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纳米 TiO, 对土壤重金属释放及形态变化的影响

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关键词:纳米二氧化钛;土壤;重金属;形态;转化

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Effect of Nano-TiO₂ on Release and Speciation Changes of Heavy Metals in Soil

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Abstract: The effects of nano-TiO₂ on migration and transformation of heavy metals in soil were investigated by outdoor flooding simulation experiments. Cr, Pb, Zn, Cd and Cu contents of different forms were determined in soil of typical fluctuating zone of Three Gorges Reservoir. The results showed that, after flooding months, both addition of 4 g·kg⁻¹ of rutile and anatase particles resulted in the release of about 30% Cr into the water. Nano-TiO₂ particles mainly promoted the dissolution of oxidizable residual Cr, and elevated its ecological risk. Thus nano-TiO₂ promoted the activation of chromium and improved the mobility of chromium in soil. 4 g·kg⁻¹ of rutile particles caused the decrease of acid exchangeable lead by 25. 92% and oxidizable lead by 33. 09%, and enhanced the mobility of Pb. However, anatase particles caused the increase of oxidizable zinc by 30% in soil, which facilitated fixing of zinc. In addition, two types of nano-TiO₂ particles had no significant effect on the speciation changes of Cu and Cd. Therefore, the effect of nano-TiO₂ on release and transformation of Cr in soil was the largest, followed by Pb and Zn. This needs special attention when using nano-TiO₂ to remediate heavy metals contaminated soil and assessing its environmental risk.

Key words: nano-TiO2; soil; heavy metal; speciation; transformation

纳米 TiO₂ 是应用最早的纳米材料之一,已经被广泛应用于各领域数十年^[1~5],其在生产、使用和废物处理过程中必将进入环境^[6]. 土壤和沉积物是环境污染物的汇聚场所,进入环境的纳米 TiO₂ 最终也汇入土壤或沉积物中^[7,8]. 有研究预测纳米 TiO₂ 在污 泥 处 理 的 土 壤 中 的 含 量 以 42 ~ 89 μg·(kg·a) ⁻¹的速度增加^[9],全球范围内预计到 2025 年高风险区域土壤中纳米 TiO₂ 的含量可达到 20 g·kg^{-1[9,10]}. 并且,利用纳米 TiO₂ 的光催化作用降解土壤中有机农药和处理重金属污染也是目前正在探索的土壤修复新方法之一. 由于具有催化活性,理论上可能改变重金属的价态和存在状态,一些

研究显示,纳米 TiO₂ 对土壤中 Zn、Pb、Cu、Cd 等有一定固定作用^[11,12]. 因此关于纳米 TiO₂ 的安全应用及对土壤环境的生态风险问题已成为关注的焦点. 有研究显示纳米 TiO₂ 颗粒影响 Cu²⁺在不同类型土壤中的迁移^[13]. 但目前关于纳米 TiO₂ 对其他重金属的迁移转化的影响还知之甚少. 重金属的形态变化是反映其迁移转化的重要指标,因此,本研究

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以三峡水库典型消落区土壤为对象,通过室外的模拟淹水实验,分析两种晶型的纳米 TiO₂ 颗粒对土壤中重金属形态变化的影响,以期为纳米 TiO₂ 的安全应用及其环境风险预测提供科学依据.

1 材料与方法

1.1 纳米 TiO₂ 颗粒与供试土壤

锐钛矿颗粒(~100 nm)和金红石颗粒(<25 nm)颗粒均购自阿拉丁试剂公司(Aladdin),纯度均

为99.8%.

供试土壤采自三峡水库干流重庆市忠县石宝寨(N30°25′5.5″, E108°10′5.5″)消落区,海拔153 m处,该消落区淹没面积较大,且随水库水位而变化,占重庆市消落区总面积的1/10,土壤类型是西南地区典型的紫色土,每年4~9月为落干期,其余时间为淹水期.样品风干后粉碎,过2 mm和0.149 mm筛,混合均匀备用.土壤基本的理化性质和Cr、Zn、Pb、Cu、Cd含量如表1.

