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### 不同环境因素下太湖中四环素的自然消减

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摘要:四环素已经广泛应用于兽药生产和疾病治疗中,并通过禽畜粪便等途径进入环境中,基于四环素残留物的危害性,采用了模拟自然环境状态的实验方法,研究了四环素在不同环境状态(光照、底泥、重金属)下的自然消减过程.结果表明,四环素在自然光照下的消减不明显;未杀菌底泥水体中,四环素的消减速率大于杀菌底泥水体;实验初期,含硝酸铅水体中四环素消减缓慢,随着时间延长,消减速率加快,而在硝酸镉水体中四环素短时间内消减趋势已经十分明显,且消减速率大于硝酸铅水体;以0.08 mmol·L<sup>-1</sup>四环素为例,四环素在光照下各环境因素中的消减速率依次为:未杀菌底泥(87.2%)>杀菌底泥(70.37%)>硝酸镉水体(64.2%)>硝酸铅水体(32.3%)>空白组(6.6%),各环境因素均促进了四环素的消减.避光时,各环境因素中四环素的消减趋势与光照组相同,但消减速率较光照组小,表明光照对四环素的自然消减具有一定的促进作用.

关键词:四环素;消减;光照;底泥;硝酸铅;硝酸镉

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# Natural Attenuation of Tetracycline in the Water of Taihu Lake Under Different Environmental Conditions

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**Abstract**: Tetracycline (TC) has been widely used in veterinary medicines and disease treatments, and has been discharged into nature system due to manure application. To know the harmfulness of TC residues, and to investigate the natural attenuation of TC under different environmental conditions, simulated attenuation experiments in the presence of light, sediment,  $Cd(NO_3)_2$  and  $Pb(NO_3)_2$  were performed. Results showed that the natural attenuation of TC was not obvious under the sunlight, and the natural attenuation rate of TC in water with unsterilized sediment was greater than that with sterilized sediment. The natural attenuation of TC in water with  $Pb(NO_3)_2$  was slow at the first stage of the experiment, and progressively speeded up as time went on. However, the natural attenuation of TC in water with  $Cd(NO_3)_2$  was more rapid at the beginning, and significantly faster than that of  $Pb(NO_3)_2$ . The natural attenuation rates of  $O(NO_3)_2$  (64.  $O(NO_3)_2$  (64

**Key words:** tetracycline; attenuation; sunlight; sediment;  $Pb(NO_3)_2$ ;  $Cd(NO_3)_2$ 

抗生素作为广谱类药物,被广泛应用于治疗人体疾病,治疗和预防畜禽、水产品的细菌性病害,在世界范围的使用量呈逐年递增趋势<sup>[1]</sup>.在美国,作为促生长作用的抗生素使用量在近 40 年內增加了 80 倍<sup>[2]</sup>.中国每年兽用抗生素的平均使用量已高达 6 000t<sup>[3,4]</sup>.随着我国畜禽养殖业的繁荣和发展,四环素类、大环内酯类、青霉素类、磺胺嘧啶类抗生素的使用日益广泛,但是抗生素的不合理应用及不遵守正确的体药期导致大部分抗生素不能完全被

机体吸收,抗生素及其衍生物作为微量污染物所造成的污染及由此产生的环境效应引起人们的关注<sup>[5~7]</sup>.

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四环素(tetracycline, TC)作为一种典型人畜共用抗生素,应用较为广泛,TC是一种稠环芳烃化合物,属于难降解物质,在环境中残留危害较大,并可致突变、致癌<sup>[8]</sup>.TC类抗生素及其中间代谢产物具有生物毒性,可以透过细菌的细胞壁,与细菌的遗传物质核糖核蛋白结合,抑制微生物的蛋白质合成,对生化处理过程的微生物都有明显的抑制作用<sup>[9]</sup>.同时其降解产物的溶血作用,对肝脏、胃肠有损害及可使牙齿染色等,长期低浓度的药物残留还会诱发病菌的耐药性<sup>[10]</sup>.因此,研究环境中残留 TC 的消减过程就显得尤为重要<sup>[11]</sup>.而目前,国内外用于 TC类药物降解技术主要包括高锰酸钾氧化法<sup>[12]</sup>、TiO<sub>2</sub>光催化降解法<sup>[13]</sup>、臭氧氧化法<sup>[14]</sup>和电化学法<sup>[15]</sup>等,但是 TC 在不同环境条件下的自然消减过程鲜有报道.

