湖^[22]中已检出4大类12种抗生素(总浓度1.06~135.40 ng·L⁻¹),大通湖^[23]已检出4大类抗生素(总浓度0.162~61.89 ng·L⁻¹),鄱阳湖^[24]中已检出18种抗生素(总浓度ND~56.2 ng·L⁻¹).湖泊中抗生素的研究多集中于其时空分布及其风险评价等^[25~27],而抗生素的源解析则较少关注.目前,抗生素源解析的方法主要包括正定矩阵因子分解(positive matrix factor, PMF)模型^[18]、Unmix模型^[19]和多元线性回归主成分分析(PCA-MLR)模型^[28]等.其中,PMF模型对解析结果具有非负约束的特点,已广泛应用于抗生素的源解析.对环境管理者而言,定量解析抗生素的污染源将有助于抗生素及其风险的科学精准管控.

白洋淀作为雄安新区的核心生态功能区,将为新区的建设提供重要生态支撑^[29].然而,新区成立前,白洋淀长期接收上游城市污水、工业废水、生活污水和周边养殖废水等,导致淀区抗生素污染形势较为严峻.如水体中SAs检出率最高(78.1%),

其浓度为0.86~1563 ng·L⁻¹;沉积物中QNs检出率最高(2.22%~100%),其含量为65.5~1166 ng·g⁻¹^[30].然而,目前有关白洋淀中抗生素的研究主要集中于其污染特征、生物累积及其风险评估等^[31,32].因此,本研究选取白洋淀为研究区,根据其土地利用和人为干扰特征,选取13个样点,分别采集水体和沉积物样品,共选取3大类26种抗生素为目标物,明晰白洋淀中典型抗生素的空间分异特征;基于PMF模型定量解析水体和沉积物中抗生素的来源;结合源解析和风险商值法(risk quotients, RQ)来评估特定源风险,以期对白洋淀抗生素及其风险的精准管控提供理论支撑和科学依据.

1 材料与方法

1.1 研究区概况与样品采集

白洋淀位于华北平原,共有8条入淀河流,包括府河、瀑河和潞龙河等.淀内主要有143个大小不等的淀泊和3700条沟壕,且此前淀内养殖业发达,人口密集,大量生活污水和养殖废水直排入淀,加剧了白洋淀中抗生素的污染形势^[33,34].

2018年4月,根据白洋淀土地利用类型和人为干扰特征并结合现场实际情况共设置13个采样点(图1),分别为:南刘庄(S1)、鸳鸯岛(S2)、烧车淀(S3)、王家寨(S4)、寨南(S5)、杨庄子(S6)、枣林庄(S7)、圈头(S8)、东田庄(S9)、后塘(S10)、采蒲台(S11)、范峪淀(S12)和金龙淀(S13).水体样

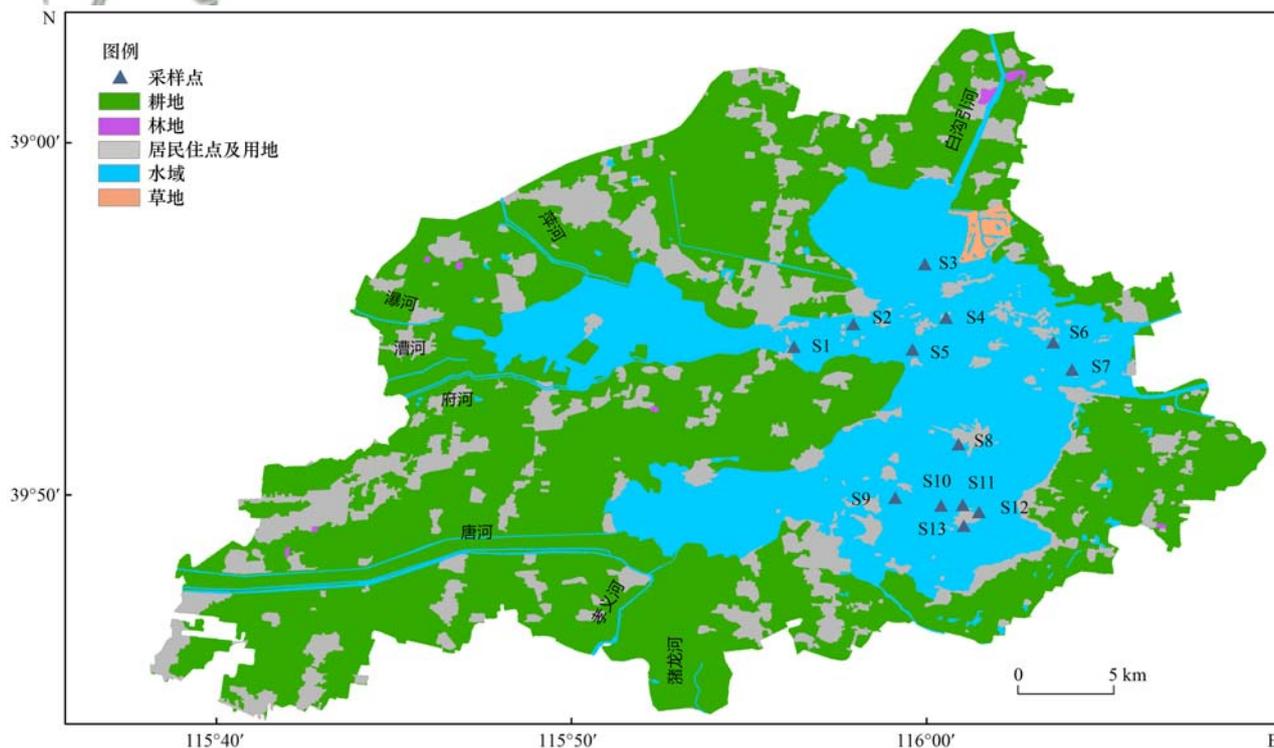


图1 白洋淀采样点

Fig. 1 Sampling sites of Baiyangdian Lake

品使用 5 L 棕色玻璃瓶采集,密封保存,使用保温箱避光运回实验室,进行适当过滤后存放在 4℃ 冰箱内,待后续分析^[35,36]. 沉积物使用彼得森采泥器在各样点 0~10 cm 处采集表层约 500 g 样品,去除碎屑和石子等异物,装入聚乙烯密封袋中并进行标记^[35,37],低温保存运回实验室,放在 -20℃ 冰箱中,保存备用. 使用便携式水质分析仪 (HYDROLAB, DS5X) 对水温 (temperature, T)、pH 和溶解性固体总量 (total dissolved solids, TDS) 等基本水质参数进行现场测定.

