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磷尾矿调控好氧堆肥微环境与强化协同去除四环素的机制

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摘要 【目的】针对磷尾矿(PT)堆存污染与有机废弃物(OWs)中四环素(TC)残留的双重环境问题,本文以实现两类固废资源化安全利用为目标,探究PT对OWs好氧堆肥微环境的调控作用,揭示其与Fe(Ⅲ)联用强化TC去除的效果与机制。【方法】本试验以PT为核心调理剂,石英砂(SiO_2)为对照,设置对照组($\text{SiO}_2 + \text{OWs}$)、PT组($\text{PT} + \text{OWs}$)和PT-Fe(Ⅲ)组[$\text{PT} + \text{Fe(Ⅲ)} + \text{OWs}$]3组好氧堆肥试验。监测堆体温度、含水率、pH、电导率(EC)等理化指标,总有机碳(TOC)、总氮(TN)等营养转化指标,分析磷素分级,测定种子发芽指数(GI)及TC去除率。【结果】PT的添加未干扰堆肥正常升温、腐熟进程,其碳酸盐缓冲体系可将堆体pH值稳定在7.5~8.5,减小EC波动幅度并维持其在腐熟安全阈值内,使最终GI稳定在127%以上,显著降低堆肥植物毒性。同时,PT也可定向调控磷形态转化,将HCl提取态磷(HCl-P)占比提升至80%~85%,为堆肥产品后续土壤应用提供长效磷素。PT对堆体中TC也具有较好的去除效果,PT组最终TC去除率达80.87%,较对照组提升8.12%。Fe(Ⅲ)与PT联用后协同效应显著,PT-Fe(Ⅲ)组TC去除率进一步升至95.24%,增幅达22.49%,核心机制为Fe(Ⅲ)水解生成的氢氧化物可强化TC在PT上的吸附,同时Fe价态循环产生活性氧化物质也能降解TC,最终形成“吸附-氧化-降解”的作用路径,显著提升TC去除效率。【结论】PT可作为高效调理剂,与Fe(Ⅲ)联用后能进一步强化TC污染控制效果,本试验结果为PT高值资源化利用与OWs堆肥抗生素风险管控提供了重要理论依据与技术参考。

关键词 磷尾矿; 固体废物; 好氧堆肥; 四环素; 资源化利用

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Mechanism of Phosphate Tailings for Microenvironment Regulation in Aerobic Composting and Enhanced Synergistic Tetracycline Removal

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Abstract [Objective] Addressing the dual environmental issues of phosphate tailings (PT) stockpile pollution and tetracycline (TC) residues in organic wastes (OWs), this paper aims to achieve the safe and resourceful utilization of both solid wastes. It investigates the regulatory effect of PT on the microenvironment in the aerobic composting of OWs and elucidates the synergistic effect and mechanism of PT combined with Fe(Ⅲ) in enhancing TC removal. [Methods] Three sets of aerobic composting experiments were established with PT as the core conditioner and quartz sand (SiO_2) as the control, including the control group ($\text{SiO}_2 + \text{OWs}$), PT group ($\text{PT} + \text{OWs}$) and PT-Fe(Ⅲ) group [$\text{PT} + \text{Fe(Ⅲ)} + \text{OWs}$]. Throughout the process, physicochemical indicators such as pile temperature, moisture content, pH and electrical conductivity (EC), along with nutrient transformation indicators including total organic carbon (TOC) and total nitrogen (TN) were monitored. Additionally, phosphorus fractionation, seed germination index (GI) and TC removal rate were determined. [Results] The addition of PT did not interfere with the normal temperature rise and maturation process of composting. Its carbonate buffer system stabilized the pile pH value within the suitable range of 7.5–8.5, reduced the

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magnitude of EC fluctuations and maintained it within the safe threshold for compost maturation, which kept the final GI above 127% and significantly reduced compost phytotoxicity. Meanwhile, PT regulated the transformation of phosphorus forms in a directional manner and increased the proportion of HCl-extractable phosphorus (HCl-P) to 80%–85%, thus providing a slow-release phosphorus source for the subsequent soil application of compost products. PT also exhibited favorable TC removal performance. The final TC removal rate of the PT group reached 80.87%, which was 8.12% higher than that of the control group. A significant synergistic effect was observed after the combination of Fe(Ⅲ) and PT, with the TC removal rate of the PT-Fe(Ⅲ) group further rising to 95.24%, representing an improvement of 22.49%. The core mechanism involved the enhancement of TC adsorption onto PT by Fe(Ⅲ)-derived hydroxides, coupled with TC degradation by reactive oxidants generated through Fe redox cycling, which collectively established an “adsorption-oxidation-degradation” removal pathway and thus significantly enhanced TC removal efficiency. [**Conclusion**] PT can serve as an effective compost conditioner, and its combination with Fe(Ⅲ) further enhances TC removal. These findings provide a crucial theoretical basis and technical reference for the high-value resource utilization of PT and antibiotic risk management in OWs composting.

