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微米 SiC/石墨烯复合物光催化降解罗丹明 B

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摘要:为发展低耗和环境友好的有机物降解技术,采用光催化还原制备微米级碳化硅(SiC)/石墨烯复合材料,XRD、FTIR、Raman 光谱、XPS 和 SEM 等手段表征其物相组成和形貌结构,并以罗丹明 B(RhB)为模拟污染物,研究了复合材料在可见光照射下的光催化活性和稳定性;通过活性物种捕获实验初步探讨了 RhB 的光催化降解机制. 结果表明,SiC 与石墨烯复合延长了光生电子和光生空穴的寿命,提高了材料的光催化活性与稳定性. 当 SiC/石墨烯配比为 1:0.8 时,光照 60 min 时 RhB 的降解率可以达到 92.7%,降解过程符合一级反应动力学方程. 光催化降解 RhB 过程中,主要活性物种的贡献依次为:光生空穴(h^+)>超氧阴离子自由基(\cdot O₂-)>光生电子(e^-)>羟基自由基(\cdot OH).

关键词:碳化硅(SiC);石墨烯;复合材料;光催化降解;罗丹明 B(RhB)

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Photocatalytic Degradation of Rhodamine B with Micro-SiC/Graphene Composite Under Visible Light Irradiation

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Abstract: To develop low consumption and an environmentally friendly degradation technology for organic pollutants, micro-SiC/graphene composite materials were synthesized by photocatalytic reduction, and the composition and morphology of the prepared materials were characterized by XRD, FTIR, Raman spectroscopy, XPS, and SEM. Rhodamine B (RhB) was selected as the simulated pollutant to investigate the photocatalytic activity and stability of composite materials under visible light irradiation. The degradation mechanism was preliminarily discussed by active species capture experiments. Results show that the lives of photogenerated electron and photogenerated hole of SiC were prolonged when combined with graphene, which improved the photocatalytic activity and stability of composite materials. The degradation efficiency of RhB reached 92.7% with the composite material of SiC/graphene ratio (1:0.8) under 60 min irradiation, and the degradation process accorded with the first-order reaction kinetic equation. The contribution of main active species for photocatalytic degradation followed with a decreasing order of photogenerated hole (h^+), superoxide anion radical (\cdot O₂⁻), photogenerated electron (e^-), and hydroxyl radical (\cdot OH).

Key words: SiC; graphene; composite materials; photocatalytic degradation; Rhodamine B (RhB)

能源过度消费和人口快速增长加剧了能源短缺与环境污染,吸附、电解、膜分离、离子交换、絮凝沉淀、氧化还原、生物处理和光催化降解等废水处理技术不断发展. 近年来,在紫外光照射下催化降解有机物取得了重大进展,但紫外光在太阳光谱中的比例不到 4%,可见光占据 43%. 要使光催化真正成为高效、低耗的处理技术,充分利用可见光尤为关键[1],半导体参与的光催化过程是利用太阳能的一种有效方式. 因此,用响应可见光的半导体光催化剂发展低耗和环境友好的有机物降解技术非常必要[2].

根据化学组成和能带结构,光催化剂包括等离子体光催化剂、含金属光催化剂和无金属光催化剂这3种类型.尽管前两类光催化剂的活性较高,但它们大多含有稀有金属,其来源少、成本高,限制了它们的实际应用.无金属光催化剂储量丰富、价格低廉,具有广阔的发展潜力.近年来发现一些具有可见

光催化活性的无金属半导体光催化剂,如红磷^[3]、 $g-C_3N_4^{[4]}$ 和 $SiC^{[5]}$ 等. 特别是 SiC, 其禁带宽度由晶型决定, 其中立方晶型 SiC(3C-SiC)的禁带宽度为2.4 eV, 可直接利用可见光, 还具有硬度大、耐磨和抗腐蚀等物理特性^[6,7]. 然而, 受表面电子结构和比表面积等因素的影响, 3C-SiC 对可见光的利用率有限, 急需提升其光催化性能. 目前, SiC 基光催化剂性能提升方法主要有形貌调控(如 SiC 纳米线^[8]、SiC 纳米薄片^[9])、离子掺杂(如 B 掺杂^[5])、金属或金属氧化物负载(如 $Pt^{[10]}$ 、 $Cu_2O^{[11]}$)、与其它半导体复合(如 $SiC/BiVO_4^{[12]}$ 、 $SiC/TiO_5^{[13]}$ 、 $SiC/TiO_5^{[13]}$

