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目次

2015~2016年北京市3次空气重污染红色预警PM_{2.5}成因分析及效果评估..... 吕喆,魏巍,周颖,程水源,王晓琦(1)

长三角地区重点源减排对PM_{2.5}浓度的影响..... 于燕,王泽华,崔雪东,陈锋,徐宏辉(11)

上海市实施清洁空气行动计划的健康收益分析..... 戴海夏,安静宇,李莉,黄成,严茹莎,朱书慧,马英歌,宋伟民,阚海东(24)

京津冀及周边地区PM_{2.5}时空变化特征遥感监测分析..... 陈辉,厉青,李莹,张连华,毛慧琴,周伟,刘伟汉(33)

MODIS C006气溶胶光学厚度产品在京津冀典型环境背景下的适用性..... 王海林,刘琼,陈勇航,孙冉,李霞,张华,魏刚,胡俊,刘统强(44)

气象因素对香港地区臭氧污染的影响..... 赵伟,高博,刘明,卢清,马社霞,孙家仁,陈米国,范绍佳(55)

天津夏季边界层低层大气中PAN和O₃的输送特征分析..... 姚青,马志强,林伟立,刘敬乐,王晓佳,蔡子颖,韩素芹(67)

成都冬季PM_{2.5}化学组分污染特征及来源解析..... 吴明,吴丹,夏俊荣,赵天良,杨清健(76)

郑州市冬季天气PM_{2.5}传输路径和潜在源分析..... 段时光,姜楠,杨留明,张瑞芹(86)

常州春季PM_{2.5}中WSOC和WSON的污染特征与来源解析..... 李清,黄雯倩,马帅帅,黄红缨,叶招莲,陈敏东(94)

天津隧道机动车VOCs污染特征与排放因子..... 孙露娜,刘妍,赵静波,孙世达,宋从波,张静,李悦宁,林应超,王婷,毛洪钧(104)

机动车源大气颗粒物粒径分布及碳组分特征..... 梅德清,朱宗宁,孙天硕,王向丽,梅丛蔚,肖政臻(114)

超低排放路线下燃煤烟气可凝结颗粒物在WFGD、WESP中的转化特性..... 杨柳,张斌,王康慧,麻丁仁,盛文(121)

超低排放燃煤电厂颗粒物脱除特性..... 阮仁晖,谭厚章,段钰锋,杜勇乐,刘鹤欣,萧嘉繁,杨富鑫,张朋(126)

北京建筑施工裸地时空变化及扬尘污染排放..... 张立坤,李令军,姜磊,赵文慧,鹿海峰,王新辉,邱昀(135)

SCR装置对焦炉煤气燃烧废气中PCDD/Fs、PCBs和PCNs的协同脱除..... 任美慧,樊芸,王胜,许亮,张宁,张雪萍,陈吉平,张海军(143)

古浪河流域大气降水稳定同位素的时空特征及其环境意义..... 桂娟,李宗省,冯起,卫伟,李永格,吕越敏,袁瑞丰,张百娟(149)

夏季闽江CDOM的空间分布与降解特征..... 程琼,庄婉娥,王辉,陈菁,杨丽阳(157)

亚热带河口陆基养虾塘水体CDOM三维荧光光谱平行因子分析..... 朱爱菊,孙东耀,谭季,黄佳芳,罗敏(164)

基于UV-vis及EEMs解析周村水库夏秋季降雨不同相对分子质量DOM的光谱特征及来源..... 周石磊,张艺冉,黄廷林,刘艳芳,张磊,李贵霞,岳琳,罗晓(172)

分层型水源水库溶解性有机物质及其膜污染特性..... 李凯,王晓东,黄廷林,李舒,刘双(185)

重庆西部山区典型湖泊水-气界面CO₂交换通量及其影响因素..... 罗佳宸,倪茂飞,李思悦(192)

基于稀疏表达的水体遥感反射率高光谱重构及其应用..... 李渊,李云梅,郭宇龙,张运林,张毅博,胡耀霖,夏忠(200)

巢湖十五里河河床地貌单元沉积物硝化速率及污染特征..... 李如忠,阙凤翔,熊鸿斌,王莉(211)

特大城市河流表层沉积物磷形态分布及有效性:以成都市为例..... 丁瑶,欧阳莉莉,石清,高平川,赖承钺,陈舒平,贾滨洋,姚刚(219)

黄河中游(渭南—郑州段)全/多氟烷基化合物的分布及通量..... 李琦路,程相会,赵祯,郭萌然,袁梦,华夏,方祥光,孙红文(228)

重工业城市岩溶地下水中多环芳烃污染特征及来源..... 苗迎,孔祥胜,李成展(239)

沈抚新城地下水中PAHs的污染特征及健康风险评价..... 张士超,姚宏,向鑫鑫,刘殷佐,刘明利,鲁根涛,于晓华(248)

多环芳烃及其衍生物在北京纳污河流中的分布及健康风险..... 付璐婧,李一兵,乔梦,赵旭(256)

原水性质对新型含Ca²⁺复合混凝剂混凝过程的影响..... 曲江东,徐慧,徐建坤,段晋明,门彬,王东升(263)

水体中磺胺甲噁唑间接光降解作用..... 李聪鹤,车潇炜,白莹,石晓勇,苏荣国(273)

MoS₂/BiOI复合光催化剂制备及其光催化氧化还原性能..... 张亮,赵朝成,高先瑶,闫青云,王帅军,董培,侯亚璐(281)

纳米零价铜活化分子氧降解水中恩诺沙星..... 倪永炯,程永清,徐梦苑,邱春根,马晓雁,李军,邓靖(293)

不同类型LDHs负载改性麦饭石对Cr(VI)吸附性能..... 张翔凌,邓礼楚,方晨佳,雷雨,何春艳,高晨光,赵双杰,向洋(300)

单层硅烷负载磁铁矿纳米颗粒的制备及除磷性能..... 邢明超,谢强,陈守慧,吴德意(310)

新生态型聚硅酸铁锰处理染料废水的优化..... 唐立朋,魏群山,吕强,张弛,刘亚男,柳建设(318)

不同外加电压下自养型生物阴极还原硫酸盐的性能及生物膜群落响应..... 胡佳萍,曾翠平,骆海萍,刘广立,张仁铎,卢耀斌(327)

碳源对O/A-F/F模式积累内源聚合物及反硝化的影响..... 崔有为,金常林,王好韩,李晶(336)

进水C/P对SNEDPR系统脱氮除磷性能的影响..... 甄建园,于德爽,王晓霞,陈光辉,都叶奇,袁梦飞,杜世明(343)

