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中国主要城市土壤重金属累积特征与风险评价

彭驰, 何亚磊, 郭朝晖, 肖细元, 张严



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洞庭湖及入湖河流中 209 种多氯联苯同类物分布特征与风险评估

黄智峰^{1,2}, 郑丙辉¹, 尹大强², 崔婷婷¹, 赵兴茹^{1*}

(1. 中国环境科学研究院国家环境保护洞庭湖科学观测研究站, 岳阳 414000; 2. 同济大学环境科学与工程学院, 上海 200092)

摘要: 为全面了解洞庭湖水域多氯联苯(PCBs)的污染情况,采用同位素稀释-高分辨质谱法对洞庭湖及入湖河流共 21 处表层水进行了 209 种 PCB 同类物测定分析. 结果表明,共检出 50 种 PCB 同类物, $\rho\left(\sum \text{PCBs}\right)$ 在 $0.077 \sim 10 \text{ ng}\cdot\text{L}^{-1}$ 之间,平均值为 $2.7 \text{ ng}\cdot\text{L}^{-1}$,中值为 $1.9 \text{ ng}\cdot\text{L}^{-1}$,污染浓度为:入湖河流 > 湖区 > 出口. 与国内外研究相比,洞庭湖水域 PCBs 污染处于较低水平. 表层水中主要污染物为二、三和四氯联苯,相对丰度分别为 67%、14% 和 16%. PCB11、PCB17、PCB18、PCB20 + 33、PCB28、PCB47 + 75、PCB52 和 PCB68 分布最为广泛,在超过 80% 的样品中均有检出. 其中, $\rho(\text{PCB11})$ 最高,平均值为 $1.6 \text{ ng}\cdot\text{L}^{-1}$,占 $\rho\left(\sum \text{PCBs}\right)$ 的 59%,来自于国产有机颜料生产过程中的无意排放. 大气传输沉降和历史残留是水中 PCBs 主要来源. 根据毒性当量因子法对研究区域生态毒性风险进行评估,结果显示水中 TEQ 远低于相关标准限值,表明洞庭湖及入湖河流表层水中 PCBs 污染不会对暴露生物体构成危害.

关键词: 多氯联苯(PCBs); 洞庭湖; 污染特征; 风险评估; 高分辨质谱法

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Distribution Characteristics and Risk Assessment of 209 Polychlorinated Biphenyls in Dongting Lake and the Inflow Rivers

HUANG Zhi-feng^{1,2}, ZHENG Bing-hui¹, YIN Da-qiang², CUI Ting-ting¹, ZHAO Xing-ru^{1*}

(1. State Environmental Protection Scientific Observation and Research Station for Lake Dongtinghu, Chinese Research Academy of Environmental Sciences, Yueyang 414000, China; 2. College of Environmental Science and Engineering, Tongji University, Shanghai 200092, China)

Abstract: In order to fully understand the pollution of polychlorinated biphenyls (PCBs) in Dongting Lake, 209 PCB congeners were quantitatively detected and analyzed by isotope-dilution high-resolution mass spectrometry in 21 surface water sampling sites of Dongting Lake and the inflow rivers of the lake. The results showed that a total of 50 PCB congeners were detected in the study area. The $\rho\left(\sum \text{PCBs}\right)$ ranged from 0.077 to $10 \text{ ng}\cdot\text{L}^{-1}$, with an average concentration of $2.7 \text{ ng}\cdot\text{L}^{-1}$ and a median concentration of $1.9 \text{ ng}\cdot\text{L}^{-1}$. The order of contamination concentrations were as follows: the inflow rivers of the lake > lake region > outlet. Compared with the reports of domestic and foreign studies, PCB pollution in Dongting Lake is at a low level. The main pollutants in the surface water were dichlorobiphenyls and tri- and tetrachlorobiphenyls, with relative abundances of 67%, 14%, and 16%, respectively. PCB11, PCB17, PCB18, PCB20 + 33, PCB28, PCB47 + 75, PCB52, and PCB68 were the most widely distributed and detected in more than 80% of samples. Among them, $\rho(\text{PCB11})$ was the highest, with an average concentration of $1.6 \text{ ng}\cdot\text{L}^{-1}$, accounting for 59% of $\rho\left(\sum \text{PCBs}\right)$, from the domestic organic pigment production process of unintentional emissions. Atmospheric transport deposition and historical residue were the main sources of PCBs in water. According to the toxicity equivalent factor method, the ecological toxicity risk of the study area was evaluated, and the results showed that the TEQ in the water was far lower than the relevant standard limit, indicating that the pollution of PCBs in the surface water of Dongting Lake and the inflow rivers would not cause harm to exposed organisms.