与抗生素相比,太湖的重金属污染问题吸引了更多学者的关注. 2012 年 Lu 等  $^{[16]}$  在太湖水体检出 Pb 的浓度为  $0.95 \sim 4.30~\mu g \cdot L^{-1}$ ; 陈璐璐等  $^{[17]}$  研究指出,太湖中 Cd 的浓度为  $0.76 \sim 1.12~\mu g \cdot L^{-1}$ ,平均值为  $0.85~\mu g \cdot L^{-1}$ ; 2012 年中国环境科学研究院秦延文等  $^{[18]}$  发现,太湖表层沉积物中 Cd 含量为  $0.20 \sim 2.88~m g \cdot k g^{-1}$ ,平均  $0.45~m g \cdot k g^{-1}$ ,超过背景

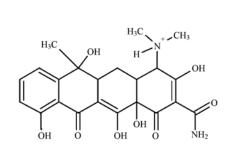
值3.46 倍. Cd 富集程度最为严重,具有较强的生态危害,其次是Pb,全湖Pb 的含量在29.20~74.48 mg·kg<sup>-1</sup>之间,平均值为38.27 mg·kg<sup>-1</sup>;2012年河海大学王沛芳等<sup>[19]</sup>报道,太湖表层沉积物中Cd污染较重且分布较广,含量为1.62~3.29 mg·kg<sup>-1</sup>,可提取态所占比例最高、迁移性最强,其次是Pb,Cd在太湖各湖区沉积物中存在中等潜在生态风险.

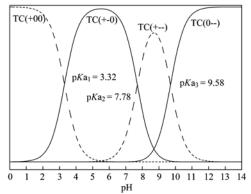
太湖流域人口密集、社会经济高速发展,是典型的水网地区,非常易于抗生素、重金属等污染物在水体中的搬运和迁移. 残留抗生素和重金属经污水排放、地表径流等途径进入太湖后,太湖水体中的 Pb、Cd 等重金属能否影响 TC 的自然消解,尚不明确. 本实验以 TC 为研究对象,主要分析在光照及避光条件下, TC 在太湖底泥、重金属影响下的自然消减过程,重点研究了 TC 在不同环境下的自然消减速率,并探讨了 TC 的消减机制,以期为不同环境下太湖水体中 TC 的自然消减提供参考.

#### 1 材料与方法

#### 1.1 实验材料与仪器

TC 为分析纯,购自上海国药集团化学试剂有限公司.TC 分子结构式及形态见图 1.





#### 图 1 四环素分子结构及形态

Fig. 1 Structure and speciation of tetracycline

TC 有酰胺基(CONH<sub>2</sub>)、二甲胺基 N(CH<sub>3</sub>)<sub>2</sub> 和 酚羟基等官能团,此外还有两个含有酮基和烯醇基 的共轭双键系统. 二甲胺基具有碱性( $pK_a = 9.6$ ),而三羰基甲烷系统和酚二酮系统具有酸性( $pK_a$  分别为 3. 3 和 7. 7). 因此四环素是一类两性化合物,它能和各种酸、碱反应形成较为稳定的盐.

底泥,2013 年 2 月采自于太湖. 将底泥在自然 条件下风干,用研钵研细. 一部分在烘箱内 170℃干燥 1 h,并在紫外灭菌灯下照射 15 min,以杀灭底泥 中的微生物;另一部分为未杀菌底泥.采集的太湖底泥中金属元素含量如表1所示.

实验所用仪器为紫外可见分光光度计(日本岛津,UV-2550);恒温干燥箱(上海精宏,DHG-9240A型);分析天平(美国丹佛,TP-214);紫外灭菌灯(ZWS 15W).

#### 1.2 TC 标准曲线

对一定浓度的 TC 溶液进行紫外光谱扫描,得到 TC 的紫外图谱,如图 2(a) 所示.

