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二级出水 DOM 自活化过硫酸盐的效能

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摘要：基于二级出水中溶解性有机物(DOM)的光敏化特性,利用太阳辐照构建了实际二级出水 DOM 自活化过硫酸盐反应体系.分别研究了不同过硫酸盐类型、初始 pH 值以及共存无机阴离子对过硫酸盐分解率的影响,解析了反应前后 DOM 的理化特性与结构变化.结果表明,二级出水各分级组分可有效活化过一硫酸盐(PMS),其中亲水性物质(HI)的活化效果最佳,在 pH 值为 7 时,PMS 分解率可达 26.7%.分解过程中产生的活性氧化物质作用于 DOM,使其芳香结构逐步被破坏、分子量减小,所含类腐殖酸等荧光物质强度显著降低,木质素、脂质、单宁酸等化合物占比发生明显变化,表明二级出水 DOM 通过有效活化过硫酸盐实现了自降解.二级出水 DOM 中的类腐殖酸和木质素作为主要电子供体,受太阳光激发后将光生电子传递至 PMS 使其活化,从而引发 DOM 自降解过程.本研究构建的实际二级出水 DOM 自催化体系无需外加催化剂,避免了高能量消耗与二次污染.

关键词：二级出水；溶解性有机物；过硫酸盐；绿色低碳
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The efficiency of peroxymonosulfate self-activation by dissolved organic matter in secondary effluent. TIAN Bao-jia, BAI Xue*, HAN Yu-jia, JIN Peng-kang** (School of Human Settlements and Civil Engineering, Xi'an Jiaotong University, Xi'an 710049, China). *China Environmental Science*, 2025,45(8)：4333~4340

Abstract：The persulfate-based advanced oxidation process demonstrates significant advantages in the removal of refractory organic pollutants. However, traditional activation methods are associated with high energy consumption and the potential generation of secondary pollution, which restricts the widespread application of this technology. Based on the photosensitive properties of dissolved organic matter (DOM) in secondary effluent, a practical self-activation peroxymonosulfate (PMS) reaction system using solar radiation was established. Systematic investigations were conducted on the effects of different persulfate types, initial pH conditions, and common inorganic anions on PMS decomposition efficiency, accompanied by comprehensive characterization of physicochemical properties and structural transformations in DOM before and after the reaction. The results indicated that the all fractions of secondary effluent could effectively activate PMS, with the hydrophilic (HI) fraction exhibiting the most efficient activation, achieving a PMS decomposition rate of up to 26.7% under neutral conditions. During the decomposition process, reactive oxidative species generated from PMS interacted with DOM, leading to the gradual degradation of its aromatic structures and a reduction in molecular weight. The intensity of fluorescent substances, such as humic-like compounds, significantly decreased, while the proportions of lignin, lipids, tannins, and other compounds had notable changes. These observations indicated that the DOM in secondary effluent achieved self-degradation through effective activation of PMS. Humic-like compounds and lignin in secondary effluent DOM served as the primary electron donors. Upon excitation by sunlight, they transferred photogenerated electrons to PMS, thereby activating it and triggering the self-degradation of DOM. The self-catalytic system of using the DOM in actual secondary effluent in this study without the need for external catalysts, avoiding high energy consumption and secondary pollution.

Key words：secondary effluent；dissolved organic matter；persulfate；green and low-carbon

二级出水中溶解性有机物(DOM)多为难降解物质,是污水深度处理的难点,也是污水资源化回用的限制性因素[1-2].新兴的过硫酸盐($SO_4^{\bullet-}$)高级氧化技术与传统以羟基自由基为基础的高级氧化技术相比,因产生氧化能力更强($SO_4^{\bullet-}$ *vs.* •OH:2.5~3.1V *vs.* 1.9~2.7V)、半衰期更长($SO_4^{\bullet-}$ *vs.* •OH:30~40μs *vs.* 20ns)的硫酸根自由基,在难降解有机物去除方面展现出良好的性能[3-7].硫酸根自由基的生成依赖于过硫酸盐的有效活化,常见活化方式包括紫外辐射、热辐射、超声辐射、过渡金属离子及其氧化物、碳基材料等,需外加能量或催化剂,导致能耗高、成本高、易引发二次污染等问题,制约该技术的推广与应用[8].

