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污水厂二级出水中难凝聚有机物的臭氧化特性

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摘要:以城市污水处理厂二级出水为原水, 研究臭氧化处理对水中难凝聚有机物的去除效果. 结果表明单独混凝对水中有机物的去除率较低, 混凝过程中未被去除的有机物属于难凝聚有机物, 加入臭氧进行氧化, 随着臭氧投加量的增加, 二级出水色度和 UV₂₅₄ 去除率逐渐增大, 而 DOC 去除率变化较小. 当臭氧投加量(以 O₃/DOC 计)增加至 1.5 mg·mg⁻¹ 时, 色度、UV₂₅₄ 和 DOC 的去除率分别为 45%、34% 和 20%, 说明臭氧易于与不饱和结构有机物反应, 而直接氧化的矿化能力较弱. 为进一步明确二级出水有机物种类和含量的变化, 测定了混凝和不同臭氧投加量下有机物的相对分子质量和三维荧光光谱. 结果表明混凝对有机物含量的影响很小, 而臭氧能够优先氧化分解难凝聚有机物中的大分子物质, 明显减弱腐殖质类物质的荧光峰强度, 但不改变荧光峰位置. 同时采用光电子能谱分析有机物结构的变化规律, 结果表明混凝能够去除含羧基类官能团有机物, 而臭氧则易于与含苯环类难凝聚有机物反应, 随着臭氧投加量的增大, 脂肪类饱和有机物含量有所升高.

关键词:难凝聚有机物; 臭氧化; 混凝; 光电子能谱; 三维荧光

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Ozonation Characteristics of Low Coagulability Organic Matter from the Secondary Effluent of WWTPs

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Abstract: The aim of this study was to investigate the effectiveness of ozonation on the removal of organic matter with low coagulability from municipal secondary effluent. The results revealed that the removal efficiency of coagulation generally remained quite low. The residual organic matter belonged to low coagulability organic matter. The presence of the ozone increased the removal efficiency of color and UV₂₅₄ gradually for low coagulability organic matter, whereas DOC had no noticeable change; the efficiencies were 45%, 34%, and 20%, respectively, at a dosage (denoted as O₃/DOC) of 1.5 mg·mg⁻¹. It could be concluded that ozone easily reacted with unsaturated organic matter, and the mineralization of organic matter was less effective. In order to further define the variation in organic matter of the secondary effluent, the differences between the relative molecular weight distribution and fluorescence characteristics of coagulation and ozonation with different zone dosages were monitored in this study. The findings showed that coagulation had little effect on organic content. Nonetheless, ozone might have preferentially reacted with high-molecular-weight substances of organic matter with low coagulability and reduced the fluorescence intensity in the humic-like regions significantly. The shift of fluorescence peak was not changed by ozonation. In addition, via X-ray photoelectron spectroscopy (XPS) analysis, it was identified that coagulation could remove carboxylic organic matter. On the other hand, with the increasing ozone dosage (from 0 to 1.5 mg·mg⁻¹), ozone could preferentially react with low coagulability organic matter with aromatic structure, thus the amount of aliphatics increased gradually.

Key words: organic matters with low coagulability; ozonation; coagulation; X-ray photoelectron spectroscopy; three-dimensional fluorescence

随着水资源的日益短缺, 城市污水再生回用受到人们的广泛关注. 城市污水中 90% 的溶解态有机质和 96% 的非溶解态有机质可经常规生物处理工艺有效去除^[1], 但是仍存在部分难降解有机物, 需要进行深度处理. 混凝作为污水深度处理方法之一, 去除溶解性有机物一般通过混凝颗粒形成过程中的吸附电中和作用以及混凝剂与有机物之间复杂的络合反应实现, 主要依赖于原水水质、混凝剂投加量和 pH 等条件^[2-4]. 然而这些难降解有机物大多为酚类、苯类等有机物, 仅仅通过混凝、过滤、吸附等常规的物理、化学方法很难将它们去除^[5].

因此, 单独混凝对溶解性有机物的去除效果较差, 再生水水质难以保证.

臭氧化技术具有杀菌性强、无药剂残留、操作简单、不产生氯化消毒副产物等优点, 在饮用水净化、污水深度处理等方面均有很好的应用前景^[6,7].