表 1 底泥中的金属元素含量/mg·kg	g -	·k	g·	mg	量/	ij	含	素	元	属	金	的	中	泥	底	1	表	
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Table 1	Original	content	of metal	in	sediment/	mg•kg-1
1 abic 1	Offigiliai	Content	or metar	111	seument	mg Kg

元素	Fe	Cu	Zn	Al	Pb	$\operatorname{Cr}$	Cd	Mn	Ni	As
含量	4. 8	ND	0.4	8. 4	ND	ND	ND	2. 1	1.8	0. 2

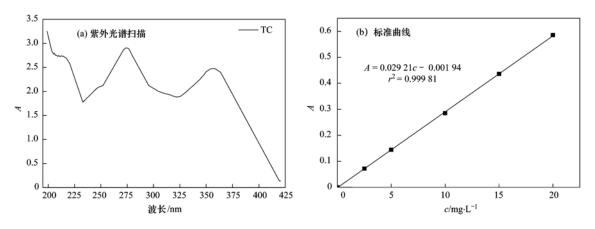


图 2 TC 溶液的紫外光谱扫描及其标准曲线

Fig. 2 UV absorbance of TC and its standard curve

由图 2(a) 可知, TC 的紫外特征吸收峰为 274 nm 和 355 nm, 本实验选取 274 nm 进行后续研究.

取 25 mg·L<sup>-1</sup>TC 标准溶液,配置 2.50、5.00、10.00、15.00、20.00 mg·L<sup>-1</sup>的 TC 溶液,在 274 nm 处测量其吸光度,重复测定 3 次,取其平均值,即可得吸光度 A 与 TC 质量浓度 c 的线性回归方程,拟合曲线如图 2(b) 所示.

由图 2(b) 可知, TC 溶液在  $0 \sim 20 \text{ mg·L}^{-1}$  时, TC 质量浓度与其吸光度之间有较好的线性关系,  $R^2$  为0.99981.

#### 1.3 实验方法

取1 L 烧杯 25 个,每 5 个分为一组,于第二、三、四、五组中分别加入杀菌底泥 100 g、未杀菌底泥 100 g、未杀菌底泥 100 g、稍酸镉 0.03 mmol·L<sup>-1</sup>、硝酸铅 0.03 mmol·L<sup>-1</sup>、硝酸铅 0.03 mmol·L<sup>-1</sup>,第一组为空白对照组.于每组中配置 1 L 浓度分别为 0.02、0.03、0.04、0.08 mmol·L<sup>-1</sup>的 TC 溶液,放置于同一光照条件下,分别称重,并记录原始重量,每天称重并补加蒸馏水,以弥补蒸发散失的水分,定期用紫外分光光度计测定溶液中 TC 的浓度. 另设避光条件下的实验组,除避光外,所有实验条件及方法同光照组.

根据公式(1),计算 TC 的消减率 r:

$$r = \frac{c_0 - c_t}{c_0} \times 100\% \tag{1}$$

式中, $c_0$  为 TC 的初始质量浓度,  $\operatorname{mg} \cdot \operatorname{L}^{-1}$ ;  $c_t$  为 TC 在 t 时刻的质量浓度,  $\operatorname{mg} \cdot \operatorname{L}^{-1[20]}$ .

#### 2 结果与讨论

#### 2.1 未杀菌底泥对 TC 自然消减的影响

图 3 为不同 TC 浓度的自然消减过程中,对照组与含未杀菌底泥水体中 TC 的浓度变化.

由图 3 可知,避光时对照组中剩余的 TC 浓度高于光照组,即表明光照下 TC 的降解速率大于避光组.光照和避光时,未杀菌底泥水体中 TC 的浓度都呈明显的降低趋势,主要是前期底泥的吸附作用所致.后期光照下 TC 呈先上升后下降的趋势,避光下的 TC 浓度趋于平稳.实验结束时,以图 3(a)为例(低 TC 浓度),在光照和避光下对照组 TC 的消减速率分别为 15.9% 和 3.3%,未杀菌底泥水体中 TC 的消减速率分别为 58.44% 和 68.7%;以图 3(d)为例(高 TC 浓度),光照和避光下,未杀菌底泥水体中的TC 的趋势与图 3(a)相似,对照组 TC 的消减速率分别为 6.6% 和 6.8%,未杀菌底泥水体中 TC 的消减速率分别为 87.2% 和 90.3%.