不同污泥龄(SRT)对SNEDPR系统脱氮除磷影响..... 王晓霞,甄建园,赵骥,于德爽,都叶奇,杜世明,袁梦飞,张帆(352)

后置短程反硝化AOA-SBR工艺实现低C/N城市污水的脱氮除磷..... 巩秀珍,于德爽,袁梦飞,王晓霞,陈光辉,王钧,毕春雪,都叶奇(360)

缺氧-好氧连续流亚硝化颗粒污泥反应器的启动及稳定运行..... 李冬,郭跃洲,劳会妹,曹美忠,张杰(369)

低温SNAD颗粒污泥工艺启动方式..... 李冬,崔雅倩,赵世勋,刘志诚,张杰(376)

不同取样尺度和数量下针阔混交林土壤呼吸的空间异质性..... 严俊霞,孙琦,李君剑,李洪建(383)

轮作方式对冬水田温室气体排放的影响..... 冯夕,江长胜,彭小乐,李彦沛,郝庆菊(392)

紫色土N₂O排放及氨氧化微生物群落结构对玉米秸秆与化肥减量配施的响应..... 黄容,高明,王莹燕,黎嘉成,徐国鑫,罗梅,徐畅(401)

岩溶地区不同土地利用方式土壤固碳细菌群落结构特征..... 张双双,靳振江,贾远航,李强(412)

丹江口库区库滨带植被土壤细菌群落多样性及PICRUSt功能预测分析..... 孙峰,田伟,张菲,陈彦,任学敏,庞发虎,李玉英,姚伦广,陈兆进(421)

硫酸盐还原菌介导的吸附态砷的迁移转化..... 贾欠欠,李伟,王亚男,段晋明,刘玉灿(430)

铅锌冶炼厂周边农田土壤重金属污染空间分布特征及风险评估..... 王洋洋,李方方,王笑阳,杨志辉,韩科,阮心玲(437)

新疆干旱区某矿冶场对周围土壤重金属累积的影响..... 杨伟光,王美娥,陈卫平(445)

九龙江流域水稻土重金属赋存形态及污染评价..... 林承奇,黄华斌,胡恭任,于瑞莲,郝春莉,林颖(453)

草海典型高原湿地食物链中汞同位素组成特征..... 许议元,何天容(461)

Cd、Zn交互作用对三七景天根系形态和重金属吸收积累的影响..... 郭俊梅,杨俊兴,杨军,陈同斌,李厚恩,徐铁兵,周小勇,叶勇,于豹(470)

不同浓度镉处理对铅胁迫下玉米生长和铅吸收的影响..... 王起凡,郭伟,常青,潘亮,周昕南,杨亮,李娥(480)

广西龙江鱼类镉含量分布特征及生物积累特性分析..... 王俊能,赵学敏,胡国成,钟松雄,姚玲爱,马千里,许振成(488)

6种消解方法对荧光测定生物体内聚苯乙烯微塑料的影响..... 邹亚丹,徐擎擎,张智,李富云,李锋民(496)

一种负载功能型微生物的营养缓释填料的制备及性能评价..... 冯克,徐丹华,成卓韦,於建明,陈建孟(504)

《环境科学》征订启事(113) 《环境科学》征稿简则(238) 信息(93, 262, 342)

SCR装置对焦炉煤气燃烧废气中PCDD/Fs、PCBs和PCNs的协同脱除

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摘要: 研究了某大型焦化厂的选择性催化还原(SCR)装置对焦炉煤气燃烧废气中典型非故意产生持久性有机污染物(UP-POPs)的脱除效果, 对SCR装置入口和出口废气和SCR装置降尘中二噁英(PCDD/Fs)、多氯联苯(PCBs)和多氯萘(PCNs)的全部异构体进行了分析. 结果表明, SCR装置对总PCDD/Fs的脱除效率最高, 为94.6%, 对总PCBs和总PCNs的脱除率分别为74.7%和78.4%. SCR装置入口和出口废气中UP-POPs同系物的分布模式明显不同, 入口处以高氯取代为主, 出口处以低氯取代为主. SCR装置对高氯取代UP-POPs有更好的脱除效果. 催化还原和催化氧化降解同时发生是SCR装置脱除废气中UP-POPs的重要机制.

关键词: 持久性有机污染物(POPs); 二噁英; 多氯联苯; 多氯萘; 选择性催化还原(SCR); 脱除

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Simultaneous Removal of Polychlorinated Dibenzo-*p*-dioxins/dibenzofurans, Polychlorinated Biphenyls, and Polychlorinated Naphthalenes From Flues Gases From Coke Gas Burning Using Selective Catalytic Reduction Equipment

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Abstract: The removal efficiencies of typical unintentionally produced persistent organic pollutants (UP-POPs) from flues gases from coke gas burning were obtained using selective catalytic reduction (SCR) equipment installed in a large coking plant. The total congeners of polychlorinated dibenzo-*p*-dioxins/dibenzofurans (PCDD/Fs), polychlorinated biphenyls (PCBs), and polychlorinated naphthalenes (PCNs) in the flue gases at the inlet and outlet of the SCR equipment and the dustfall were analyzed. The results show that the removal efficiency of the total PCDD/Fs was the highest (94.6%). The removal efficiencies of total PCBs and total PCNs were 74.7% and 78.4%, respectively. The homologue profile of UP-POPs in the flue gases at the inlet of the SCR equipment notably differed from that at the outlet. Highly chlorinated UP-POPs predominated over the homologue profile of UP-POPs in the inlet flue gas, while low-chlorinated UP-POPs were predominant in the outlet flue gas. The SCR equipment achieved a better removal efficiency with respect to highly chlorinated UP-POPs. Catalytic reduction and catalytic oxidation degradation are both important mechanisms for the removal of UP-POPs from flue gas using SCR equipment.

Key words: persistent organic pollutants (POPs); dioxins; polychlorinated biphenyls; polychlorinated naphthalenes; selective catalytic reduction (SCR); removal

焦炉煤气是炼焦过程副产品, 作为燃料燃烧时产生的废气中通常含有较高浓度的氮氧化物(NO_x), 同时也会包含多氯代二苯并二噁英和多氯代二苯并呋喃(PCDD/Fs, 统称为二噁英)、多氯萘(PCNs)和多氯联苯(PCBs)等非故意产生持久性有机污染物(UP-POPs)^[1~7]. 这些UP-POPs具有环境持久性、远距离迁移特性、生物蓄积性和高毒性, 可引起较高的生态风险和人体健康风险, 被联合国环境署《关于持久性有机污染物的斯德哥尔摩公约》所管控^[8]. 为实现废气的达标排放, 国内焦炉煤气燃烧炉已普遍开始采用选择性催化还原(SCR)技术消减废气中的 NO_x ^[9]. 此技术的应用也可能会

同时显著减少UP-POPs的排放. 已有研究表明, SCR装置在脱硝过程中可大幅脱除废气中的二噁英^[10~13]. 因此, SCR技术也被联合国环境署推荐为减少城市生活垃圾焚烧过程二噁英排放的最佳可行性技术/最佳环境实践(BAT/BEP), 但其脱除二噁英的机制尚不清晰.