Key words: polychlorinated biphenyls (PCBs); Dongting Lake; pollution characteristics; risk assessment; high-resolution mass spectrometry

多氯联苯(polychlorinated biphenyls, PCBs)是一类人工合成的氯代芳香族有机化合物,共有 209 种单体分子. PCBs 具有“三致效应”,在生态系统中能够对水生生物体产生毒害,也能通过食物链累积与传递作用影响到高营养级生物体包括人类^[1]. 尽管 PCBs 已被禁止使用和生产超过 40 a,但由于 PCBs 的高迁移性、持久性和难降解性,在各种环境介质中几乎无处不在,在大气、土壤、水、沉积物、动植物甚至人类母乳中均有检出报道^[2~4]. 然而,受限于

仪器与检测方法,大多数研究是在低分辨率的 GC-MS 基础上进行的,难以排除其他化合物的干扰,存在假阳性或者漏测的问题,不能体现 PCBs 总浓度,环境中的 PCBs 污染水平及生态风险可能被低

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作者简介: 黄智峰(1993~),男,博士研究生,主要研究方向为持久性污染物的迁移转化, E-mail: 1810329@tongji.edu.cn

* 通信作者, E-mail: zhaoxr@cras.org.cn

估^[5,6].

洞庭湖位于湖南省北部,是我国第二大淡水湖.湖区大致可分为东洞庭湖、南洞庭湖和西洞庭湖三部分,南和西接湘、资、沅、澧四水及汨罗江等小支流,由岳阳市城陵矶注入长江.作为长江流域重要的调蓄湖泊和饮用水源地,保障洞庭湖水质和维护生态系统健康具有重要意义.近年来,已有研究关注了洞庭湖中 PCBs 的残留情况. Wei 等^[7]的研究在 2014 年调查了东洞庭湖区水体中的 7 种指示性 PCBs(In-PCBs)单体,结果表明, ρ (PCBs)处于较低水平($0.50 \sim 2.8 \text{ ng} \cdot \text{L}^{-1}$). 胡余明^[8]的研究调查了洞庭湖 13 种鱼类组织中 12 种二噁英类 PCBs(dl-PCBs),发现 PCB118 浓度最高,PCB126 毒性当量占比最大.然而,此前的研究只针对洞庭湖部分湖区或者湖区内生物进行研究,且检测目标仅限于极少部分 PCBs,对于洞庭湖全湖及入湖河流中全部 209 种 PCBs 的污染研究尚未见报道.

本文选取洞庭湖及入湖河流作为研究对象,采用高分辨质谱仪对表层水中全部 209 种 PCB 同类物进行检测分析,分析湖中 PCBs 的浓度水平与分布特征,探讨其潜在来源,评估其毒性风险,以期对洞庭湖有机污染防治与生态安全保障工作提供数据支撑.

1 材料与方法

1.1 研究区域与样品采集

在洞庭湖湖区、入长江口及 4 条入湖河流布设共 21 个采样点(图 1),包括东洞庭湖(S1~S5)、南洞庭湖(S6~S11)、西洞庭湖(S12~S15)、湖区出口(S16~S17)和入湖四水(S18~S21).样品采集于 2018 年 9 月,表层水样($0 \sim 50 \text{ cm}$)保存在 1 L 棕色玻璃瓶中,采集水样前加入 0.5 mL 甲醇,防止微生物生长.水样经玻璃纤维滤膜过滤去除悬浮颗粒物,4℃冷藏保存待处理分析.

