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NiFe layered double-hydroxide nanoparticles for efficiently enhancing performance of BiVO₄ photoanode in photoelectrochemical water splitting

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ABSTRACT

A bismuth vanadate (BiVO₄) photoanode with a cocatalyst consisting of NiFe layered double-hydroxide (NiFe-LDH) nanoparticles was fabricated for photoelectrochemical (PEC) water splitting. NiFe-LDH nanoparticles, which can improve light-absorption capacities and facilitate efficient hole transfer to the surface, were deposited on the surface of the BiVO₄ photoanode by a hydrothermal method. All the samples were characterized using X-ray diffraction, scanning electron microscopy, and diffuse-reflectance spectroscopy. Linear sweep voltammetry and current-time plots were used to investigate the PEC activity. The photocurrent response of NiFe-LDH/BiVO₄ at 1.23 V vs the reversible hydrogen electrode was higher than those of Ni(OH)₂/BiVO₄, Fe(OH)₂/BiVO₄ and pure BiVO₄ electrodes under visible-light illumination. NiFe-LDH/BiVO₄ also gave a superior PEC hydrogen evolution performance. Furthermore, the stability of the NiFe-LDH/BiVO₄ photoanode was excellent compared with that of the bare BiVO₄ photoanode, and offers a novel method for solar-assisted water splitting.

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In recent years, solar-driven photoelectrochemical (PEC) water splitting, which is an efficient, environmentally friendly, and sustainable technique, has attracted considerable attention [1,2]. Improvements in the sunlight-absorption properties, conversion efficiencies, and catalytic activities of photoelectrode materials are crucial in the use of PEC for transforming solar energy into chemical energy [3,4]. Various photoelectrode materials such as TiO₂ [5,6], WO₃ [7,8], and α -Fe₂O₃ [9,10] have been widely studied and used for efficient production of hydrogen at the photocathodes of PEC cells. The use of BiVO₄ in PEC water splitting is increasing because of its small bandgap ($E_g = 2.4\text{--}2.5$ eV), which enables significant visible-light absorp-

tion, and its high valence band, which provides an overpotential that is sufficient for water oxidation [11–13]. However, weak electron–hole separation and a poor transport yield restrict the overall quantum efficiency in PEC processes when pure BiVO₄ is used as the photoanode [14].

To address these limitations, diverse strategies for modification of BiVO₄, such as doping [15,16], addition of a cocatalyst [17,18], and construction of heterojunction structures [19,20] have been developed to improve the PEC properties of BiVO₄ photoanodes. Transition-metal (oxy)hydroxides such as Ni(OH)₂ [21], FeOOH [22], and NiFe layered double-hydroxide (NiFe-LDH) materials [23,24] have been widely explored for

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use in electrochemical oxygen evolution reactions (OERs). An operando Mössbauer spectroscopic study showed that Fe^{4+} was formed in the electrochemical OER process and the active sites were Fe^{4+} species generated within an Fe -doped NiOOH lattice [25], suggesting that Fe plays a critical role. In situ surface-enhanced Raman spectroscopy showed that NiOO^- generated from $\text{Ni}(\text{Fe})\text{OOH}$ acted as an electrochemical OER precursor [26]. It is therefore believed that doping with Fe or Ni is an effective method for enhancing PEC activity.

In this work, a BiVO_4 film was prepared by electrochemical deposition, and used as a precursor in hydrothermal production of $\text{Ni}(\text{OH})_2/\text{BiVO}_4$, $\text{Fe}(\text{OH})_2/\text{BiVO}_4$, and $\text{NiFe-LDH}/\text{BiVO}_4$ films [27]. The $\text{NiFe-LDH}/\text{BiVO}_4$ composite gave the best PEC performance. The photocurrent response of $\text{NiFe-LDH}/\text{BiVO}_4$ in the high-potential region (>0.5 V vs Ag/AgCl) was better than that of pure BiVO_4 . Hydrogen production of $1260 \mu\text{mol}$ was achieved in 3 h under visible-light illumination; this is 2.5 times that produced with the pristine BiVO_4 photoanode. Ni can facilitate efficient hole transfer to the surface and Fe increases the light-absorption capacity. NiFe-LDH nanoparticles are therefore not only an excellent electrochemical catalyst, but also an excellent PEC catalyst, and these nanoparticles have potential applications in solar water splitting.

