

Y³⁺ 掺杂的钼酸铋钠电极制备 及降解西地那非反应机理

孙嘉鸿¹, 李佳依¹, 付中东², 王洪宁², 冯威¹

(1. 吉林大学 新能源与环境学院, 长春 130021; 2. 吉林省拓达环保设备工程有限公司, 长春 130062)

摘要: 通过压片法制备 NaBi(MoO₄)₂ 电极, 通过掺杂 Y³⁺ 对其进行改性, 采用 X 射线衍射 (XRD)、扫描电子显微镜 (SEM)、透射电子显微镜 (TEM)、紫外可见漫反射光谱 (UV-Vis-DRS) 和电子自旋共振 (ESR) 等方法对电极形貌、结构和电化学性能进行表征, 并研究光-电协同催化氧化降解西地那非的性能和反应机理. 实验结果表明: NaBi(MoO₄)₂ 基电极为片层结构, 具有较高的纯净度, 尺寸约为 37.5~24.6 nm; 在浓度为 0.5 mol/L 的最佳电解质 Na₂SO₄ 溶液中, 最佳偏压为 1.5 V, 70 min 时可完全去除西地那非.

关键词: 钼酸铋钠; 西地那非; 光电催化; 压片电极

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Preparation of Y³⁺-Doped Sodium Bismuth Molybdate Electrode and Degradation Mechanism of Sildenafil

SUN Jiahong¹, LI Jiayi¹, FU Zhongdong², WANG Hongning², FENG Wei¹

(1. College of New Energy and Environment, Jilin University, Changchun 130021, China;

2. Jilin Tuoda Environmental Protection Equipment Engineering Co., Changchun 130062, China)

Abstract: The NaBi(MoO₄)₂ electrode was prepared by the pressed sheet method, and the material was modified by doping with Y³⁺. The morphology, structure and electrochemical properties of the electrodes were characterized by X-ray diffraction (XRD), scanning electron microscope (SEM), transmission electron microscope (TEM), ultraviolet-visible-diffuse reflectance spectrum (UV-Vis-DRS) and electron spin resonance (ESR), and the performance and reaction mechanism of the photocatalytic degradation of sildenafil were also investigated. The experimental results show that the NaBi(MoO₄)₂-based electrode has a lamellar structure and high purity, with the size ranging from about 37.5 nm to 24.6 nm. In the optimal electrolyte Na₂SO₄ solution with a concentration of 0.5 mol/L and the optimal bias voltage of 1.5 V, sildenafil can be completely removed after 70 min.

Keywords: sodium bismuth molybdate; sildenafil; photocatalysis; pressed sheet electrode

西地那非(C₂₂H₃₀N₆O₄S)为白色结晶粉末, 密度为 1.39 g/cm³, 主要用于治疗心血管疾病^[1-2]. 人体服用后, 在其尿液和粪便中含有约 80% 给药量的西地那非. 若将未完全处理的西地那非废水排放

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第一作者简介: 孙嘉鸿(2000—), 男, 汉族, 硕士研究生, 从事环境功能材料的研究, E-mail: sjh22@mails.jlu.edu.cn. 通信作者简介: 冯威(1972—), 男, 汉族, 博士, 教授, 从事环境功能材料的研究, E-mail: weifeng@jlu.edu.cn.

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到环境中,将对生态环境产生不良影响^[3].

与物理、化学和生物等现代处理方法相比,光催化氧化技术具有反应条件温和、操作简单、无二次污染等优点,是解决环境污染的有效途径^[4-5].利用半导体光/电催化的方法,半导体光催化产生的光生电子和空穴可有效生成具有强氧化作用的羟基自由基($\cdot OH$)和超氧自由基($O_2^{\cdot -}$)等基团,从而有效降解有机污染物^[6-9].但常规的光催化反应具有反应效率低和光生电子-空穴对复合率高等缺点,从而降低了光催化反应效率.通过施加偏压,可有效抑制光生电子-空穴复合,施加的电势场可诱导光生电子-空穴向相反方向移动,提高分离效率,有效实现污染物的高效降解^[10].压片电极法是制备电极的常用方法之一,通过将电极材料用聚四氟乙烯粘合在不锈钢网状集流体上,用压片机压合,即可制成电子高性能传输电极^[11-12].本文通过对 $NaBi(MoO_4)_2$ 进行稀土钇离子掺杂,破坏其电偶极矩的稳定性,以增强其压电性能,并通过 X 射线衍射(XRD)、扫描电子显微镜(SEM)、透射电子显微镜(TEM)、紫外可见漫反射光谱(UV-Vis-DRS)和电子自旋共振(ESR)等表征方法,分析其元素组成和晶体结构,进一步研究压片电极对西地那非的光-电协同降解性能.