1.2 抗生素分析方法

1.2.1 化学试剂

目标抗生素共 3 大类 26 种:包括 6 种 QNs [氧氟沙星 (ofloxacin, OFL)、恩诺沙星 (enrofloxacin, ENR)、氟甲喹 (flumequine, FLU)、氟罗沙星 (fleroxacin, FLE)、诺氟沙星 (norfloxacin, NOR) 和马波沙星 (marbofloxacin, MAR)], 11 种 SAs [磺胺嘧啶 (sulfadiazine, SDZ)、磺胺噻唑 (sulfathiazole, STZ)、磺胺吡啶 (sulfapyridine, SPY)、磺胺甲基嘧啶 (sulfamerazine, SMR)、磺胺二甲嘧啶 (sulfamethazine, SMT)、磺胺对甲氧嘧啶 (sulfameter, SME)、磺胺间甲氧嘧啶 (sulfamonomethoxine, SMM)、磺胺氯吡嗪 (sulfachloropyridazine, SCP)、磺胺甲基异噁唑 (sulfamethoxazole, SMX)、磺胺二甲异噁唑 (sulfisoxazole, SIZ) 和磺胺甲氧嘧啶 (sulfamethoxypridazine, SMP)] 以及 9 种 TCs [四环素 (tetracycline, TC)、强力霉素 (doxycycline, DC)、土霉素 (oxytetracycline, OTC)、去甲金霉素 (demeclocycline, DMC)、金霉素 (chlortetracycline, CTC)、盐酸甲烯土霉素 (metacycline, MET)、米诺环素 (minocycline, MCN)、4-差向脱水四环素 (4-epi-anhydrotetracycline, EATC) 和脱水四环素 (anhydrotetracycline, ATC)]. 标准品购自 Sigma-Aldrich (steinheim, Germany), 纯度均大于 95%.

1.2.2 样品前处理

准确量取 1 L 水样经 0.45 μm 玻璃纤维滤膜过滤,加入 0.2 g 的乙二胺四乙酸二钠 (Na_2EDTA), 用 1 $\text{mol}\cdot\text{L}^{-1}$ 的硫酸溶液调节 pH 为 3.0. 用 6 mL 甲醇、3 mL 盐酸 (0.5 $\text{mol}\cdot\text{L}^{-1}$) 和 6 mL 超纯水液使 HLB 柱活化, 然后以 2~5 $\text{mL}\cdot\text{min}^{-1}$ 的流速通过 HLB 小柱进行萃取. 上样后, 用 10 mL 超纯水淋洗并弃去淋洗液, 负压条件下抽空干燥 30 min, 然后依次用 6 mL 体积比为 2% 的氨水甲醇溶液和 6 mL 纯甲醇溶液进行洗脱, 洗脱液经氮吹 (40℃) 至近干后, 用甲醇水溶液 (甲醇: 水 = 1:1, 体积比) 定容至 1

mL, 过 0.22 μm 滤膜并转移至棕色瓶中, 待上机分析^[38,39].

取部分冷冻干燥后的沉积物样品进行粉碎和研磨, 过 40 目筛, 放入 10 mL 离心管中. 准确称取样品 1 g, 与适量干燥的硅藻土 (Na_2EDTA 处理过的) 充分混合, 并以乙腈-磷酸盐缓冲液 (pH = 3) 作为萃取液. 使用 ASE 350 快速溶剂萃取仪 (Thermo, Germany) 进行萃取, 循环 2 次, 再用平行浓缩蒸发仪 (Buchi, Switzerland) 将萃取液浓缩至萃取剂小于 1 mL, 转移至锥形瓶中, 使用超纯水稀释至 200 mL, 其他操作遵循上面水样品的步骤^[35,40].

1.2.3 样品分析

采用超高效液相色谱-三重四级杆串联质谱联用仪 (HPLC-MS/MS) 对样品进行测定, 使用 Agilent 1200 系列 HPLC (色谱柱: C18, 2.1 $\text{mm}\times 50\text{mm}$, 1.8 μm), 质谱为安捷伦 6470 三重四级杆质谱系统. 流动相 A 为 0.1% 的甲酸水溶液, 流动相 B 为甲醇和 0.1% 的甲酸溶液, 流速 0.3 $\text{mL}\cdot\text{min}^{-1}$, 进样量 5 μL . 质谱条件为电喷雾离子源, 采用多重选择检测模式 (MRM), 干燥气温度为 350℃, 干燥气体流速为 11 $\text{L}\cdot\text{min}^{-1}$, 毛细管电压为 $\pm 3\ 500\text{V}$, 雾化气压力为 45 psi (310.5 kPa)^[32].

1.2.4 质量控制

采用内标法定量. 配制浓度分别为 0.1、0.5、1.0、5.0、10.0 和 100 $\text{ng}\cdot\text{mL}^{-1}$ 的 6 个系列标准溶液, 并设置空白组. 经 HPLC-MS 分析获得质量浓度与峰面积的标准曲线, 相关系数均 ≥ 0.99 , 各目标抗生素的回收率为 72.4%~104.6%.

1.3 源解析方法

PMF 模型通过将样品数据分解成两个矩阵: 因子贡献矩阵 (\mathbf{G}) 和因子分布矩阵 (\mathbf{F}), 以及一个残差矩阵 (\mathbf{E})^[41], 其计算公式如式 (1):

$$\mathbf{X}_{ij} = \sum_{k=1}^p \mathbf{G}_{ik} \mathbf{F}_{jk} + \mathbf{E}_{ij} \quad (1)$$

式中, i 为样品数; j 为污染物种类; p 为污染源数量.

通过 PMF 模型最小化累积残差 Q 值得到因子贡献与分布, 如公式 (2) 所示:

$$Q = \sum_{i=1}^n \sum_{j=1}^m \left(\frac{\mathbf{E}_{ij}}{\mathbf{u}_{ij}} \right) \quad (2)$$

式中, n 为样本数量; m 为抗生素数量; \mathbf{u}_{ij} 为抗生素 j 的不确定度, 计算公式如下

$$\mathbf{u}_{ij} = \begin{cases} \frac{5}{6} \text{MDL}, & c \leq \text{MDL} \\ \sqrt{(\sigma_i \times x_{ij})^2 + \text{MDL}^2}, & c > \text{MDL} \end{cases} \quad (3)$$

式中, σ_i 为抗生素浓度的相对标准偏差; c 为抗生

素浓度; MDL 为方法的检出限。

本研究选取水体和沉积物中检出率大于 30% 的抗生素,按(3)进行计算,输入 PMF 模型进行源解析。随机选取 20 作为初始起点进行迭代计算,取 3~8 个因子分别运算,对比发现选择因子数为 4 时, R^2 均大于 0.60 且所有抗生素的残差都在 -3 和 3 之间并服从正态分布,表明所选抗生素能够很好地被模拟。

1.4 生态风险评估

多种抗生素同时存在于水体中会导致毒性作用加强^[42],因此本研究采取联合风险商(RQ_{sum})来表征抗生素的生态风险,其计算公式如下:

$$RQ_{sum} = \sum RQ \quad (4)$$

$$RQ = MEC/PNEC \quad (5)$$

$$PNEC = L(E)C_{50}/AF \quad (6)$$

式中, RQ 为单一抗生素的生态风险商^[43,44]; RQ_{sum} 为联合风险商; MEC 为实测浓度; PNEC (predicted no-effect concentration) 为无效应浓度; LC_{50} 为半数致死浓度; EC_{50} 为半数有效浓度; AF (assessment factor) 为评价因子。 RQ_{sum} 的分类标准: $0.01 < RQ_{sum} \leq 0.1$ 为低风险; $0.1 < RQ_{sum} \leq 1$ 为中风险; $RQ_{sum} > 1$ 为高风险。已有研究的相关数据见表 1。