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C₃N₄^[14])和负载石墨烯(纳米 SiC/石墨烯^[15])等. 石墨烯的电子迁移率高[20 000 cm²·(V·s)⁻¹]及比表面积大(~2 600 m²·g⁻¹)^[16,17],在光催化降解有机废水中能充分发挥其优势.将 SiC 与石墨烯构建复合异质结,可以解决光生电子和光生空穴的复合问题,提高其对污染物的吸附能力. Yang 等^[18]用纳米 SiC 与被还原的氧化石墨烯构建复合物,提高了可见光催化分解水的产氢能力,但光催化剂易失活,限制了其实际应用. Zhu 等^[19]用热解石墨烯包覆的六方晶型 SiC(6H-SiC, 60~120 μm)催化降解罗丹明 B,但是 6H-SiC 的禁带宽度为 3. 45 eV,只能响应紫外光. 若能用响应可见光的微米级 3C-SiC 半导体与石墨烯形成光催化活性强的复合材料,解决纳米材料在实际应用中易失活、难分散和造成二次污染的问题^[20,21],具有重要的理论意义与实用价值.

本研究以 5 μm 的 SiC 和氧化石墨烯为原料,采用光催化还原法制备 SiC/石墨烯复合材料,以染料废水中常见的罗丹明 B 为模拟污染物,考察复合材料的光催化活性,通过活性物种捕获实验初步分析了 SiC/石墨烯对罗丹明 B 的光催化降解机制.

1 材料与方法

1.1 主要试剂与材料

SiC(粒径为 5 μm,陕西西科博尔有限公司);氧化石墨烯(GO,纯度≥98%,中科院成都有机化学有限公司);罗丹明 B(RhB,分析纯,天津市光复精细化工研究所). 其它试剂均为分析纯(中国国药集团).

1.2 主要仪器

紫外可见分光光度计(TU1901,北京普析通用仪器有限责任公司);氙灯光源(300 W,配 420 nm截至滤光片,北京中教金源科技有限公司);X-射线衍射仪(XRD,3 kW,Rigaku Smartlab,日本);傅里叶变换红外光谱仪(FTIR,PerkinElmer,美国);拉曼显微光谱仪(Raman, XploRA PLUS HORIBA Scientific,美国);场发射扫描电子显微镜(SEM,Quanta 400 FEG,美国);X-射线光电子能谱仪(XPS,ESCALAB 250XI,美国);电化学工作站(CHI 660E,上海辰华仪器公司).

1.3 SiC/石墨烯复合材料的制备

将30 mg SiC 微米粒子超声分散 1 h,得到 SiC 分散液;分别取 3、9、15、24 和30 mg GO 于200 mL 20%的乙醇-水溶液中,超声分散 1 h,得到均匀的 GO 分散液.在剧烈搅拌下,分别将 GO 分散液缓慢滴入 SiC 分散液中,在高压汞灯下光催化还原 4 h,用 0.45 μm 滤膜减压抽滤,蒸馏水、无水乙醇洗涤,

得到的固体产物在 60℃ 真空干燥 12 h, 研磨, 获得的 SiC/石墨烯复合材料分别记为 SiC/G(1: 0. 1)、SiC/G(1: 0. 3)、SiC/G(1: 0. 5)、SiC/G(1: 0. 8) 和 SiC/G(1: 1).

1.4 材料表征与光电性能测试

用 XRD 分析复合材料的物相组成和微观结构, 以 Cu 靶 Kα 为 X-射线源; FTIR 和 Raman 光谱分析 复合物的物质组分; SEM 观察复合物的微观形貌; XPS 定性分析复合物表面元素.

用电化学工作站测试复合材料的光电流. 取 10 mg SiC/石墨烯复合材料,超声分散于 100 mL 的 0.5 mol·L $^{-1}$ Na₂SO₄ 溶液中,氙灯照射,以 Pt 片为工作电极和对电极,饱和甘汞电极为参比电极,偏压为 0.6 V,在时间-电流模式下进行测试.