1 实验

1.1 试剂、材料和仪器

五水合硝酸铋($Bi(NO_3)_3 \cdot 5H_2O$),六水合硝酸钇($Y(NO_3)_3 \cdot 6H_2O$),钼酸钠($Na_2MoO_4 \cdot 2H_2O$),无水乙醇, Na_2SO_4 , 聚四氟乙烯乳液,乙炔炭黑,硝酸, $NaCl$ 和高氯酸 $HClO_4$ 均为市售分析纯试剂;去离子水为实验室自制;不锈钢丝网(200目),铂电极夹,铂电极和 $AgCl$ 参比电极均购于江苏环亚电热仪表有限公司.

X 射线粉末衍射仪(德国 Bruker 公司); XL-30 ESEM FEG 型场发射扫描电子显微镜(美国 FEI 公司); Tecnai G220 型场发射透射电子显微镜(美国 FEI 公司);压片机(天津天光光学仪器有限公司); ESC ALAB 250 型 X 射线光电子能谱仪(美国 Thermo 公司); UV3600 型紫外-可见分光光度计(日本岛津公司); API3200 型液相质谱仪(日本岛津公司); SBW 型直流电源(上海稳压器厂).

1.2 钼酸铋钠压片电极的制备

1.2.1 $NaBi(MoO_4)_2$ 电极材料的制备

向 20 mL 纯净水中加入 2.5 mmol $Na_2MoO_4 \cdot 2H_2O$ 和 5 mmol $Bi(NO_3)_3 \cdot 5H_2O$, 搅拌混合至混合物为乳白色混悬液.用 HNO_3 将混合溶液的 pH 值调至 6.0 后转移到 50 mL 水热反应釜中.在 200 °C 水热反应 24 h. 去除上清液,将沉淀的白色固体用无水乙醇离心循环洗涤 3 次.上述步骤完成后,将所得白色固体放入烘箱,80 °C 干燥 6 h, 即可得纯净 $NaBi(MoO_4)_2$ 压片电极材料,命名为 NBMO.

1.2.2 Y^{3+} 掺杂 $NaBi(MoO_4)_2$ 电极材料的制备

向 1.2.1 中乳白色混悬液中加入物质的量比(基础参照物为 $Na_2MoO_4 \cdot 2H_2O$)为 1.5 : 100 的 $Y(NO_3)_3 \cdot 6H_2O$, 与 1.2.1 中相同条件和步骤,用水热法即可制得 Y^{3+} 掺杂的 $NaBi(MoO_4)_2$ 压片电极材料,命名为 1.5Y-NBMO.

1.2.3 压片电极的制备

先将不锈钢丝网(200目)剪成直径为 4 cm 的圆形,再将前两步制成的电极材料、聚四氟乙烯乳液和乙炔炭黑以 m (电极材料) : V (聚四氟乙烯乳液) : m (乙炔炭黑) = 0.2 : 200 : 0.06(g, μL , g) 制成黏稠的糊状电极材料前驱体,并将其均匀涂抹在圆形不锈钢丝网中间.在烘箱中 50 °C 干燥 30 min 后取出,前后两面垫上称量纸后,放入压片机中.在 10 kPa 的压力下压 30 s 后将材料翻转,继续压片 30 s,即可得到力学性能良好的压片电极.

1.3 实验方法

将西地那非用超纯水配制成 10 mg/L 的溶液.先将制备的压片电极与直流电源正极相连,作为光阳极;再将 1 cm \times 1 cm 的铂电极与直流电源负极相连,作为对电极.以 1.5 V 电压, 0.5 mol/L Na_2SO_4 电解质为催化测试背景,在透光率为 85% 的石英电解池中,进行光电催化降解测试.每隔一段时间,抽取 10 mL 反应溶液,经半透膜过滤后,存入测试管中待检测.