Scheme 1 shows a schematic diagram of the synthesis of the pure BiVO_4 , $\text{Ni}(\text{OH})_2/\text{BiVO}_4$, $\text{Fe}(\text{OH})_2/\text{BiVO}_4$, and $\text{NiFe-LDH}/\text{BiVO}_4$ photoanodes. A BiVO_4 electrode was synthesized by a previously reported method [28]. In short, after electrodeposition of a BiOI film, vanadium was added, followed by calcination; excess V_2O_5 was removed by soaking, to give pure BiVO_4 . The composite photoanodes were prepared by a traditional hydrothermal method. An aqueous solution (30 mL) containing $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (0.1047 g), $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (0.1454 g), and urea (0.2162 g) was transferred to a 50-mL Teflon-lined autoclave in which BiVO_4 was placed with the BiVO_4 side facing the wall of the autoclave; the mixture was heated at 120°C for 6 h. The formed composite was thoroughly rinsed with purified water and dried at room temperature before use.

The morphologies of the photoelectrodes were examined using scanning electron microscopy (SEM) and high-resolution transmission electron microscopy (HRTEM); the images are shown in Fig. 1. The surface of the bare BiVO_4 photoanode consisted of nanoparticles of uniform size. Ni was loaded on the BiVO_4 surface in the form of nanospheres. After Fe deposition, the BiVO_4 particle size decreased, and NiFe-LDH nanoparticles

Scheme 1. Synthesis schematic diagram of pure BiVO_4 , $\text{Ni}(\text{OH})_2/\text{BiVO}_4$, $\text{Fe}(\text{OH})_2/\text{BiVO}_4$ and $\text{NiFe-LDH}/\text{BiVO}_4$ photoanodes.

Fig. 1. (a–d) SEM mapping of BiVO_4 , $\text{Ni}(\text{OH})_2/\text{BiVO}_4$, $\text{Fe}(\text{OH})_2/\text{BiVO}_4$ and $\text{NiFe-LDH}/\text{BiVO}_4$; (e) HRTEM images of $\text{NiFe-LDH}/\text{BiVO}_4$ photoanode.

were wrapped around the BiVO_4 particle surfaces. Fig. 1(e) shows that the NiFe-LDH nanoparticles were deposited on the BiVO_4 surface. The interplanar distances were estimated to be about 0.363 and 0.318 nm, corresponding to the (511) and (421) planes, respectively, of the NiFe-LDH nanoparticles. Fig. S1 confirms that a composite film was prepared.

The crystallographic structure of all the prepared photocatalysts were determined using X-ray diffraction (XRD; Fig. 2). The results confirm that all the BiVO_4 photoanodes have a single monoclinic scheelite structure. However, diffraction peaks corresponding to $\text{Ni}(\text{OH})_2$, $\text{Fe}(\text{OH})_2$, and NiFe-LDH nanoparticles were not observed. This may be because the contents of $\text{Ni}(\text{OH})_2$, $\text{Fe}(\text{OH})_2$, and NiFe-LDH nanoparticles were low, possibly below the XRD detection limit.

Ultraviolet-visible (UV-vis) spectroscopy was used to determine the bandgap values; the spectra are shown in Fig. 3. The absorption for the $\text{NiFe-LDH}/\text{BiVO}_4$ photoanode was slightly higher than that for the BiVO_4 photoanode. This can be attributed to a larger absorption efficiency and narrower bandgap in the case of the NiFe-LDH nanoparticles. Fig. S2 shows the photoluminescence (PL) emission spectra of the samples at an excitation wavelength of 350 nm. The PL intensity for the $\text{NiFe-LDH}/\text{BiVO}_4$ film was lower than that for the pure BiVO_4 film, showing that the electrons and holes in the $\text{NiFe-LDH}/\text{BiVO}_4$ film were easily separated.

Linear current-voltage curves for the BiVO_4 , $\text{Ni}(\text{OH})_2/\text{BiVO}_4$, $\text{Fe}(\text{OH})_2/\text{BiVO}_4$, and $\text{NiFe-LDH}/\text{BiVO}_4$ photoanodes were recorded (Fig. 4). The photocurrent densities of the

Fig. 2. XRD patterns of the BiVO_4 , $\text{Ni}(\text{OH})_2/\text{BiVO}_4$, $\text{Fe}(\text{OH})_2/\text{BiVO}_4$ and $\text{NiFe-LDH}/\text{BiVO}_4$ photoanodes.

Fig. 3. UV-Vis diffused reflectance spectra of BiVO_4 and NiFe-LDH/BiVO_4 (a) and the energy gap of BiVO_4 and NiFe-LDH/BiVO_4 (b).