自然状态下,一般认为光化学降解反应机制在于分子吸收光能变成激发态,从而引发各种反应,太阳光对 TC 的消减具有一定促进作用[21]. 有的研究表明,TC 在水中还可通过与 $O_2$ 和  $O_2$  发生反应进行光解[22~24]. Castillo 等[25]研究发现,在可见光照射下,TC 可通过基态电子转移产生阴离子自由基 $(RF^{--})$ ,并且进一步产生超氧阴离子自由基 $O_2$  ,同时激发三重态 $^3RF^*$  可将能量传递给溶解氧产

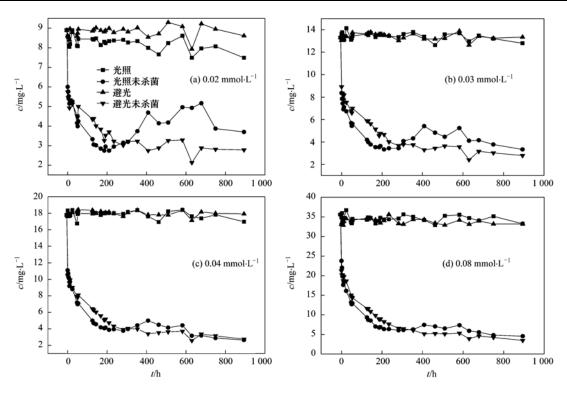


图 3 对照组与含未杀菌底泥水体中 TC 的浓度变化

Fig. 3 Changes of the concentration of tetracycline in the blank group and the unsterilized sediment

生¹O₂,并通过实验证明,强力霉素、甲稀土霉素、去甲金霉素和土霉素都可与¹O₂和 O₂⁻ 发生反应进行光解;而 Addamo 等[²6]研究发现,直接光解对TC 的降解和矿化作用不大. 本实验同样得到了上述结论,TC 的直接光解速率非常缓慢,而且降解时间较长.

除长时间的光解和底泥的吸附外,底泥中的某些物质在受到光照时,会形成大量的自由基、过氧化物和单重态氧,这些物质加速了 TC 的降解[<sup>27]</sup>.同时底泥中的微生物也促进了 TC 的降解,TC 残留物的结构和理化性质在微生物作用下发生改变,从大分子化合物降解为小分子化合物,最终转变为 H<sub>2</sub>O和 CO<sub>2</sub>,因而 TC 在含未杀菌底泥水体中的消减过程较快.而光照下 TC 浓度出现上升后又下降的现象,可能有 3 种原因:一是光照下 TC 的原有降解产物再次转化成母药而造成总量增长<sup>[28]</sup>;二是底泥中TC 的吸附与解吸平衡被打破,解吸速率大于吸附速率,出现上升趋势;三是随着时间的延长,出现了耐药微生物,TC 对底泥呼吸的影响降低,从而加快了TC 的降解<sup>[11]</sup>.

#### 2.2 杀菌底泥对 TC 自然消减的影响

图 4 为不同浓度下的 TC 自然消减过程中,对 照组与含杀菌底泥水体中 TC 的浓度变化. 由图 4 可知,不同浓度时(0.08 mmol·L<sup>-1</sup> 除外),避光下对照组中剩余的 TC 浓度高于光照组.由图 4(a)和 4(b)可知,含杀菌底泥的水体中,TC的浓度出现了先上升后下降的趋势,且避光下的浓度大于光照组,均大于对照组.由图 4(c)和 4(d)可知,含杀菌底泥的水体中 TC 的浓度具有明显的下降趋势,且光照下的下降速率大于避光下,随着时间的延长趋于平衡.在光照和避光下,以图 4(a)为例(低 TC 浓度),杀菌底泥中 TC 并没有消减;以图 4(d)为例(高 TC 浓度),杀菌底泥中 TC 的消减速率分别为 70.37%和 59.04%.