Keywords phosphate tailing (PT); solid waste; aerobic composting; tetracycline (TC); resource recovery

磷资源的持续开采产生了大量的磷尾矿(PT)^[1]。我国年PT产量约为700万t,历史累积堆存量已超过12亿t,综合利用率仅为10%~17%^[2]。PT的累积可能会严重污染附近的土地和水体^[3]。目前,PT主要用于矿山回填,也用作缓释磷肥和土壤改良剂等^[4-5],但整体消纳量与利用效率仍有待提升^[6]。与此同时,城市与农业生产中产生的有机废弃物(OWs)如污泥、园林废物与畜禽粪便等作为1种“放错位置的资源”,富含氮、磷、钾等营养元素,也可作为有潜力的土地利用基质^[7-9]。将PT与OWs协同处置,用于制备生态有机基质,不仅能为PT的大规模消纳提供资源化路径,还能实现OWs的价值转化。

然而,畜禽粪便等OWs中常检测到高含量的抗生素,其中四环素类抗生素(TCs)残留物质量分数甚至可达数百毫克每千克^[10]。施肥已成为抗生素进入农业土壤的重要途径。据报道^[11],施用粪肥后,土壤中TCs增加了176.5%。而四环素(TC)因使用广泛、环境残留量高常被视为TCs的代表性物质,若不经有效去除,其生态利用存在一定的环境风险^[12]。已有研究证实,在堆肥过程中添加特定矿物材料,能改善堆体理化性质并去除部分有机污染物。如赤铁矿和高岭土在堆肥过程中可促进有机质稳定化和腐殖化^[13];硫酸亚铁可以增加牛粪和玉米秸秆堆肥过程中的腐殖质含量并减少氮损失^[14];添加硅藻土和膨润土可以使总抗生素耐药基因的相对丰度分别下降53.72%和59.54%^[15]。PT的主要矿物组分为白云石[CaMg(CO₃)₂],其是1种天然的吸附材料^[16],对

Co(Ⅱ)、Zn(Ⅱ)、Cu(Ⅱ)、Pb(Ⅱ)等多种金属离子^[17-18]和有机酸^[19]均具有一定吸附能力。然而,PT作为堆肥添加剂对OWs堆肥进程的影响和对TC的调控效应的系统性研究较少。同时,市政污泥也是OWs的常见组分,其在脱水处理过程中常投加铁盐(如FeCl₃)作为调理剂,致使脱水污泥中含有一定量的Fe(Ⅲ)^[20]。Fe(Ⅲ)与PT在堆肥过程中存在何种效应仍有待探究。

因此,本文旨在探究PT对OWs好氧堆肥过程的调控作用,系统评估PT对堆体温度、pH、电导率(EC)、碳氮转化及腐熟度等关键指标的影响,并阐明PT及其与外源Fe(Ⅲ)联用对堆肥中TC的强化去除效果与内在机理。结果可为PT高值资源化利用和OWs堆肥抗生素风险管控提供理论依据与技术参考。

1 材料与方法

1.1 试验材料

本试验使用的PT采自云南云天化股份有限公司。PT与SiO₂经研磨、60目筛网筛分后自然风干备用。OWs包括食物残渣、园艺废弃物等,经混合均匀后过2mm筛网。TC试剂购自上海阿拉丁生化科技股份有限公司,FeCl₃·6H₂O试剂购自上海创赛科技有限公司。

1.2 试验方法

本试验在14L泡沫保温箱(30cm×21.5cm×22.5cm)中进行,由于堆体体积较小、散热较快,泡沫箱内外均包裹2cm厚泡沫层,泡沫箱底部开2个小孔以便堆肥过程中的废液排出,总共设置3个试验组:对照组(SiO₂+OWs)、PT组(PT+OWs)、PT-