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团队前期研究表明,光敏有机染料受可见光激发后,自身的光生电子可有效转移至溶液中共存的过硫酸盐,使其活化生成 $SO_4^{\bullet-}$、•OH、$^1O_2$ 等多种活性氧化物质,从而使染料高效降解[9].二级出水 DOM 主要由微生物代谢产物、天然有机物、难降解人工合成有机物以及尚未辨识的有机污染物组成,多为芳香烃和脂肪族化合物,富含多种发色官能团,使 DOM 具有一定的光吸收特性[10-11].

基于光敏染料自活化过硫酸盐的反应原理,结合二级出水 DOM 结构特征,设计基于太阳辐射的二级出水 DOM 自活化过硫酸盐的自降解路径.本文构建了二级出水/过硫酸盐/太阳光自催化体系,探究二级出水不同分级组分中 DOM 对过硫酸盐的活化效能差异,解析反应前后 DOM 的理化特性与结构变化,探明该自催化过程的应用潜能.

1 材料与方法

1.1 实验试剂

过一硫酸氢钾($KHSO_5{\cdot}0.5KHSO_4{\cdot}0.5K_2SO_4$)、过硫酸钾($K_2S_2O_8$)、氢氧化钠(NaOH)、盐酸(HCl)、甲醇($CH_3OH$)购于上海麦克林,碳酸氢钠($NaHCO_3$)、碳酸钾($K_2CO_3$)、碘化钾(KI)、硫酸钾($K_2SO_4$)、氯化钾(KCl)、硝酸钾($KNO_3$)购于天津科密欧,XAD-8 大孔吸附树脂购于 Sigma-Aldrich,实验用水均为超纯水.

1.2 实验方法

研究所用二级出水取自西安市某生活污水处理厂二沉池,水样经 0.45μm 滤膜过滤后,采用反渗透装置浓缩 10 倍.通过 XAD-8 大孔吸附树脂分级为疏水酸性物质(HOA)、疏水碱性物质(HOB)、疏水中性物质(HON)和亲水性物质(HI)[12].取二级出水各分级组分 50mL,添加 1mmol/L 过硫酸盐,使其充分混合,随后将样品置于带有冷却循环系统的双层玻璃烧杯中,使用氙灯(CEL-HXF300,中教金源)模拟太阳光源并将其置于反应器上方一定高度,设定太阳辐照强度为 150mW/cm^2,在固定时间点取样进行后续分析,每个样品设置三个平行样.

1.3 分析方法

采用紫外分光光度计(TU-1901,北京普析)测定水样过硫酸盐浓度、溶解性有机物 UV_{254} 和 UV_{365},采用总有机碳分析仪(TOC-VCPH,岛津)测定水样中溶解性有机碳浓度,采用配有凝胶色谱柱(Zenix SEC-100,美国赛分科技)和紫外可见吸收检测器的高效液相色谱仪(LC-2010AHT,日本岛津)、荧光光谱仪(F-7000,日立)和傅里叶回旋离子共振高分辨质谱仪(7.0T SolariX,布鲁克)分析反应前后水样中物质的分子量分布和组成差异.

2 结果与讨论

2.1 二级出水 DOM 自活化过硫酸盐效能差异

2.1.1 过硫酸盐类型的影响 分别以过一硫酸盐(PMS)和过二硫酸盐(PDS)为研究对象,考察太阳光照条件下二级出水不同分级组分活化过硫酸盐的潜力,以过硫酸盐分解率为评价依据,结果如图 1(a)和图 1(b)所示.二级出水中疏水酸性物质(HOA)、疏水碱性物质(HOB)、疏水中性物质(HON)对 PDS 几乎没有活化效果,而亲水性物质(HI)可分解约 5%的 PDS.相比之下,二级出水各分级组分对 PMS 的活化效果较为显著,由 HOA、HOB、HON、HI 构成的体系中,PMS 分解率分别为 23.0%、22.5%、14.7%和 26.7%.同时,反应后各体系的 SO_4^{2-} 浓度明显增大(图 1(c)),验证了 PMS 的有效分解.