1.5 光催化活性测试

取 10 mg 复合材料于 100 mL 浓度为 10 mg·L⁻¹的 RhB 中,磁力搅拌下暗反应 10 min,加入 0.5 mL H_2O_2 , 氙灯($\lambda \ge 420$ nm) 照射, 每隔 10 min 取反应液样品,离心分离,在 554 nm 处测定上清液的吸光度,计算 RhB 的去除率,考察复合材料的光催化活性. 每个实验重复 3 次,用平均值加减标准偏差表示结果.

1.6 活性物种捕获实验

为研究复合材料对 RhB 的光催化降解机制,以 EDTA 二钠盐(EDTA-2Na, 5 mmol·L⁻¹)、2-丙醇 (IPA, 2.5 mL·L⁻¹)、1, 4-苯醌(BQ, 2 mmol·L⁻¹) 和 $AgNO_3(0.025 \text{ mmol·L}^{-1})$ 分别为活性物种光生空穴(h⁺)、羟基自由基(·OH)、超氧阴离子自由基(·O₂⁻)和电子(e⁻)的捕获剂,加入到光催化降解 RhB 体系中,确定催化降解过程中的主要活性物种.

2 结果与讨论

2.1 材料表征与光电性能测试

2.1.1 XRD 分析

图 1 为 SiC 和 SiC/石墨烯复合材料的 XRD 图谱. 可以看出, 2θ 为 35. 6° 、41. 5° 、60. 1° 和 71. 8° 分别对应 3C-SiC 的(111)、(200)、(220)、(311)和(222)晶面衍射峰 $^{[22]}$;在 2θ 为 10. 3° 处出现明显的GO(001)特征衍射峰,峰强随复合材料中 GO 含量的增加而增加. 在复合材料的 XRD 图谱中,除 GO 的特征衍射峰外,其余基本与纯 SiC 一致,说明 SiC 与石墨烯成功复合,且复合后其晶型未发生明显变化.

2.1.2 FTIR 光谱分析

图 2 为 SiC、GO 和 SiC/石墨烯复合材料的红外光谱图. 可以看出,所有材料在3 455 cm⁻¹和1 636 cm⁻¹均出现—OH 的振动吸收峰,可能来自于材料

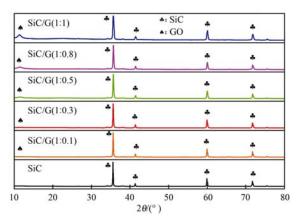


图 1 SiC 和 SiC/石墨烯复合材料的 XRD 图

Fig. 1 XRD patterns of SiC and SiC/graphene composite materials

表面 C—OH 和吸附水中的—OH^[23];871 cm⁻¹处的 宽峰对应 SiC 的 Si—C 振动,随着石墨烯含量的增加,SiC 振动吸收峰逐渐减弱,说明 SiC 表面逐渐被石墨烯包裹.与 GO 比较,可明显观察到复合材料振动吸收峰减少,其原因是复合材料表面 GO 被还原,官能团减少.

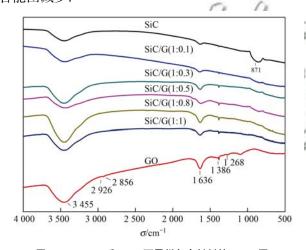


图 2 SiC、GO 和 SiC/石墨烯复合材料的 FTIR 图 Fig. 2 FTIR spectra of SiC, GO and SiC/graphene composite materials

2.1.3 Raman 光谱分析

Raman 光谱进一步证明 SiC 和石墨烯复合物的存在及 GO 的还原情况(图 3). 可以看出,在1 361.2 cm⁻¹和1 585.9 cm⁻¹处 SiC/G(1:0.8)和 GO 都分别出现显著的 D 带和 G 带峰,D 峰代表无序的碳原子,G 峰代表 sp^2 杂化轨道的碳原子,D 峰和 G 峰强度比(I_D/I_G)可用于评估石墨烯的还原程度^[24].与GO 比较,在光催化还原石墨烯过程中 sp^2 杂化轨道的碳原子含量增加和无序碳原子的减少,使得 SiC/G(1:0.8)的 G 峰强度大大增加;根据谱图的 D 峰和 G 峰强度,计算获得 GO 和 SiC/G(1:0.8)的 I_C/I_D 分别为 1.06 和 1.20,表明光照后复合物中的 GO 被成功还原. SiC/G(1:0.8)在 789.8 cm⁻¹和 969 cm⁻¹