Fe(Ⅲ)组[PT + Fe(Ⅲ) ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) + OWs]。每堆堆体总质量为 2.5 kg, 矿物添加比例为 20% (以干重计), Fe(Ⅲ)添加比例为 5% (以干重计), 初始含水率调整至 60%, 初始碳氮比设定为 25 : 1, 初始 TC 添加质量分数为 20 mg/kg (以干重计), 通气速率为 0.3 L/(min·kg) (以干重计), 堆肥过程持续 35 d。其中, SiO_2 作为惰性无机调节剂, 满足堆肥过程中无机组分的基本要求, 确保试验差异源于 PT 的化学性质及界面相互作用。

1.3 取样与分析方法

堆肥过程中, 每日 10:00 使用温度计测量每堆堆肥的中心温度, 并定期从顶层、中层和底层采集复合样品。每份样品经均质化处理后分为 3 份: 一份立即分析, 一份经风干研磨后保存, 另一份储存在 $-80\text{ }^\circ\text{C}$ 冰箱中, 用于 TC 含量的测定。

按照《有机肥料》(NY/T 525—2021)中所规定的方法进行含水率、pH、EC 和种子发芽指数(GI)等指标的检测。采用《有机肥料中土霉素、四环素、金霉素与强力霉素的含量测定 高效液相色谱法》(GB/T 32951—2016)测定 TC 含量。每个指标的测定均设置 3 个平行组。

2 结果和讨论

2.1 堆体温度与含水率的变化

堆肥过程中温度、含水率的变化如图 1 所示。堆肥温度常用于评估堆肥效率和堆肥产品的稳定性^[21]。一方面, 温度会影响微生物的代谢速率与堆

肥腐熟进程; 另一方面, 抗生素的官能团(如磺胺类抗生素中的杂环结构、氯霉素中的硝基苯环和氯乙酰基)在堆肥温度高于 $55\text{ }^\circ\text{C}$ 时容易降解^[22-23]。图 1(a)展示了 3 个试验组堆肥过程中堆体温度的变化情况。在 35 d 的堆肥周期内, 堆体依次经历升温期(0~2 d, $22\text{ }^\circ\text{C}$ 升至 $35\text{ }^\circ\text{C}$)、高温期(3~6 d, $52\text{ }^\circ\text{C}$ 升至 $63\text{ }^\circ\text{C}$)、降温期(7~21 d, $46\text{ }^\circ\text{C}$ 降至 $27\text{ }^\circ\text{C}$)与腐熟期(22~35 d, $27\text{ }^\circ\text{C}$ 降至 $23\text{ }^\circ\text{C}$) 4 个典型阶段。由于试验堆体较小, 热量散失较快, 且试验过程中天气骤变, 高温期内对照组堆体温度 $\geq 55\text{ }^\circ\text{C}$ 的持续时长为 4 d, PT 组与 PT-Fe(Ⅲ)组的持续时长均为 3 d。

除了堆肥温度, 含水率也是影响微生物代谢的重要指标。它不仅可以调节堆体温度、溶解性有机物(DOM)含量, 还直接参与微生物代谢过程^[24]。堆肥过程中, 堆体含水率过高或过低均不利于微生物活动。含水率过高会影响堆肥的通气效果, 导致堆体局部厌氧发酵产生酸性环境使得养分损失。含水率太低会抑制微生物的代谢活性从而影响好氧堆肥效果^[25]。如图 1(b)所示, 3 组堆体初始含水率均调节为 55%~59%, 随着堆肥的进行, 3 组含水率均下降且下降趋势相似。由于堆体较小, 在 0~7 d 下降最快, 在第 22 d 下降趋势明显减弱, 最终含水率稳定在 22% 左右。由于 3 组体系温度、含水率的变化趋势较为一致, PT 与 Fe(Ⅲ)的引入未显著扰动堆肥微环境。

