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高铁酸钾对水中藻类及其次生嗅味污染物二甲基三硫 醚同步去除研究

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摘要:模拟高藻期碱性水源水,采用高铁酸钾对水中以颤藻和二甲基三硫醚为代表的藻类及微量嗅味污染物进行同步控制研究.在高铁酸钾与聚合氯化铁(PFC)单独混凝对藻类的控制效果对比的基础上,展开了高铁酸钾与高锰酸钾预氧化-PFC 联用方法对藻类及嗅味污染物的控制效果对比,探讨了 pH、预氧化时间和浊度等条件对控制效果的影响.结果表明,PFC 单独混凝除藻率最高为 90.6%,以 Fe 计的等量投加条件下,高铁酸钾控藻效果较 PFC 混凝好,除藻率可达 92.4%.高锰酸钾对 PFC 具有强化混凝效果,可明显提高除藻率(94.5%).高铁酸钾较高锰酸钾预氧化对二甲基三硫醚的去除效果理想,且氧化时间大大缩短,高铁酸钾氧化时间 1 min 可去除 92.5%二甲基三硫醚,高于高锰酸钾预氧化 10 min 后达到的去除率(74.6%).

关键词:高藻水; 次生嗅味污染; 二甲基三硫醚; 高铁酸钾; 混凝; 2-甲基异莰醇; 土臭素

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Simultaneous Removal of Algae and Its Odorous Metabolite Dimethyl Trisulfide in Water by Potassium Ferrate

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Abstract: Co-removal of oscillatoria algae and its potential odorous metabolite dimethyl trisulfide (DMTS) in simulated algae-laden alkaline source water by potassium ferrate (K_2FeO_4) was investigated in contrast to potassium permanganate (KMnO $_4$) pre-oxidation followed by polyferric chloride (PFC) under varying conditions, including pH, initial oxidant dosage and turbidity. Based on the precomparison with PFC, the optimal dosage of PFC in the combined KMnO $_4$ pre-oxidation-PFC treatment was determined. Potassium ferrate resulted in 92.4% removal of algae, higher than PFC when the dosage was equivalent as measured by Fe and KMnO $_4$ showed obviously positive effect as a coagulation aid. Degradation of dimethyl trisufide (92.5%) by potassium ferrate was better than the preoxidation of potassium permanganate (74.6%), and the treatment time was decreased from 10 min to 1 min.

Key words; algae; taste and odor; dimethyl trisulfide; potassium ferrate; coagulation; 2-MIB; geosmin

我国城市周边水源污染及其衍生的供水安全问 题已成为亟待解决的难题. 水源水中周期性、突发 性的藻类水华及以藻毒素、藻嗅和藻类胞外分泌物 为代表的次生物会严重干扰饮用水的净水过程和产 水质量,其中嗅味次生物极易穿透水厂常规水处理 工艺进入供水管网,由于其嗅阈值多为ng·L-1,在水 中即使含量很低也可被人们直接感知. 由嗅味次生 物激发的公众反馈最迅速,引发的质疑和恐慌最普 遍,因此针对饮用水中频繁发生的藻致嗅味污染的 控制逐渐成为国内、外学者关注和研究的重 点[1~4]. 藻致嗅味的检测和控制研究多集中于 2-甲 基异莰醇(2-MIB)、土臭素(geosmin)等,近几年由 于国内水污染事件中检出大量 β-环柠檬醛、硫醚 等,且推测此类嗅味物质可能与藻类暴发有关,因此 相关研究逐渐兴起[5,6]. 水中藻致嗅味的控制方法 多以氧化控制为主,常见氧化剂氯、二氧化氯、臭 氧、高锰酸钾和高级氧化对藻致嗅味均有一定程度的去除,通常臭氧和高级氧化治嗅效果较好^[7~11].由于氧化工艺往往导致藻细胞破碎,从而引发次生嗅味污染物的二次释放^[12,13],所以进行原水除嗅处理前需要先进行除藻处理,大多数学者进行相关研究时,往往将原水中藻类及与之相关的嗅味污染物作为相对独立的目标进行有序控制和效果探讨.