本研究中,将结合不同样点的 RQ_{sum} 和源的贡献率 C_p 进行特定源风险评估,如式(7)所示,

$$RQ_p = RQ_{sum} \times C_p \quad (7)$$

式中, C_p 为水体中不同污染源的贡献率。

表 1 不同抗生素对应最敏感生物毒理数据¹⁾

Table 1 Data of the most sensitive biotoxicology for different antibiotics

抗生素	对应最敏感生物	E(L)C ₅₀ /mg·L ⁻¹	评价因子(AF)	PNEC /ng·L ⁻¹	文献
OFL	<i>P. subcapitata</i>	14.400	1 000	14 400.0	[45]
ENR	<i>Microcystis aeruginosa</i>	0.049	1 000	49.0	[45]
FLU	藻类	1.960	1 000	1 960.0	[45]
FLE	藻类	1 128.329	1 000	1 128 329	[45]
MAR	藻类	148.890	1 000	148 890.0	[45]
SDZ	<i>S. capricornutum</i>	2.2	1 000	2 200	[46]
SMT	藻类	1.56	1 000	1 560	[47]
SCP	水蚤	8.03	1 000	8 030	[48]
SMP	藻类	3.82	1 000	3 820	[48]
SPD	<i>Lminor</i>	0.46	1 000	460	[49]
SMM	<i>Lemna minor</i>	8.56	1 000	8 560	[50]
SMX	<i>S. leopoliesis</i>	0.027	1 000	27	[51]
TC	<i>P. subcapitata</i>	3.31	1 000	3 310	[51]
DC	<i>Green algae</i>	0.316	1 000	316	[52]
OTC	<i>P. subcapitata</i>	1.04	1 000	1 040	[23]
CTC	<i>Chlorella vulgaris</i>	0.005	1 000	5	[52]

1) SME、DMC、MET、EATC、ATC 和 MCN 的 E(L)C₅₀、AF 和 PNEC 值暂缺

1.5 数据分析

使用 Microsoft Excel 2016 和 SPSS 26 软件进行数据处理和统计分析;用 EPA PMF 5.0 模型对抗生素进行源解析;使用 ArcGIS 10.7 和 Origin pro 2021 软件进行绘图。

2 结果与分析

2.1 白洋淀中典型抗生素的污染特征

在水体中(表 2),共检出 23 种抗生素,11 种抗生素的检出率高达 100%,总浓度范围为 252.1~2 957 ng·L⁻¹,均值为 35.57 ng·L⁻¹。就各类抗生素的平均检出率而言,TCs 的检出率最高(100%),其次为 QNs(76.9%)和 SAs(26.9%)。就各类抗生素的浓度而言, ρ (QNs) 范围为 243.3~2 946 ng·L⁻¹,平均值为 154.1 ng·L⁻¹; ρ (SAs) 范围为 ND~9.75

ng·L⁻¹,平均值为 0.25 ng·L⁻¹; ρ (TCs) 范围为 8.48~13.77 ng·L⁻¹,平均值为 1.14 ng·L⁻¹。就单种抗生素的浓度平均值而言,呈 FLU > OFL > FLE > MAR > ENR > ATC > DMC > CTC > MCN > SMX 的趋势。其中, ρ (FLU) 最高,其范围为 144.5~2635 ng·L⁻¹,占抗生素总浓度的 78.2%。

在沉积物中(表 2),26 种抗生素均检出,检出率均在 40% 以上,总含量范围为 26.14~346.8 ng·g⁻¹。其中,QNs 的平均检出率最低(87.7%),但其含量平均值最高(141.1 ng·g⁻¹),远高于 SAs(0.24 ng·g⁻¹)和 TCs(1.27 ng·g⁻¹)。就单个抗生素的含量平均值而言,呈 OFL > FLU > FLE > ENR > MAR > OTC > DMC > ATC > CTC > MCN 的趋势。其中 ω (OFL) 最高,其范围为 4.54~259.8 ng·g⁻¹,占抗生素总含量的 59.8%。

表 2 白洋淀水体和沉积物中抗生素的检出情况¹⁾

Table 2 Detection of antibiotics in surface water and sediments in Baiyangdian Lake

抗生素	水体				沉积物				
	检出率 /%	最小值 /ng·L ⁻¹	最大值 /ng·L ⁻¹	平均值 /ng·L ⁻¹	检出率 /%	最小值 /ng·g ⁻¹	最大值 /ng·g ⁻¹	平均值 /ng·g ⁻¹	
QNs	MAR	76.9	ND	75.60	21.94	76.9	ND	6.01	2.82
	FLE	69.2	ND	151.5	33.84	92.3	ND	11.26	5.55
	OFL	100	ND	428.1	109.2	100	4.54	259.8	32.47
	ENR	38.5	ND	110.7	13.17	69.2	ND	12.96	4.14
	FLU	100	144.5	2 635	602.6	100	0.54	46.15	9.28
TCs	MCN	100	1.01	1.64	1.16	100	1.29	1.56	1.38
	TC	100	0.63	0.67	0.60	100	0.42	0.85	0.69
	OTC	100	0.46	2.39	0.87	100	0.49	7.53	1.74
	DMC	100	1.00	3.54	1.48	100	1.55	3.53	1.71
	CTC	100	1.26	2.00	1.15	100	1.25	2.12	1.46
	MTC	100	0.84	2.71	0.90	100	0.82	1.48	0.96
	DC	100	1.03	1.45	1.15	100	1.03	1.32	1.11
	EATC	100	0.60	2.71	1.11	100	0.59	1.54	0.89
ATC	100	1.24	2.02	1.57	100	1.22	2.56	1.54	
SAs	SDZ	61.5	ND	2.45	0.55	100	ND	1.94	0.46
	STZ	0.0	ND	ND	0	100	ND	0.41	0.18
	SPY	23.1	ND	0.31	0.02	100	ND	5.59	0.81
	SMR	0.0	ND	ND	0	100	ND	0.28	0.11
	SMT	7.7	ND	0.35	0.03	100	ND	0.32	0.19
	SME	15.4	ND	0.12	0.02	92.3	ND	0.22	0.08
	SMP	23.1	ND	0.47	0.08	61.5	ND	0.24	0.09
	SMM	23.1	ND	0.58	0.11	100	ND	0.25	0.09
	SCP	7.7	ND	0.40	0.01	46.2	ND	0.29	0.16
	SMX	53.8	ND	6.47	1.16	100	ND	0.81	0.26
	SIZ	0.0	ND	ND	0	100	ND	0.34	0.18

1) ND 表示未检出

2.2 抗生素的空间分异特征

抗生素在水体和沉积物中的空间分布特征如图 2 和图 3 所示,相关分析表明水体中各类抗生素均无相关关系,而沉积物中 QNs 和 TCs 显著正相关($P < 0.01$).