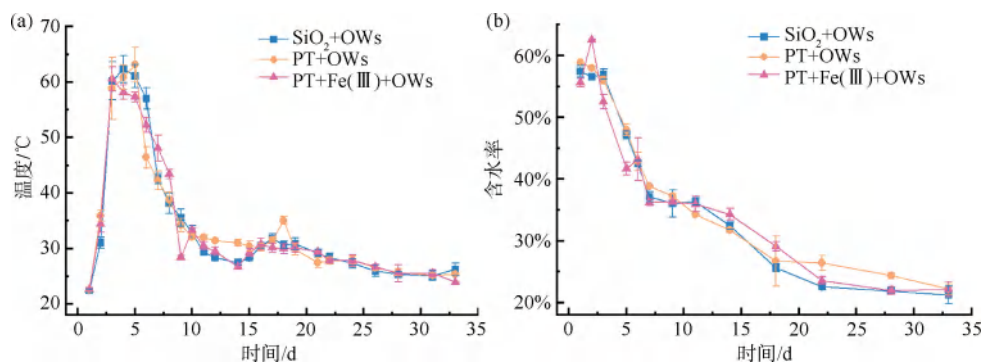


图 1 好氧堆肥过程中(a)温度;(b)含水率的变化曲线

Fig. 1 (a) Temperature; (b) Moisture Content Variation Curves during Aerobic Composting

2.2 堆体 pH 与 EC 的变化

堆肥过程中 pH 值与 EC 的变化如图 2 所示。堆体 pH 的动态变化反映了堆肥理化环境的稳定性。由图 2(a)可知, 3 组堆体的 pH 变化差异显

著。对照组 pH 值在 0~6 d 内快速波动, 受升温期氨氮大量释放的影响迅速升至 9.2, 后期维持在 8.5~9.5 的高碱度区间, 说明 SiO_2 不具备酸碱缓冲能力, 堆肥过程中无法抑制 pH 的剧烈波动^[26]。

相比之下,PT 组的 pH 值在 0~6 d 上升后稳定在 7.5~8.5 的弱碱性区间,波动幅度明显减小,这源于 PT 主要成分 $[\text{CaMg}(\text{CO}_3)_2]$ 提供的碳酸盐缓冲体系,可有效调节 pH,缓解氨氮积累引发的 pH 骤变。在 PT 组中添加 Fe(Ⅲ)后,PT-Fe(Ⅲ)组 pH 值整体稳定在 7.0~7.8 的近中性区间,波动最小,这是因为 Fe(Ⅲ)水解消耗体系中的氢氧根离子,并与 PT 碱性组分形成络合物,进一步增强了体系的酸碱缓冲能力,减少了碱度对微生物活性的抑制。

EC 反映了堆体中可溶性盐离子浓度的变化,由

图 2(b)可知,红色虚线标注的 4.0 mS/cm 为堆肥腐熟的安全盐分阈值^[27]。对照组初期 EC 高达 5.34 mS/cm,0~5 d 快速下降后波动上升,后期稳定在 3.96 mS/cm。PT 组的 EC 在前期呈迅速波动状态,后期逐步趋于平稳,稳定在 3.87 mS/cm,这是由于添加 PT 可固定部分可溶性盐离子,减少盐分流失,从而降低 EC 波动幅度。PT-Fe(Ⅲ)组初期 EC 显著偏高,这与 Fe(Ⅲ)水解产生的离子直接相关;EC 整体呈波动下降趋势,说明 Fe(Ⅲ)与有机质、PT 组分发生络合或沉淀反应,有效降低了可溶性盐离子浓度,最终稳定在 4.05 mS/cm。