1.2 实验试剂与仪器

主要仪器:高分辨气质联用仪(HRGC-HRMS, Agilent 6890-GC, Waters Micromass, Manchester, UK);主要试剂:农残级正己烷(*n*-Hexane)、二氯甲烷(Dichloromethane)、甲醇(Methanol)和丙酮(Acetone)购自 Fisher(Fair Lawn, NJ, USA)和 Tedia(Fairfield, OH, USA).标准溶液 EC-4977(^{13}C 标记,添加标)由 EC-4979(^{13}C 标记,注射标)Cambridge 同位素实验室(Andover, MA, USA)提供,209 种 PCB 单体标准品购自美国 Accusand 公司.

1.3 样品预处理与分析

采用固相萃取法对 1 L 水样进行富集后,经旋

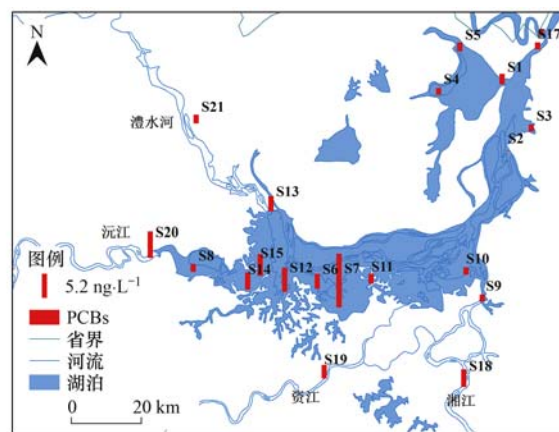


图 1 洞庭湖水域采样点分布示意

Fig. 1 Map of Dongting Lake area and sampling sites

转蒸发、氮吹浓缩至 20 μL .样品通过同位素稀释-高分辨气相色谱-高分辨质谱法测定分析 PCBs 的组成.色谱柱 DB-5MS($60 \text{ m} \times 250 \mu\text{m} \times 0.25 \mu\text{m}$),仪器分析条件详见文献[9].

1.4 质量控制

分析样品前,进行精密度、回收率和实验室空白实验,包括样品富集提取与仪器分析.在测样时进行实验中精密度和回收率实验,每 12 个样品做一个全过程空白.本实验中 PCBs 检出限为 $0.060 \sim 0.10 \text{ pg} \cdot \text{L}^{-1}$,回收率为 $56\% \sim 80\%$,精密度为 $7.0\% \sim 23\%$.空白实验中的检出浓度低于样品的 10% 才有效.

1.5 数据分析

对水中 PCBs 浓度之间进行关联分析, Pearson 相关性分析在 SPSS v22.0 (IBM Corp., Armonk, NY, USA)中完成,对分析结果进行热图绘制,其中 $P < 0.05$ 表明具有显著相关性.使用单因素方差分析检测了不同区域 PCBs 浓度差异.

2 结果与讨论

2.1 PCBs 浓度水平与空间分布

PCBs 在洞庭湖水环境中普遍存在,各采样点均有不同浓度检出.水中总 PCBs ($\sum \text{PCBs}$) 和 In-PCBs 浓度水平如图 2 和表 1 所示.水样中共有 50 种 PCBs 同系物检出, $\rho(\sum \text{PCBs})$ 在 $0.077 \sim 10 \text{ ng} \cdot \text{L}^{-1}$ 之间,平均值为 $2.7 \text{ ng} \cdot \text{L}^{-1}$,中值为 $1.9 \text{ ng} \cdot \text{L}^{-1}$.其中 S7 采样点浓度最高, S2 采样点浓度最低.总体上来看,洞庭湖流域水相环境中 PCBs 浓度水平在西洞庭湖及入湖支流要高于东洞庭湖与南洞庭湖,在湖区出口处(入长江口)最低. $\rho(\sum \text{PCBs})$ 呈现入湖支流 [$(3.2 \pm 2.2) \text{ ng} \cdot \text{L}^{-1}$] > 湖区 [$(2.7 \pm 2.4) \text{ ng} \cdot \text{L}^{-1}$] > 湖区出口 [$(1.3 \pm$

0.085) ng·L⁻¹] 的趋势. 入湖河流中浓度相对较高, 可能是由于入湖口水流流速较快, 岸边土壤冲刷等作用携带了颗粒物吸附 PCBs 进入湖区, 这与徐磊等^[10]在太湖竺山湾的研究结论是一致的.