2.2.1 水体中抗生素的空间分异特征

就水体中抗生素的空间分布而言,QNs 在 S5 浓度最高($2\,946\text{ ng}\cdot\text{L}^{-1}$),最小值出现在 S12($243.3\text{ ng}\cdot\text{L}^{-1}$),总体呈“西高东低”的分布特征. SAs 在 S6 浓度最高($9.75\text{ ng}\cdot\text{L}^{-1}$),而在 S12 未检出,呈“中部高,南北低”的分布特征. TCs 浓度总体呈“中部低,南北高”的分布特征,其浓度最大和最小值分别出现在 S13($13.8\text{ ng}\cdot\text{L}^{-1}$)和 S8($8.48\text{ ng}\cdot\text{L}^{-1}$).

2.2.2 沉积物中抗生素的空间分异特征

就沉积物中抗生素的空间分布而言, ω (QNs)最大值在 S2($334.6\text{ ng}\cdot\text{g}^{-1}$),最小值出现在 S5($15.68\text{ ng}\cdot\text{g}^{-1}$),总体呈“中部高,东西低”的空间分布规律. SAs 和 TCs 均呈“西高东低”的空间分布规律,且最大值均出现在 S1($8.54\text{ ng}\cdot\text{g}^{-1}$ 和 $19.47\text{ ng}\cdot\text{g}^{-1}$),而最小值分别出现在 S12($0.69\text{ ng}\cdot\text{g}^{-1}$)和 S5($9.20\text{ ng}\cdot\text{g}^{-1}$).

2.3 抗生素源解析及相对贡献

2.3.1 水体中抗生素源解析

水体中抗生素源解析结果如图 4 和图 5 所示. 因子 1 对 CTC 的贡献相对较高,贡献率为 56.8%,且相关性分析显示 CTC 与其他抗生素相关性较弱,则其可能具有单一来源. CTC 作为抗菌药物在渔业养殖中广泛应用,检出率较高^[53~55]. 丁慧君对环鄱阳湖水产养殖区的研究表明 ρ (CTC) 高达 $162.68\text{ ng}\cdot\text{L}^{-1}$ ^[56],而淀内有众多养殖区,因子 1 推断为水产养殖.

因子 2 对 SDZ 和 SMX 的贡献率较高,分别为 62.4% 和 62.8%,且二者显著正相关($P < 0.01$),表明其可能具有相同的来源. 其中,SDZ 是家用抗菌药的主要成分,已有研究表明 SDZ 主要通过生活污水进入环境,其浓度高达 $5.0 \times 10^4\text{ ng}\cdot\text{L}^{-1}$ ^[57]. 而 SMX 用于治疗人类尿道感染,其在各种废水中均占主导地位^[58~60]. 淀内有众多的村落且人口密集,此前大量生活污水直接排入淀^[61],因子 2 推断为生活污水.

因子 3 对 DMC 和 MCN 的贡献率相对较高,分别为 58.4% 和 44.3%,且二者显著正相关(P

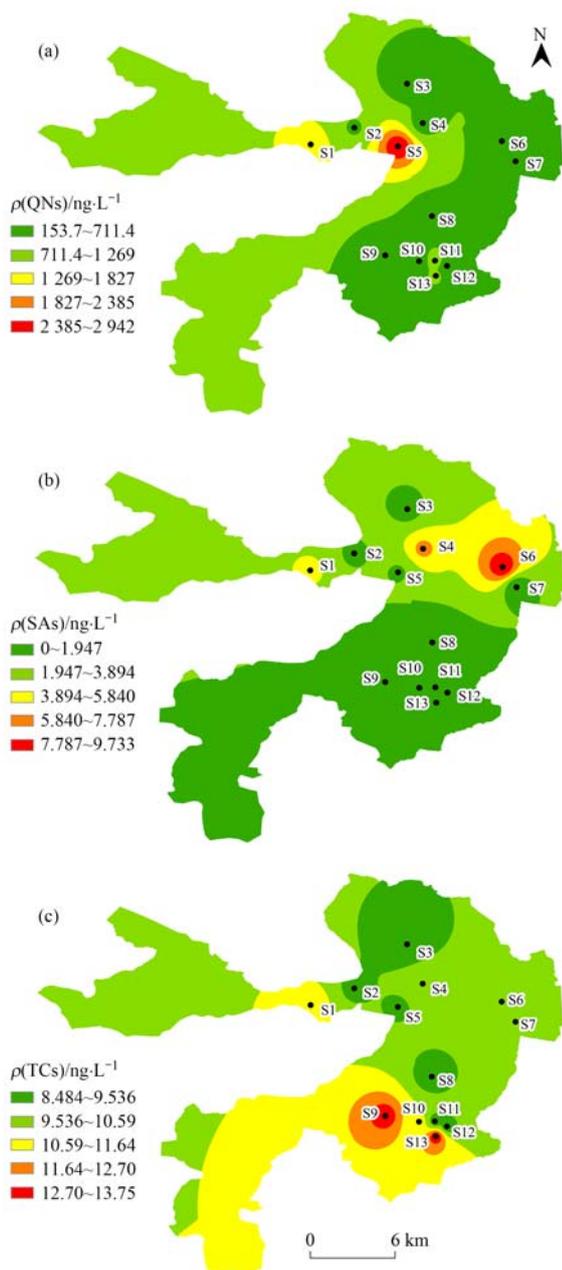


图2 白洋淀水体中喹诺酮类、磺胺类和四环素类抗生素的空间分布

Fig. 2 Spatial distribution of QNs, SAs, and TCs in the surface water of Baiyangdian Lake

< 0.01), 则其可能具有相同的来源. 研究表明 DMC 和 MCN 在制药工艺中常作为原料药和中间体^[62,63], 而河北省作为制药大省且研究区长期接收来自上游城市医药和工业等废水^[64], 加之, 目前的污水处理技术不完善, 使得未完全降解的抗生素排入自然水体^[65,66], 因子 3 推断为污水处理厂.