Fig. 4. Linear current-voltage curves of BiVO_4 , $\text{Ni(OH)}_2/\text{BiVO}_4$, $\text{Fe(OH)}_2/\text{BiVO}_4$ and NiFe-LDH/BiVO_4 photoanode with visible light irradiation (a), without illumination (b), respectively.

$\text{Ni(OH)}_2/\text{BiVO}_4$ and $\text{Fe(OH)}_2/\text{BiVO}_4$ films were 0.81 and 1.98 mA, respectively, at 0.6 V (vs Ag/AgCl). The BiVO_4 film coated with NiFe-LDH nanoparticles gave the highest photocurrent density, i.e., 2.49 mA, at 0.6 V (vs Ag/AgCl), and its photoactivity was 217% higher than that of the bare BiVO_4 film. Fig. 4(b) shows that the onset potentials of the BiVO_4 and NiFe-LDH/BiVO_4 photoanodes in water oxidation were 1.2 and 0.8 V (vs Ag/AgCl), respectively. The clear cathodic shift in its onset potential and overpotential in water oxidation compared with those for pure BiVO_4 indicate that the NiFe-LDH nanoparticles had a catalytic effect. Photogenerated holes accumulate on the BiVO_4 photoanode surface, resulting in significant surface charge recombination. Loading with NiFe-LDH nanoparticles could reduce the amount of accumulated holes, and this would prevent recombination during water oxidation; this is supported by the much less pronounced photocurrent transients (Fig. S3).

Electrochemical impedance spectroscopy (EIS) was used to evaluate the charge-transfer kinetics at the semiconductor-electrolyte interface (Fig. 5). The Nyquist plots for the BiVO_4 and NiFe-LDH/BiVO_4 electrodes were consistent with the

linear sweep voltammetry results, with and without light irradiation. The semicircle for NiFe-LDH/BiVO_4 was smaller than that for the BiVO_4 electrode under both dark and illuminated conditions. This indicates that NiFe-LDH/BiVO_4 gave highly efficient electron and hole transport. Further analysis to clarify the mechanism of the PEC water-splitting reaction was performed by obtaining Mott-Schottky curves, shown in Fig. S4. Fitting of the linear parts of the plots showed that the carrier density in the BiVO_4 electrode was increased by surface modification with NiFe-LDH nanoparticles, and superior PEC hydrogen activity was achieved as a result of effective separation and transfer of charge carriers.

Fig. 6 shows that hydrogen generation with the NiFe-LDH/BiVO_4 photoanode under visible-light illumination for 3 h was higher than that with the pure BiVO_4 electrode. The reactor was filled with flowing argon gas for 30 min to exclude air from the system. The PEC reaction was then started with the assistance of light from a light-emitting diode lamp (CEL-LED100). A 1-mL injector was used to extract the evolved hydrogen for analysis with a gas chromatography (GC-9560) online system at set intervals of 30 min during light irradiation.

Fig. 5. Nyquist plots of EIS measurements on the pure BiVO_4 and NiFe-LDH/BiVO_4 photoanodes (a) in the dark at the open circuit potential in 0.5 mol/L Na_2SO_4 ; (b) Under the visible light irradiation at the open circuit potential.

Fig. 6. Time course of produced H_2 at 1.23 V vs. RHE in 0.5 mol/L Na_2SO_4 by a self-made H-type photo-reactor (a) and amperometric $I-t$ curves for 3 h PEC hydrogen production (b).

Fig. 6 shows that the NiFe-LDH/BiVO_4 photoanode gave an excellent PEC water-splitting performance. Hydrogen production was 1260 μmol in 3 h under light illumination; this is 2.5 times that produced with the pristine BiVO_4 photoanode. Furthermore, the NiFe-LDH/BiVO_4 photoanode showed excellent stability compared with the bare BiVO_4 photoanode. NiFe-LDH/BiVO_4 therefore has potential applications in the development of new solar water-splitting techniques.

A possible mechanism for the reaction at the NiFe-LDH/BiVO_4 photoelectrode is proposed on the basis of the above results and previously reported results (Scheme 2). First, the NiFe-LDH nanoparticles promote visible-light absorption by NiFe/BiVO_4 . The NiFe-LDH nanoparticles tend to shift to the positive potential region and generate positive charges, and then accept electrons from the semiconductor to revert to their original state. This is similar to cocatalysis with Ag and Bi [29,30]. The transfer of holes from the semiconductor to the metal suppresses recombination of the photogenerated electrons and holes, and this increases the lifetimes of the carriers.