1.4 分析方法

用 XRD 法分析电极材料的晶体结构及晶相组成,用 SEM-EDS 法分析材料的表面形貌和元素组成,用 TEM 法分析材料的微观形貌和晶格结构,用 UV-Vis-DRS 法分析材料光学性质,用液相色谱仪监测西地那非在光电催化降解过程中的质量浓度变化.

2 结果与讨论

2.1 物相表征

2.1.1 XRD 结果

图 1 为 NBMO 和 1.5Y-NBMO 的 XRD 谱. 由图 1 可见, 1.5Y-NBMO 的特征峰分别对应纯 $\text{NaBi}(\text{MoO}_4)_2$ 的 (112), (004), (200), (211), (114), (213), (204), (220), (116), (312) 和 (224) 等晶面, 表明 $\text{NaBi}(\text{MoO}_4)_2$ 的晶格结构未被破坏. 此外, 还出现了对应 Y_2O_3 晶面的 (20-2), (202), (310), (11-3), (004), (114), (71-2) 衍射峰和对应 $\text{YO}_{1.458}$ 晶面的 (302), (003), (112) 衍射峰. 可见, 1.5Y-NBMO 在合成过程中产生了钇的氧化物. 同时 $\text{NaBi}(\text{MoO}_4)_2$ 主要衍射峰强度随 Y^{3+} 的掺杂而下降, 表明掺杂的 Y^{3+} 有效取代了 $\text{NaBi}(\text{MoO}_4)_2$ 中 Bi 的晶格位, 使 $\text{NaBi}(\text{MoO}_4)_2$ 主要衍射峰强度变弱^[13-14]. 根据 Scherrer 公式估算纯 $\text{NaBi}(\text{MoO}_4)_2$ 材料和 $\text{NaBi}(\text{MoO}_4)_2$ 基材料的晶粒尺寸分别为 NBMO (37.5 nm) 和 1.5Y-NBMO (24.6 nm). 因此, Y^{3+} 掺杂有效抑制了晶粒的生长.

2.1.2 SEM 结果

图 2 为 NBMO 和 1.5Y-NBMO 的 SEM 照片. 由图 2 可见, $\text{NaBi}(\text{MoO}_4)_2$ 基电极为片层结构, 呈块状分布, 掺杂 Y^{3+} 对电极的形貌影响较小. 纯 $\text{NaBi}(\text{MoO}_4)_2$ 电极含 Na, Bi, Mo, O 4 种元素, 掺杂 Y^{3+} 后, 电极中仅多了一种元素 Y, 可见掺杂后的电极在保持原有层状结构的基础上, 纯度仍然很高.

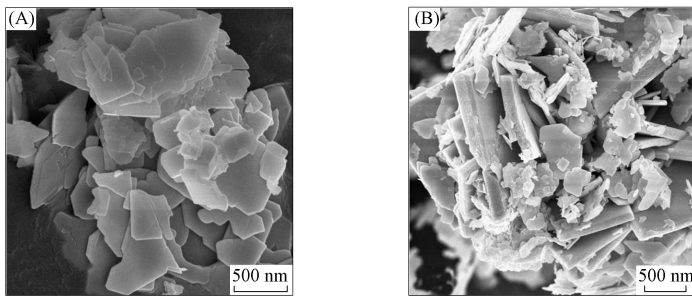


图 2 NBMO(A)和 1.5Y-NBMO(B)的 SEM 照片

Fig. 2 SEM images of NBMO (A) and 1.5Y-NBMO (B)

2.1.3 TEM 结果

图 3 为 NBMO 和 1.5Y-NBMO 的 TEM 照片. 由图 3 可见, $\text{NaBi}(\text{MoO}_4)_2$ 电极呈片状分布, 与 SEM 结果一致. TEM 照片中宽度为 0.313, 0.289, 0.264 nm 的晶格条纹分别对应 $\text{NaBi}(\text{MoO}_4)_2$ 的 (112), (004), (200) 晶面, 与 XRD 结果相符. 衍射花样表明, NBMO 和 1.5Y-NBMO 均为混晶结构. 由图 3(B) 可见, 掺杂 Y^{3+} 对 $\text{NaBi}(\text{MoO}_4)_2$ 电极的形貌影响较小, 仍可观察到原有的 $\text{NaBi}(\text{MoO}_4)_2$ 晶格条纹. 此外, 还可观察到与钇的氧化物相对应的晶格条纹^[15]. 宽度为 0.393, 0.293, 0.306 nm 的晶格条纹分别对应 Y_2O_3 的 (202), (003), (302) 晶面, 与 XRD 结果相符.