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$V_2O_5-WO_3/TiO_2$ 是使用最多的一种 SCR 催化剂^[14-17], 在欧洲和日本, 商业化的 $V_2O_5-WO_3/TiO_2$ 催化剂已经被应用到部分城市生活垃圾焚烧处理系统, 通常设置在余热锅炉后或布袋除尘器后, 对焚烧废气中的二噁英脱除效率可达 90% 以上^[18-20]. 目前, 有关 SCR 技术应用对焦炉煤气燃烧废气中二噁英脱除效果的研究尚未见报道. 在本研究中通过采集国内某大型焦化厂焦炉煤气燃烧系统的 SCR 装置入口和出口的废气样品、以及 SCR 装置的降尘灰, 分析了 SCR 装置对废气中二噁英的脱除效果, 同时也考察了 SCR 装置对废气中多氯联苯和多氯萘的协同脱除情况. 210 种二噁英异构体、209 种多氯联苯和 75 种多氯萘异构体被全部分析, 通过对比 SCR 装置进出口废气中 UP-POPs 异构体分布模式, 探索 SCR 装置对废气中 UP-POPs 的脱除机制及共性规律, 以期对废气中 NO_x 和 UP-POPs 协同脱除技术的研发提供理论基础.

1 材料与方法

1.1 仪器与试剂

自制烟道气等动力采样器(中国科学院大连化学物理研究所), 符合国家标准 HJ 77.2-2008^[21]; R-205 型旋转蒸发器购于瑞士 Buchi 公司, Waters AutoSpec Premier 高分辨气相色谱-高分辨质谱联用仪(HRGC/HRMS)购于美国 Waters 公司. XAD-2 树脂和碱性氧化铝分别购于美国 Supelco 公司和美国 MP Biomedicals 公司; 农残级正己烷和二氯甲烷购于美国 J&T Baker 公司; 硅胶(75 ~ 150 μm , 德国 Merck 公司), 经 650 $^\circ\text{C}$ 活化 4 h; 无水硫酸钠(分析纯, 天津大茂), 正己烷超声清洗后晾干. 二噁英、多氯联苯和多氯萘分析标样均购于加拿大 Wellington Laboratories 公司和美国 Cambridge Isotope Laboratories 公司, 包括: ① ^{13}C 同位素标记的二噁英混合标准溶液 MDDF-MDT、EDF-8999 和 EDF-5999; ②非同位素标记的二噁英标准样品 DDF-MDT; ③非同位素标记的二噁英标准曲线溶液 EDF-9999; ④ ^{13}C 同位素标记的多氯联苯混合标准溶液 P48-W-ES、P48-M-ES 和 P48-RS; ⑤非同位素标记的多氯联苯标准曲线溶液 P48-W-CS 和 P48-M-CS; ⑥ ^{13}C 同位素标记的多氯萘标准溶液 CN-2、CN-6 和 ECN-5102(混合标样); ⑦非同位素标记的多氯萘标准样品 PCN-MXA 和 PCN-MXC. 其中二噁英混合标准溶液 MDDF-MDT 和 DDF-MDT 中包含二苯并二噁英(DD)和二苯并呋喃(DF).

1.2 样品采集

在国内某大型焦化厂焦炉煤气燃烧系统的

SCR 装置入口和出口各采集 3 个废气样品, 同时采集 2 个 SCR 装置降尘样品. 该燃烧系统的废气量为 17 000 $\text{m}^3 \cdot \text{h}^{-1}$. SCR 催化剂为 $V_2O_5-WO_3/TiO_2$ 蜂窝状催化剂脱硝模块, 通入氨水作为还原剂, 运行温度为 280 ~ 350 $^\circ\text{C}$. 废气采样方法遵照国家标准 HJ 77.2-2008^[21], 采用等动力模式采样, 采样体积大于 1.2 $\text{m}^3 \cdot \text{h}^{-1}$, 采样时间大于 1 h. 在废气采样前后, 分别采取 SCR 装置的降尘, 混匀后装在密封袋中, 带回实验室后研磨过 60 目筛, 待分析.

1.3 样品前处理

废气和降尘样品经冷冻干燥后加入 ^{13}C 同位素标记的 UP-POPs 提取内标, 采用 250 mL 正己烷和二氯甲烷(1:3)混合溶液进行索氏提取, 提取时间为 18 h; 样品提取液经旋转蒸发浓缩后过多层复合硅胶柱, 采用正己烷洗脱, 洗脱液经旋转蒸发浓缩后过氧化铝柱, 采用不同配比的正己烷和二氯甲烷进行洗脱, 分别收集含有二噁英、多氯联苯和多氯萘组分的洗脱液. 将洗脱液旋转蒸发浓缩、用氮气吹至近干、加回收率内标复溶, 密封后待分析.

1.4 仪器分析

采用同位素稀释-高分辨气相色谱-高分辨质谱联用技术(HRGC/HRMS)分别分析样品中的二噁英、多氯联苯、多氯萘、以及 DD 和 DF. 色谱柱选用 Rtx-5MS(60 m \times 0.25 mm ID \times 0.25 μm df, 美国 Restek 公司), 色谱进样口温度为 280 $^\circ\text{C}$ 、传输线温度 260 $^\circ\text{C}$, 进样体积为 1 μL , 不分流进样. 二噁英、DD 和 DF 分析时色谱柱升温程序为: 初始温度 120 $^\circ\text{C}$ (1 min), 43 $^\circ\text{C} \cdot \text{min}^{-1}$ 升到 220 $^\circ\text{C}$ (15 min), 2.3 $^\circ\text{C} \cdot \text{min}^{-1}$ 升到 250 $^\circ\text{C}$, 0.9 $^\circ\text{C} \cdot \text{min}^{-1}$ 升到 260 $^\circ\text{C}$, 20 $^\circ\text{C} \cdot \text{min}^{-1}$ 升到 310 $^\circ\text{C}$ (20 min). 多氯联苯分析时色谱柱升温程序为: 初始温度 90 $^\circ\text{C}$ (2 min), 20 $^\circ\text{C} \cdot \text{min}^{-1}$ 升到 180 $^\circ\text{C}$ (15 min), 3 $^\circ\text{C} \cdot \text{min}^{-1}$ 升到 300 $^\circ\text{C}$ (15 min). 多氯萘分析时色谱柱升温程序为: 初始温度 90 $^\circ\text{C}$ (2 min), 20 $^\circ\text{C} \cdot \text{min}^{-1}$ 升到 200 $^\circ\text{C}$, 3 $^\circ\text{C} \cdot \text{min}^{-1}$ 升到 280 $^\circ\text{C}$ (5 min), 20 $^\circ\text{C} \cdot \text{min}^{-1}$ 升到 300 $^\circ\text{C}$. 质谱条件为: 电子轰击离子源(EI)、离子源温度 260 $^\circ\text{C}$ 、电离能 37 eV、选择离子检测(SIM)、质谱分辨率 $\geq 10\,000$, 分别检测 DD 和 DF 所对应的 M^+ 和 $(M+1)^+$, 二噁英、多氯联苯和多氯萘所对应的 M^+ 和 $(M+2)^+$ 或 $(M+2)^+$ 和 $(M+4)^+$ 的质量色谱峰及其对应的同位素质量色谱峰, 利用同位素稀释内标法及色谱保留规律定性定量.