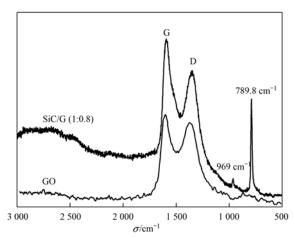


图 3 GO 和 SiC/G(1:0.8)的 Raman 光谱图

Fig. 3 Raman spectra of GO and SiC/G (1:0.8)

出现 3C-SiC 的特征峰,表明复合材料中 SiC 和石墨 烯的同时存在.

2.1.4 XPS 分析

图 4 表示 SiC/G(1:0.8)的 XPS 谱图. C 1s 谱图中 284.8 eV 对应 C—C 键, 286.8 eV 和 288.6 eV 分别对应 C—O 和 O =C—O 键^[25], 283.2 eV 对应 3C-SiC 中的 C—Si 键^[26]; Si 2p 谱图中 101.2 eV 和 103.5 eV 分别对应 Si—C 和 Si—O 键. XPS 分析表明 SiC/G 复合材料被成功制备.

2.1.5 SEM 分析

图 5 表示 SiC 和 SiC/石墨烯复合材料的 SEM 图. 可以看出, SiC 的粒径约为 5 μm, 表面光滑且无杂质[图 5(a)和 5(b)];与石墨烯复合后, SiC 表面不再光滑,少量褶皱状石墨烯负载在 SiC 上[图 5(c)和 5(d)];随着复合材料中石墨烯含量的增加, SiC 颗粒被成片的石墨烯包裹,分散独立的 SiC 颗粒逐渐减少[图 5(e)和 5(h)];当 SiC/G 配比达到1:0.8时, SiC 颗粒分散在石墨烯结成的大网上,但可清晰看到石墨烯的褶皱状和其上镶嵌的 SiC 轮廓[图 5(i)和 5(j)];当 SiC/G 配比达到1:1时, 石墨烯的褶皱状几乎消失,包覆 SiC 的石墨烯也变得更加致密且厚实[图 5(k)和 5(1)],这可能影响 SiC 对光的吸收,进而降低光催化效率.

2.1.6 光电性能分析

在可见光(λ≥420 nm)照射下测试 SiC、SiC/G (1:0.8)和 SiC/G(1:1)的光电流,获得如图 6 所示的结果. 光照时 3 种材料均产生稳定的光电流,但复合材料的光电流明显高于纯 SiC,表明复合材料中SiC 产生的光生电子与光生空穴得到有效分离. SiC/G(1:1)产生的光电流低于 SiC/G(1:0.8),其原因可能是 SiC 的有效受光面积减少,进而降低光电流的产生.

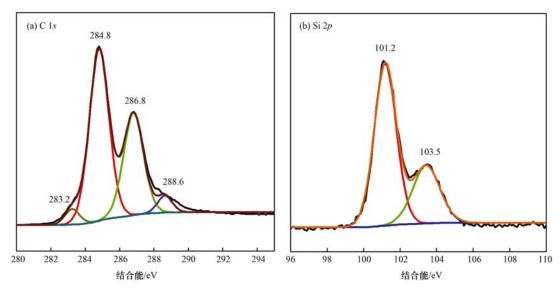
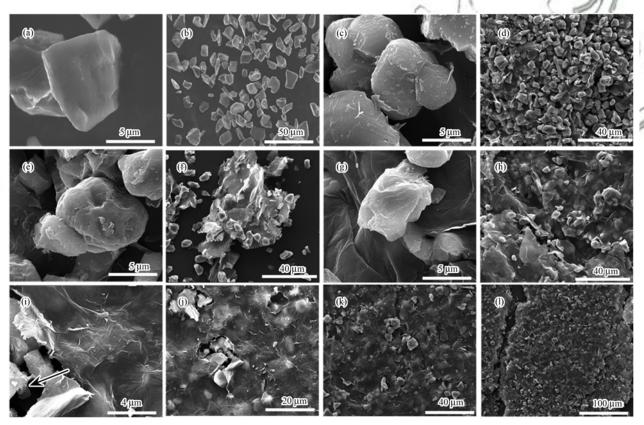