In summary, a high-performance BiVO_4 photoanode with a NiFe-LDH nanoparticle cocatalyst was developed for PEC water splitting. The photocurrent density and hydrogen production achieved with the optimized NiFe-LDH/BiVO_4 were respec-

tively approximately 217% and 2.5 times the values achieved with pristine BiVO_4 . The NiFe-LDH nanoparticles facilitate efficient hole transfer to the surface and enhance the light-absorption capacity. The optimized NiFe-LDH/BiVO_4 photoanode also showed excellent stability in PEC water splitting.

Scheme 2. The possible PEC hydrogen generation mechanism of NiFe-LDH/BiVO_4 photoanode at 1.23 V vs. RHE in 0.5 mol/L Na_2SO_4 solution under visible light irradiation.

Graphical Abstract

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A high-performance BiVO₄ photoanode with a NiFe layered double-hydroxide nanoparticle cocatalyst was fabricated for photoelectrochemical water splitting. The cocatalyst boosted the photocatalytic activity, hydrogen production, and photoanode stability.

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NiFe双氢纳米粒子有效提高BiVO₄光阳极光电化学水分解性能

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摘要: 近年来, 太阳能驱动的光电化学水分解作为一种高效、环保、可持续的技术, 已经引起了广泛的关注. 为了更好地使用光电化学技术将太阳能转化为化学能, 至关重要是提高光电极材料的光吸收和光转化效率. BiVO₄禁带宽度($E_g = 2.4\text{--}2.5\text{ eV}$)小, 具有很好的可见光响应能力, 因此BiVO₄光电极材料引起了广泛关注. 但是, 当单独BiVO₄作为光电阳极材料时, 电子-空穴对分离弱、载流子传输慢, 从而使BiVO₄不能很好地在光电化学水分解中发挥作用. 为了缓解或解决此类限制性因素, 本课题组通过水热法合成了NiFe双氢纳米粒子, 并将其负载于BiVO₄电极表面, 光电催化分解水实验表明其产氢效率得到大幅度提高. 同时制备了Ni(OH)₂/BiVO₄和Fe(OH)₂/BiVO₄电极并用于研究NiFe/BiVO₄电极的反应机理. 在上文基础上, 本文采用电子扫描电镜(SEM)、高分辨投射电镜(HRTEM)、X射线衍射(XRD)、紫外可见漫反射(UV-Vis DRS)等表征手段和线性扫描伏安法(LSV)和电流时间($I-t$)等对其光电化学活性进行了测试, 研究了NiFe/BiVO₄电极在发生水氧化时的反应机理.

SEM结果表明, Ni(OH)₂是以纳米片组成的纳米球负载于多孔BiVO₄表面; 而当Fe(OH)₂负载于BiVO₄表面时, BiVO₄的纳米尺寸减小; NiFe-LDH纳米粒子负载于BiVO₄表面时, 可以明显看见BiVO₄纳米颗粒表面包裹着一层更小的纳米粒子. 这证明了Ni(OH)₂, Fe(OH)₂和NiFe-LDH纳米粒子均成功负载于BiVO₄表面. 这也得到HRTEM结果的确认. UV-Vis DRS结果表明NiFe-LDH纳米粒子能有效拓宽BiVO₄的吸收边, 从而增加对可见光的吸收, 增加了对光的利用率.

LSV测试结果表明, 暗反应条件下Ni(OH)₂/BiVO₄比NiFe/BiVO₄和Fe(OH)₂/BiVO₄电极的起始电位更低, 说明Ni(OH)₂有更好的传输电子性能; 而在光照条件下, 在同一电位时NiFe/BiVO₄比Ni(OH)₂/BiVO₄和Fe(OH)₂/BiVO₄电极的光电流值更高. 值得注意的是, 此时Ni(OH)₂/BiVO₄比Fe(OH)₂/BiVO₄电极的光电流值低, 这又说明Fe(OH)₂比Ni(OH)₂对光更敏感. 因此当NiFe-LDH纳米粒子负载于BiVO₄表面时, 不仅提高了BiVO₄光电极的光吸收效率, 而且加速了载流子的传输从而抑制了光生电子-空穴的复合, 使反应过程中的量子效率得到提高.

关键词: NiFe双氢纳米粒子; BiVO₄光阳极; 光电化学水分解; 光电催化

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