



Surface modification of hematite photoanode by NiFe layered double hydroxide for boosting photoelectrocatalytic water oxidation



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ABSTRACT

The photoelectrocatalytic (PEC) oxygen evolution reaction (OER) is one of the most efficient ways for utilizing solar energy for water electrolysis. Nevertheless, up to now, the PEC conversion efficiency of the established photoanode is still low. In this work, α -Fe₂O₃/NiFe layered double hydroxide (LDH) photoanodes were synthesized by electrodeposition of LDH on α -Fe₂O₃. Compared with bare α -Fe₂O₃, the α -Fe₂O₃/Ni_{0.5}Fe_{0.5}-LDH photoanode displays about 3 times photocurrent enhancement and excellent long-term stability. The enhanced PEC activity of α -Fe₂O₃/NiFe-LDH is ascribed to the interface between α -Fe₂O₃ and NiFe-LDH which can facilitate charge transfer and improve carrier density. Simultaneously, NiFe-LDH as a co-catalyst can accelerate the surface OER kinetics.

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1. Introduction

Converting and utilizing solar energy into the useable energy through photoelectrocatalytic (PEC) water splitting is one of the most challenges nowadays [1,2]. The crucial step in PEC water splitting is oxygen evolution reaction (OER) occurred at the photoanode, but it is restricted by a multistep four electrons transfer that requiring large overpotentials [3]. For decades, great efforts have been focused on the transition metal oxides as promising photoanode materials, such as TiO₂ [4], WO₃ [5], α -Fe₂O₃ [6], and BiVO₄ [7]. Among them, hematite (α -Fe₂O₃) was extensively studied and has shown interesting OER performance due to the favourable optical bandgap and appropriate valence band position. However, several inherent drawbacks impede their PEC efficiency,

such as poor electrical conductivity, short hole diffusion lengths and slow surface OER kinetics [8]. Therefore, many efforts have been devoted to overcome these limitations by morphology engineering [9], facet engineering [10] and surface modification. Generally, surface modification of α -Fe₂O₃ with oxygen evolution co-catalyst (OEC) (e.g. IrO₂/RuO₂ [11], Ni(OH)₂ [12], NiOOH [13] and Co-Pi [14]) can improve its PEC activities by accelerating the occurrence of surface reaction. Recently, layered double hydroxide (LDH) materials that are permeable to electrolyte have been introduced on photoelectrode as promising OEC showing greatly enhanced photoelectrocatalytic OER performances, such as ZnO@CoNi-LDH [15], TiO₂/ZnFe-LDH [16], WO₃@NiFe-LDH [17], α -Fe₂O₃/ZnCo-LDH [18] and BiVO₄/Fe based-LDH [19] photoelectrode. Thus, it may be an effective strategy to improve the PEC activity through decorating α -Fe₂O₃ photoanode with NiFe-LDH as a kind of OEC.

Herein, we develop a facile electrodeposition synthesis method to fabricate NiFe-LDH modified α -Fe₂O₃ photoanodes with enhanced PEC OER performance. The α -Fe₂O₃/Ni_{0.5}Fe_{0.5}-LDH electrode shows 3-fold higher photocurrent densities at 1.23 V versus reversible hydrogen electrode (RHE) than bare α -Fe₂O₃, and a very

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long-term durability. Mott-Schottky (MS) and Electrochemical impedance spectroscopy (EIS) measurements reveal that the greatly enhanced PEC performances of α -Fe₂O₃/NiFe-LDH come from the reduced charge transfer resistance and improved carrier density.

2. Material and methods

2.1. Synthesis of α -Fe₂O₃ photoanode

The α -Fe₂O₃ crystals were synthesized by a hydrothermal method [20,21]. 1.5 mmol KF·2H₂O and 1.5 mmol FeCl₃·6H₂O were dissolved in 60 mL of deionized water under stirring. A clear solution was obtained and transferred to a Teflon-lined stainless steel autoclave and reacted at 220 °C for 24 h. After the hydrothermal reaction, the precipitation was filtered, washed with distilled water and dried at 60 °C. Before the Ti foil was used as substrate to deposit α -Fe₂O₃ powder. It was immersed in concentrated HCl solution (37%) and etched for 30 min at 90 °C. Then, α -Fe₂O₃ photoanodes were prepared by electrophoretic deposition on the pre-treated Ti foil (Fig. S1) [22]. Firstly, 50 mg iodine and 20 mg α -Fe₂O₃ powder were dispersed in 30 mL acetone by sonication. The Ti foil and Pt foil were immersed in the above solution, and 50 V of bias potential was applied between them for 40 s using a potentiostat (ITECH IT6720). Lastly, the α -Fe₂O₃ coated Ti foil substrate was washed by ethanol and calcined at 400 °C for 3 h in air.