1.5 质量控制

对所有烟道气和降尘样品进行加标回收率测定, 并进行程序空白分析, 所有测定样品中添加的 21 种 ^{13}C 标记二噁英内标、18 种 ^{13}C 标记多氯联苯内

标、8 种¹³C 标记多氯萘内标的回收率分别在 40% ~ 90%、30% ~ 130%、40% ~ 90% 之间,符合美国 EPA 建议的回收率标准范围。

2 结果与分析

2.1 SCR 装置对废气中 UP-POPs 的总体脱除效果

SCR 装置对焚烧废气中典型 UP-POPs 具有很好的脱除效果。如图 1 所示,废气中全部多氯代二苯并二噁英异构体总浓度(\sum PCDDs)和全部多氯代二苯并呋喃异构体总浓度(\sum PCDFs)在 SCR 装置入口处分别为 $109.0 \text{ ng}\cdot\text{m}^{-3}$ 和 $685.4 \text{ ng}\cdot\text{m}^{-3}$,流经 SCR 装置后分别降至 $5.3 \text{ ng}\cdot\text{m}^{-3}$ 和 $37.7 \text{ ng}\cdot\text{m}^{-3}$,二者在废气中的脱除率分别高达 95.2% 和 94.5%,二噁英的总体(\sum PCDD/Fs)脱除率为 94.6%。根据二噁英的国际毒性当量因子(I-TEF)计算废气中二噁英的毒性当量浓度(I-TEQ)。SCR 装置进出口废气中二噁英的 I-TEQ 浓度分别为 $13.84 \text{ ng}\cdot\text{m}^{-3}$ 和 $0.315 \text{ ng}\cdot\text{m}^{-3}$ 。流经 SCR 装置后废气中的二噁英 I-TEQ 浓度降低了 97.7%,但出口废气中二噁英的 I-TEQ 浓度仍高于我国大型城市生活垃圾焚烧企业的二噁英排放标准 $0.1 \text{ ng}\cdot\text{m}^{-3}$ [22]。

废气中全部多氯联苯异构体(\sum PCBs)和全部多氯萘异构体(\sum PCNs)的总浓度在 SCR 入口处分别为 $216.7 \text{ ng}\cdot\text{m}^{-3}$ 和 $552.6 \text{ ng}\cdot\text{m}^{-3}$,在出口处分别为 $54.7 \text{ ng}\cdot\text{m}^{-3}$ 和 $119.5 \text{ ng}\cdot\text{m}^{-3}$ (图 1)。SCR 装置对 \sum PCBs 和 \sum PCNs 的脱除率分别为 74.7% 和 78.4%,明显低于对 \sum PCDD/Fs 的脱除率。SCR 装置对各类污染物脱除效率的不同显著改变了入口和出口废气中不同类别 UP-POPs 的相对含量(图 1)。在 SCR 装置入口废气中不同类别 UP-POPs 相对含量由高到低的顺序为: \sum PCDFs (43.8%) > \sum PCNs (35.3%) > \sum PCBs (13.9%) > \sum PCDDs (7.0%),而在出口废气中以 \sum PCNs (55.0%) > \sum PCBs (25.2%) > \sum PCDFs (17.4%) > \sum PCDDs (2.4%) 顺序递减。流经 SCR 装置后,废气中 \sum PCDD/Fs 的相对含量显著降低,而 \sum PCBs 和 \sum PCNs 的相对含量显著增加。

2.2 SCR 装置进出口废气中 UP-POPs 的同系物分布特征

为洞悉流经 SCR 装置前后废气中不同 UP-POPs 同系物(具有相同分子量的异构体的总和)分布的差

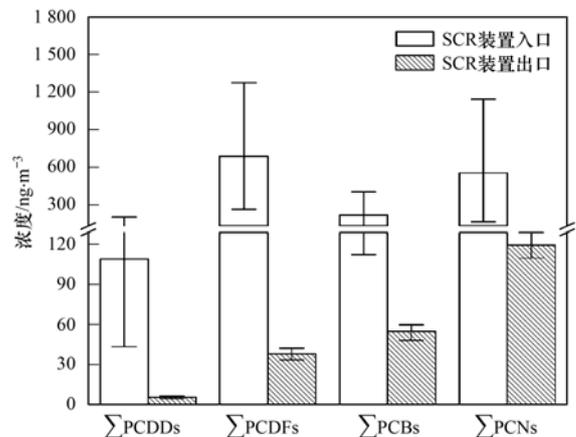


图 1 SCR 装置进出口废气中不同种类 UP-POPs 的浓度

Fig. 1 Concentrations of different categories of UP-POPs in the flue gases at the inlet and outlet of the SCR equipment

别与联系,对浓度总和归一化处理后的二噁英、多氯联苯和多氯萘同系物数据集进行正交偏最小二乘法判别分析(OPLS-DA)。结果表明,SCR 装置入口废气和出口废气在 OPLS-DA 得分图上能清晰分开,说明入口废气和出口废气中 UP-POPs 同系物的分布模式显著不同。在 OPLS-DA 的载荷上(图 2),一氯和二氯代二噁英(MoCDD/Fs 和 DiCDD/Fs)、二氯至四氯代联苯(Di-TeCBs)、以及二氯至五氯代萘(Di-PeCNs)分布在 SCR 装置出口废气一侧,而其它高氯取代的 UP-POPs 分布在 SCR 装置入口废气一侧,说明 SCR 装置对废气中高氯取代和低氯取代 UP-POPs 的脱除行为和脱除效率明显不同。