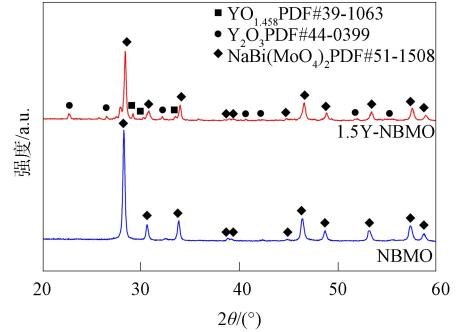


图 1 NBMO 和 1.5Y-NBMO 的 XRD 谱

Fig. 1 XRD patterns of NBMO and 1.5Y-NBMO

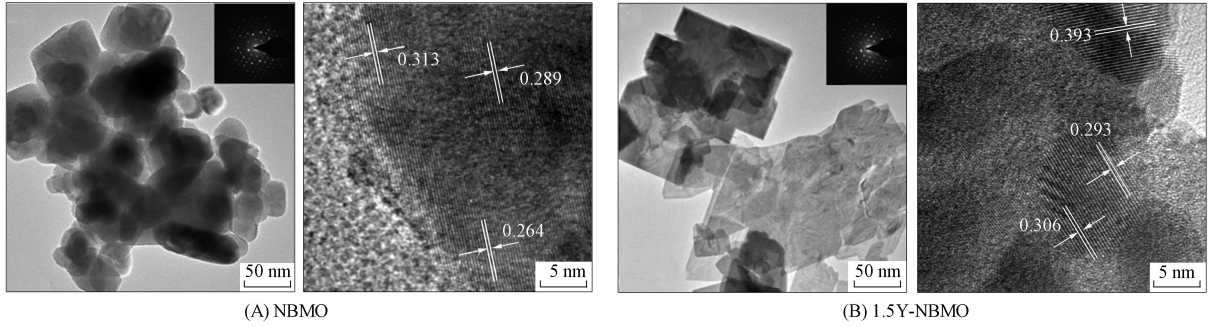


图 3 NBMO 和 1.5Y-NBMO 的 TEM 照片
Fig. 3 TEM images of NBMO and 1.5Y-NBMO

2.1.4 UV-Vis-DRS 结果

图 4 为 NBMO 和 1.5Y-NBMO 的 UV-Vis-DRS 谱. 由图 4 可见: NaBi(MoO₄)₂ 电极的吸收带边为 421.77 nm, 表明该电极对可见光有较强的吸收能力; 1.5Y-NBMO 电极的吸收带边为 821.19 nm, 可见光的吸收范围变大, 表明掺杂适量的 Y³⁺ 后, NaBi(MoO₄)₂ 基电极的可见光响应能力显著提升^[11].

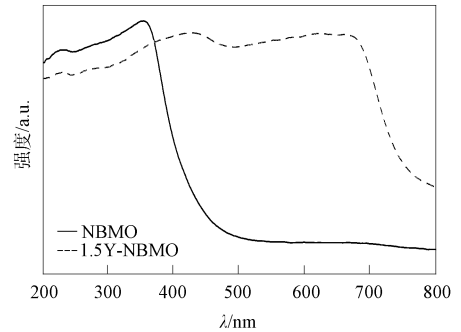


图 4 NBMO 和 1.5Y-NBMO 的 UV-Vis-DRS 谱
Fig. 4 UV-Vis-DRS of NBMO and 1.5Y-NBMO

2.2 降解性能的影响因素

2.2.1 降解性能

将西地那非的光电催化降解过程进行一级反应动力学拟合, 结果如图 5 所示, 其中 PS, PC, EC 和 PEC 分别表示光降解实验值、光催化降解实验值、电催化降解实验值和光电催化降解实验值. 由图 5 可见, R² 最小值为 0.990, 表明西地那非的质量浓度变化与时间呈良好的线性关系, 可认为西地那非的光电催化降解反应遵循一级反应动力学过程. 对应的光电催化反应的快慢速率常数分别为 0.05(1.5Y-NBMO)和 0.000 2(NBMO). 可见, 以摩尔分数为 1.5% 的 Y³⁺ 掺杂的 NaBi(MoO₄)₂ 电极催化降解效果最优, 且光电催化反应速率常数 K 值为纯 NaBi(MoO₄)₂ 电极 K 值的 260 倍.