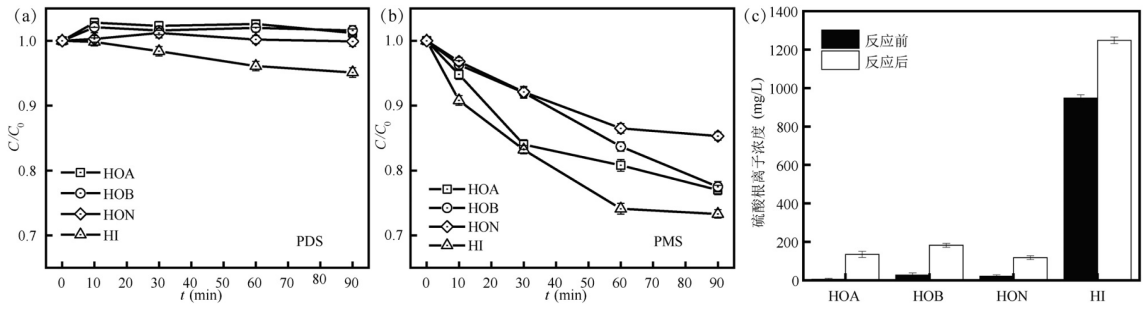


图 1 不同 DOM 对过硫酸盐的活化效果

Fig.1 Activation effects of persulfate by different DOM

[PMS]=1mmol/L,[PDS]=1mmol/L,pH=7,温度为 20℃

相同分级组分对不同过硫酸盐的活化效果差异显著,这与过硫酸盐的结构密切相关[13-14].PDS为对称结构,其过氧键的电荷均匀分布,而具有非对称结构的 PMS,与 H 原子相连的过氧键上带有部分正电荷,使之易于受到亲核进攻[15-19].因此,光照后产生的光激发态 DOM 分子作为电子供体,更容易将光生电子传递至 PMS,促使其分解产生多种活性氧化物质[20-21].基于良好的活化效果,后续研究围绕 PMS 展开.

2.1.2　初始 pH 值的影响　如图 2 所示,4 种分级组分构成的反应体系在中性条件下对 PMS 的活化效果优于酸性和碱性条件,这是由于 pH 值较低的条件下,H^+浓度高,导致 H^+与 HSO_5^-之间更容易产生静电引力,从而抑制 HSO_5^-与环境中 DOM 的接触,使活化效果减弱[22].而在 pH 值较高的环境中,羧基和酚羟基去质子化,导致 DOM 中负电基团含量增加, DOM 与 HSO_5^-间的静电斥力作用,同样不利于光生电子的转移[23].在 pH 值为 7 的条件下 DOM 结构能够充分扩展,使具有反应活性的官能团更易接触环境中的氧化剂[24].所以在中性条件下,更有利于对 PMS 的活化,因此本研究选择在 pH 值为 7 的条件下展开.

2.1.3　共存离子的影响　如图 3 所示,碳酸根离子的存在使 HOA、HOB、HON、HI 4 种分级组分构成的反应体系对 PMS 的活化效果显著增强,其 PMS 分解率分别提升至 48.4%、74.9%、36.6%、60.0%.这是由于碳酸根离子能够活化 PMS,通过亲核反应攻击 PMS 使其分解,使 PMS 的分解率进一步提高[25].而硫酸根离子、氯离子、硝酸根离子的存在对反应体系中 PMS 的活化几乎无影响,这说明构建的太阳光照下 DOM 自活化 PMS 反应体系,在具有上述离子存在的较为复杂的水质中,仍能保持良好的稳定性.