2.2. Synthesis of α -Fe₂O₃/NiFe-LDH nanostructure

The α -Fe₂O₃/NiFe-LDH nanostructures were prepared via a fast electrodeposition method [19,23]. Typically, the α -Fe₂O₃ photoelectrode, saturated calomel electrode (SCE) and Pt foil were used as the working, reference and counter electrodes, respectively. Ni(NO₃)₂·6H₂O (0.10 M) and FeSO₄·7H₂O (0.10 M) were dissolved in 30 mL of water which purged with N₂ to make the electrolyte for the electrosynthesis of Ni_{0.5}Fe_{0.5}-LDH. The electrodeposition was carried out at -1.00 V vs. SCE for 50 s. To optimize the composition

of the Ni²⁺ and Fe²⁺, the total moles of them in the electrolyte were maintained at 0.20 M.

2.3. Characterization

X-ray diffraction (XRD) was carried out with DX2700 operating at 40 kV and 30 mA equipped with Cu K α radiation ($\lambda = 1.5418 \text{ \AA}$). The morphology of the samples were characterized by using a JSM-6390LV scanning electron microscope (SEM). Transmission electron microscopy (TEM) and high-resolution TEM (HRTEM) images were obtained using a JEM-2100F electron microscope. The X-ray photoelectron spectroscopy (XPS) were determined on a RBD 147 upgraded PHI 5000C ESCA system equipped with an Mg K α anode.

2.4. PEC measurement

The PEC performance was evaluated using CHI760E Electrochemical Workstation in a three-electrode cell with 0.5 M Na₂SO₄ solution as the electrolyte. The simulated solar illumination (100 mW/cm²) was provided by a PLS-SXE 300C Xe arc lamp (Perfect Light) with an AM 1.5G filter (Fig. S2). I-t curves were measured at 1.23 V vs. RHE and linear sweep voltammetry (LSV) curves were recorded with a scan rate of 10 mV/s. EIS was carried out at the open circuit potential with the frequency range of 10⁵ to 10⁻² Hz and a 5 mV amplitude. The MS measurements were performed at a fixed frequency of 500 Hz with 10 mV amplitude. The measured potentials vs. Ag/AgCl were converted to the RHE with the Nernst equation ($E_{\text{RHE}} = E_{\text{Ag/AgCl}} + 0.059 \times \text{pH} + 0.1976$).

3. Results and discussion

The α -Fe₂O₃/NiFe-LDH films were fabricated via a three-step process. Firstly, the polyhedral α -Fe₂O₃ crystals were synthesized by a hydrothermal process, with a particle size of 0.5–2 μm (Fig. 1a). Subsequently, the α -Fe₂O₃ powders were assembled on Ti foil by an electrophoretic deposition method. Lastly, a fast electrodeposition method was adopted in aqueous solution with Ni²⁺ and Fe²⁺

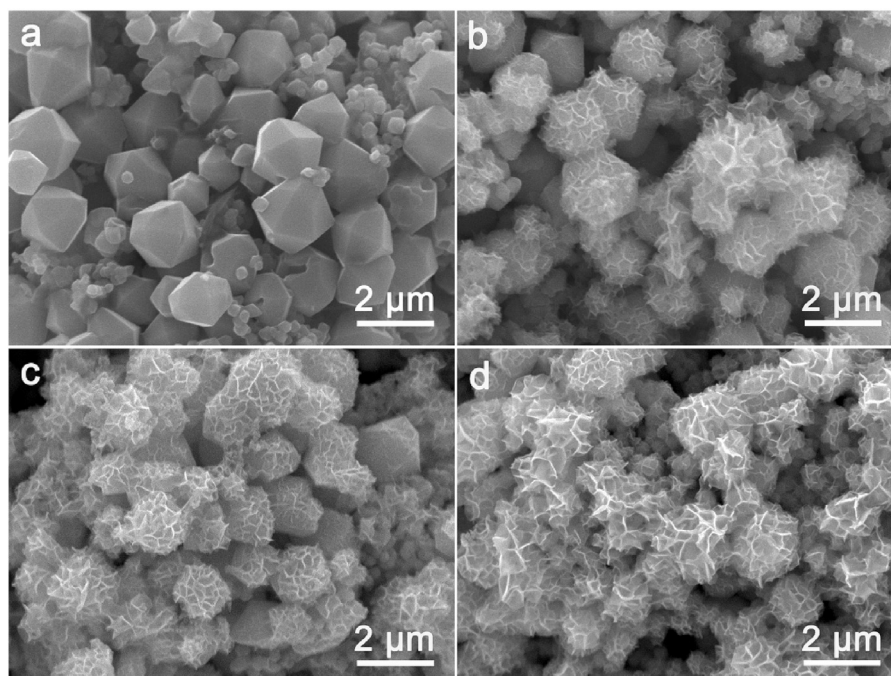


Fig. 1. SEM images of (a) bare α -Fe₂O₃, (b) α -Fe₂O₃/Ni_{0.8}Fe_{0.2}-LDH, (c) α -Fe₂O₃/Ni_{0.5}Fe_{0.5}-LDH and (d) α -Fe₂O₃/Ni_{0.2}Fe_{0.8}-LDH film.

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