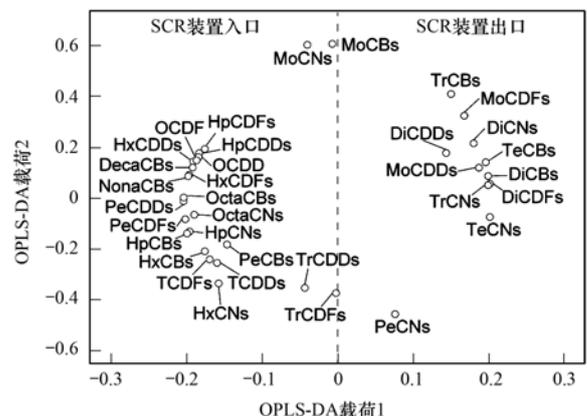


图 2 SCR 装置进出口废气中 UP-POPs 同系物的正交偏最小二乘法判别分析(OPLS-DA)载荷

Fig. 2 OPLS-DA loading plot of the UP-POPs homologue in SCR inlet and outlet flue gases

从不同 UP-POPs 同系物浓度分布数据可见,在 SCR 装置入口废气中高氯取代 UP-POPs 同系物的含量较高,这些高氯取代 UP-POPs 同系物包括三至八氯代二苯并二噁英(Tr-OcCDDs)、三至七氯代二苯并呋喃(Tr-HpCDFs)、四至九氯代联苯(Te-NoCBs)和三至七氯代萘(Tr-HpCNs)(图 3)。而在

SCR 出口废气中低氯代 UP-POPs 的相对含量较高 (图 3), 尤其是二氯代二苯并二噁英 (DiCDDs)、二氯代二苯并呋喃 (DiCDFs)、三氯代二苯并呋喃

(TrCDFs)、Di-TeCBs、三氯代萘 (TrCNs) 和四氯代萘 (TeCNs). SCR 装置对废气中 UP-POPs 同系物的脱除率表现出随氯取代度增加而增加的趋势.

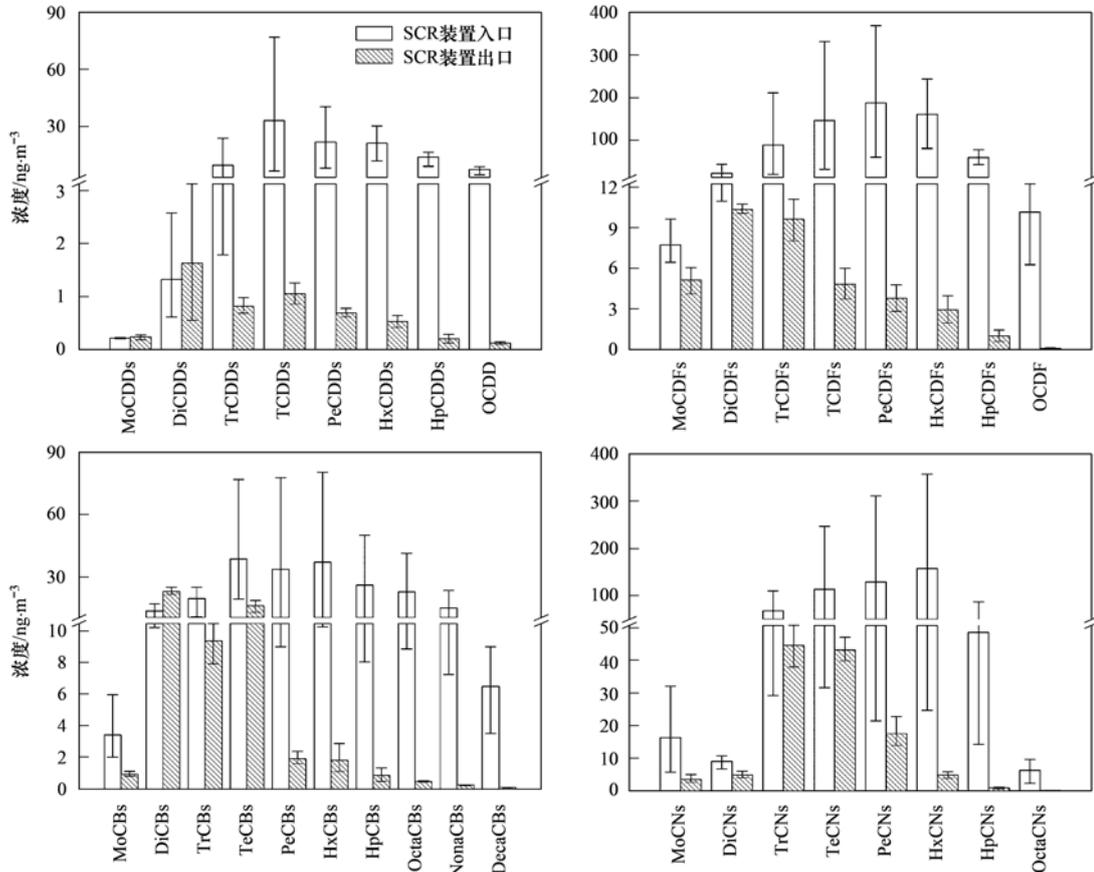


图 3 SCR 装置进出口废气中 UP-POPs 同系物的分布特征

Fig. 3 Homologue profiles of UP-POPs in SCR inlet and outlet flue gases

2.3 SCR 装置降尘中 UP-POPs 的水平及同系物分布特征

废气流经 SCR 装置蜂窝状催化剂模块后, 部分废气中的颗粒物会被 SCR 装置截获, 形成降尘; 部分废气中的 UP-POPs 会随着降尘的分流得以从废气中脱除. 从图 4 可见, 沉降灰中 \sum PCDDs、

\sum PCDFs、 \sum PCBs 和 \sum PCNs 的含量分别为 1.8、4.2、14.8 和 4.1 $\text{ng}\cdot\text{g}^{-1}$, 以 \sum PCBs (59.4%) > \sum PCDFs (16.9%) > \sum PCNs (16.4%) > \sum PCDDs (7.3%) 的顺序呈递减趋势. 降尘中二噁英的 I-TEQ 含量为 0.14 $\text{ng}\cdot\text{g}^{-1}$.