本研究以藻及藻致嗅味污染为控制目标,拟利用高铁酸钾 (K_2FeO_4) 的氧化性及其产物的混凝性实现水中藻及其次生嗅味污染的同步去除. Park 等 $^{[14]}$ 探讨了Fe(VI)对 2-MIB 和 geosmin 的氧化控

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作者简介: 马晓雁(1978~), 女, 博士, 副教授, 主要研究方向为饮用 水中藻致嗅味污染控制, E-mail: mayaner620@163. com 制效果,由于污染有机物结构的稳定性及氧化剂的选择性影响,去除率较低,在模拟水环境 pH为6~8时均不超过25%,研究未提及Fe(VI)对藻类的去除.本研究选择了分子结构不同的典型嗅味污染物二甲基三硫醚(DMTS)^[15]及蓝藻中常见的颤藻为控制对象,试验条件中pH为碱性,更接近实际高藻水源水环境条件.此外,通过与单纯聚合氯化铁(PFC)混凝和高锰酸钾(KMnO₄)预氧化强化混凝比较,对 K_2 FeO₄ 控藻和治臭效果进行了评价.

1 材料与方法

1.1 试剂与仪器

DMTS (纯度 \geq 95%, 色谱纯, Tokoy Chemic Company, 日本),正己烷(\geq 99%, 色谱纯, CNW, 德国), 氯化钠(\geq 99.5%, 分析纯, 南京化学试剂有限公司), PFC(B=1.0, 自制), KMnO₄(\geq 99.5%, 分析纯, 南京化学试剂有限公司), K₂FeO₄(纯度 \geq 50%, 自制),纯水(18 M Ω ·cm).

气相色谱仪(GC-2014, 岛津,日本),光学显微镜(XSP-2C,光学仪器厂,上海),便携式浊度仪(2100P,哈希,美国),便携式 pH 测定仪(SensION,哈希,美国).

1.2 模拟高藻水源水

试验所用的颤藻购自中国科学院武汉水生生物研究所,采用 BG11 培养基培养. 试验时将颤藻水样用纯水稀释,使水样中的颤藻细胞浓度含量在 10^5 ~ 10^6 个·L⁻¹范围之内,DMTS 为标准品,试验中调节其浓度为 555. 3 ~ 600. 0 μ g·L⁻¹. 采用碳酸氢钠-碳酸钠缓冲液调节试验用水至所需 pH 值,以高岭土配置原水浊度为 62 ~ 65 NTU,各项水质指标见表 1.

表 1 模拟高藻水水质指标

Table 1 Water quality index of artificial water with

algae of high concentration							
颤藻	DMTS	UV_{254}	浊度/NTU	На			
/个·L ⁻¹	/μg•L ⁻¹	$/cm^{-1}$	供及/NIU	рп			
6.4×10^5	555. 3 ~ 600. 0	0. 035	62 ~65	10.8			

1.3 试验方法

- (1)调节模拟高藻水呈碱性,投加一定量的PFC(以 Fe 计),250 r·min⁻¹快搅 1 min 后,50 r·min⁻¹慢搅 10 min,沉淀 30 min,取上清液测定藻细胞浓度和嗅味污染物的含量.
- (2) 调节模拟高藻水呈碱性,投加一定量的 KMnO₄,以 100 r·min⁻¹接触氧化 10 min 后,投加一定量的 PFC 进行混凝试验. 取上清液测定藻细胞浓

度和嗅味污染物的含量.

(3) 调节模拟高藻水呈碱性,投加一定量的 K_2 FeO₄(以 Fe 计),以 100 r·min⁻¹接触氧化 1 min 后,250 r·min⁻¹快搅 1 min,50 r·min⁻¹慢搅 10 min, 沉淀 30 min,取上清液测定藻细胞浓度和嗅味污染物的含量.