图 4 SiC/G(1:0.8)的 XPS 图谱

Fig. 4 XPS spectra of SiC/G (1:0.8)



 $\label{eq:continuous} \begin{tabular}{ll} (a) \sim (b) : &SiC; (c) \sim (d) : &SiC/G(1:0.1); (e) \sim (f) : &SiC/G(1:0.3); \\ (g) \sim (h) : &SiC/G(1:0.5); (i) \sim (j) : &SiC/G(1:0.8); (k) \sim (l) : &SiC/G(1:1) \\ \end{tabular}$

图 5 SiC 和 SiC/石墨烯复合材料的 SEM 图

Fig. 5 SEM images of SiC and SiC/graphene composite materials

2.2 罗丹明 B 的光催化降解

比较不同条件下石墨烯、SiC、SiC/石墨烯复合材料对 RhB 的作用情况,获得如图 7 所示的结果.可以看出,纯 SiC 对 RhB 的降解率仅为 20% (曲线 2);随着材料中石墨烯含量的增加,RhB 浓度的下降趋势逐渐增大(曲线 3、4、6、9 和 10),说明提高SiC 与石墨烯的配比可以显著提升材料的光催化降

解能力,SiC/G(1:0.8)和 SiC/G(1:1)的光催化活性非常接近,光照 60 min 时 RhB 的去除率达到92.7%. 在纯石墨烯存在下,光照和黑暗反应时 RhB浓度显著降低,且变化趋势一致(曲线 7 和 8),表明石墨烯强烈吸附 $RhB^{[27]}$,但无明显的光催化降解作用. 在只加 H_2O_2 光照 60 min 时,RhB 溶液的降解率约为 10% (曲线 1);在 SiC/G(1:0.8)存在下,不加

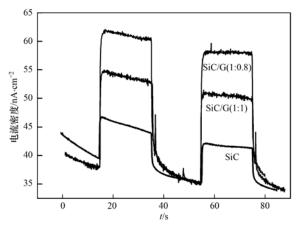


图 6 可见光照射下 SiC、SiC/G(1:0.8)和 SiC/G(1:1)的瞬态光电流曲线

Fig. 6 Transient photocurrent curves of SiC, SiC/G (1:0.8), and SiC/G (1:1) under visible light radiation

 H_2O_2 光照时 RhB 浓度下降缓慢(曲线 5),说明 H_2O_3 对 RhB 光解具有辅助作用.

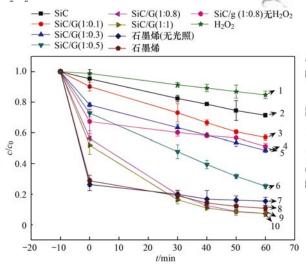


图 7 SiC、石墨烯、SiC/G 对 RhB 的可见光降解

Fig. 7 Degradation of RhB with SiC, graphene, and SiC/G composites under visible light irradiation

拟合实验数据,获得不同配比复合材料催化降解 RhB 的动力学特征(图 8). 可以看出,SiC、SiC/石墨烯复合材料催化降解 RhB 均符合一级反应动力学方程,决定系数(R^2)均大于 0.97,获得 SiC/G (1:0.1)、SiC/G(1:0.3)、SiC/G(1:0.5)、SiC/G (1:0.8)和 SiC/G(1:1)催化降解 RhB 的速率常数分别为: 0.0077、0.0079、0.0179、0.0340和0.0309 min -1,远高于纯 SiC 催化降解的速率常数(0.0048 min -1). 比较发现,SiC/G(1:0.8)与FeOCl/H₂O^[28]、Fe₃O₄@ ZnO-Ce^[29]催化降解 RhB的能力相当,高于 Zhu 等^[19]用热解石墨烯包覆的6H-SiC 在紫外光照下的降解速率(0.021 min -1).