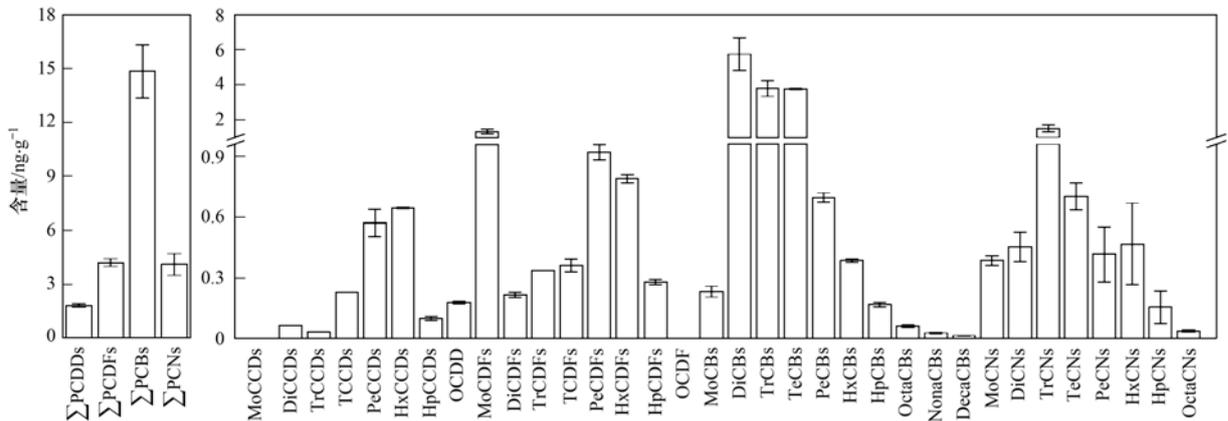


图 4 SCR 装置降尘中 UP-POPs 的含量和同系物分布特征

Fig. 4 Contents and homologue profiles of UP-POPs in SCR dustfall

在 SCR 装置降尘中,五氯和六氯代二苯并二噁英 (PeCDDs 和 HxCDDs)、一氯二苯并呋喃 (MoCDFs)、五氯和六氯代二苯并呋喃 (PeCDFs 和 HxCDFs)、Di-TeCBs、TrCNs 和 TeCNs 的含量相对较高. 降尘中二噁英同系物的分布模式与入口废气更为相似,而降尘中多氯联苯和多氯萘的同系物分布模式与出口废气基本一致.

3 讨论

以上结果表明,SCR 装置对焦炉煤气燃烧废气中的二噁英、多氯联苯和多氯萘具有较高脱除能力,对二噁英的脱除率接近 95%. 为实现对废气中 NO_x 的高效脱除,SCR 装置前通入氨水经蒸发产生氨气作为还原剂. 在氨气还原 NO_x 为 N_2 的同时,也会使废气中的 UP-POPs 发生催化还原反应. 在本研究中,SCR 装置入口废气中高氯取代的 UP-POPs 的相对含量较高,而出口废气中低氯取代的 UP-POPs 含量较高(图 2 和图 3),这意味着废气中的 UP-POPs 在 SCR 催化剂作用下发生了催化脱氯反应,催化还原脱氯是 SCR 装置脱除废气中 UP-POPs 的重要机制.

如果仅发生催化还原反应,SCR 装置出口废气中 UP-POPs 的同系物浓度分布应该表现出随氯取代数减少而升高的趋势,或者会有较高的非氯取代母体化合物生成. 在本研究中,SCR 装置出口废气中一氯取代 UP-POPs 同系物浓度低于二氯取代 UP-POPs,且二者均低于三氯取代 UP-POPs. 这意味着,通过催化还原反应生成的低氯取代 UP-POPs 也在不断地从废气中脱除. 为进一步了解低氯取代 UP-POPs 的脱除机制,本研究分析了废气中二噁英的母体化合物 DD 和 DF. 结果表明,废气中 DD 和 DF 浓度在 SCR 装置入口处分别为 $2.8 \text{ ng}\cdot\text{m}^{-3}$ 和 $203.2 \text{ ng}\cdot\text{m}^{-3}$,在出口处分别为 $1.3 \text{ ng}\cdot\text{m}^{-3}$ 和 $75.9 \text{ ng}\cdot\text{m}^{-3}$ (图 5). SCR 装置出口废气中 DD 和 DF 的浓度明显低于入口废气,说明废气中的 DD 和 DF 在 SCR 催化剂的作用下被不断地催化氧化脱除,催化氧化降解也是 SCR 装置脱除废气中 UP-POPs 的重要机制. 蜂窝状 SCR 催化剂模块中的 TiO_2 和 V_2O_5 均具有催化氧化能力,应该是废气中 UP-POPs 得以脱除的重要原因^[14, 23-25].

SCR 装置降尘的分流也应该对废气中 UP-POPs 的脱除有所贡献. 根据废气中的颗粒物浓度、SCR 装置对颗粒物的截获率及实际观察结果,笔者估算 SCR 装置降尘产生速率约为 $1.5 \text{ kg}\cdot\text{h}^{-1}$. 根据废气和降尘中 UP-POPs 的浓度数据,SCR 装置降尘分流对废气中二噁英、多氯联苯和多氯萘的贡献率分

别仅为 0.07%、0.80% 和 0.07%. 可见,降尘分流对废气中 UP-POPs 的脱除贡献极小. 降尘中的 DD 和 DF 也被定量分析,其含量分别为 $0.5 \text{ ng}\cdot\text{g}^{-1}$ 和 $14.2 \text{ ng}\cdot\text{g}^{-1}$ (图 5),分别高于一氯代二苯并二噁英 (MoCDDs) 和 MoCDFs. 这可能是因为氨气氛围中降尘颗粒表面吸附的二噁英发生了催化还原反应.

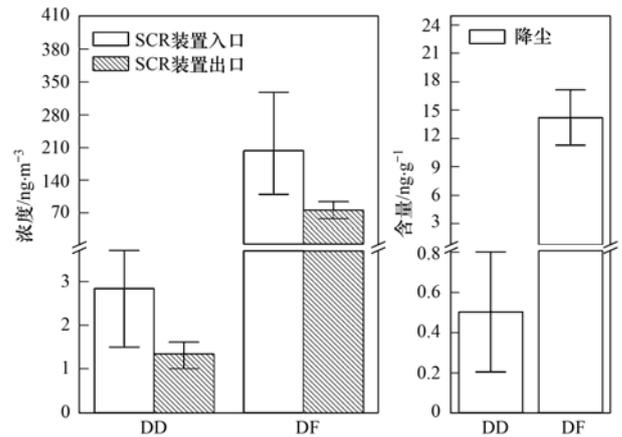


图 5 SCR 装置进出口废气和降尘中 DD 和 DF 的浓度和含量

Fig. 5 Concentrations of DD and DF in SCR inlet and outlet flue gas and dustfall

4 结论

在氨气还原条件下,SCR 装置对焦炉煤气燃烧废气中的典型 UP-POPs 实现了很好的协同脱除效果,对二噁英的总体脱除率接近 95%,对多氯联苯和多氯萘的总体脱除率高于 70%. SCR 装置入口和出口废气中 UP-POPs 同系物的分布模式截然不同,入口处以高氯取代为主,出口处以低氯取代为主;SCR 装置对高氯取代的 UP-POPs 有更高的脱除率. 催化还原反应和催化氧化反应同时发生是 SCR 装置脱除焦炉煤气燃烧废气中 UP-POPs 的主要机制,降尘分流对废气中 UP-POPs 的脱除贡献极小.