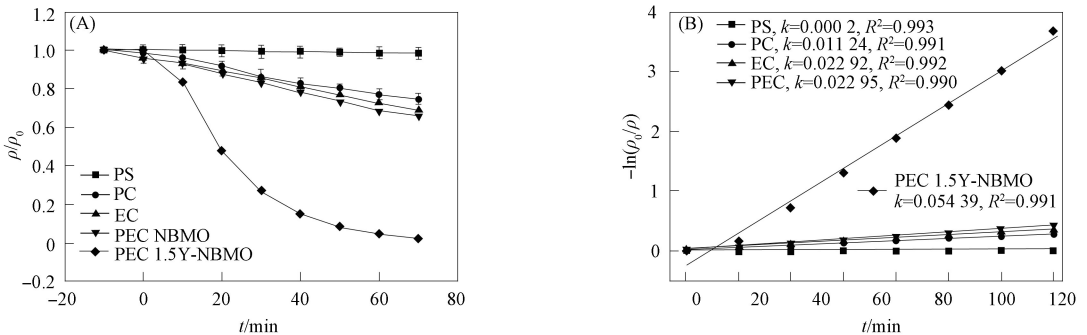


图 5 NBMO 和 1.5Y-NBMO 对西地那非的光电催化降解曲线(A)和动力学拟合结果(B)

Fig. 5 Photocatalytic degradation curves (A) and kinetic fitting results (B) of sildenafil by NBMO and 1.5Y-NBMO

2.2.2 电解质溶液影响

在西地那非质量浓度为 10 mg/L, 电解质溶液浓度为 0.5 mol/L, 偏压为 1.5 V 的条件下进行光电催化降解实验, 考察不同种类电解质对西地那非降解性能的影响, 结果如图 6(A)所示. 由 6(A)可见, 1.5Y-NBMO 压片电极在 HClO₄ 电解质溶液中的效率最高, 仅 20 min 时, 西地那非的降解率已达 95%, 40 min 时几乎完全降解. 降解速率最快的原因是 HClO₄ 的强氧化性破坏了西地那非的分子结构. Na₂SO₄ 电解质的降解能力次之, 约 70 min 将西地那非完全去除. NaCl 溶液对西地那非的降解效率最低, 约 120 min 时将西地那非完全去除, 原因可能为 Cl⁻ 占据了电极的活性位点^[16].

不同浓度的 Na_2SO_4 作为电解质溶液对西地那非降解性能的影响如图 6(B) 所示. 由图 6(B) 可见: 当 Na_2SO_4 浓度为 0.1 mol/L 时, 1.5Y-NBMO 电极光电催化降解西地那非的效率最低, 110 min 时降解率约为 77%; 当 Na_2SO_4 电解质溶液浓度为 0.5 mol/L 时, 1.5Y-NBMO 电极光电催化降解西地那非的效率最高, 70 min 时降解率约为 100%; 当 Na_2SO_4 电解质溶液浓度为 1.0 mol/L 时, 1.5Y-NBMO 电极完全降解西地那非需约 120 min. 可见当电解质浓度过高时, 电极材料位点会被结晶占据, 从而影响降解率^[17]. 因此, 确定溶液最佳电解质浓度为 0.5 mol/L.

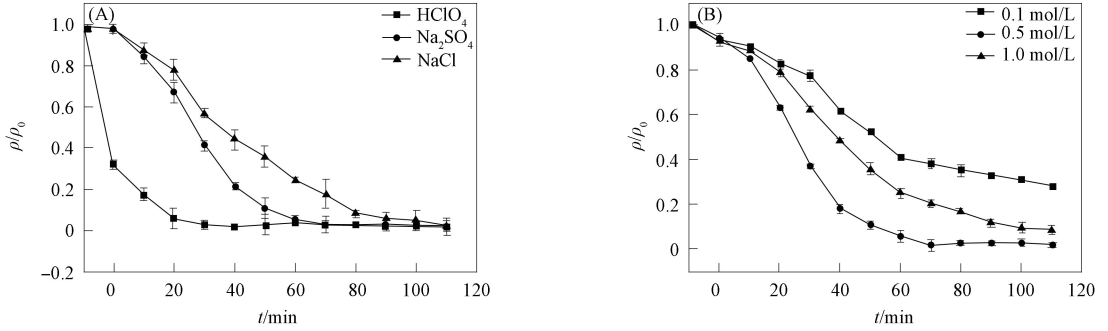


图 6 电解质种类(A)和浓度(B)对西地那非降解性能的影响

Fig. 6 Effect of electrolyte type (A) and concentration (B) on degradation performance of sildenafil