表 1 供试土样的基本特性

Table 1 Physicochemical properties of soil for test

рН	有机质	CEC		机械组成/%		Cr	Pb	Zn	Cd	Cu
рп 	/g•kg ⁻¹	/cmol⋅kg ⁻¹	<0.02 mm	0. 02 ~ 0. 25 mm	>0. 25 mm	/mg·kg -1	$/\mathrm{mg}\cdot\mathrm{kg}^{-1}$	/mg·kg ⁻¹	/mg·kg -1	$/mg \cdot kg^{-1}$
8. 04	15.76	7. 12	45.88	51.00	3. 12	25. 14 ± 0. 51	73. 84 ± 3.25	133. 29 ± 10. 81	2.83 ± 0.06	32. 91 ± 1. 14

1.2 实验方法

用超纯水配制 300 mL 0. 2 g·L⁻¹和 0. 4 g·L⁻¹的纳米 TiO_2 颗粒悬液,超声 30 min. 称 30 g 土样 (0. 149 mm),加到纳米 TiO_2 颗粒悬液中,摇匀,土样中纳米 TiO_2 颗粒的添加量分别为 2 g·kg⁻¹和 4 g·kg⁻¹,金红石处理组编号为 $R2(2 g·kg^{-1})$ 和 $R4(4 g·kg^{-1})$,锐钛矿处理组编号为 $T2(2 g·kg^{-1})$ 和 T4

(4 g·kg⁻¹),以不加纳米 TiO₂ 处理为对照,每个处理重复 3 次. 实验采用磨口硼硅玻璃瓶,加盖,置于室外环境中,60 d 后,分离上覆水,测定水样中 Cr、Pb、Zn、Cd 和 Cu 的浓度,土样冻干后粉碎,过筛(0.149 mm),混合均匀. 采用改进的 BCR 连续提取法^[14]分析样品中各形态 Cr、Pb、Zn、Cd 和 Cu 的含量,具体步骤见表 2.

表 2 各形态重金属的提取方法

Table 2 Extraction method of various forms of heavy metals in soil

步骤	提取方法	形态
1	称取 1.00 g 样品到 100 mL 聚丙烯离心管中,加入 0.11 mol·L ⁻¹ HAc 40 mL,室温下振荡 16 h(250 r·min ⁻¹),离心分离(4 000 r·min ⁻¹ ,20 min),取上层清液到聚乙烯瓶中,置在 4℃ 冰箱中待测,残渣用超纯水清洗两次	可交换态
2	向上述残渣中加入 $0.5~\mathrm{mol}\cdot\mathrm{L^{-1}~NH_2OH}\cdot\mathrm{HCl}$ 40 mL,振荡 $16~\mathrm{h}$,离心分离. 其余操作同第一步	可还原态
	向第二步提取后的残渣中缓慢加入 $10\mathrm{mL}\mathrm{H_2O_2}$,盖上表面皿,偶尔振荡,室温下消解 $1\mathrm{h}$,然后水浴加热到 $85\mathrm{℃}$ 消解	
3	1 h,去表面皿,升温加热至溶液近干,再加入 10 mL $\rm H_2O_2$,重复以上过程. 冷却后,加 1 mol·L $^{-1}$ NH $_4$ OAc 50 mL,其余操作同第一步	可氧化态
4	将第三步提取后的残渣转到 50 mL 聚四氟乙烯烧杯中,加入 10 mL HNO $_3$ 、10 mL HF 和 3 mL HClO $_4$,盖上盖子后放到电热板上低温加热 1 h,再中温加热 1 h 之后开盖除去硅. 至冒浓厚白烟时就盖上盖子. 等到坩埚壁上的黑色有机物消失后,打开盖驱赶白烟直到内容物呈黏稠状. 取下稍冷,用水冲洗坩埚盖和内壁,并加入 1 mL $V(HNO_3):V(H_2O)$ = 1: 1的溶液低温加热溶解残渣. 消解液冷却后将其转移至 25 mL 容量瓶中,定容后摇匀待测	残渣态

实验采用超纯水(Minipore 18.2 $M\Omega \cdot cm$),所用试剂均为优级纯,样品采用电感耦合等离子体质谱 ICP-MS(Thermo,iCAP-Q)测定. 实验过程采用空白实验、标准工作曲线、加标回收、标准物质(GBW 07406)进行质量控制,回收率在 90.6% ~ 106.8% 之间.