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臭氧不仅可以和水中有机物发生反应, 改变有机物的结构和性质^[8]; 还可以破坏溶解性有机物中大分子结构, 生成小分子有机物, 提高有机物的可生化性^[9,10]. 尽管近年来, 臭氧氧化二级出水的研究逐渐增多, 然而涉及混凝无法去除的难凝聚有机物与臭氧作用的研究并不多, 且大多集中在单独臭氧与溶解性有机物的反应^[11]或预臭氧与其他工艺联用以提高有机物去除效率^[12]的研究上, 对于臭氧化工艺去除二级出水中难凝聚有机物的特性研究较少.

因此, 本研究将污水厂二级出水先经过混凝处理, 混凝之后的水再经过臭氧化处理后, 分析水中有机物色度、DOC、UV₂₅₄和SUVA等的变化情况, 并进一步讨论不同处理方式和不同臭氧投加量对难凝聚有机物相对分子质量、荧光特性和官能团种类

的影响.

1 材料与方法

1.1 实验材料及臭氧水制备

水样来自西安市某污水处理厂 A²/O 工艺的二沉池出水, 该污水处理厂进水主要来自城市生活污水. 采用图 1(a) 所示装置制备臭氧原液, 制备装置中加入适量超纯水, 用磷酸调 pH 至 3. 氧气源臭氧发生器(济南三康, SK-CFQ-5P)出口流量通过气体流量计控制在 20 L·h⁻¹, 溢出的气体通入 KI 吸收瓶. 制备时温度通过水浴控制在 0~4℃之间, 曝气约 2 h 后使用紫外分光光度计(UNIC, UV-4802)在波长 258 nm 下测定吸光度值 [$\epsilon_{258} = 3\,000\text{ L}\cdot(\text{mol}\cdot\text{cm})^{-1}$], 其浓度可达到 80 mg·L⁻¹.

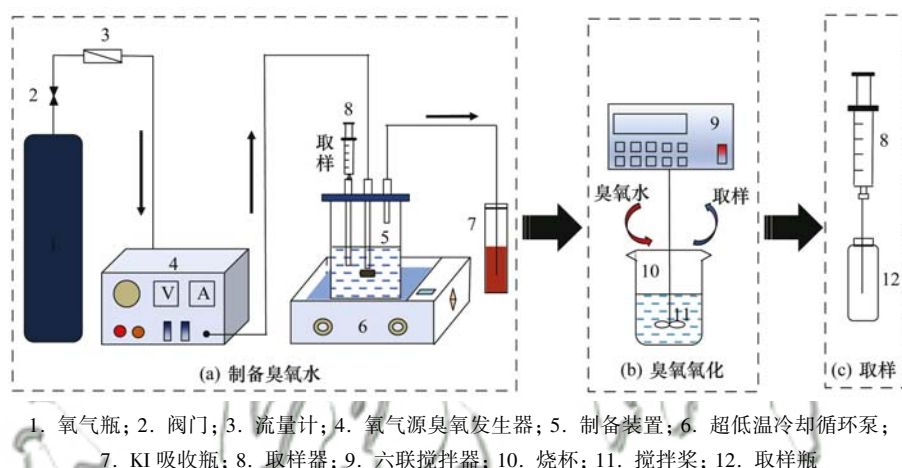


图 1 实验流程示意

Fig. 1 Diagram of the batch experiment

1.2 实验方法

1.2.1 混凝实验

将盛有原水的 500 mL 烧杯置于混凝搅拌器上(武汉梅宇, 3000-6), 使用 0.1 mol·L⁻¹ NaOH 和磷酸调节 pH 至 5, 加入 2 mmol·L⁻¹ 磷酸盐缓冲液以控制 pH 在 5±0.5 附近, 使用 6 mg·L⁻¹ 聚合氯化铝(以 Al 计)进行混凝实验, 如图 1(b) 所示. 混凝条件为 300 r·min⁻¹ ($G = 670.4\text{ s}^{-1}$) 快搅 1 min, 60 r·min⁻¹ ($G = 59.9\text{ m}\cdot\text{s}^{-2}$) 慢搅 30 min, 结束后静置沉淀 30 min, 并于液面下 2 cm 处取上清液过滤后测 DOC、UV₂₅₄、UV₂₈₀、色度、三维荧光、相对分子质量和 X 射线光电子能谱(XPS)等水质指标.