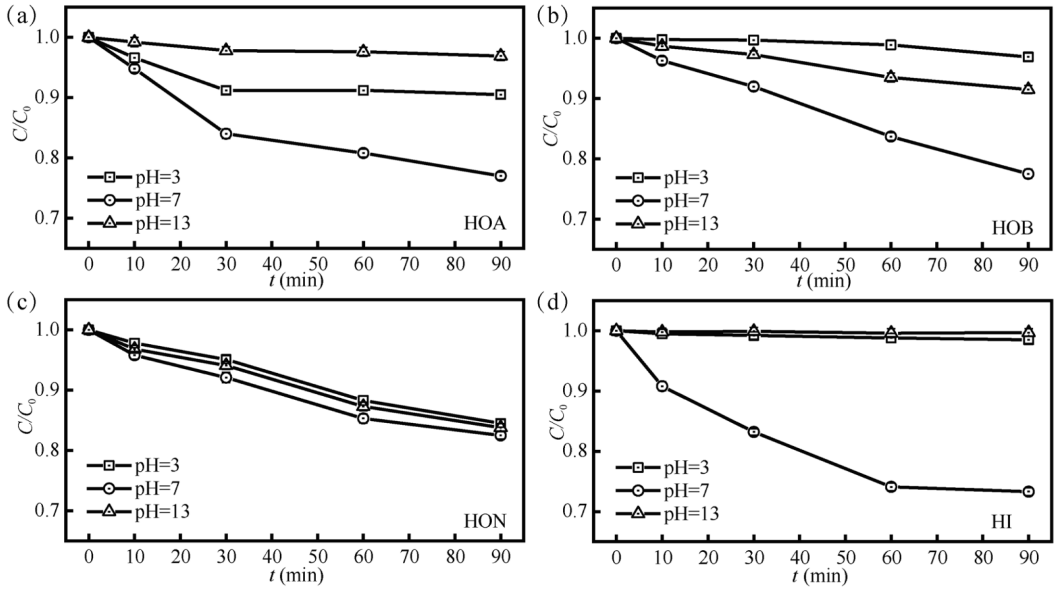
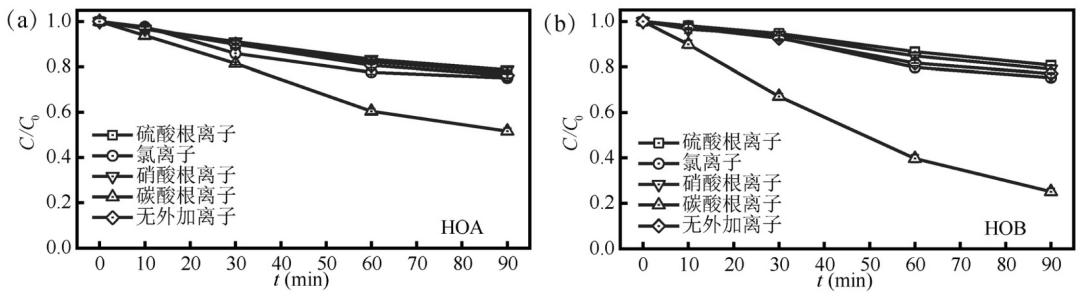