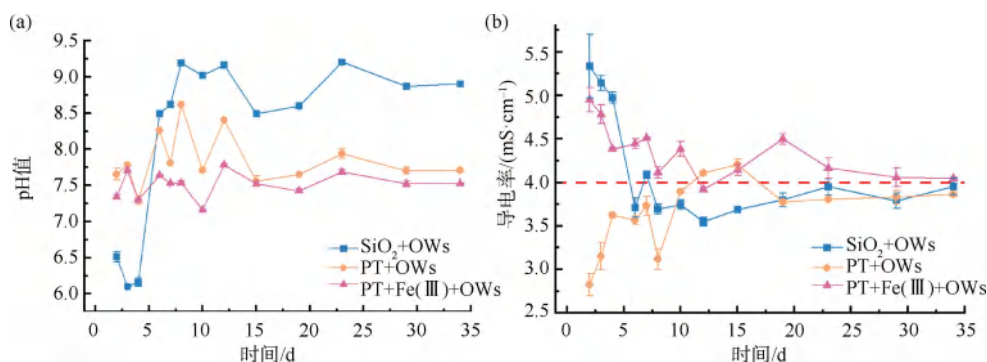


图 2 好氧堆肥过程中 (a) pH 值; (b) EC 的变化

Fig. 2 Changes of (a) pH Values; (b) EC during Aerobic Composting

2.3 堆体总有机碳 (TOC)、总氮 (TN) 的变化

堆肥过程中 TOC 与 TN 的变化如图 3 所示。TOC 是衡量有机物降解和稳定性的重要指标^[28]。3 组处理的 TOC 均随堆肥进程呈下降趋势,且变化速率与堆肥阶段高度关联[图 3(a)],在 0~10 d 内,对照组的 TOC 从初始 27.5%快速降至 22.0%,这一阶段微生物优先利用易降解有机质代谢。PT 组与 PT-Fe(Ⅲ)组初始 TOC 均低于对照组 (23.13%~23.73%),10 d 后降至 20.73%~21.00%。10~35 d

进入降温期与腐熟期,3 组 TOC 下降速率均降低,最终稳定在 18.7%~19.1%。

TN 是定量反映堆体肥力重要指标之一,可体现堆肥中有效养分的含量及堆肥的质量水平^[29]。如图 3(b)所示,对照组 TN 的质量分数始终处于最高水平,初始值为 2.17%,在堆肥过程中呈缓慢下降趋势,但在 32 d 时仍维持在 1.9% 以上。PT 组的 TN 初始质量分数为 1.47%,前期经历小幅波动后逐渐下降,全程维持在 1.39%~1.50%,为 3

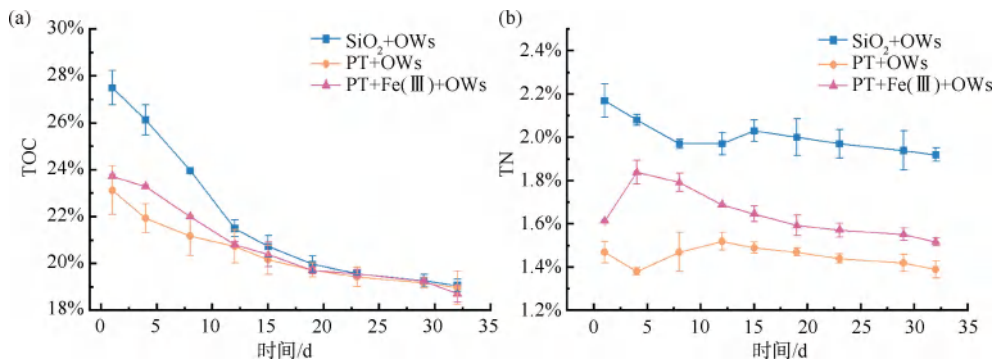


图 3 好氧堆肥过程中 (a) TOC; (b) TN 的变化

Fig. 3 Changes of (a) TOC; (b) TN during Aerobic Composting

组中最低。PT-Fe(Ⅲ)组中TN初始质量分数为1.61%,在堆肥前5d呈上升趋势并达到1.84%,随后持续下降,至32d时降至1.52%左右,整体变化趋势介于对照组与PT组之间。PT的引入会降低堆肥初始TN含量,而Fe(Ⅲ)的添加在堆肥初期可通过其水解产物固持部分溶解态有机氮,减缓氮素损失,使TN含量出现阶段性上升,但随着堆肥进程推进,各组TN含量均呈下降趋势,推测主要与堆肥过程中的氨挥发、反硝化作用及氮素矿化损失相关^[30]。

2.4 堆体磷组分的变化

根据Hedley等^[31]的修正法,堆肥中的磷可分为7种形态。由图4可知,PT的添加显著改变了磷的形态分布。在整个堆肥过程中PT组[图4(b)]

的速效磷(水溶性磷与NaHCO₃提取态无机磷之和)占比持续低于对照组[图4(a)],而HCl提取态磷(HCl-P)的占比(80%~85%)显著高于对照组(68%~79%)。HCl-P主要为与钙、镁结合的磷(Ca/Mg-P),是潜在的缓释磷源^[32]。这一差异主要源于PT自身含一定量的氟磷灰石[Ca₅(PO₄)₃F]等原生钙磷矿物^[33],其在分级提取中属于HCl-P组分^[34]。同时,PT组中主要组分白云石释放的Ca²⁺、Mg²⁺与堆体速效磷反应,生成的钙镁磷酸盐沉淀,在分级提取中也归入HCl-P,进一步提高了其占比^[8]。PT的加入对堆肥中磷素循环的定向调控作用实现了磷素从活性形态向稳定性形态的转变,缓释磷源的高占比可为堆肥产品后续土壤应用提供长效磷素供应。