1.4 分析方法

1.4.1 微量 DMTS 检测方法

取 100 mL 待测水样,置于 100 mL 容量瓶中,操作过程中尽量使水样沿瓶壁流下,以避免搅动造成的曝气影响. 根据试验需要加入 5% 经烘干后的NaCl 固体,然后加入 1 mL 正己烷试剂,磁力搅拌器以1 200 r·min ⁻¹搅拌 10 min 后,静置一定时间,取上层正己烷溶液经 GC 进样分析,测定目标嗅味污染物含量. 色谱条件:进样口温度 250℃,无分流进样;程序升温初始温度 50℃,以 15℃·min ⁻¹的速率升温至 150℃,再以 30℃·min ⁻¹速率升温至 240℃; FID 温度为 250℃;载气流速为 1.5 mL·min ⁻¹;进样体积为 1 μ L; DMTS 的保留时间为 3.419 min.

1.4.2 藻类计数方法

水样用鲁哥氏液进行固定后,采用浮游植物计数框在光学显微镜下计数,2次计数取平均值,平均偏差控制在15%以内.

2 结果与讨论

2.1 K₂FeO₄ 控制高藻水中藻类和藻嗅

由图 1 可见, K_2 FeO₄ 对藻类及藻嗅均有良好的去除效果,其中投加量对藻嗅的去除率影响较大,当投加量为 0.05 mmol·L⁻¹时,DMTS 的去除率仅为 44.5%,增加到 0.20 mmol·L⁻¹时达到 92.5%,之后投加量的增加对去除率的影响逐渐减弱,最高为 93.3%. K_2 FeO₄ 有极强的氧化性,在酸性和碱性条件下其氧化势分别为 2.2V 和 0.7V^[16],图 1 表明 K_2 FeO₄ 可将 DMTS 氧化去除,氧化去除效果较好. 当 K_2 FeO₄ 投加量大于 0.2 mmol·L⁻¹后去除效果趋于平缓,可能是相对于氧化反应而言, K_2 FeO₄ 投加处于足量或过量范围,0.2 mmol·L⁻¹为氧化控制的特征投加点. K_2 FeO₄ 的 Ames 测试(沙门氏菌回复突变试验)显示 K_2 FeO₄ 并不产生任何诱导有机体突变的副产物,是一种"绿色友好"的氧化剂^[16,17].

 K_2 FeO₄ 对藻类去除效果稳定,投加量为 0.05 mmol·L⁻¹即可获得 87.5%的藻类去除效率,随着投加量的增加,去除效果略微增加,但增幅不大,最高

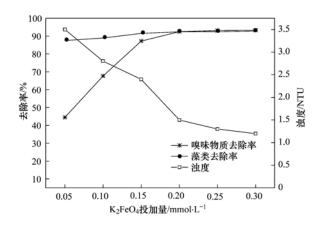


图 1 K_2 FeO₄ 投加量对藻及藻嗅去除效果的影响

Fig. 1 Removal of algae and its odor metabolites $\label{eq:K2FeO4} \mbox{with different dosage of } \mbox{K_2 FeO_4$}$

去除率为 92. 8%. K_2 FeO₄ 的混凝作用主要依靠其还原产物 Fe³⁺形成的具有絮凝作用的 Fe(OH)₃,吸附到藻细胞表面改变藻细胞表面属性,增加其沉降性能,使得藻类混凝去除^[18,19]. 此外,絮体可去除无机颗粒造成的浊度,降低出水浊度. 随投加量增加剩余浊度逐渐减小, K_2 FeO₄ 投加量 0. 30 mmol·L⁻¹时剩余浊度为 1. 5 NTU.