因子 4 对 MAR、FLE 和 OTC 的贡献率相对较高, 分别为 63.5%、64.0% 和 35.7%, 且 MAR、FLE 和 OTC 两两之间显著正相关 ($P < 0.01$), 表明其可能具有相同的来源. 作为典型的

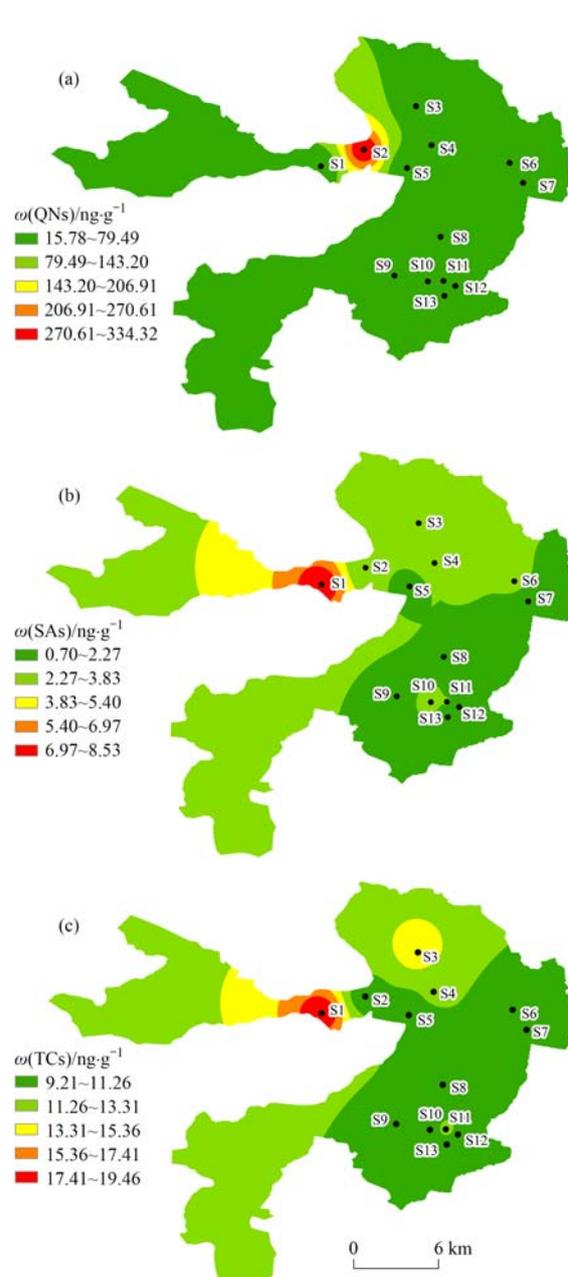


图3 白洋淀沉积物中喹诺酮类、磺胺类和四环素类抗生素的空间分布

Fig. 3 Spatial distribution of QNs, SAs, and TCs in the sediments of Baiyangdian Lake

兽用药物, MAR、FLE 和 OTC 在畜禽养殖中常用作饲料添加剂来预防和治疗疾病^[67-69]. 淀区内家禽畜牧业的废弃物、粪便直接或间接入淀^[70], 因子 4 推断为畜禽养殖.

2.3.2 沉积物中抗生素源解析

沉积物中抗生素源解析结果如图 6 和图 7 所示. 因子 1 对 SMX (64.9%) 贡献较高, 与水体中因子 2 相同, 且相关性分析显示 SMX 与其他抗生素相关性较弱, 则其可能具有单一来源, 因子 1 推断为生活污水.

因子 2 对 FLE (71.0%) 和 ENR (50.3%) 的贡

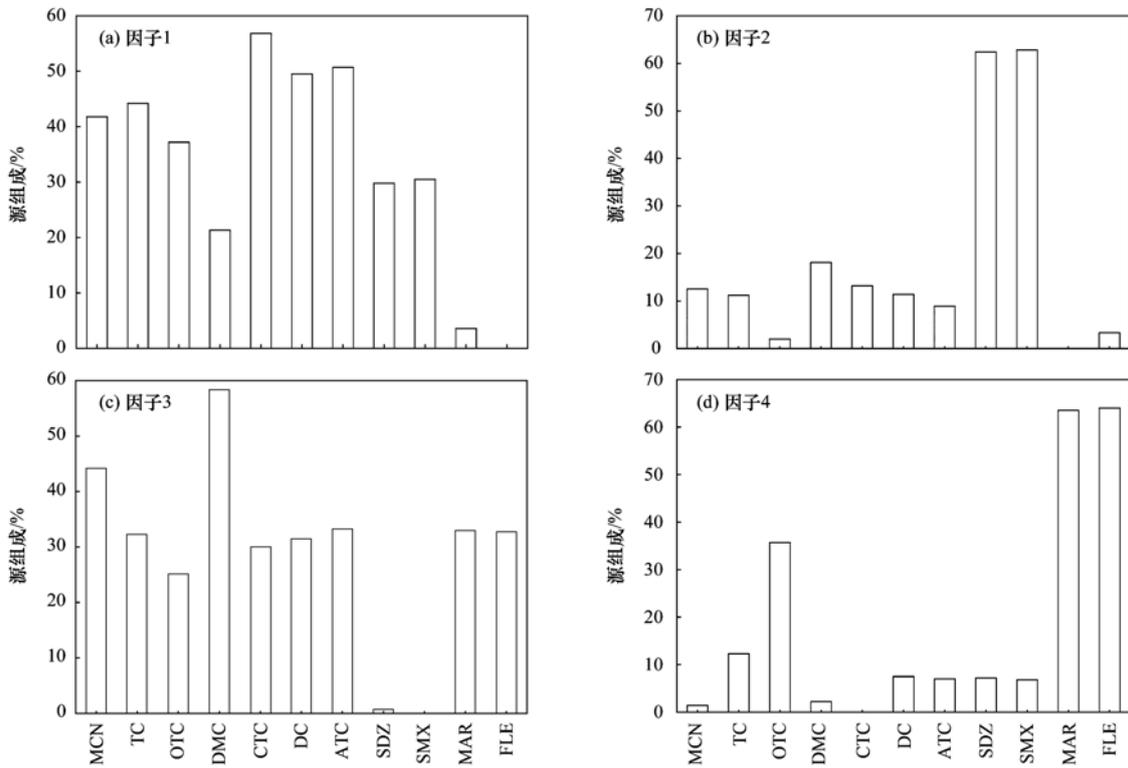


图 4 基于 PMF 模型水体中抗生素源解析

Fig. 4 Source profiles of antibiotics in surface water obtained with the PMF model

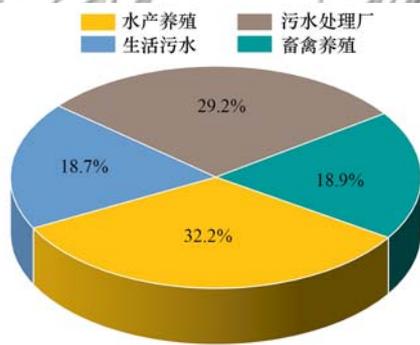


图 5 水体中各因子对抗生素总浓度的贡献

Fig. 5 Contribution of each factor to total antibiotic concentrations in surface water

相对较高,且二者显著正相关($P < 0.01$). 作为典型的 QNs,在养殖场常用来预防家禽疾病和感染,研究表明 ENR 在养殖场中的检出量 ($1.27 \times 10^4 \text{ ng} \cdot \text{g}^{-1}$) 远高于其他的抗菌药物^[71],因子 2 推断为畜禽养殖.

因子 3 对 SPY (93.0%) 的贡献较高,且相关性分析显示 SPY 与其他抗生素相关性较弱,则其可能具有单一来源. 因 SPY 本身很少用作抗菌,多源于其相关的代谢物^[72]. 而目前的处理工艺对其去除率较低,研究表明 SPY 在污水处理厂中的检出浓度高达 $35.9 \sim 64.8 \text{ ng} \cdot \text{L}^{-1}$ ^[73],因子 3 推断为污水处理厂.