图 4(a) 和 4(b) 中低浓度组的 TC 浓度出现上升趋势,可能是因为杀菌底泥中的重金属由于扩散、解吸、溶解、氧化还原和络合作用,以及在物理、化学等因素的作用下,从底泥向水相释放,造成了水体中重金属含量的升高,而在取样测定吸光度时,部分金属加强了 TC 在该波长下的响应,同时部分金属也会在该波长下具有自身的响应,导致了测定时数据增大;而后 TC 浓度又出现下降趋势,除光照因素外,可能由于底泥中如光合菌、乳酸菌、放线菌、酵母菌、发酵丝状菌、芽孢杆菌、枯草杆菌、硝化细菌、酵母等具有降解抗生素功能的微生物并没有被完全杀灭<sup>[29]</sup>,而是以休眠体的形式存在于底泥中,随着时间的延长,微生物进而苏醒、繁殖,同时也对

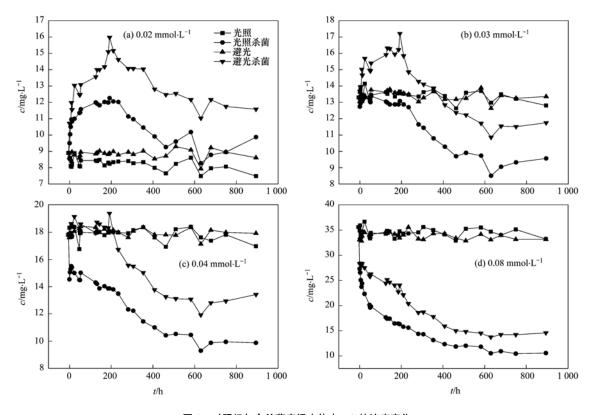


图 4 对照组与含杀菌底泥水体中 TC 的浓度变化

Fig. 4 Changes of the concentration of tetracycline in the blank group and the sterilized sediment

TC 产生了耐药性,加快了 TC 的降解. 而避光下的 TC 浓度大于光照组,也表明光照可以加速 TC 的消减.

图 4(c)和 4(d)表明,光照下含杀菌底泥的水体中,四环素的浓度低于避光组,且光照下的消减速率明显大于避光组.在底泥的吸附作用、自身的降解以及重金属的络合等作用下,四环素的浓度呈降低趋势.

#### 2.3 水中硝酸镉对 TC 消减过程的影响

图 5 为不同 TC 浓度下,对照组与含硝酸镉水体 TC 消减过程中浓度的变化.

由图 5 可知,光照与避光下,对照组中 TC 浓度的变化趋势同以上实验组.不同 TC 浓度时,含硝酸镉水体中,TC 的浓度均表现为下降趋势,且避光下TC 浓度要大于光照组,即避光下 TC 的消减速率要低于光照组,即光照促进了 TC 的消减.在光照和避光下,以图 5(a)为例(低 TC 浓度),含硝酸镉水体中 TC 的消减速率分别为 70.7% 和 52.7%;以图 5(d)为例(高 TC 浓度),含硝酸镉水体中 TC 的消减速率分别为 64.2% 和 48.1%.

TC 是两性化合物,其分子含有 1 个碱性基团、2 个酸性官能团,在酸性、中性和碱性条件下,通常

以正离子、中性和负离子形态存在<sup>[30]</sup>. 本实验水体呈中性,此时 TC 以兼性离子存在,水体中加入镉离子后,为 TC 提供了较多的反应位点. 随着 TC 浓度的增加,自身所提供的反应位点相应增多,可能发生了镉离子与 TC 的配位反应,其与镉离子通过络合作用,形成 2:1 的配合物,导致 TC 聚合后沉淀<sup>[31-33]</sup>. Dulisio等<sup>[34]</sup>认为在形成 2:1配合物的过程中,这些金属离子与 TC 的络合主要是发生与二甲基氨基或者是羟基基团的配合. TC 与金属离子的络合过程中,只有官能团发生了电离. TC 在亲核和亲电试剂的作用下,容易发生水解并引起分子的重排,转化为容易降解的物质. 某些金属离子,如镉离子对 TC 的水解和分子重排起到了催化作用.