2.2 PFC 控制高藻水中藻类和藻嗅

PFC 是一种具有多羟基氧化桥、高正电荷的聚合无机高分子絮凝剂^[18],具有很好的电中和能力,常用于水中藻类的混凝去除。图 2 探讨了 PFC 不同投加量情况下,藻及藻嗅的去除情况。由于 PFC 与 K_2 FeO₄ 还原产物同为铁源性混凝剂,可进行与 PFC 等量投加试验,通过检验藻类去除效果,评价 K_2 FeO₄ 还原产物的混凝性。

由图 2 可见, PFC 混凝对藻类去除效果良好,投加量为 0.125 mmol·L⁻¹时,藻类去除率可达 85.3%,随着 PFC 投加量的增多,藻类去除率略有上升,最高去除率可达 90.6%,经过一段相对稳定的短暂区,随着投加量继续增多,去除率呈下降趋势.该现象主要由藻类表面电荷变化引起,随着 PFC 的投加,藻类表面带的负电荷会与 PFC 水解产生的 Fe(Ⅲ)电中和,使得藻体系脱稳被去除,但当投加量继续升高时,过量的 PFC 导致藻类等表面附着反电荷并重新稳定,使得混凝效果变差[20,21],浊度的类似变化趋势也可表明混凝剂投加量拐点.单独 PFC 混凝对藻嗅的去除甚微,由于 DMTS 是一种小分子的硫醚类物质,不易被吸附及混凝去除"15.22],单独的投加 PFC 对 DMTS 基本没有去除效果,图 2 中表征的 27.2%的去除率主要是由于混凝

过程中的搅拌等导致的嗅味损失. 从中可知, PFC 投加量为 $0.25 \, \text{mmol} \cdot \text{L}^{-1}$ 时, 藻类去除率最高 90.6%, 剩余浊度 $0.76 \, \text{NTU}$.

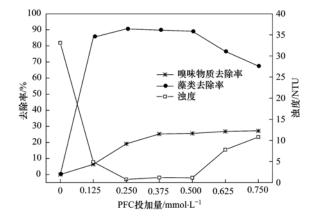


图 2 不同投加量 PFC 对藻及藻嗅去除效果

Fig. 2 Removal of algae and its odor metabolites with ${\rm different\ dosage\ of\ PFC}$

2.3 KMnO₄-PFC 联用控制高藻水中藻类和藻嗅

图 3 为 PFC 投加量 $0.25 \text{ mmol} \cdot \text{L}^{-1}$, $KMnO_4$ 投加量 $0.0125 \sim 0.0750 \text{ mmol} \cdot \text{L}^{-1}$ 时, 嗅味和藻类等指标去除率情况.

由图 3 可见, $KMnO_4$ -PFC 联用方法对 DMTS 有较好的氧化去除效果, 去除率随投加量的增加而上升, 最高达 90.9%. $KMnO_4$ 有较强的氧化性, 在酸性和碱性条件下其氧化势分别为 1.7 V 和 0.59 V, 可以氧化硫醇类物质[23,24], 随着 $KMnO_4$ 投加量的增加, 藻嗅的去除效果逐步提高, 未出现类似 K_2FeO_4 的氧化饱和点, 表明试验中 $KMnO_4$ 投加量仍属于不足的情况.

KMnO₄-PFC 联用方法对藻类去除效果稳定,去除率始终保持在90%以上,最高可达94.8%.与单

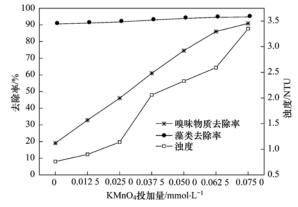


图 3 KMnO₄ 投加量对藻及藻嗅去除效果的影响

Fig. 3 Removal of algae and its odor metabolites with different dosage of ${\rm KMnO_4}$

纯 PFC 相比,去除率增加了 4.2 个百分点. 有研究 认为投加 $KMnO_4$ 预氧化时 $^{[25]}$,藻体系的 Zeta 电位 并不发生改变,但 $KMnO_4$ 可能导致藻细胞释放胞外分泌物(EOM),EOM 协同 $KMnO_4$ 的氧化产物 MnO_2 形成络合物覆盖在藻细胞表面改变藻细胞表面属性 使后续的处理效果加强.