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CONTENTS

Cause and Effect Evaluation of PM _{2.5} During Three Red Alerts in Beijing from 2015 to 2016	LÜ Zhe, WEI Wei, ZHOU Ying, <i>et al.</i> (1)
Effects of Emission Reductions of Key Sources on the PM _{2.5} Concentrations in the Yangtze River Delta	YU Yan, WANG Ze-hua, CUI Xue-dong, <i>et al.</i> (11)
Health Benefit Analyses of the Clean Air Action Plan Implementation in Shanghai	DAI Hai-xia, AN Jing-yu, LI Li, <i>et al.</i> (24)
Monitoring and Analysis of the Spatio-temporal Change Characteristics of the PM _{2.5} Concentration Over Beijing-Tianjin-Hebei and Its Surrounding Regions Based on Remote Sensing	CHEN Hui, LI Qing, LI Ying, <i>et al.</i> (33)
Applicability of MODIS C006 Aerosol Products in a Typical Environmental Area of the Beijing-Tianjin-Hebei Region	WANG Hai-lin, LIU Qiong, CHEN Yong-hang, <i>et al.</i> (44)
Impact of Meteorological Factors on the Ozone Pollution in Hong Kong	ZHAO Wei, GAO Bo, LIU Ming, <i>et al.</i> (55)
Transport Characteristics of PAN and O ₃ in the Lower Atmosphere of the Boundary Layer in Tianjin in Summer	YAO Qing, MA Zhi-qiang, LIN Wei-li, <i>et al.</i> (67)
Analysis of Pollution Characteristics and Sources of PM _{2.5} Chemical Components in Chengdu in Winter	WU Ming, WU Dan, XIA Jun-rong, <i>et al.</i> (76)
Transport Pathways and Potential Sources of PM _{2.5} During the Winter in Zhengzhou	DUAN Shi-guang, JIANG Nan, YANG Liu-ming, <i>et al.</i> (86)
Characteristics and Sources of Water-soluble Organic Carbon/Nitrogen in PM _{2.5} During Spring in Changzhou	LI Qing, HUANG Wen-qian, MA Shuai-shuai, <i>et al.</i> (94)
Pollution Characteristics and Emission Factors of VOCs from Vehicle Emissions in the Tianjin Tunnel	SUN Lu-na, LIU Yan, ZHAO Jing-bo, <i>et al.</i> (104)
Size Distribution and Carbon Component Characteristics of Atmospheric Particulate Matter from Motor Vehicles	MEI De-qing, ZHU Zong-ning, SUN Tian-shuo, <i>et al.</i> (114)
Conversion Characteristics of Combustible Particles from Coal-fired Flue Gas in WFGD and WESP	YANG Liu, ZHANG Bin, WANG Kang-hui, <i>et al.</i> (121)
Particle Removal Characteristics of an Ultra-low Emission Coal-fired Power Plant	RUAN Ren-hui, TAN Hou-zhang, DUAN Yu-feng, <i>et al.</i> (126)
Spatial and Temporal Distribution Characteristics and Fugitive Dust Emission of Building Sites in Beijing	ZHANG Li-kun, LI Ling-jun, JIANG Lei, <i>et al.</i> (135)
Simultaneous Removal of Polychlorinated Dibenzo- <i>p</i> -dioxins/dibenzofurans, Polychlorinated Biphenyls, and Polychlorinated Naphthalenes From Flues Gases From Coke Gas Burning Using Selective Catalytic Reduction Equipment	REN Mei-hui, FAN Yun, WANG Sheng, <i>et al.</i> (143)
Space-Time Characteristics and Environmental Significance of the Stable Isotopes in Precipitation in the Gulang River Basin	GUI Juan, LI Zong-xing, FENG Qi, <i>et al.</i> (149)
Spatial Distribution and Degradation of CDOM in the Minjiang River in Summer	CHENG Qiong, ZHUANG Wan-e, WANG Hui, <i>et al.</i> (157)
Parallel Factor Analysis of Fluorescence Excitation Emission Matrix Spectroscopy of CDOM from the Mid-culture Period of Shrimp Ponds in a Subtropical Estuary	ZHU Ai-ju, SUN Dong-yao, TAN Ji, <i>et al.</i> (164)
Spectral Characteristics and Sources of Dissolved Organic Matter With Different Relative Molecular Weight from Rainwater From Summer and Autumn in the Zhoucun Reservoir Based on UV-Vis and EEMs	ZHOU Shi-lei, ZHANG Yi-ran, HUANG Ting-lin, <i>et al.</i> (172)
Characteristics and Fouling Potential of Dissolved Organic Matter in a Stratified Source Water Reservoir	LI Kai, WANG Xiao-dong, HUANG Ting-lin, <i>et al.</i> (185)
Water-Air Interface CO ₂ Exchange Flux of Typical Lakes in a Mountainous Area of the Western Chongqing and Their Influencing Factors	LUO Jia-chen, NI Mao-fei, LI Si-yue (192)
Reconstruction of Water Hyperspectral Remote Sensing Reflectance Based on Sparse Representation and Its Application	LI Yuan, LI Yun-mei, GUO Yu-long, <i>et al.</i> (200)
Nitrification Rates and Pollution Characteristics of Sediments with Different Geomorphic Features in the Shiwuli Stream, Chaohu Lake Basin	LI Ru-zhong, QUE Feng-xiang, XIONG Hong-bin, <i>et al.</i> (211)
Distribution and Bioavailability of Phosphorus in Surface Sediments in Megalopolis: A Case Study of Chengdu	DING Yao, OUYANG Li-li, SHI Qing, <i>et al.</i> (219)
Distribution and Fluxes of Perfluoroalkyl and Polyfluoroalkyl Substances in the Middle Reaches of the Yellow River (Weinan-Zhengzhou Section)	LI Qi-lu, CHENG Xiang-hui, ZHAO Zhen, <i>et al.</i> (228)
Distribution and Sources of Polycyclic Aromatic Hydrocarbons in a Karst Groundwater System in a Strongly Industrial City	MIAO Ying, KONG Xiang-sheng, LI Cheng-zhan (239)
Pollution Characteristic and Risk Assessment of Polycyclic Aromatic Hydrocarbons in the Groundwater of Shen-Fu New City in the Hunhe River Basin	ZHANG Shi-chao, YAO Hong, XIANG Xin-xin, <i>et al.</i> (248)