2 结果与分析

2.1 上覆水中各重金属元素的浓度

淹水 60 d 后,上覆水中各重金属元素的浓度如表 3.与对照相比,添加纳米 TiO_2 颗粒处理后,上覆水中 Cr 浓度均明显升高(P < 0.05),且均随纳米

TiO₂ 颗粒浓度的增加而升高,4 g·kg⁻¹处理组 Cr 浓度均大幅升高,说明两种纳米 TiO₂ 颗粒均促进土壤中 Cr 的释放,提高水体污染风险,且浓度越高风险越大.上覆水中 Pb 浓度仅 R4 处理组明显升高,表明 4 g·kg⁻¹金红石促进土壤中 Pb 的释放.与 Cr 和 Pb 不同的是,T2 和 T4 处理组上覆水中 Zn 浓度明显低于对照组,说明锐钛矿纳米 TiO₂ 抑制土壤 Zn 的溶出,具有一定的固定作用.此外,上覆水中 Cd 浓度均非常低,差异也较小,可能是由于土壤中 Cd 含量很低或纳米 TiO₂ 对土壤 Cd 影响很小;各处理组Cu含量与对照相比也没有明显的变化,显示纳

表 3 上覆水中各重金属元素的浓度 $^{1)}/\mu g \cdot m L^{-1}$

Table 3 Concentrations of heavy metal elements in overlying water/ $\mu g \cdot m L^{-1}$

元素	对照	R2	T2	R4	T4
Cr	0. 021 ± 0. 002	0. 111 ± 0. 013 *	0. 051 ± 0. 008 *	0. 644 ± 0. 018 *	0. 675 ± 0. 051 *
Pb	0.268 ± 0.054	0.227 ± 0.002	0.324 ± 0.003	0. 561 ± 0.006 *	0.233 ± 0.021
Zn	1.457 ± 0.144	1.552 ± 0.066	0.892 ± 0.066 *	1. 343 ± 0.021	0.312 ± 0.056 *
Cd	0.006 ± 0.001	0.001 ± 0.000	0.012 ± 0.001	0.004 ± 0.000	0.002 ± 0.000
Cu	0.179 ± 0.030	0.178 ± 0.023	0.197 ± 0.010	0.213 ± 0.018	0.180 ± 0.024

1) * 表示 P < 0.05(与对照相比),下同

米 TiO₂ 对土壤 Cu 释放的影响很小.

2.2 土壤中重金属的总量变化

经过反复淹水和落干过程,三峡水库消落带土壤重金属含量相对较稳定.淹水60 d后,与原样相比,对照组各重金属元素的总量均略有降低,但没有明显的差异(表4).与对照相比,4 g·kg⁻¹纳米 TiO₂颗粒处理组 Cr 含量均明显下降,分别降低 30.20%

和30.85%,这与上覆水中该处理组 Cr浓度急剧升高是一致,R4和T4处理组上覆水中 Cr浓度显著升高,进一步证明土壤中 Cr释放到水中.金红石颗粒组土壤 Pb含量呈下降趋势,但没有达到显著水平.锐钛矿颗粒处理组土壤 Zn呈升高趋势,且T4处理组明显高于对照组.此外,土壤中 Cd和 Cu均没有明显变化.