1.2.2 臭氧实验

取上述实验中的上清液分别置于 3 个 100 mL 烧杯中, 加入适量臭氧原液 ($\text{O}_3/\text{DOC} = 0.5, 1.0, 1.5$) 后立即开始搅拌, 实验条件为 60 r·min⁻¹, 搅

拌 20 min. 反应结束后静置 10 min, 取样测定 DOC、UV₂₅₄、UV₂₈₀、色度、三维荧光、相对分子质量和 XPS 等水质指标.

1.3 分析方法

(1) 水样预处理

污水处理厂沉淀池出水在进行实验分析前均需使用 0.45 μm 滤膜过滤, 过滤后的水质如表 1 所示. 样品的后续分析均使用 3~5 个平行样.

(2) pH 的测定

采用上海雷磁 PHSJ-3F 型精密酸度计测定.

(3) 色度的测定

采用分光光度铂钴比色法^[13]测定.

(4) UV₂₅₄、UV₂₈₀ 及 SUVA 的测定

采用紫外分光光度计分别在 254 nm 和 280 nm 下测定吸光度值. SUVA ($100 \times \text{UV}_{254}/\text{TOC}$) 表示单位浓度 TOC 的紫外吸光度值.

表 1 二级出水反应前后水质特征
Table 1 Water quality variation before and after reaction

项目	原水	混凝	臭氧化/ $\text{mg}\cdot\text{mg}^{-1}$		
			0.5	1.0	1.5
DOC/ $\text{mg}\cdot\text{L}^{-1}$	7.72 ± 1.57	6.97 ± 1.23	6.62 ± 1.01	6.35 ± 1.12	6.17 ± 1.25
色度	1.02 ± 0.05	0.87 ± 0.03	0.77 ± 0.02	0.66 ± 0.05	0.56 ± 0.03
UV ₂₅₄ / cm^{-1}	0.163 ± 0.003	0.144 ± 0.004	0.135 ± 0.003	0.121 ± 0.002	0.108 ± 0.004
UV ₂₈₀ / cm^{-1}	0.138 ± 0.002	0.125 ± 0.001	0.118 ± 0.003	0.109 ± 0.004	0.089 ± 0.002
SUVA/ $\text{L}\cdot(\text{mg}\cdot\text{cm})^{-1}$	2.11 ± 0.32	2.07 ± 0.27	2.04 ± 0.24	1.91 ± 0.26	1.75 ± 0.24

(5)DOC 的测定

采用日本岛津生产的 TOC-VCPH 分析仪测定. 测定前先用硫酸对水样进行酸化氮吹处理, 除去水中碱度保证测定值的准确性.

(6)液态臭氧浓度的测定

采用靛蓝比色法^[14]测定.

(7)三维荧光光谱

采用日本 JASCO 公司生产的 FP6500 型荧光分光光度计进行分析, 原水和混凝后的二级出水均稀释 1.5 倍测定. 激发波长范围为 220 ~ 480 nm, 发射波长范围为 280 ~ 550 nm. 激发和发射扫描间隔分别为 5 nm 和 2 nm, 扫描速度为 2 000 nm·min⁻¹. 测定前使用空白溶液校准, 排除水的散射峰影响.

(8)相对分子质量

相对分子质量由岛津公司生产的 LC-2010AHR 型高效液相色谱测定. 采用紫外检测器, 检测波长为 254 nm 和 280 nm, 色谱柱为 Zenix SEC-100 凝胶色谱柱, 流动相为 150 mmol·L⁻¹磷酸盐缓冲溶液, 流速 0.8 mL·min⁻¹. 不同聚合度聚乙二醇(PEG)标准品用于测定腐殖质类物质的标线.

(9)XPS 分析

利用 X 射线光电子能谱(英国热电, K-α)探究二级出水经混凝和臭氧化处理后的碳元素价键形式. XPS 分析前, 需将样品放置在冷冻干燥机(北京博益康, FD-1C-50)中干燥成粉末后进行测定. 实验得到的元素电子结合能以 C1s(285 eV)进行校正. C1s 谱图应用 XPSPEAK41 专业软件进行高斯分峰拟合, 对不同形态的碳原子进行定量分析.