Distribution and Risk Assessment of Polycyclic Aromatic Hydrocarbons and Their Derivatives in Wastewater-Receiving Rivers in Beijing	FU Lu-jing, LI Yi-bing, QIAO Meng, <i>et al.</i> (256)
Influence of the Coagulation Mechanism on the Coagulation Performances Using New Composite Coagulants: Role of the Raw Water Characteristics	QU Jiang-dong, XU Hui, XU Jian-kun, <i>et al.</i> (263)
Indirect Photodegradation of Sulfamethoxazole in Water	LI Cong-he, CHE Xiao-wei, BAI Ying, <i>et al.</i> (273)
Fabrication of the Heterojunction Photocatalyst MoS ₂ /BiOI and Its Investigation of Its Photocatalytic Reduction and Oxidation Activities	ZHANG Liang, ZHAO Chao-cheng, GAO Xian-yao, <i>et al.</i> (281)
Nanoscale Zero-valent Copper-Activated Molecular Oxygen for the Degradation of Enrofloxacin in Water	NI Yong-jiong, CHENG Yong-qing, XU Meng-yuan, <i>et al.</i> (293)
Adsorption of Cr(VI) in Water by Malfanite Modified with Different LDHs Coatings	ZHANG Xiang-ling, DENG Li-chu, FANG Chen-jia, <i>et al.</i> (300)
Preparation of the Silane Monolayer on Magnetite Nanoparticles and Its Performance with Respect to Phosphate Removal from Water	XING Ming-chao, XIE Qiang, CHEN Shou-hui, <i>et al.</i> (310)
Optimization of Dyeing Wastewater Treatment with New Eco-friendly Polysilicate Ferromanganese	TANG Li-peng, WEI Qun-shan, LÜ Qiang, <i>et al.</i> (318)
Sulfate Reduction and Microbial Community of Autotrophic Biocathode in Response to Externally Applied Voltage	HU Jia-ping, ZENG Cui-ping, LUO Hai-ping, <i>et al.</i> (327)
Effect of Carbon Sources on the Accumulation of Endogenous Polymers and Denitrification in the O/A-F/F Mode	CUI You-wei, JIN Chang-lin, WANG Hao-han, <i>et al.</i> (336)
Effect of the Influent C/P Ratio on the Nutrient Removal Characteristics of the SNEDPR System	ZHEN Jian-yuan, YU De-shuang, WANG Xiao-xia, <i>et al.</i> (343)
Effect of Different Sludge Retention Time (SRT) Operations on the Nutrient Removal Characteristics of a SNEDPR System	WANG Xiao-xia, ZHEN Jian-yuan, ZHAO Ji, <i>et al.</i> (352)
Denitrification and Phosphorus Removal from Low C/N Urban Sewage Based on a Post-Partial Denitrification AOA-SBR Process	GONG Xiu-zhen, YU De-shuang, YUAN Meng-fei, <i>et al.</i> (360)
Startup and Stabilization of Nitrosation in an Anoxic-aerobic Continuous-flow Reactor with Granules	LI Dong, GUO Yue-zhou, LAO Hui-mei, <i>et al.</i> (369)
Startup Strategies for the SNAD Granular Sludge Process at Low Temperature	LI Dong, CUI Ya-qian, ZHAO Shi-xun, <i>et al.</i> (376)
Effect of the Sampling Scale and Number on the Heterogeneity of Soil Respiration in a Mixed Broadleaf-conifer Forest	YAN Jun-xia, SUN Qi, LI Jun-jian, <i>et al.</i> (383)
Effects of the Crop Rotation on Greenhouse Gases from Flooded Paddy Fields	FENG Xi, JIANG Chang-sheng, PENG Xiao-le, <i>et al.</i> (392)
Response of the Soil N ₂ O Emission and Ammonia-oxidizing Microorganism Community to the Maize Straw Return with Reducing Fertilizer in Purple Soil	HUANG Rong, GAO Ming, WANG Ying-yan, <i>et al.</i> (401)
Community Structure of CO ₂ -fixing Soil Bacteria from Different Land Use Types in Karst Areas	ZHANG Shuang-shuang, JIN Zhen-jiang, JIA Yuan-hang, <i>et al.</i> (412)
Composition and Predictive Functional Analysis of Rhizosphere Bacterial Communities in Riparian Buffer Strips in the Danjiangkou Reservoir, China	SUN Feng, TIAN Wei, ZHANG Fei, <i>et al.</i> (421)
Migration and Transformation of Adsorbed Arsenic Mediated by Sulfate Reducing Bacteria	JIA Qian-qian, LI Wei, WANG Ya-nan, <i>et al.</i> (430)
Spatial Distribution and Risk Assessment of Heavy Metal Contamination in Surface Farmland Soil Around a Lead and Zinc Smelter	WANG Yang-yang, LI Fang-fang, WANG Xiao-yang, <i>et al.</i> (437)
Effect of a Mining and Smelting Plant on the Accumulation of Heavy Metals in Soils in Arid Areas in Xinjiang	YANG Wei-guang, WANG Mei-e, CHEN Wei-ping (445)
Assessment of the Speciation and Pollution of Heavy Metals in Paddy Soils from the Jiulong River Basin	LIN Cheng-qi, HUANG Hua-bin, HU Gong-ren, <i>et al.</i> (453)
Characteristics of Stable Mercury Isotopic Compositions in the Food Web of the Caohai Lake	XU Yi-yuan, HE Tian-rong (461)
Interaction of Cd and Zn Affecting the Root Morphology and Accumulation of Heavy Metals in <i>Sedum aizoon</i>	GUO Jun-mei, YANG Jun-xing, YANG Jun, <i>et al.</i> (470)
Effects of Different Concentrations of Lanthanum on the Growth and Uptake of Pb by Maize Grown Under Moderate Lead Stress	WANG Qi-fan, GUO Wei, CHANG Qing, <i>et al.</i> (480)
Distribution and Bioaccumulation Characteristics of Cadmium in Fish Species from the Longjiang River in the Guangxi Autonomous Region	WANG Jun-neng, ZHAO Xue-min, HU Guo-cheng, <i>et al.</i> (488)
Influence of Six Digestion Methods on the Determination of Polystyrene Microplastics in Organisms Using the Fluorescence Intensity	ZOU Ya-dan, XU Qing-qing, ZHANG Ge, <i>et al.</i> (496)
Preparation of a Nutritional Slow-release Packing Material with Function Microorganisms and Its Characteristics Evaluation	FENG Ke, XU Dan-hua, CHENG Zhuo-wei, <i>et al.</i> (504)