在所有检出的 PCB 同类物中, 检出率高于 80% 的包括 PCB11、PCB17、PCB18、PCB20 + 33、PCB28、PCB47 + 75、PCB52 和 PCB68. 其中, 检出率最高的是 PCB11(95%), 且 $\rho(\text{PCB11})$ 最高, 平均值为 1.6 ng·L⁻¹. 7 种 In-PCBs 中有 5 种检出, 分别为 PCB28、PCB52、PCB101、PCB118 和 PCB153. $\rho(\text{In-PCBs})$ 在 0.018 ~ 1.4 ng·L⁻¹ 之间, 仅占 $\rho(\sum \text{PCBs})$ 的 1.4% ~ 13% (图 2), 而 12 种 dl-PCBs 中仅有 PCB118 检出, 浓度范围在 ND ~ 0.026 ng·L⁻¹ 之间. 因此, 仅检测 In-PCBs 和 dl-PCBs 并不能全面反映水中 PCBs 的污染水平, PCBs 污染程度也受到一些其他单体的影响, 这也表明在相关水域研究中可能低估了 PCBs 的总浓度.

表 2 汇总了国内外部分地区地表水环境中 PCBs 的检出情况. 与我国其他地区地表水中 PCBs 总检出浓度相比, 本研究中 $\rho(\sum \text{PCBs})$ (0.076 ~

10 ng·L⁻¹) 与洪湖 (0.16 ~ 11 ng·L⁻¹) 和东洞庭湖 (0.50 ~ 2.8 ng·L⁻¹) 相当, 明显低于淀山湖 (19 ~ 39 ng·L⁻¹) 和白洋淀 (19 ~ 132 ng·L⁻¹), 但高于长江中段 (0.002 0 ~ 0.16 ng·L⁻¹)、黄河 (0.64 ~ 2.3 ng·L⁻¹) 和太湖 (ND ~ 1.0 ng·L⁻¹). 与国外相关水域相比, 洞庭湖 $\rho(\sum \text{PCBs})$ 较低, 远低于美国密西西比河 (22 ~ 163 ng·L⁻¹), 略低于意大利亚诺河

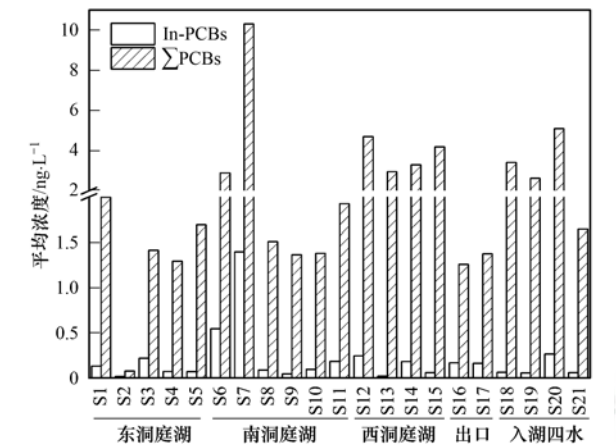


图 2 洞庭湖水域 $\sum \text{PCBs}$ 与 In-PCBs 浓度分布

Fig. 2 Concentrations of $\sum \text{PCBs}$ and In-PCBs in the Dongting Lake areas

表 1 研究区域检出 PCB 单体及浓度分布¹⁾/ng·L⁻¹

Table 1 Distribution of PCB congeners detected and concentrations in the study area/ng·L⁻¹