联用工艺中,随着 $KMnO_4$ 投加量的不断增大,水中剩余浊度也不断上升,分析原因可能是由于过量的 $KMnO_4$ 还原后的 MnO_2 等残存导致.考虑到剩余浊度和过量 $KMnO_4$ 引起水中色度的上升,试验中 $KMnO_4$ 投加量不再增加.

2.4 K_2 FeO₄ 及 $KMnO_4$ -PFC 联用工艺除藻及藻嗅效果对比

由图 $1 \sim 3$ 可知,藻类去除主要依靠混凝作用,单纯投加 0.25 mmol·L⁻¹ PFC 去除率为 90.6%, KMnO₄-PFC 去除率为 94.8%, K₂FeO₄ 除藻率为 92.4%, K₂FeO₄ 比单纯 PFC 混凝的效果好,可见 K₂FeO₄ 和藻类接触氧化的过程中会破坏藻细胞结构,同时一些细胞内和胞外分泌物被释放到水体中充当了助凝剂的作用,促进了混凝除藻效果. 3 种方法除藻效果的优劣顺序为 $KMnO_4$ -PFC > K₂FeO₄ > PFC,剩余浊度高低顺序为: $KMnO_4$ -PFC > K₂FeO₄ > PFC.

PFC 对 DMTS 基本没有去除效果, K_2 FeO₄ 的除嗅率92.5%,远高于 KMnO₄-PFC 的 74.6%. KMnO₄ 投加量为 0.05 mmol·L⁻¹, K_2 FeO₄ 投加量为 0.25 mmol·L⁻¹,为 KMnO₄ 的 5 倍,此外Fe(VI) 的氧化还原电位高于 Mn(VII),因此 K_2 FeO₄ 对 DMTS 的氧化效果要优于 KMnO₄. 参见图 1 和图 3,对氧化剂同等量浓度条件下的除藻和除嗅效果进行对比,可见,KMnO₄ 和 K_2 FeO₄ 投加量均为 0.05 mmol·L⁻¹时,DMTS 的去除率分别为 74.6% 和 44.5%,KMnO₄ 控制藻嗅效果好于 K_2 FeO₄. 天然高藻水由于藻类的大量生长,水体呈现碱性,pH 值上升至 10 以上,KMnO₄ 和 K_2 FeO₄ 的氧化能力随 pH 值上升减弱,而 K_2 FeO₄ 在酸性条件下易分解在碱性条件下稳定的特性使得其氧化应用性反而有所提升^[23,26],这有利于其在高藻水中的应用.

2.5 K_2 FeO₄ 与 KMnO₄-PFC 工艺除藻除嗅影响因素对比试验

由 2. 1 ~ 2. 3 节确定 K_2FeO_4 与 $KMnO_4$ -PFC 联用 2 种方法除藻及藻嗅的对比工艺条件, K_2FeO_4 投加量为 0. 25 $mmol \cdot L^{-1}$, $KMnO_4$ -PFC 工艺中 PFC 投加量(以 Fe 计)为 0. 25 $mmol \cdot L^{-1}$, $KMnO_4$ 投加量 0. 05 $mmol \cdot L^{-1}$.

2.5.1 pH 值的影响

由图 4 可见,2 种方法对藻类都有良好的去除效果,且受 pH 影响甚微. KMnO₄ 预氧化在不同 pH 值条件下对藻嗅去除效果稳定,维持在 70% 以上,最高达 75. 2%. K_2 FeO₄ 对藻嗅去除受 pH 值影响较大,pH 为 5. 24 时 DMTS 的去除率仅为 35. 4%,pH 升高至 9. 13 时藻嗅的去除率为 78. 5%,当 pH 偏碱性时,高于同条件下 KMnO₄ 预氧化的去除效果,这与碱性条件下 K_2 FeO₄ 分解缓慢且氧化还原电位高相符合^[16]. 同等原始浊度条件下, K_2 FeO₄ 处理后高藻水的浊度小于 KMnO₄,原因可能是由于 K_2 FeO₄ 还原产物转化为絮体参与混凝反应,较 PFC 的混凝效果好,此外,KMnO₄ 还原产物 MnO₂ 部分残存导致浊度升高.