2 结果与讨论

2.1 反应前后二级出水水质特性

由表 1 可以看出, 混凝对二级出水有机物的去除是十分有限的, 各项指标的去除率均在 15% 以下. 原因在于混凝主要去除水中悬浮颗粒和胶体微粒等大分子有机物(相对分子质量在 5 000 ~ 50 000 之间)^[15], 而污水厂二级出水中有机物分子量普遍

较低, 主要以腐殖酸形式存在^[16].

混凝无法去除的难凝聚有机物经臭氧化后 UV₂₅₄ 和 UV₂₈₀ 均有了明显去除, 且随着臭氧投加量的增加, UV₂₅₄ 和 UV₂₈₀ 的去除率逐渐增大. 这是因为 UV₂₅₄ 代表水中芳香族有机物以及含共轭双键结构的有机物, 特别是腐殖质类物质的含量, 而具有亲电特性的臭氧能够与有机物的不饱和键迅速发生反应, 使有机物芳香性降低或消失, 在短时间内即将不饱和有机物大量去除^[17,18], 从而有效降低水的紫外吸收值. 同时, 臭氧化后色度也随着臭氧投加量的增加而逐渐降低, 当臭氧投加量(以 O₃/DOC 计, 下同)增加至 1.5 mg·mg⁻¹ 时, 色度的去除率已达到 45%, 体现了臭氧良好的脱色性能^[19]. 相比于臭氧对 UV₂₅₄ 的去除效果, 臭氧化后 DOC 的去除率仅由 9% 增加到 20%, 说明臭氧对有机物直接氧化的矿化能力较弱. 刘建红等^[20]研究也认为, 臭氧完全氧化去除二级出水有机物的效果有限, 但能够有效氧化分解具有强烈紫外吸收的有机物.

SUVA 可以从某种程度上反映水中有机物的共轭不饱和及芳香程度^[21]. 由于臭氧对二级出水 UV₂₅₄ 的去除效果明显优于对 DOC 的去除效果, 因此臭氧化后 SUVA 随臭氧投加量的升高呈现逐渐降低的趋势, 说明经过臭氧化后有机物的不饱和性及芳香程度得以降低, 臭氧改变了有机物的结构. 这与 Gong 等^[22]和 Wu 等^[23]的研究结果一致, 臭氧对二级出水中 DOC 的去除效果不明显, 但水中的 SUVA 值却快速下降.

与此同时, 前期的研究结果显示^[24,25], 金属盐混凝剂及其水解产物能够作为臭氧化的催化剂, 引发链式反应, 产生强氧化性的·OH, 从而进一步氧化有机物. 本研究中混凝后水中残留的铝盐可能在臭氧化过程中起到促进臭氧分解, 产生·OH 的作用, 从而提高了难凝聚有机物的去除效率.