目标物	检出 PCBs 单体	浓度范围	均值
二氯联苯 (Di-CBs)	PCB4、PCB5、PCB9 + 7、PCB11 和 PCB15	ND ~ 4.2	1.7
三氯联苯 (Tri-CBs)	PCB16、PCB17、PCB18、PCB19、PCB20 + 33、PCB22、PCB26、PCB28、PCB35 和 PCB37	ND ~ 0.23	0.38
四氯联苯 (Tetra-CBs)	PCB40、PCB42、PCB44、PCB45、PCB46、PCB47 + 75、PCB49、PCB51、PCB52、PCB53、PCB60、PCB68、PCB70、PCB71、PCB74 和 PCB80	ND ~ 0.55	0.60
五氯联苯 (Penta-CBs)	PCB86、PCB87、PCB88、PCB99、PCB101、PCB110、PCB118 和 PCB121	ND ~ 0.29	0.040
六氯联苯 (Hexa-CBs)	PCB139 和 PCB153	ND ~ 0.094	0.002 1
dl-PCBs	PCB118	ND ~ 0.026	0.001 9
In-PCBs	PCB28、PCB52、PCB101、PCB118 和 PCB153	0.018 ~ 1.4	0.20
$\sum \text{PCBs}$	—	0.077 ~ 10	0.27

1) “ND” 表示低于检出限

表 2 洞庭湖与国内外部分地区地表水环境中 PCBs 浓度水平比较¹⁾

Table 2 Comparison of PCBs concentration levels in the surface water environment between Dongting Lake and some areas at home and abroad

研究对象	地区	采样年份	检测 PCB 数 (检出数)	$\rho(\sum \text{PCBs})$ /ng·L ⁻¹	文献
洞庭湖	中国	2018	209 (50)	0.076 ~ 10	本研究
太湖	中国	2012 年 6 月	7 (7)	ND ~ 1.0	[11]
淀山湖	中国	2015 ~ 2016	18 (18)	19 ~ 39	[12]
长江中段	中国	2010	12 (12)	0.002 0 ~ 0.16	[13]
白洋淀	中国	2008	41 (39)	19 ~ 132	[14]
洪湖	中国	2014	7 (7)	0.16 ~ 11	[7]
东洞庭湖	中国	2014	7 (7)	0.50 ~ 2.8	[7]
黄河	中国	2008	7 (7)	0.64 ~ 2.3	[15]
Mississippi River	美国	2004	28 (28)	22 ~ 163	[16]
Sarno River	意大利	2008	32 (32)	1.4 ~ 25	[17]
River Ravi	巴基斯坦	2015	209 (35)	1.9 ~ 12	[18]
Selangor River	马来西亚	2009	44 (44)	0.13 ~ 7.7	[19]

1) “ND” 表示低于检出限

(1.4 ~ 25 ng·L⁻¹) 和巴基斯坦拉维河 (1.9 ~ 12 ng·L⁻¹), 与马来西亚雪兰莪州河 (0.13 ~ 7.7 ng·L⁻¹) 相当. 值得一提的是, 不同研究中所检测的目标 PCB 同类物数不同, 且多数研究仅检测了几种至多几十种 PCBs, 这会造成 PCBs 总浓度比较上的偏差. 本研究检测了全部 209 种同类物, 但总体来说, 洞庭湖水体中 PCBs 的浓度在国内外水域中仍处于较低水平.

2.2 PCBs 组成与潜在来源分析

环境介质中 PCBs 同类物的组成通常能反映它们在环境中的来源、迁移和归趋等信息^[20]. 我国 PCBs 的生产和使用时间相较于国外尤其是发达国家较晚, 且历时较短 (20 世纪 60 年代开始生产使用至 20 世纪 80 年代停产), PCBs 多用于电力容器和润滑剂等. 但由于生产和使用量较大, 造成管理难度大. 洞庭湖是我国农工业重点发展区域, 历史遗留机械设备如果处置不当会对环境产生威胁. 本研究中, 不同氯代 CBs 的浓度与相对丰度见表 1 和图 3. 从中可知, 洞庭湖水域表层水中 PCBs 以低氯代为主, 只有二~六氯代 CBs 检出. 二氯联苯 (Di-CBs) 浓度最高 (平均值 1.7 ng·L⁻¹), 在 80% 的样品中相对丰度 > 50%. 其次是: 三氯联苯 (Tri-CBs) > 五氯联苯 (Penta-CBs) > 四氯联苯 (Tetra-CBs) > 六氯联苯 (Hexa-CBs). PCBs 类似的组成分布也在太湖^[10] 和白洋淀^[21] 等研究中报道. 低氯代 CBs 在洞庭湖水环境中为主, 一方面是由于低氯代 CBs 的水溶性高于高氯代 CBs, 且具有较高的蒸气压, 可以通过大气干湿沉降进入湖泊中^[22]. 另一方面, 环境中某些微生物多有 PCBs 的降解脱氯作用, 使得高氯代 CBs 逐渐转向低氯代 CBs^[23]. 所有目标物中, PCB11 在检出中浓度最高, PCB11 是一种典型的非商业化的 PCB 单体, 在国产有机颜料生产过程中会无意排放