图 2　不同 pH 值对自活化 PMS 的影响

Fig.2　Effect of pH on the self-activation of PMS

[PMS]=1mmol/L,pH=7,温度为 20℃



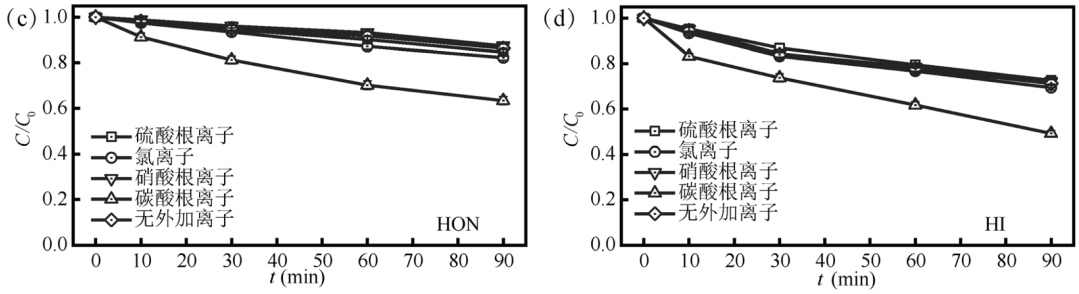


图 3 共存离子对自活化 PMS 的影响

Fig.3 Effect of coexisting ions on the self-activation of PMS

[PMS]=1mmol/L,各离子浓度为 10mmol/L,pH=7,温度为 20℃

2.2 二级出水各组分理化与结构特征变化

2.2.1 二级出水各组分理化特征解析 由图 4 可知,4 种分级组分中 HOA 和 HI 的溶解性有机碳(DOC)含量最高,分别为 32.9%和 36.0%,说明该种二级出水的主要成分为疏水酸性物质和亲水性物质.UV_{254} 能够反应水中芳香化合物的含量[26].DOC 值的大小能够衡量各组分中 DOM 浓度的高低.$SUVA_{254}$ 是指 UV_{254} 与 DOC 的比值,可反应 DOM 的腐殖化程度,其值越大,腐殖化程度越高[27-28]. E_2/E_3 为溶解性有机物 UV_{254} 与 UV_{365} 的比值,能够指示有机物的分子量大小,其值与分子量大小呈负相关[28-29].由表 1 可知,4 种分级组分中,HI 和 HOA 的 UV_{254}、$SUVA_{254}$ 较大,DOC 值较高,E_2/E_3 较小,说明相比于 HOB 和 HON,这两类 DOM 浓度高,芳香族化合物含量高,腐殖化程度高,平均分子量较大.Nie 等[11]指出,随着 DOM 浓度由 0mg/L 逐步增至 50mg/L,PMS 的降解速率逐渐增大,同时,PMS 的分解率也随之升高.在本研究中 DOM 浓度较高的组分对应更高的 PMS 活化率,表明 DOM 浓度的差异是影响 PMS 活化的因素之一.此外,Li 等[30]的研究表明腐殖化程度较高的 DOM 具有更强的光反应活性,并且腐殖化程度能通过影响 DOM 光转化模式,使腐殖化程度较高的 DOM 在光照后能够活化氧化剂,产生活性氧化物质.同时腐殖化程度越高的 DOM,芳香性越强,其电子交换能力和供电子能力也越强[31].各组分对应的 $SUVA_{254}$ 与其 PMS 活化率呈正相关,说明不同腐殖化程度是造成二级出水各组分活化 PMS 效能差异的原因.

2.2.2 二级出水各组分 UV_{254} 和 E_2/E_3 变化 图 5(a)中不同组分 DOM 的 UV_{254} 随反应的进行逐渐降低,说明其中所含物质的芳香结构逐步被破坏,致使其含量下降.E_2/E_3 值逐渐升高(图 5(b)),意味着反应过程中各组分的分子量逐步减小,说明基于 PMS 有效分解产生的多种活性氧化物质促使 DOM 发生转化和降解.

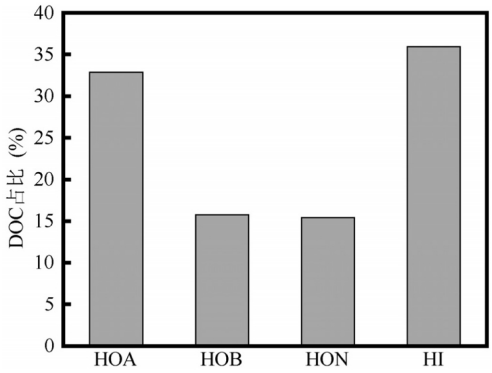


图 4 二级出水各分级组分中溶解性有机碳分布情况

Fig.4 Distribution of dissolved organic carbon in fractionated components of secondary effluent

表 1 二级出水各分级组分理化特征

Table 1 Physicochemical characteristics of fractionated components in secondary effluent

组分	DOC (mg/L)	UV_{254} (cm^{-1})	$SUVA_{254}$ (L·mg/m)	E_2/E_3
HOA	17.35	0.376	2.17	10.8
HOB	8.32	0.089	1.07	11.8
HON	8.14	0.033	0.405	15.4
HI	18.98	0.423	2.23	12.5

2.2.3 二级出水各组分分子量变化 采用凝胶色谱法对 DOM 分子量分布进行表征,从而揭示自催化过程中有机物的转化特性.HOA 和 HI 的分子量主要集中于 1.5~20kDa(图 6(a)、(d)),说明两组分主要由大分子有机物组成;而 HOB 和 HON 的分子量集中于 10Da~1.5kDa,说明其主要由较小分子量的有机

物构成(图 6(b)、(c)).反应后相同位置的峰值明显降低,表明部分有机物发生转化与降解.通常芳香族化合物难以转化降解,HON 的芳香族化合物含量低,腐殖化程度低,缩合芳香结构比例低,所以 HON 遭受 PMS 分解产生的活性氧化物质进攻时,其所含化合物相对易于发生转化和降解.此外,腐殖化程度低,缩合芳香结构比例低的 DOM,在光照下不稳定,易被光解,所以 HON 除受到体系中产生的活性氧化物质攻击外,亦可能发生光解,转化为分子量更小的有机物,使 10Da~1.5kDa 分子量范围内的 HON 由较大分子物质向小分子物质转化,导致较小分子量物质对应的峰强度显著升高[32-33].