图 9 考察了复合材料的光催化稳定性,发现重复使用 SiC/G(1:0.8)3次,RhB 的降解率保持

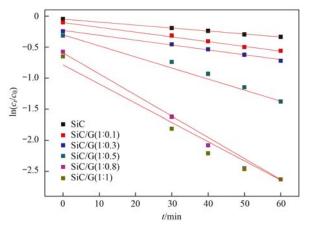


图 8 SiC、SiC/G 光催化降解 RhB 的动力学曲线

Fig. 8 Photocatalytic degradation kinetic curves of RhB ${\rm with~SiC~and~SiC/graphene~composites}$

在 90% 左右, 说明复合材料具有较高的光催化稳定性.

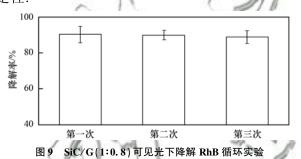


Fig. 9 Cycling runs for RhB degradation with SiC/G
(1:0.8) under visible light irradiation

2.3 罗丹明 B 的光催化降解机制

图 10 为活性物种捕获实验结果. 可以看出,当加入 EDTA-2Na、IPA、BQ 和 AgNO₃ 降解 60 min时,RhB 的降解效率从 92.7% 分别下降至 49.5%、86.2%、61.1% 和 67.3%,说明在光降解过程中起作用的活性基团依次为 $h^+ > \cdot O_2^- > e^- > \cdot OH$.

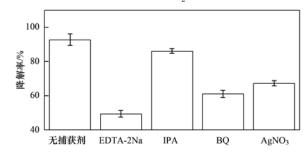


图 10 SiC/G(1:0.8) 光催化降解 RhB 过程中活性物种的捕获

Fig. 10 Trapping experiments of active species in photocatalytic degradation of RhB with SiC/G (1:0.8)

基于实验和材料表征结果,推测 SiC/石墨烯复合材料光催化降解 RhB 的可能机制(图 11). SiC 表面被大量石墨烯包覆,在可见光照射下 SiC 被激发产生 e⁻和 h⁺,e⁻转移到 SiC 表面的石墨烯上并通过石墨烯进行传导,有效地避免了 e⁻和 h⁺复合,提

高了 e^- 的分离效率,延长了 e^- 和 h^+ 的寿命. 部分裸露 SiC 表面的 h^+ 可直接氧化石墨烯表面的 RhB;被激发产生的 e^- 可以与石墨烯表面吸附的 RhB 和 O_2 反应,降解 RhB 或生成 $\cdot O_2^-$. H_2O_2 在整个反应体系中,可辅助复合材料产生更多的 $\cdot O_2^-$ 和 $\cdot OH$,共同作用于 RhB,提高了 RhB 的降解效率.

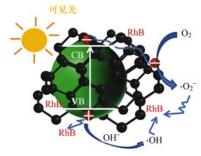


图 11 SiC/石墨烯光催化降解 RhB 的可能机制

Fig. 11 Possible photocatalytic degradation mechanism of RhB with SiC/graphene composite

3 结论

通过光催化还原法成功制备 SiC/G 复合材料, 在可见光照射下 SiC/G(1:0.8)对 RhB 降解具有明显的催化效果,降解过程符合一级反应动力学特征. SiC 与石墨烯复合有效提高了光生电子和光生空穴的分离效率,延长了它们的寿命,显著提升了 RhB的光催化降解.

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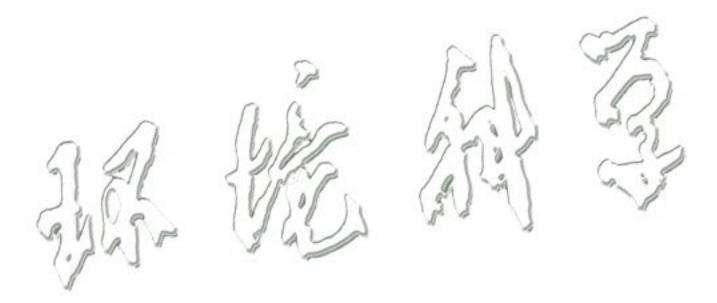
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