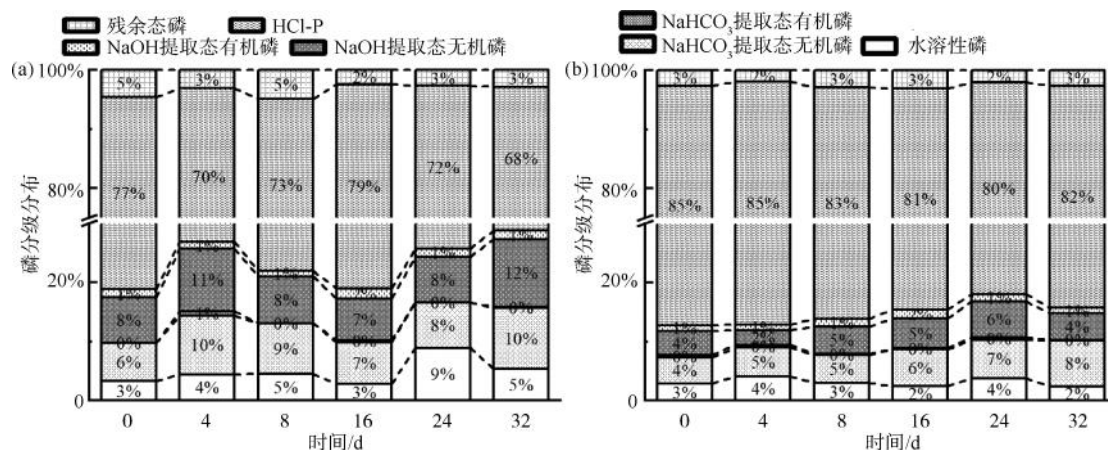


图4 好氧堆肥过程中(a)对照组;(b)PT组的土壤磷素分级情况

Fig. 4 Soil Phosphorus Classification in (a) Control Group; (b) PT Groups during Aerobic Composting

2.5 堆体GI变化

GI是反映腐熟程度与堆肥产物植物毒性的重要生物指标,一般认为GI值为80%表示腐熟^[35]。未腐熟的堆肥物料通常含有较高的氨、有机酸或其他DOM,可能抑制种子发芽和植物发育,同时抗生素残留也会显著抑制植物根系生长^[36]。如图5所示,3组堆体的GI值均随堆肥进程呈上升趋势,但组间差异较为明显。对照组前期上升缓慢,直至25d才升至94.33%,说明SiO₂对堆肥毒性的缓解作用较弱。PT组初始GI值约为30.10%,是3组中上升速率最快的处理,20d后GI值稳定在130%~146%的较高水平,表明PT的添加可有效降低堆肥中的毒性物质,加速堆体腐熟。PT-Fe(Ⅲ)组初始GI值约为20%,32d时GI值稳定在127.54%,说明

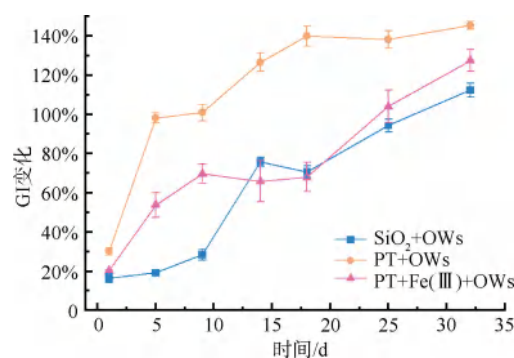


图5 好氧堆肥过程中GI变化

Fig. 5 Changes of Seed GI during Aerobic Composting

Fe(Ⅲ)的引入未增加堆肥毒性,反而与PT协同促进了堆体腐熟。整体来看,PT组及PT-Fe(Ⅲ)组的GI值均显著高于对照组,且最终均远超80%的腐熟

安全阈值,其中 PT 组的腐熟效果最优。

2.6 堆体中 TC 的去除情况

结合前文堆肥理化性质的演变规律,PT 与 Fe(Ⅲ)的引入未显著扰动堆肥温度、含水率等微环境,排除环境波动干扰后,TC 去除率的差异源于堆体物料本身。从堆肥进程来看,在 0~5 d 内 3 组 TC 去除率均呈快速上升趋势(图 6),其中 PT-Fe(Ⅲ)组增幅最显著,这可能是由于 PT 表面 Ca^{2+} 、 Mg^{2+} 等活性位点,可以通过表面吸附作用、静电吸附等有效富集堆肥体系中的 DOM,TC 易与 DOM 络合,从而在矿物界面形成局部高浓度微环境^[37-38],堆肥快速升温产生的高温效应可进一步强化去除富集态的 TC。