表 4 土壤中各重金属元素的总量变化/mg·kg-1

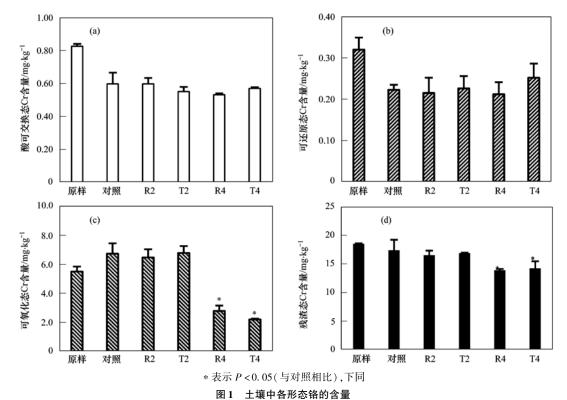
Table 4 Changeof the total amounts of heavy metals in soils/mg·kg⁻¹

处理	Cr	Pb	Zn	Cd	Cu
原样	25. 14 ± 0. 51	73.84 ± 3.25	133. 29 ± 10. 81	2.83 ± 0.06	32.91 ± 1.14
对照	24.93 ± 2.65	71.43 ± 3.03	120.07 ± 3.67	2.72 ± 0.16	30.74 ± 1.08
R2	23.82 ± 1.44	69.47 ± 1.81	119.97 ± 4.32	2.82 ± 0.05	31.00 ± 0.75
T2	24.44 ± 0.65	70.07 ± 4.24	126.17 ± 5.55	2.70 ± 0.08	30.48 ± 1.71
R4	17.40 ± 0.65 *	66.53 ± 4.04	116. 17 ± 6.55	2.78 ± 0.14	30.35 ± 2.21
T4	17.24 ± 1.33 *	71.40 ± 2.23	128.41 \pm 0.87 *	2.81 ± 0.09	30.62 ± 1.08

2.3 土壤中重金属的形态变化

从形态分布来看,供试土壤 Cr 主要以残渣态和

可氧化态形式存在(图 1),两种形态占总量的 95% 以上. 值得注意的是,两种纳米 TiO₂ 颗粒均促进土



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Fig. 1 Contents of different speciation of chromium in soil

壤中这两种形态 Cr 的释放,R4 和 T4 处理组可氧化态 Cr 含量分别下降 58.79% 和 67.40%,残渣态含量分别下降 20.08% 和 18.11%,土壤 Cr 的活性增强,环境风险增大. 纳米 TiO₂ 处理组酸可交换态和可还原态 Cr 与对照相比没有明显变化,说明释放出来的可氧化态和残渣态 Cr 没有转化成其他形态,全部进入水中.

土壤中 Pb 主要以残渣态存在(图 2),所占比例 为 51.77% ~ 61.87%,且淹水后略有升高,其次是

可还原态,淹水后,可还原态 Pb 含量均明显低于原样,但纳米 TiO₂ 对这两种形态 Pb 均没有明显的影响. 土壤中酸可交换态和可氧化态 Pb 含量的变化规律类似,仅 R4 处理组明显低于对照组,其他处理组都没有明显的变化,值得注意的是,虽然 R4 处理组土壤中总 Pb 含量下降没有达到显著水平,但实际上仍有少量 Pb 释放到水中,只是本研究采用的土壤中酸可交换态和可氧化态 Pb 含量相对较低,所以对总量变化影响较小.