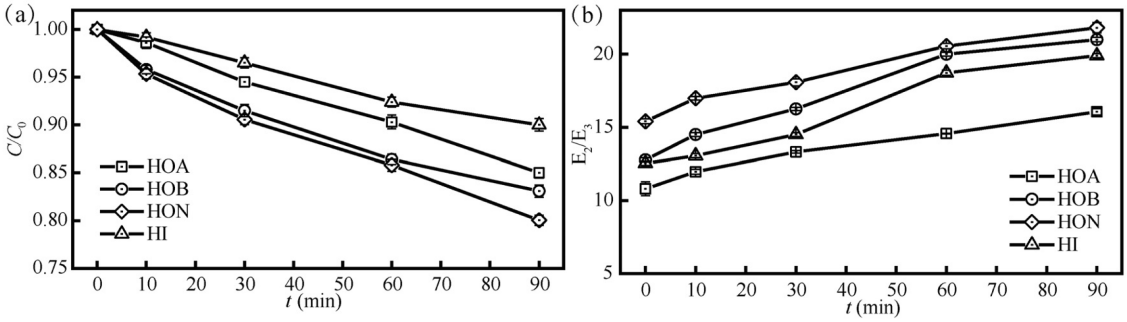


图 5　自活化 PMS 反应中二级出水各分级组分(a)UV_{254} 变化(b)E_2/E_3 变化

Fig.5　Changes in (a) UV_{254} and (b) E_2/E_3 ratio of fractionated components in secondary effluent during PMS self-activation