因子 4 对 STZ (64.9%) 和 SCP (54.7%) 的贡献

相对较高,且二者显著正相关($P < 0.01$). STZ 和 SCP 因其成本低,常作为鱼用饲料以治疗和预防水产品疾病^[74,75]. 阮悦斐等^[76]已在天津近郊水产养殖区沉积物中检出 STZ 和 SCP,因子 4 推断为水产养殖. 综上所述,白洋淀中抗生素的主要来源为水产养殖.

2.4 风险评价

2.4.1 生态风险评价

本研究对水体中抗生素进行风险评估(图 8). 就各抗生素的生态风险而言,FLU 在 S5 ($RQ > 1.0$) 处于高风险水平,其余样点均为中低风险水平; ENR 在 S1 ($RQ > 1.0$) 为高风险水平, S4、S5、S9 和 S11 的 RQ 处于 $0.1 \sim 1.0$ 之间,为中风险水平; CTC 的 RQ 均处于 $0.1 \sim 1.0$ 之间,为中风险水平; SMX 在 S4 和 S6 的 RQ 处于 $0.1 \sim 1.0$ 之间,为中风险水平,其余样点均为低风险水平; 其余抗生素均处于低风险水平.

就抗生素联合生态风险而言, S1、S5 和 S11 样点的 $RQ_{\text{sum}} > 1.0$, 处于高风险水平,最大值出现在 S1 (3.03); 其余样点的 RQ_{sum} 处于 $0.1 \sim 1.0$ 之间,为中风险水平.

2.4.2 特定源生态风险评价

就各特定源生态风险的空间分布而言,水产养殖在 S1 处的 $RQ_p > 1.0$, 处于高风险水平,其余样点均为中风险水平; 生活污水和畜禽养殖在 S1、S2、

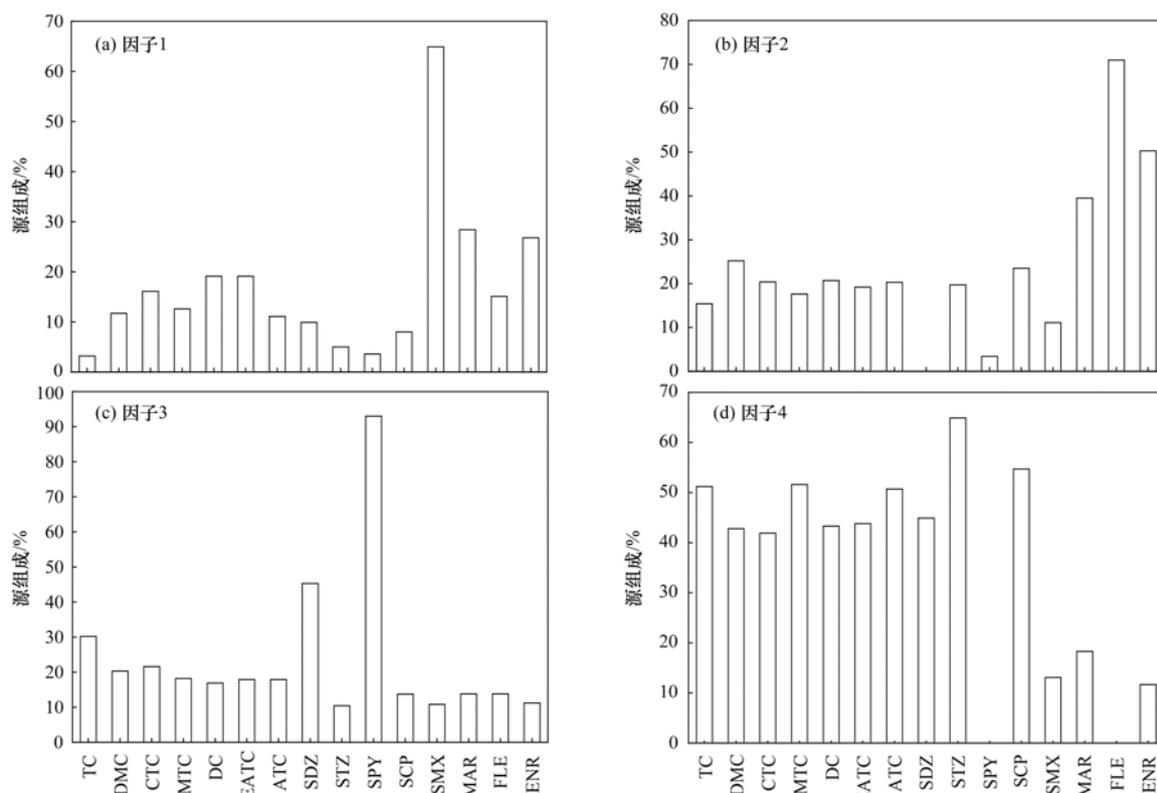


图 6 基于 PMF 模型沉积物中抗生素源解析

Fig. 6 Source profiles of antibiotics in sediments obtained with the PMF model



图 7 沉积物中各因子对抗生素总含量的贡献

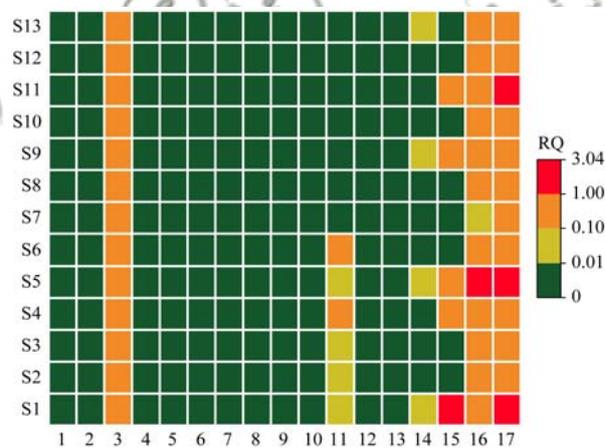
Fig. 7 Contribution of each factor to total antibiotic contents in sediment

S4、S5、S6、S9、S10 和 S11 样点的 RQ_p 处于 0.1 ~ 1.0 之间,为中风险水平,其余样点均为低风险水平;而污水处理厂在所有样点的 RQ_p 均处于 0.1 ~ 1.0 之间,为中风险水平。

3 讨论

3.1 国内外湖泊和河流中抗生素的污染特征

目前,在国内外河流和湖泊中 QNs、TCs 和 SAs 均已有检出。就水体中各抗生素的浓度而言(表 3), OFL 浓度最大值远高于洞庭湖($0.53 \text{ ng}\cdot\text{L}^{-1}$)^[78]、南四湖($10.30 \text{ ng}\cdot\text{L}^{-1}$)^[26]和骆马湖($13.30 \text{ ng}\cdot\text{L}^{-1}$)^[25]; FLU 浓度(最大值为 $2635 \text{ ng}\cdot\text{L}^{-1}$)远