2.2.3 偏压影响

在西地那非质量浓度为 10 mg/L, 0.5 mol Na_2SO_4 电解质的条件下, 通过添加不同的直流电压考察偏压对降解西地那非的影响, 结果如图 7 所示. 由图 7 可见: 当外加偏压为 0.5 V 时, 西地那非的降解率最低, 110 min 时降解率仅为 75%; 当偏压为 1.5 V 时, 降解率最高, 70 min 时, 西地那非几乎完全降解; 继续增加偏压, 电极表面开始析出气泡, 降解率逐渐降低, 当外加偏压为 2.5 V 时, 电极表面出现大量气泡, 溶液浑浊, 100 min 降解率小于 90%. 这是因为通过外加偏压可将电子-空穴进行分离, 加快西地那非降解, 当偏压为 1.5 V 时, 反应中电子-空穴对分离最彻底, 氧化和还原位点相互分隔开, 互不影响, 此时降解率最高^[18]. 继续增大偏压, 光电极的空间电荷层可能发生重新排列和分布, 导致光电催化反应中光生载流子的数量减少, 从而西地那非降解率下降^[19]. 并且当外加偏压增加到一定程度时, 电极表面会产生大量气泡, 将西地那非与光电极上活性位点隔绝, 使降解率明显下降^[20]. 因此, 反应的最佳偏压为 1.5 V.

2.2.4 稳定性

图 8 为 1.5Y-NBMO 电极对西地那非的 9 次循环降解率. 由图 8 可见, 1.5Y-NBMO 电极在 9 次循环实验中均表现出良好的降解性能. 9 次循环降解实验后, 1.5Y-NBMO 电极的西地那非降解率由 99.5% 下降至 97.4%, 下降幅度较小, 这是由于 1.5Y-NBMO 电极具有良好的机械稳定性, 因此在循环降解过程中保持了良好的降解效率.

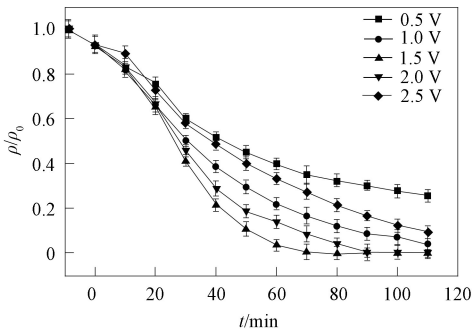


图 7 偏压对西地那非降解性能的影响

Fig. 7 Effect of bias voltage on degradation performance of sildenafil

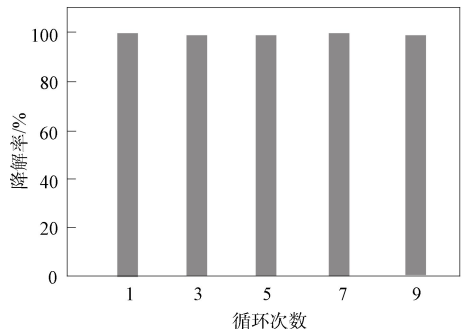


图 8 1.5Y-NBMO 电极对西地那非的 9 次循环降解率

Fig. 8 Nine cycles degradation rate of sildenafil by 1.5Y-NBMO electrode

2.2.5 降解机理

为考察 1.5Y-NBMO 电极在光电催化过程中的降解机理, 对其进行电子自旋共振(ESR)实验, 结果如图 9 所示. 由图 9 可见, 1.5Y-NBMO 电极比 NBMO 电极具有更明显的 $\cdot OH$ 和 $O_2^{\cdot -}$ 信号, 表明掺杂 Y^{3+} 增强了 NBMO 电极产生 $\cdot OH$ 和 $O_2^{\cdot -}$ 能力, 从而有助于提升降解效果.