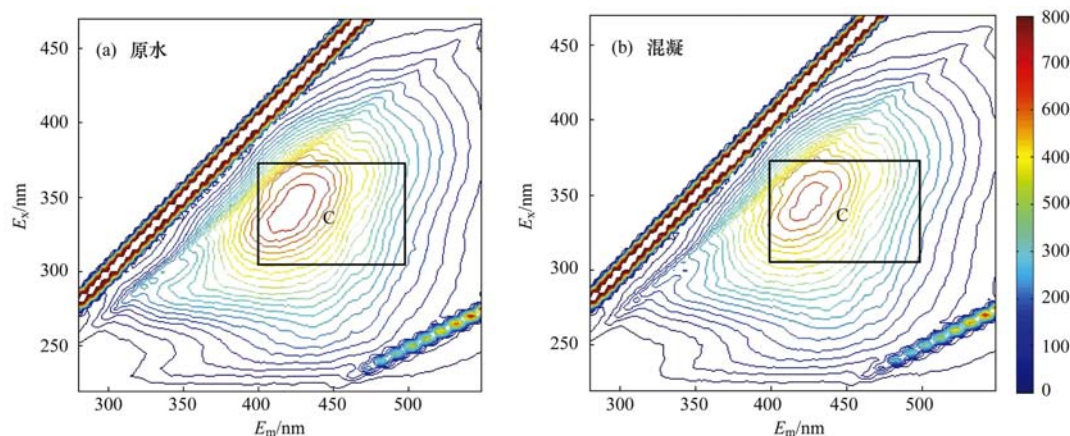
2.2 三维荧光 EEM 谱图

三维荧光光谱能够揭示多组分复杂体系中有机污染物种类及含量等信息^[26], 具有准确度高、灵敏

度高、所需样品少等优点,也被称为“荧光指纹”^[27]. 本实验采用三维荧光光谱法对原水及臭氧化前后二级出水的荧光特性进行表征,结果如图2和图3所示. 原水荧光光谱图中含有明显的荧光峰 $E_x/E_m = 350/440$ nm(峰C),根据已有的研究表明该处荧光峰表示腐殖质类物质^[28~30]. 经过混凝后峰C的荧光强度减弱了4.7%,但位置没有发生变化.

图3为不同臭氧投加量下的荧光谱图,经过臭

氧化后,难凝聚有机物的荧光峰强度迅速减弱且随着臭氧投加量的增加呈现大幅降低趋势. 臭氧投加量为 $0.5 \text{ mg} \cdot \text{mg}^{-1}$ 时,荧光峰C的强度降低了14.7%,说明较低浓度的臭氧就能与难凝聚有机物的荧光基团发生反应,破坏荧光基团结构. 当臭氧投加量升至 $1.5 \text{ mg} \cdot \text{mg}^{-1}$ 时,荧光峰强度已降至600以下,说明难凝聚有机物中共轭键数量明显减少,与前述的 UV_{254} 和色度降低结果一致.



均稀释1.5倍

图2 原水和混凝后的EEM谱图

Fig. 2 EEM spectra of raw water and after coagulation

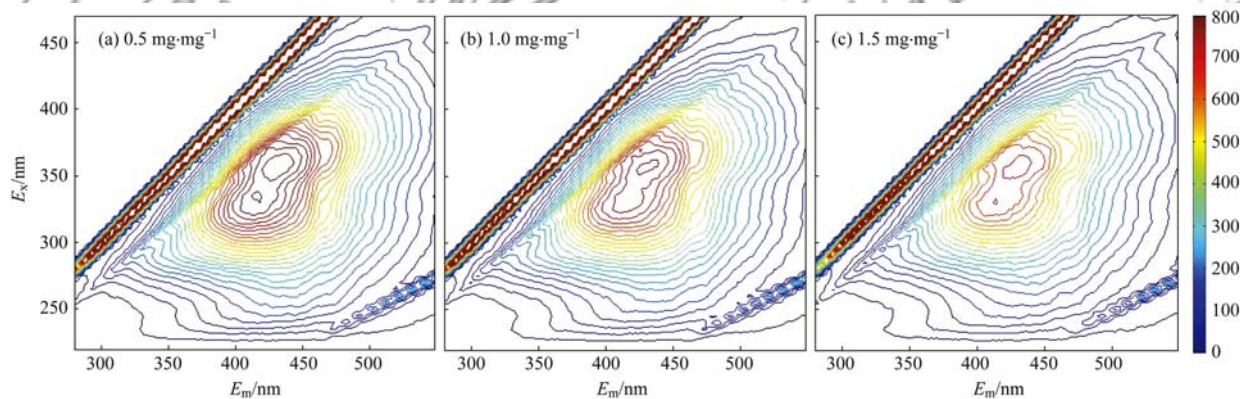


图3 二级出水臭氧化后EEM谱图

Fig. 3 EEM spectra of raw water after ozonation at three different ozone dosages

2.3 相对分子质量分析

由图4可以看出,二级出水相对分子质量主要集中在 $300 \sim 10\,000$ 范围内,混凝后相对分子质量分布并没有发生明显变化,只是紫外吸收强度略有降低. 混凝无法去除的有机物为难凝聚有机物,该类物质相对分子质量分布范围为 $300 \sim 10\,000$,主要为腐殖酸等小分子有机物^[31]. 关于难凝聚有机物的相对分子质量也有一些研究. 刘成等^[32]研究天然水体表明,混凝沉淀过程所去除有机物的相对分子质量主要集中在大于 $6\,000$ 的范围内,这是由于混