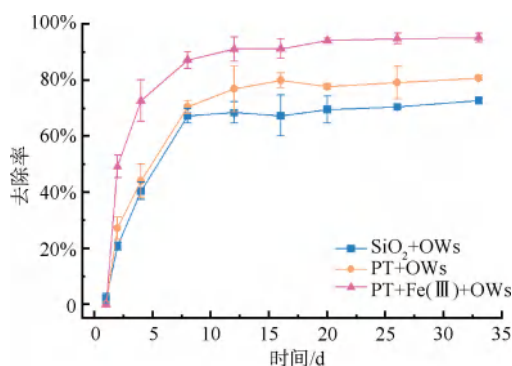


图 6 好氧堆肥过程中 TC 去除率的变化

Fig. 6 Changes of TC Concentration during Aerobic Composting

而 Fe(Ⅲ)的引入可通过在 PT 表面迅速水解产生铁的氢氧化物强化对 TC 的吸附,同时堆肥体系中 DOM(如腐植酸中的醌基)的还原作用可驱动 Fe(Ⅲ)/Fe(Ⅱ)价态循环,通过氧化作用或直接产生自由基来进一步加速 TC 的去除^[39-40]。在高温期,PT 组与 PT-Fe(Ⅲ)组的去除率增速明显高于对照组,结合前文 pH 值稳定在 7.5~8.5 的弱碱性区间、EC 维持在安全阈值内的结果,表明 PT 优化的堆肥理化环境可显著提升微生物活性,强化 PT 界面上富集的 TC 生物降解。在腐熟期,PT 组 TC 去除率稳定在 80.87%,较对照组提升 8.12%,而 PT-Fe(Ⅲ)组进一步升至 95.24%,增幅达 22.49%,这一差异不仅证实了 PT 对 TC 去除的促进作用,更揭示了 Fe(Ⅲ)与 PT 的协同机制:PT 通过吸附富集 TC 分子形成局部高含量反应区,Fe(Ⅲ)则在 PT 构建的微碱性区域中水解强化 TC 吸附并氧化部分 TC,形成“吸附-氧化-降解”的生化协同去除路径,促进有机污染物去除,更体现了“以废治废”的资源

化理念,为 OWs 堆肥的抗生素污染控制提供了切实可行的技术路径。

3 结论

本文系统探究了 PT 及 Fe(Ⅲ)对 OWs 好氧堆肥的调控效应,明确了 PT 在优化堆肥微环境、定向调控磷形态转化及强化 TC 去除中的核心作用,主要结论如下。

(1)PT 可有效优化堆肥微环境并加速腐熟进程。PT 通过自身碳酸盐缓冲体系将堆体 pH 值稳定在 7.5~8.5 的适宜区间,PT 组的 EC 值最终稳定在腐熟安全阈值,减少盐分累积风险;同时显著提升 GI,20 d 后稳定在 130%~146%,远高于 80%的腐熟阈值,有效加速堆体腐熟并降低植物毒性。Fe(Ⅲ)的引入未增加堆肥毒性,反而与 PT 协同进一步提升了体系的酸碱缓冲能力。

(2)PT 可定向调控磷素形态向缓释磷转化。PT 的引入显著改变堆肥磷素形态分布,一方面通过自身含有的氟磷灰石直接提升 HCl-P 占比;另一方面通过释放的 Ca^{2+} 、 Mg^{2+} 与速效磷发生沉淀反应,促进速效磷向潜在缓释磷源 HCl-P 转化。

(3)PT 与 Fe(Ⅲ)可协同强化 TC 的高效去除。PT 通过表面活性位点富集 TC 分子,并优化堆肥微环境以提升微生物活性,将 TC 去除率提升至 80.87%;Fe(Ⅲ)与 PT 联用后,可通过“吸附-氧化-降解”的作用路径实现 95.24%的 TC 高效去除率。

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