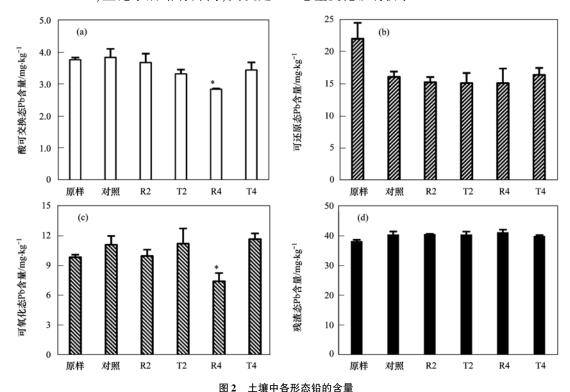


Fig. 2 Contents of different speciation of lead in soil

土壤中4种形态 Zn 的含量分布相对比较均匀(图3),淹水后呈现酸可交换态和可还原态向可氧化态和残渣态转化的趋势,纳米 TiO₂ 颗粒对酸可交换态、可还原态和残渣态 Zn 含量的影响不明显;可氧化态 Zn 淹水后也呈升高趋势,其中锐钛矿颗粒处理组(T2 和 T4)达到显著水平(P < 0.05),分别比对照高 42.65%(T2)和 44.25%(T4),说明锐钛矿颗粒促进了可氧化态 Zn 的形成,使部分溶解的 Zn 转化成可氧化态滞留在土壤中,这也是 T2 和 T4 处理组上覆水 Zn 含量明显低于对照组,且土壤中 Zn 总量高于对照组的主要原因.

此外,供试土壤 Cd 含量较低,各形态的含量变化不明显,纳米 TiO_2 对 Cu 的形态和总量也没有明显的影响(图 4).

3 讨论

综合上覆水、土壤重金属总量和各形态的含量变化可以看出,土壤重金属总量的变化能反映出不同处理之间的变化趋势,上覆水的变化能够较准确地反映出不同处理之间的差异,各形态含量的变化才能判断纳米 TiO, 对土壤重金属的影响过程.

土壤中重金属的存在形态与 pH、氧化还原电位、铁锰氧化物、有机质、微生物等因素有关. 本研究土壤淹水后处于还原条件,通常在还原条件下,重金属和土壤中的硫容易形成硫化物沉淀,因此一般残渣态含量会升高. 本研究中 Pb、Zn 和 Cu 也有类似的规律,各处理组残渣态含量均略有升高. 但Cr 的变化差异较大,各处理组残渣态含量均降低,其中对照组和2 g·kg⁻¹纳米 TiO₂ 处理组(R2 和 T2)

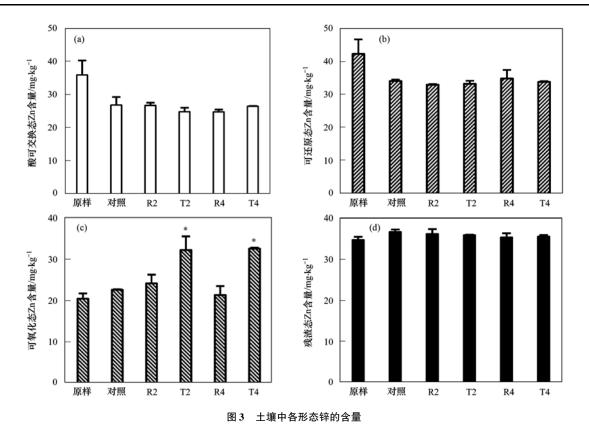


Fig. 3 Contents of different speciation of zinc in soil

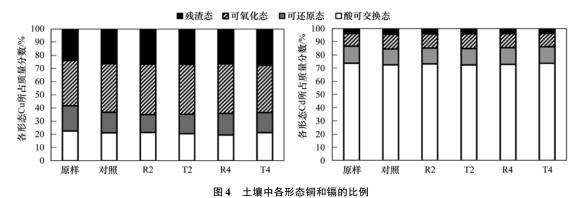


Fig. 4 Proportions of different speciation of copper and cadmium in soil

的变化与陈英旭等^[15]的研究结果相似,还原条件下土壤中铬有从残渣态逐渐向有机结合态(可氧化态)转化的趋势. 但 4 g·kg⁻¹纳米 TiO₂ 处理组(R4和T4)残渣态和可氧化态都大幅降低,并且对应的上覆水中 Cr浓度大幅升高,说明 4 g·kg⁻¹纳米 TiO₂处理促进残渣态和可氧化态 Cr的溶出,这可能与Cr的价态变化有关. 纳米 TiO₂与铬可以发生复杂化学反应, Cr(Ⅵ)可被纳米 TiO₂ 光催化还原成Cr(Ⅲ)^[16~22],但在 Mn²⁺存在时,不仅能使催化剂中毒^[23],还能氧化 Cr(Ⅳ)[Cr(Ⅵ)光催化还原的中间产物]而阻碍Cr(Ⅵ)的还原^[24]. Mn²⁺是土壤中普遍存在的元素,阻碍Cr(Ⅵ)的光催化还原过程是

可能的. 此外,Cr(VI)的光催化反应还受 pH、有机物等多种因素的影响 $[^{25,26]}$. 土壤是一个复杂的体系,纳米 TiO_2 与土壤中铬是直接发生反应,还是通过改变土壤环境条件间接导致其溶出,还需进一步研究确定.