凝过程形成的絮体对大分子有机污染物吸附作用强于小分子有机物的吸附作用,因此混凝沉淀后形成的有机物相对分子量大多小于 $6\,000$. 张月^[33]研究腐殖酸与铝盐在混凝反应前后的GC-MS图谱时表明,混凝能够有效去除相对分子量在 $10\,000 \sim 30\,000$ 范围的腐殖酸,而相对分子量在 $5\,000 \sim 10\,000$ 的腐殖酸基本不与铝盐反应.

经过臭氧化后,一方面难凝聚有机物紫外吸收峰强度减弱更加明显,另一方面相对分子量位于 100 处出现一个明显的紫外吸收峰,且随着臭氧投

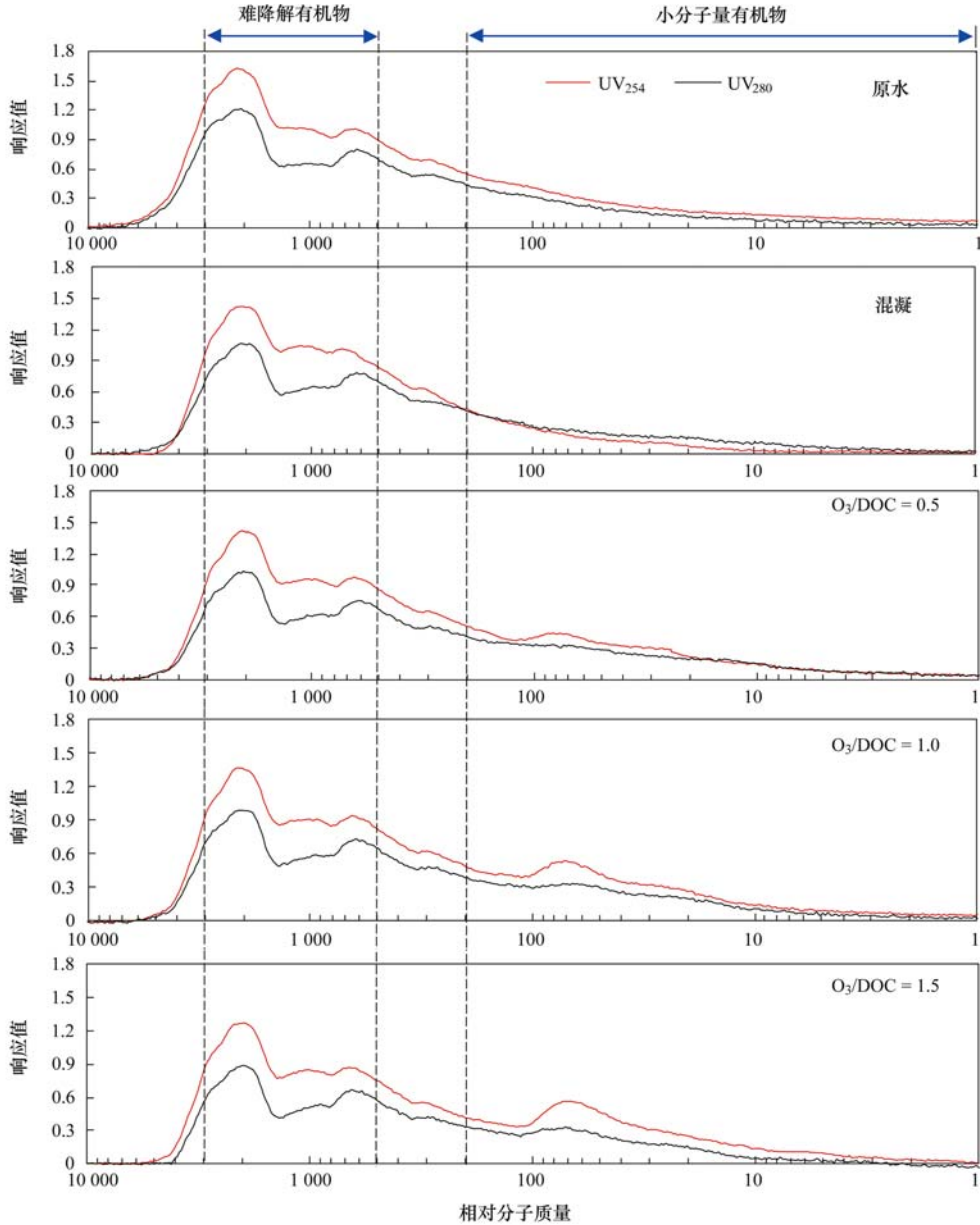


图 4 二级出水反应前后相对分子质量分布

Fig. 4 MWD of WWTP effluent before and after reaction

加量的增加,该处吸收峰强度逐渐增加,说明臭氧能够将大分子有机物氧化分解为小分子有机物,但难以将其完全氧化为 CO_2 和 H_2O ,只是生成了一些中间物质^[34],这也是臭氧化后 DOC 降低不明显的原因.

2.4 X 射线光电子能谱分析

为了进一步明确二级出水经混凝和臭氧化后难凝聚有机物结构的改变,利用 XPS 分析处理后碳元素 $\text{C}1\text{s}$ 的谱图,结果如图 5 和图 6 所示.图 5 的 XPS 谱图显示二级出水中存在 4 种官能团,分别为芳香碳、脂肪碳、羧基碳和羰基碳^[35,36].根据高斯分峰结果可以得到不同官能团所占的比例,结果如

图 7 所示.