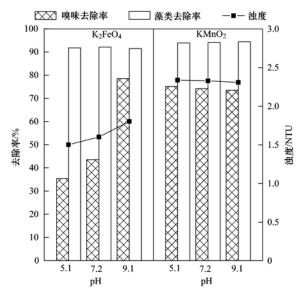


图 4 pH 对 2 种工艺除藻除嗅的影响

Fig. 4 Influence of pH value on the removal of algae and its odor metabolites by the two methods

2.5.2 预氧化时间的影响

由图 5 可见, K_2 FeO₄ 预氧化 1 min 除藻除嗅效果分别为 92.4%和 92.5%,且出水浊度低,而 $KMnO_4$ 预氧化 20 min 除藻除嗅效果仅为 94.5%和 79.5%. 之前的研究表明 K_2 FeO₄ 氧化藻嗅主要依靠反应的前 30 s,此阶段藻嗅被迅速降解,而接触反应 60~600 s 时,反应速率降低,藻嗅去除效果不明显,整体反应符合三级反应动力学[27]. K_2 FeO₄ 在时效性上高于 $KMnO_4$ 预氧化法,达到同等除藻除嗅效果 K_2 FeO₄ 所需预氧化时间大大少于 $KMnO_4$ 预氧化 所需时间.

2.5.3 浊度的影响

由图 6 可见, 浊度对 2 种方法的去除效果影响

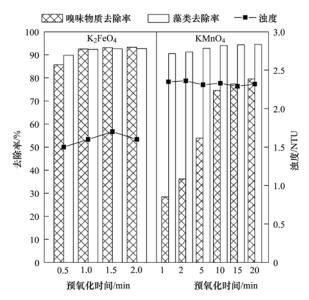


图 5 预氧化时间对 2 种工艺除嗅除藻的影响

Fig. 5 Influence of pre-oxidation time on the removal of algae and its odor metabolites by the two methods

微弱,高岭土表面呈现电负性类似于藻细胞^[18],对于藻类去除影响不大。在同等浊度条件下, K_2FeO_4 对藻类和藻嗅的共去除效果显著高于 $KMnO_4$ 预氧化,同时剩余浊度低于同水平的 $KMnO_4$ 预氧化法.对比 $KMnO_4$ 预氧化法, K_2FeO_4 具有氧化速度快、负面影响小、剩余浊度低等优点。

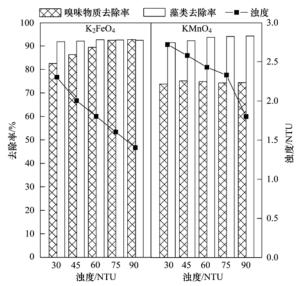


图 6 浊度对 2 种工艺除嗅除藻的影响

Fig. 6 Influence of turbidity of raw water on the removal of algae and its odor metabolites by the two methods

3 结论

(1) K₂FeO₄ 的强氧化性及其水解后生成的 Fe(OH)₃胶体颗粒对于高藻水中的藻类及嗅味物质 二甲基三硫醚有良好的去除效果,对浊度也有一定 的控制效果.

- (2)与直接投加 PFC 混凝相比,在相同投加量的情况下, K_2 FeO₄ 除藻效果明显优于 PFC.与 KMnO₄-PFC 联用法相比, K_2 FeO₄ 除藻嗅效果明显,除藻效果相差不大,但对剩余浊度的控制效果更好.
- (3)为同时获得藻及与其相关嗅味污染物的高效去除,K₂FeO₄的投加量较大,尽管大部分铁离子将形成絮体参与沉降过程,但不可避免地仍有部分铁离子游离于水中,因此高铁酸钾除藻控嗅的具体应用及其用于饮用水处理的安全性仍有待进一步探索.

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