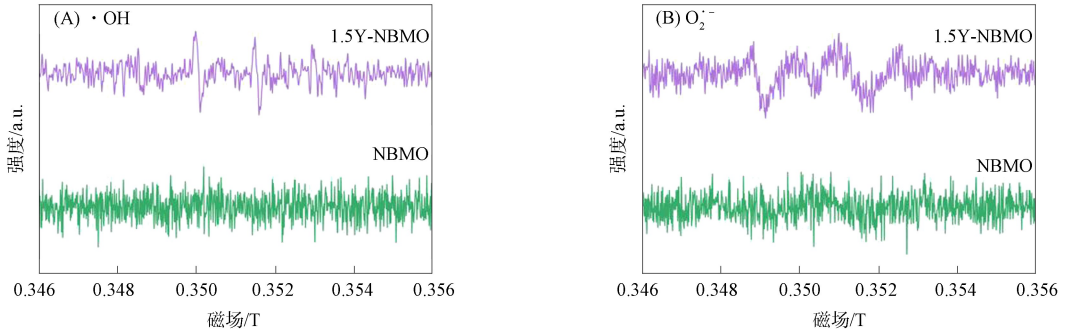


图 9 NBMO 和 1.5Y-NBMO 电极在光电过程中的 ESR 谱

Fig. 9 ESR spectra of NBMO and 1.5Y-NBMO electrodes during photoelectric process

1.5Y-NBMO 电极的光电催化降解机理如图 10 所示. 由图 10 可见, 光阳极在可见光照射下, 发生电子-空穴分离, 电子在偏压作用下, 由阳极转移到阴极与氧气结合生成 $O_2^{\cdot -}$, 将西地那非降解; 一部分空穴直接降解西地那非, 另一部分空穴氧化 OH^- 生成 $\cdot OH$, $\cdot OH$ 氧化西地那非实现降解. 在偏压 1.5 V, 0.5 mol/L Na_2SO_4 为电解质的条件下, 1.5Y-NBMO 电极明显高于纯 $NaBi(MoO_4)$ 电极的光电催化降解性能. 原因可能为: 1) Y^{3+} 替换了 $NaBi(MoO_4)$ 晶格中的 Bi^{3+} , 稀土离子的上转换效应有效压缩了半导体能带; 2) 钇作为稀土元素具有多电子能级, 可形成多电子阱对光生电子进行捕获, 从而促进光生空穴-电子对的分离^[17,21-22]. 这两方面均可强化光电催化体系中电子-空穴分离, 提高 1.5Y-NBMO 光电极对西地那非的降解率.

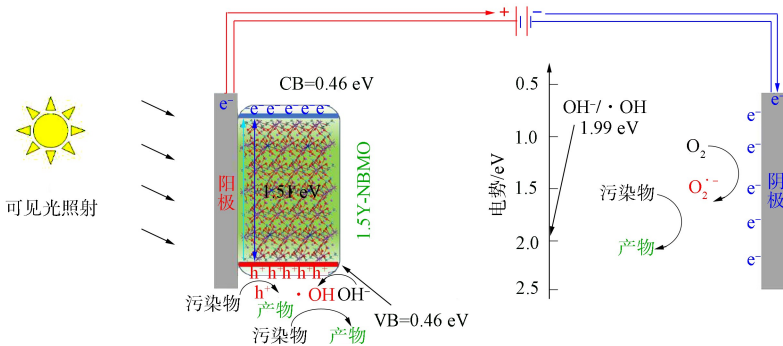


图 10 1.5Y-NBMO 电极的光电催化降解机理示意图

Fig. 10 Schematic diagram of photocatalytic degradation mechanism of 1.5Y-NBMO electrode

综上所述, 本文对 $NaBi(MoO_4)$ 及其掺杂 Y^{3+} 后的压片电极进行了光电催化降解新型污染物西地那非的环境净化性能研究. 结果表明: 以摩尔分数为 1.5% 的 Y^{3+} 掺杂 $NaBi(MoO_4)$ 形成的电极对降解性能有显著提升; 在偏压 1.5 V, 0.5 mol/L Na_2SO_4 为电解质的条件下, 1.5Y-NBMO 电极明显高于纯 $NaBi(MoO_4)$ 电极的光电催化降解性能.

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The editorial board of the journal consists of 52 members who are all well-known professors. Among them there are five Academicians of the Chinese Academy of Sciences, they are Professors FENG Shouhua (chemist), SHEN Jiacong (chemist), XU Ruren (chemist), ZOU Guangtian (physicist) and YU Jihong (chemist). Professor FENG Shouhua holds the post of the editor-in-chief.

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