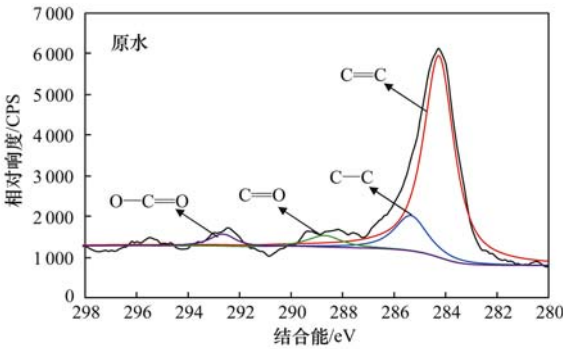


图 5 二级出水 XPS $\text{C}1\text{s}$ 图谱

Fig. 5 $\text{C}1\text{s}$ spectra of XPS from WWTP effluent

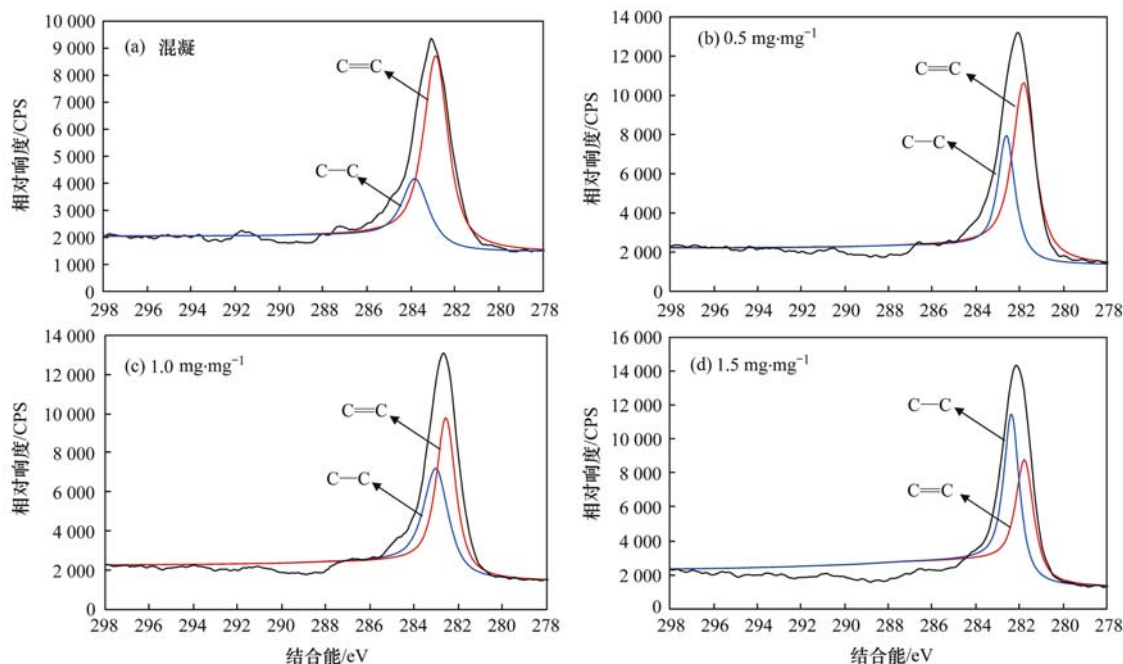


图6 臭氧化前后 XPS C1s 图谱

Fig. 6 C1s spectra of XPS before and after ozonation

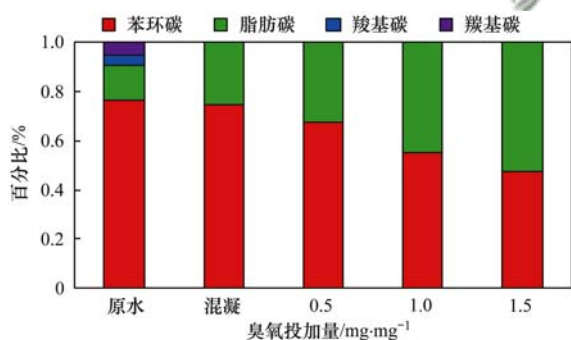


图7 臭氧化前后碳元素价键形态

Fig. 7 Carbon species before and after ozonation

二级出水中含有大量的苯环结构, 该类有机物占到大约 76.3%, 经过混凝处理后, 含氧官能团(羰基和羧基)数量明显减少, 脂肪碳数量基本无变化. 这是因为在弱酸性条件下, 金属盐混凝剂易于与有机物发生共聚络合反应, 优先与极性高、化学活性高的官能团发生络合, 而羧基类有机物较链烃类有机物极性较高, 因此混凝去除了大部分羰基和羧基类含氧官能团. 金鹏康^[37]利用 GC-MS 和 FT-IR 两种方法研究混凝前后腐殖酸中官能团的比较得到类似结论. 混凝工艺之后, 随着臭氧投加量的增加, 苯环碳类有机物含量逐渐减少, 当臭氧投加量增加至 $1.5 \text{ mg} \cdot \text{mg}^{-1}$ 时, 苯环类有机物减少 47.3%, 而脂肪碳类有机物数量增大至 52.7%, 证明臭氧易于与苯环结构有机物反应. 这与王树涛等^[9]的研究结果一致. 他们利用 GC-MS 检测到臭