至大气, 经大气传输在环境中远距离迁移^[24,25]. 我国是世界上最大的有机颜料生产国, 因此有机颜料生产所带来的大气中 PCBs 污染应当引起关注. 另外, Tri-CBs 浓度较高可能是源于该区域电容器和变压器等无意间的泄漏而产生的历史残留. Tri-CBs 中 PCB52 占比最高, PCB52 是我国历史上生产的三氯联苯商业混合物 (Aroclor1242) 中的主要成分. 综上, 洞庭湖水域 PCBs 主要来自于大气传输沉降及历史残留.

此外, S7 点位 $\rho(\sum \text{PCBs})$ (10 ng·L⁻¹) 显著高于其他采样点 (图 2), 经实地调查发现, S7 采样点位于村庄附近且周边无正在运营的工厂, 因此高浓度的 PCBs 可能源自于居民日常生活中产生的电子垃圾排放以及变压器等的无意释放.

对洞庭湖水中 PCBs 进行 Pearson 相关性分析,

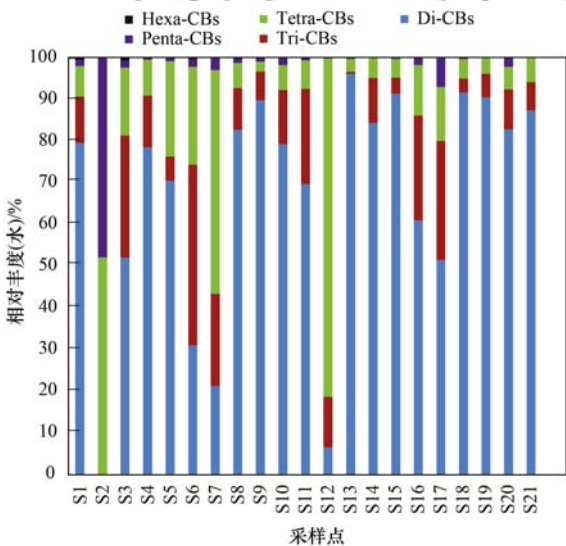
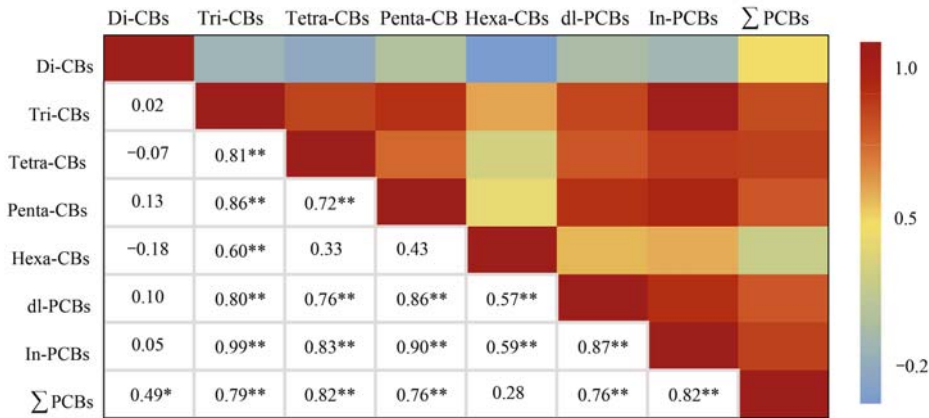


图 3 洞庭湖表层水中各氯代 PCBs 相对丰度
Fig. 3 Relative abundance of PCB homologs in the surface water of the Dongting Lake area