[PMS]=1mmol/L,pH=7,温度为 20℃

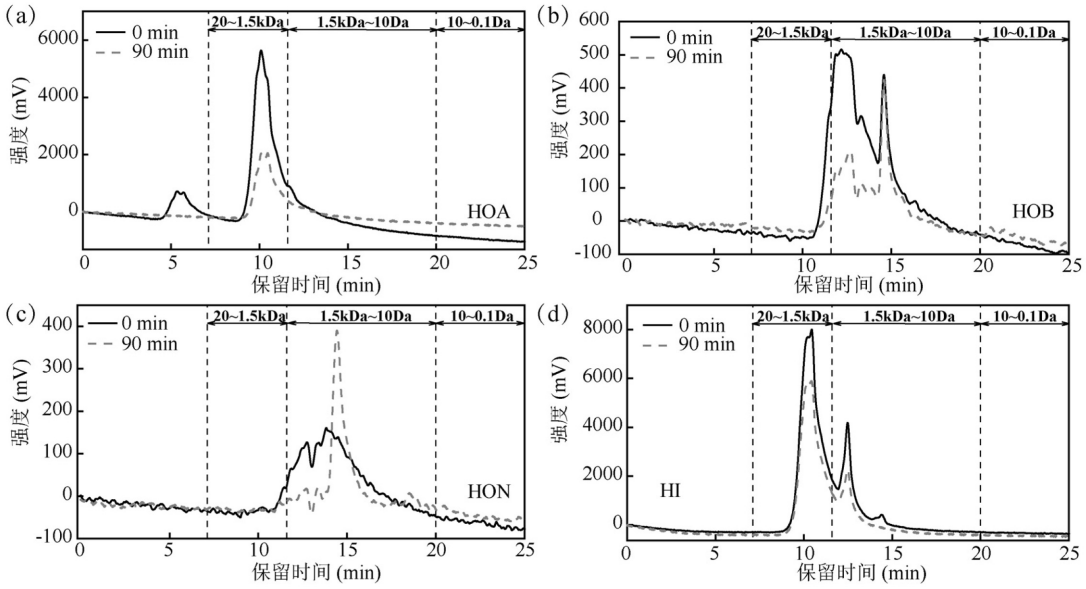


图 6　自活化反应前后不同 DOM 分子量变化

Fig.6　Molecular weight changes of DOM before and after self-activation

[PMS]=1mmol/L,pH=7,温度为 20℃,检测波长为 254nm

2.2.4　二出级水各组分三维荧光光谱变化　三维荧光光谱可表征 DOM 的化学特性,其中荧光峰的位置、强度与所含有机物的种类、含量密切相关[34].通常,谱图分为 5 个代表性区域,I 区(E_x:220~250nm,E_m:280~330nm)和 II 区(E_x:220~250nm, E_m:330~380nm)代表不同数量芳香环的芳香族蛋白质,III 区(E_x:220~250nm,E_m:380~500nm)为类富里酸区,IV 区(E_x:250~280nm,E_m:280~380nm)为溶解性微生物代谢产物,V 区(E_x:250~400nm, E_m:380~500nm)代表类腐殖酸[9,35].图 7 显示 4 个组分均以类腐殖酸物质为主,除此之外,HOB 中含有类富里酸,HON 含有芳香族蛋白质和溶解性微生物代谢产物.反应 90min 后,

各组分的荧光峰强度均显著降低,说明构建的 DOM 活化 PMS 反应体系有良好的自净化效果.

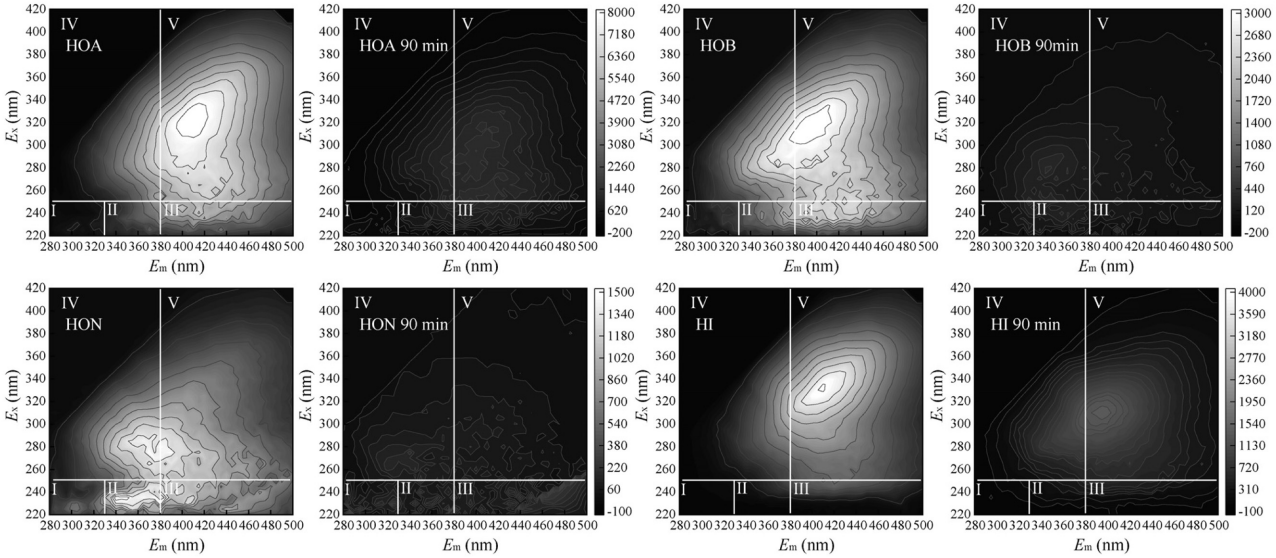


图 7 二级出水各分级组分反应前后三维荧光光谱变化

Fig.7 Evolution of EEM spectra of fractionated components in secondary effluent before and after reaction

[PMS]=1mmol/L,pH=7,温度为 20℃

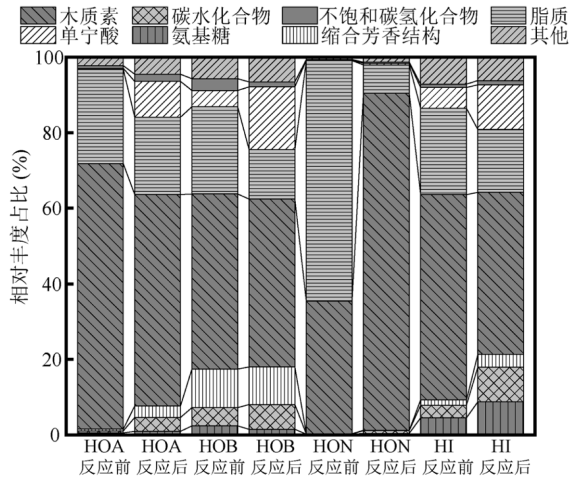


图 8 二级出水不同分级组分反应前后各类化合物占比

Fig.8 Percentage of various compounds in secondary effluent before and after reaction of different graded components