1. TC, 2. OTC, 3. CTC, 4. DC, 5. SDZ, 6. SPY, 7. SMT, 8. SMP, 9. SMM, 10. SCP, 11. SMX, 12. MAR, 13. FLE, 14. OFL, 15. ENR, 16. FLU, 17. RQ_{sum}

图 8 白洋淀水体中抗生素的生态风险评价

Fig. 8 Ecological risk assessment of antibiotics in the surface water of Baiyangdian Lake

高于石家庄河流(最大值 $645.7 \text{ ng}\cdot\text{L}^{-1}$)^[45]、潮白河(最大值 $95.59 \text{ ng}\cdot\text{L}^{-1}$)^[80]和塞纳河(最大值 $32.0 \text{ ng}\cdot\text{L}^{-1}$)^[82]; ENR 的浓度则略高于巢湖(最大值 $82.7 \text{ ng}\cdot\text{L}^{-1}$)^[77]。此外, SMX、SDZ、DC 和 CTC 的浓度均低于其它河流,如白洋淀中 SMX 浓度最大值($6.47 \text{ ng}\cdot\text{L}^{-1}$)远低于大通湖($50.90 \text{ ng}\cdot\text{L}^{-1}$)^[23]、洞庭湖($47.41 \text{ ng}\cdot\text{L}^{-1}$)^[78]、潮白河($63.78 \text{ ng}\cdot\text{L}^{-1}$)^[80]和辽河($16.40 \text{ ng}\cdot\text{L}^{-1}$)^[81],而与



1. 水产养殖, 2. 生活污水, 3. 污水处理厂, 4. 畜禽养殖

图9 白洋淀水体中特定源的生态风险 (RQ_p)

Fig. 9 RQ_p of antibiotics in the surface water of Baiyangdian Lake

鄱阳湖浓度大致相近 ($5.10 \text{ ng}\cdot\text{L}^{-1}$)^[24]; DC 的浓度最大值 ($1.45 \text{ ng}\cdot\text{L}^{-1}$) 与巢湖 ($5.70 \text{ ng}\cdot\text{L}^{-1}$)^[77]、鄱阳湖 ($8.10 \text{ ng}\cdot\text{L}^{-1}$)^[24] 处于一个量级, 但远低于南四湖 ($49.20 \text{ ng}\cdot\text{L}^{-1}$)^[26]; CTC 的浓度最大值为 $2.00 \text{ ng}\cdot\text{L}^{-1}$, 与巢湖 ($4.00 \text{ ng}\cdot\text{L}^{-1}$)^[77]、南四湖 ($3.24 \text{ ng}\cdot\text{L}^{-1}$)^[26]、洞庭湖 ($6.50 \text{ ng}\cdot\text{L}^{-1}$)^[78]、鄱阳湖 ($8.40 \text{ ng}\cdot\text{L}^{-1}$)^[24] 和辽河 ($9.50 \text{ ng}\cdot\text{L}^{-1}$)^[81] 相当。

就沉积物中各抗生素含量而言 (表 4), OFL 含量最大值 ($259.8 \text{ ng}\cdot\text{g}^{-1}$) 与滇池 ($108.9 \text{ ng}\cdot\text{g}^{-1}$)^[79] 大致接近, 远高于太湖 ($16.50 \text{ ng}\cdot\text{g}^{-1}$)^[84]、南湖 ($5.56 \text{ ng}\cdot\text{g}^{-1}$)^[85]、辽河 ($51.60 \text{ ng}\cdot\text{g}^{-1}$)^[89] 和黄河 ($49.69 \text{ ng}\cdot\text{g}^{-1}$)^[90] 流域^[90], 而低于海河 ($653 \text{ ng}\cdot\text{g}^{-1}$)^[88], 王同飞等^[83] 也证实白洋淀沉积物中

表3 国内外河流和湖泊水体中典型抗生素浓度比较¹⁾

Table 3 Comparison of typical antibiotic concentrations in the surface water of global rivers and lakes

位置	抗生素浓度/ $\text{ng}\cdot\text{L}^{-1}$							文献
	DC	CTC	SMX	SDZ	OFL	ENR	FLU	
白洋淀	1.03 ~ 1.45	1.26 ~ 2.00	ND ~ 6.47	ND ~ 2.45	ND ~ 428.1	ND ~ 110.7	144.5 ~ 2635	本研究
巢湖	ND ~ 5.70	ND ~ 4.00	—	ND ~ 8.40	ND ~ 50.60	ND ~ 82.70	—	[77]
南四湖	ND ~ 49.20	ND ~ 3.24	—	ND	ND ~ 10.3	ND	—	[26]
骆马湖	ND	—	2.95 ~ 20.24	ND ~ 87.26	ND ~ 13.30	ND ~ 9.39	—	[25]
大通湖	—	ND ~ 10.44	ND ~ 50.9	11.65 ~ 100.2	—	ND ~ 38.14	—	[23]
鄱阳湖	ND ~ 8.10	ND ~ 8.40	ND ~ 5.10	ND	≤LDQ	≤LDQ	—	[24]
洞庭湖	—	ND ~ 6.50	ND ~ 47.41	ND ~ 61.28	ND ~ 0.53	ND ~ 4.61	—	[78]
滇池	—	—	17.6 ~ 499	—	ND ~ 713.6	—	—	[79]
潮白河	—	—	ND ~ 63.78	—	ND ~ 137.8	—	ND ~ 95.59	[80]
辽河	—	ND ~ 9.50	ND ~ 16.40	ND ~ 2.50	2.00 ~ 32.00	ND	—	[81]
塞纳河	—	—	40	ND	30.00	ND ~ 19.33	12.00	[82]

1) “—”表示该抗生素未检出; ND 表示为未检出; ≤LDQ 表示低于定量限 (limit of quantitation)

OFL 含量最高 ($52.90 \text{ ng}\cdot\text{g}^{-1}$). OTC 含量最大值为 $7.53 \text{ ng}\cdot\text{g}^{-1}$, 与南湖 ($4.71 \text{ ng}\cdot\text{g}^{-1}$)^[85]、黄河 ($5.33 \text{ ng}\cdot\text{g}^{-1}$)^[90] 和乌伦古湖 ($6.60 \text{ ng}\cdot\text{g}^{-1}$)^[87] 含量水平相当, 而低于太湖 ($52.80 \text{ ng}\cdot\text{g}^{-1}$)^[84]、东洞庭湖 ($98.50 \text{ ng}\cdot\text{g}^{-1}$)^[86]、洪湖 ($74.73 \text{ ng}\cdot\text{g}^{-1}$)^[78] 和辽河 ($384.6 \text{ ng}\cdot\text{g}^{-1}$)^[89]. SMX 和 CTC 含量均处于较低水平 ($0.81 \text{ ng}\cdot\text{g}^{-1}$ 和 $2.12 \text{ ng}\cdot\text{g}^{-1}$), 远低于太湖 ($16.10 \text{ ng}\cdot\text{g}^{-1}$ 和 $19.00 \text{ ng}\cdot\text{g}^{-1}$)^[84]、洪湖 ($115.8 \text{ ng}\cdot\text{g}^{-1}$ 和 $55.57 \text{ ng}\cdot\text{g}^{-1}$)^[78] 和海河 ($2.59 \text{ ng}\cdot\text{g}^{-1}$ 和 $10.9 \text{ ng}\cdot\text{g}^{-1}$)^[88]; SPY 含量最大值为 $5.59 \text{ ng}\cdot\text{g}^{-1}$, 远高于太湖 ($0.32 \text{ ng}\cdot\text{g}^{-1}$)^[84] 和辽河 ($0.68 \text{ ng}\cdot\text{g}^{-1}$)^[89].