#### 2.4 水中硝酸铅对 TC 消减过程的影响

图 6 为不同 TC 浓度下,对照组与含硝酸铅水体中 TC 消减过程的浓度变化.

由图 6 可知,光照和避光下,对照组 TC 浓度的变化趋势同以上实验组.不同 TC 浓度时,含硝酸铅水体中 TC 浓度均表现为下降趋势,且 600 h 之前,光照和避光下四环素浓度的变化趋势几乎吻合;600 h 之后,光照下四环素的消减速率大于避光组,且趋势逐渐增大.在光照和避光下,以图 6(a)为例

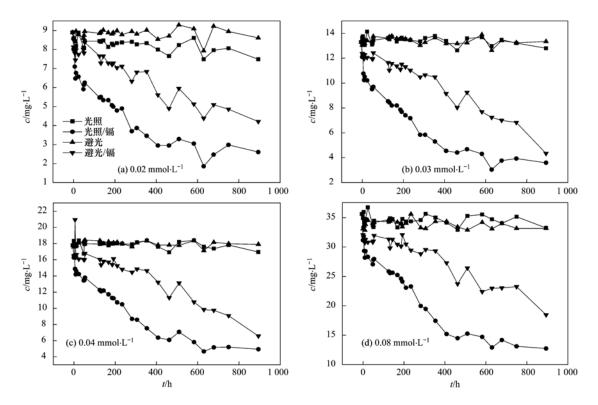


图 5 对照组与含硝酸镉水体中 TC 的浓度变化

Fig. 5 Changes of the concentration of tetracycline in the blank group and Cd(NO<sub>3</sub>)<sub>2</sub>

(低 TC 浓度),含硝酸铅水体中 TC 的消减速率分别为 29.3%和 14.8%;以图 6(d)为例(高 TC 浓度),硝酸铅水体中 TC 的消减速率分别为 32.3%和

#### 15.7%.

硝酸铅与 TC 的作用机制与硝酸镉相同,都是形成了重金属-TC 配合物. 但与硝酸镉相比,硝酸铅

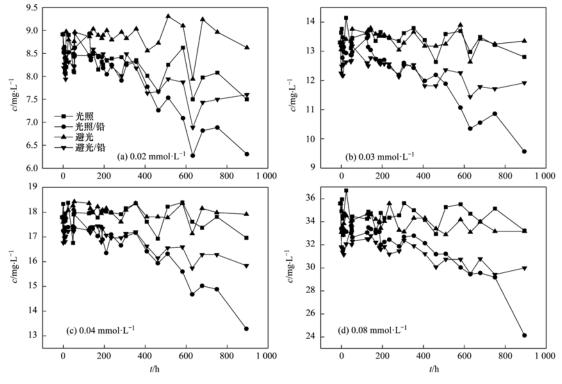


图 6 对照组与含硝酸铅水体中 TC 的浓度变化

Fig. 6 Change of the concentration of tetracycline in the blank group and  $Pb(NO_3)_2$ 

对 TC 消减的促进作用较小,这主要是由于 TC 与不同金属离子形成配合物的难易程度不同<sup>[35]</sup>,以及提供的反应位点的活性不同,水体中镉离子比铅离子更容易与 TC 形成稳定的配合物.

#### 3 结论

- (1)自然光照下,TC 的降解缓慢;未杀菌底泥水体中,TC 的消减速率大于杀菌底泥,微生物起到了重要的作用;含硝酸镉水体中 TC 的消减速率大于含硝酸铅水体,硝酸镉对 TC 消减的促进作用大于硝酸铅.以 0.08 mmol·L<sup>-1</sup>TC 为例,TC 在各环境因素中的消减速率依次为:未杀菌底泥(87.2%)>杀菌底泥(70.37%)>硝酸镉(64.2%)>硝酸铅(32.3%)>空白组(6.6%),各环境因素都促进了TC 的消减.
- (2)避光下,空白组 TC 及高浓度 TC 的消减趋势与光照组相似. 低浓度 TC 的消减受环境中金属离子及其他因素的影响较大. 避光下的 TC 消减速率小于光照组,表明光照促进了 TC 的消减.

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