[PMS]=1mmol/L,pH=7,温度为 20℃

2.2.5 二级出水各组分有机物组成变化 如图 8 所示,各组分含量最高的化合物普遍为木质素.相关研究表明,木质素作为一种难降解的复杂芳香性有机聚合物,其中含有大量酚羟基,具有良好的供电子能力,受到适当波长的光激发后,能将光生电子传递给周围的受体,这是各组分能够活化 PMS 的主要原因[36-39].自活化反应后,HOA、HOB 和 HI 中木质素类化合物的占比分别降低了 14%、2%和 11%,说明木质素作为电子供体的同时能够被氧化降解.此外,由于脂质中的不饱和碳碳键与氧化性物质具有较高的反应活性[40],从而导致 4 种组分中脂质类化合物的占比降低 4%~56%.HOA、HOB 和 HI 中单宁酸类化合物的占比分别升高 9%、12%和 6%,HON 中的单宁酸类化合物占比升高不明显,说明 HOA、HOB 和 HI 中有机物转化生成酸类物质.反应前后各组分物质组成的显著变化,进一步表明太阳光照条件下二级出水 DOM 自活化 PMS 体系具有良好的污染物转化与降解效果.

2.3 DOM 自活化过一硫酸盐体系的反应机理

上述研究结果表明,二级出水 DOM 中含有大量类腐殖酸和木质素化合物.通常,这两类物质具有较强的供电子能力,其中所含的酚类基团起到主要供电子作用[41-42].基于团队先前围绕光敏有机染料自活化过硫酸盐的研究基础,推测该自活化体系的反应机理如下[9]:二级出水 DOM 中含有大量类腐殖酸和木质素可充当电子供体,受太阳光中相应波长辐射激发后,有机物的电子由最高占据分子轨道(HOMO)跃迁至其最低未占有分子轨道(LUMO),随后可转移至 PMS,使其经电子介导活化后分解产生多种活性氧化物质,作用于体系中共存的各类 DOM,使之实现不同程度的降解[11,36,43].

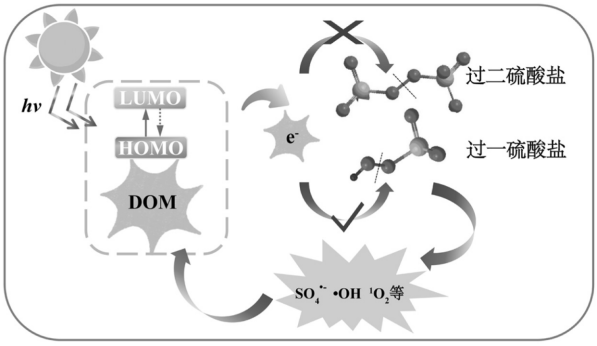


图 9　太阳光照条件下二级出水 DOM 自活化 PMS 体系反应机理

Fig.9　Reaction mechanism of PMS self-activation mediated by DOM in secondary effluent under solar irradiation

3　结论

3.1　HOA、HOB、HON、HI 4 种分级组分可有效活化 PMS,其中 HI 的活化效果最佳,PMS 分解率可达 26.7%;不同组分的 PMS 活化效果差异与其物质结构紧密相关,腐殖化程度越高的组分具有更显著的活化效果.

3.2　二级出水 DOM 受太阳光激发后,可将光生电子转移至 PMS,促使其分解产生多种活性氧化物质,无差别攻击废水中共存的有机污染物,使 DOM 的化合物类型、分子量、荧光特性等产生显著变化,实现二级出水自降解.

3.3　构建的二级出水 DOM 自活化过硫酸盐体系,无需添加催化剂或输入能量,利用废水中原生污染物实现自净化,节约成本、避免二次污染.

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