就各类抗生素而言, QNs 为主要抗生素, 其次为 TCs 和 SAs. 在郴州市东江湖中, QNs 亦为主要抗生素^[91], 与本研究的結果一致. QNs 作为一种人畜共用抗生素, 其消费量占位居抗菌药物前列, 因其具有较强的抗菌能力且价格低廉而被广泛应用^[92]. 加之, QNs 还具有较高的沉积物-水分配系数, 因此在

水体和沉积物中被广泛检出^[93]. 此外, 本研究中 TCs 的检出率均高达 100%, 这可能与 TCs 不仅用于人类和动物疾病防治, 还广泛用于水产养殖等有关^[94]; 且污水处理厂仅可去除废水中 24% 的 TCs^[95]. 其次, TCs 具有一定持久性, 其稳定性与光照、微生物和沉积物的吸附作用等多种因素有关^[88].

3.2 不同湖泊和河流源解析结果的成因与比较

此前河湖中抗生素的源解析主要集中于水体, 因此将本研究水体中抗生素的源解析结果与其他研究进行比较 (表 5). 当地政府自 2018 年 9 月开始禁止水产养殖, 而本研究结果表明水产养殖 (33.2%) 为白洋淀中抗生素主要来源, 这一现象可能与其周边密集的养殖区有关. 与其它结果比较, 不同河流或湖泊中抗生素的主要来源存在显著差异. 例如: 在东洞庭湖^[28] 中, 抗生素的主要来源为畜禽养殖 (79.6%), 这可能与洞庭湖周围有众多畜禽生产基地有关. 岳阳市作为洞庭湖第二大畜禽生产基地;

表 4 国内外河流和湖泊沉积物中主要抗生素的含量比较¹⁾

Table 4 Comparison of major antibiotic contents in the sediments of global rivers and lakes

位置	抗生素含量/ng·g ⁻¹								文献
	OTC	CTC	SMX	SDZ	SPY	OFL	FLE	FLU	
白洋淀	0.49~7.53	1.25~2.12	ND~0.81	ND~1.94	ND~5.59	4.54~259.8	ND~11.26	0.54~46.15	本研究
太湖	0.30~52.80	0.55~19.00	0.01~16.10	ND~0.39	0.01~0.32	2.18~16.50	1.59	—	[84]
南湖	ND~4.71	ND	ND~2.53	—	ND	1.32~5.56	—	—	[85]
东洞庭湖	7.48~98.5	ND~83.48	ND~15.43	1.54~38.6	—	—	—	—	[86]
洪湖	0.72~74.73	ND~55.57	ND~115.8	0.81~77.26	—	—	—	—	[78]
乌伦古湖	ND~6.60	2.79~42.83	ND	ND~1.52	—	0.65~6.47	—	—	[87]
海河流域	ND	ND~10.90	ND~2.59	ND	—	ND~653	—	—	[88]
辽河流域	ND~384.6	—	ND~2.63	—	ND~0.68	ND~51.6	ND~25.67	—	[89]
黄河流域	1.51~5.33	—	—	—	—	5.14~49.69	—	—	[90]
滇池	—	ND~92.10	—	—	—	ND~108.9	—	—	[79]

1) “—”表示该抗生素未检测; ND表示为未检出

而东洞庭湖作为洞庭湖中最大的湖区,会接收大量畜禽养殖废水^[22]。在汾河^[57]中,制药废水为主要源(30%)。据统计2020年山西省医疗机构已达14 343个^[96],汾河作为山西省内最大河流,接收大量的制药废水。而在潮白河中,生活污水(31.5%)是主要源^[100]。此外,湘江^[98]和上海市周边河流^[99]均以污水处理厂为主要源,其贡献率分别为40.0%和66.8%。据统计,上海每年处理的废水量高达26.6亿^[95]。而与传统污染物(有机物和氧化物等)相比,现有的污水处理工艺对抗生素的去除率较低,使得污水处理厂也是抗生素的主要来源。此前,基于

PCA-MLR模型对白洋淀流域中SAs、QNs、TCs以及其他药物等PPCPs进行源解析,结果表明生活污水为其主要污染源(63.5%)^[97]。因此,源解析的方法也可能会影响抗生素的源解析结果;此外,PPCPs与抗生素的种类也可能导致源解析的结果出现差异性。

综上所述,不同区域抗生素生产和使用情况存在差异,导致不同河湖污染源不同;不同的源解析方法,也会造成结果存在差异。因此,在进行源解析和污染防治时应根据实际情况选取和制定合适的方法和防治措施。

表 5 不同湖泊和河流中抗生素的源贡献率与方法比较

Table 5 Comparison of source contribution rate and methods for antibiotics in different lakes and rivers

湖泊和河流	主要源(贡献率)	源解析方法	文献
白洋淀	水产养殖(33.2%)、污水处理厂(29.2%)、畜禽养殖(18.9%)和生活污水(18.7%)	PMF	本研究
东洞庭湖	畜禽养殖场(79.6%)、污水处理排放(0.3%)和畜禽水产养殖(19.8%)	PCA-MLR	[28]
汾河	制药废水(30.0%)、水产畜禽养殖(32.0%)、污水处理厂(15.0%)、农业排水(12.0%)和生活污水(11.0%)	PMF	[57]
湘江	污水处理厂(40.0%)、处理过的废水(29.0%)、医院废水(21.0%)和畜禽水产养殖(10.0%)	PCA-MLR	[98]
上海市周边河流	污水处理厂(66.8%)、水产养殖和养牛场(21.2%)、人用药(11.9%)	PCA-MLR	[99]
潮白河	生活污水(31.5%)、鸡粪(26.4%)、污水处理厂(22.2%)和混合源(20.0%)	PMF	[100]

4 结论

(1) 各类抗生素含量在水体和沉积物中存在显著差异,QNs为白洋淀主要抗生素;各类抗生素具有不同的空间分布特征。

(2) 源解析研究表明,水体和沉积物中抗生素各来源占比存在差异。水产养殖、污水处理厂、生活污水和畜禽养殖是其主要来源,且均以水产养殖的贡献率最高。

(3) 除FLU和ENR处于高风险水平,白洋淀水体中抗生素的生态风险整体处于中低风险水平。

(4) 特定源的风险评估结果表明,除水产养殖源为中高风险水平,白洋淀中其余各源整体处于中

低风险水平。

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