氧化后有机物种类和数量明显增多, 其中烷烃类物质增多较明显, 而环状结构、羧酸和醇类化合物明显减少.

3 结论

(1) 单独的混凝处理对二级出水溶解性有机物的去除是十分有限的. 在混凝工艺后, 加入臭氧进行氧化, 二级出水中的色度、 UV_{254} 、SUVA 和 DOC 都得到一定程度的去除, 且随着臭氧投加量的增加, 去除率逐渐增大, 其中色度和 SUVA 的效果去除较明显, 而 DOC 的去除率较低.

(2) 二级出水中具有荧光特性的物质主要为腐殖质类物质. 臭氧能够改变二级出水中难凝聚有机物的荧光特性, 使得荧光强度明显降低. 随着臭氧投加量增加, 腐殖质类荧光峰强度减弱明显.

(3) 分子量分析结果表明, 二级出水经混凝后形成的难凝聚有机物相对分子量分布范围为 300 ~ 10 000, 该类物质主要为腐殖质类有机物. 臭氧化一方面能够降低该类有机物的紫外吸收峰强度, 另一方面会改变吸收峰的分布位置. 臭氧优先与大分子量难凝聚有机物反应, 生成小分子量有机物.

(4) 光电子能谱分析结果表明, 混凝能够去除二级出水中含羰基和羧基等极性较强的有机物, 臭氧化则主要去除含共轭双键结构的难凝聚有机物, 随着臭氧投加量的增加, 苯环类有机物数量减少明

显, 脂肪类有机物逐渐增加.

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