可氧化态重金属一般氧化环境下易分解释放,还原条件下有利于其积累,因而一般在还原条件下,可氧化态重金属含量会升高.本研究中除了 R4 和 T4 处理组可氧化态 Cr 含量明显降低外,金红石颗粒处理组可氧化态 Pb 含量也随颗粒浓度升高而降低,其中 R4 处理明显降低,表明金红石颗粒抑制了可氧化态 Pb 的形成,其他各处理组可氧化态含量均

呈上升趋势. 与 Cr 类似,金红石颗粒对可氧化态 Pb 的抑制可能与其价态变化有关,Pb 的价态有 +4 和 +2,结合土壤可氧化态 Cr 和 Pb 的结果,推测在本 实验条件下,纳米 TiO。可能促进土壤中低价的 Cr 和 Pb 向高价转化,起了光催化氧化的作用,高价态 Cr和Pb的活性和溶解性均高于低价态,毒性也高 于低价态,因此纳米 TiO,颗粒在提高土壤 Cr 和 Pb 的迁移性的同时可能也提高了生态毒性,特别是 Cr. 此外,与之相反的是,锐钛矿颗粒处理显著提高 了可氧化态 Zn 的含量,将淹水过程释放出来的可还 原态和酸可交换态 Zn 部分转化成可氧化态而截留 在土壤中,相应的上覆水中 Zn 浓度显著低于对照组 也证明了锐钛矿颗粒抑制土壤 Zn 的溶出,降低水体 污染风险. 其原因可能是由于纳米 TiO, 颗粒易与 土壤中有机胶体类物质(如腐殖质)结合,因其具有 较强的吸附作用,从而提高土壤中有机物对溶出 Zn2+的捕获能力,由此可以推测锐钛矿颗粒比金红 石颗粒的吸附作用更强,本研究中纳米 TiO,对 Hg^{2+} 的吸附作用的结果证明了这一推测,锐钛矿颗 粒对 Hg²⁺的吸附作用远高于金红石颗粒^[27].

土壤中可还原态重金属的含量均没有明显的变化,虽然纳米 TiO₂ 对土壤 Fe 和 Mn 形态影响的研究结果显示,2 g·kg⁻¹金红石颗粒和4 g·kg⁻¹锐钛矿颗粒促进了少量可还原态 Fe 和 Mn 的转化^[28],但并没有影响到本研究中 5 种重金属铁锰氧化物结合态的释放,对氧化态 Hg 的影响更大^[29],这可能与本研究中几种重金属可还原态含量相对较低有关,而该土壤中氧化态汞的含量较高.酸可交换态重金属的含量变化,除了 R4 处理组酸可交换 Pb 含量有明显降低以外,其他处理组与对照相比均没有明显变化.

4 结论

本实验条件下,纳米 TiO₂ 对土壤 Cr、Zn、Pb、Cu 和 Cd 的形态转化存在较大的差异. 对 Cr 的影响最大,锐钛矿和金红石两种晶型均可促进土壤中残渣态和可氧化态 Cr 的释放,提高其生态风险;金红石型纳米 TiO₂ 促进土壤中可氧化态和酸可交换态 Pb 的释放;在采用纳米 TiO₂ 材料对 Cr 和 Pb 污染土壤进行修复时需要特别注意. 而锐钛矿颗粒对土壤中 Zn 具有一定的固定作用;两种纳米 TiO₂ 颗粒对土壤中 Cu 和 Cd 则几乎没有影响.

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Environmental Science (monthly)

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