* 和 ** 分别表示在 0.05 和 0.01 水平 (双尾) 相关性显著

图 4 地表水中 PCBs 之间的相关性热图

Fig. 4 Heatmap of correlation of PCBs concentrations among surface water samples

分析结果见图 4. 水中 Tri-CBs 和 Tetra-CBs ($R = 0.81$, $P < 0.01$)、Penta-CBs ($R = 0.86$, $P < 0.01$) 和 Hexa-CBs ($R = 0.60$, $P < 0.01$) 之间, Tetra-CBs 和 Penta-CBs ($R = 0.72$, $P < 0.01$) 之间均呈现出显著的正相关性, 表明他们可能存在共同的来源. 此外, 尽管水中 In-PCBs 和 dl-PCBs 浓度很低(图 2 和表 1), 但水中 $\sum$ PCBs 与 In-PCBs ($R = 0.82$, $P < 0.01$) 和 dl-PCBs ($R = 0.76$, $P < 0.01$) 之间仍呈现出强正相关性, 这和文献[6]的研究结果一致. 这种显著的强相关性表明当实验条件受限无法检测 209 种 PCBs 时, 可以考虑使用 In-PCBs 或 dl-PCBs 来估算样品中 PCBs 总浓度.

2.3 风险评估

水源地的 PCBs 污染会影响供水安全与生态系统健康, 考虑到 PCBs 的毒性和生物积累性, 评价洞庭湖地区 PCBs 潜在的生态风险具有重要意义. 我国《地表水环境质量标准》(GB 3838-2002) 和 US. EPA(美国环保署)规定 PCBs 在水中的浓度限值分别为 $20 \text{ ng} \cdot \text{L}^{-1}$ 和 $14 \text{ ng} \cdot \text{L}^{-1}$, 洞庭湖水体中 PCBs 的浓度 ($0.076 \sim 10 \text{ ng} \cdot \text{L}^{-1}$) 远低于我国地表水环境质量标准与 EPA 限值. 因此, 洞庭湖地区的地表水环境质量受多氯联苯污染的影响较小, 表明水生态系统中目前的 PCBs 浓度水平可能不会对生态系统健康产生不利影响.

进一步地, 为了评估水样潜在的二噁英类毒性, 使用毒性当量因子(toxic equivalent factors, TEFs)计算每个采样点 dl-PCBs 的毒性当量(toxic equivalency, TEQ). 计算公式为:

$$\text{TEQ} = c \times \text{TEFs}$$

式中, c 为 PCB 浓度, TEFs 取自世卫组织 2005 年修订的数值^[26]. 本研究洞庭湖水样中 dl-PCBs 的 TEQ 范围在 $\text{ND} \sim 7.8 \times 10^{-4} \text{ pg} \cdot \text{L}^{-1}$, 平均值为 $5.8 \times 10^{-5} \text{ pg} \cdot \text{L}^{-1}$, 均远低于日本环境质量标准 ($1 \text{ pg} \cdot \text{L}^{-1}$) 及美国 EPA 标准限值 ($30 \text{ pg} \cdot \text{L}^{-1}$). 因此, 洞庭湖水域中 PCBs 污染产生毒性很小, 对水环境及水生生物几乎不产生危害.

3 结论

(1) 首次对洞庭湖及入湖河流中的 209 种 PCBs 进行了全面调查. 共检出 50 种 PCB 同类物, $\rho(\sum \text{PCBs})$ 在 $0.076 \sim 10 \text{ ng} \cdot \text{L}^{-1}$ 之间, 平均浓度呈现: 入湖支流 > 湖区 > 湖区出口的趋势. 与国内外研究相比, 洞庭湖水域 PCBs 污染处于较低水平.

(2) 研究区域表层水中的 PCBs 以低氯代为主, 尤其是 Di-CBs、Tri-CBs 和 Tetra-CBs 居多.

$\rho(\text{PCB11})$ 最高, 平均值为 $1.6 \text{ ng} \cdot \text{L}^{-1}$, 占 $\rho(\sum \text{PCBs})$ 的 59%. PCBs 主要来自于大气传输沉降和历史残留.

(3) 利用毒性当量因子法对研究区域生态毒性风险进行评估, 结果显示洞庭湖及入湖河流表层水中 PCBs 污染不会对暴露生物体构成危害.

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