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Enhanced photocatalytic activity and stability of the reduced graphene oxide loaded potassium niobate microspheres for hydrogen production from water reduction

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ABSTRACT

A facile approach to synthesize reduced graphene oxide (RGO) loaded potassium niobate microspheres was reported. The composition, microstructure and electron-transfer properties of the obtained product were characterized. Compared to pure potassium niobate microspheres and commercial P25 TiO₂, the as-prepared potassium niobate microspheres/RGO composite showed much higher photocatalytic activity for generating hydrogen under UV irradiation. It was ascribed to the enhanced separation efficiency of electron/hole pairs as testified by electrochemical impedance spectrum and fluorescence spectrum. Importantly, the composite photocatalyst was stable and easy to recycle, and the amount of hydrogen evolution did not decrease after six recycles. The results are potentially applicable to a range of semiconductors useful in water reduction as well as other areas of heterogeneous photocatalysis.

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Introduction

The depletion of fossil fuel reserves is one of the most urgent issues in modern society. Using clean, abundant and sustainable energy without fossil fuel consumption and CO₂ emission is of great significance. As a kind of storable and environmentally benign fuel, hydrogen energy is considered as an ideal energy source [1]. An important technique of

producing hydrogen is the electrolysis of water. Although electrolysis of water would be preferred to produce for high-quality hydrogen, this technique is costly [2]. It is found that using solar energy to produce clean hydrogen through photocatalysis is a good choice [3]. It is the key to design and synthesize ideal catalysts for photocatalytic hydrogen evolution.

Due to their electronic features, some semiconductor materials are mostly used as photocatalysts. An ideal

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semiconductor photocatalyst for hydrogen evolution should be highly active, stable and low cost. So, it is significantly meaningful to design and prepare a cheap and efficient semiconductor photocatalyst. Among a diverse set of photocatalysts, the active materials being employed are mainly TiO_2 and titanates [4]. Therefore, further development of the photocatalytic materials for water splitting is indispensable. Recently, for their availability and low toxicity, some niobates with perovskite structure have been intensively investigated in pollutant degradation and water splitting, and the relationship between the crystal structure and photocatalytic activities was also discussed [5]. However, photocatalytic water splitting cannot achieve a desired level due to fast recombination of photo-generated electron/hole pairs. In order to optimize the photocatalytic efficiency and provide a sufficient base for the future design and development of high performance photocatalysts, great efforts have been made to probe the underlying principles and photocatalytic performance of materials [6,7]. Jonker et al. found that efficient electrical injection of spin-polarized carriers can be achieved from a non-lattice-matched magnetic contact into a semiconductor heterostructure [8]. Besides, morphology has an important influence on properties of potassium niobates [9–12]. Especially, the morphology has a direct influence on the photoactivity by affecting the specific surface area, the hydrophilic character or the availability of charge carrier by modifying the internal electric field close to the surface. Therefore, enlarging surface area and increasing the number of active sites is an efficient way to improve the separation efficiency of electron/hole pairs through adjusting the morphology of photocatalysts [13]. Zhou et al. prepared the porous $\text{K}_4\text{Nb}_6\text{O}_{17}$ microspheres via a simple homogeneous precipitation method, and the microspheres show a high activity for generating hydrogen [14]. It was mainly attributed to the porous structure and high specific surface area of the $\text{K}_4\text{Nb}_6\text{O}_{17}$ microspheres. Ma et al. found that photocatalytic activity of hydrogen evolution over the exfoliated-scrolled $\text{K}_4\text{Nb}_6\text{O}_{17}$ is obviously higher than that of the synthetic $\text{K}_4\text{Nb}_6\text{O}_{17}$ crystals. It is demonstrated that the unmodified nanoscrolls are better catalysts for UV light driven hydrogen evolution from aqueous methanol solutions [15].

Mobility of the charges affects the probability of electrons reaching the reaction sites on the surface of the photocatalyst, so it affects the photocatalytic activity and is important to a photocatalyst. Integrated with noble-metal with low over potential (Pt, Pd, Au or Ag) is an efficient way to improve the charge mobility of niobates [16,17]. But noble-metals are very expensive for industrial applications. Nowadays, it is found that the mobility of charge can be improved by integrated with graphene [18], or carbon nanotube [19]. Owing to its unique structure and electronic properties, graphene has drawn the attention of scientists in recent years [20]. Since the reduced graphene oxide with huge specific surface area and high mobility of charge carriers exhibits many physical properties similar to those of graphene, it is being considered in electronic, sensor, and catalytic applications [21]. Here, we propose a green and facile way to synthesize reduced graphene oxide (RGO) loaded potassium niobate microspheres. The purpose is to improve the separation efficiency of electron/hole pairs of the potassium niobate microspheres by using

RGO served as a cheap cocatalyst. The UV photocatalytic activity for hydrogen evolution over the potassium niobate microspheres/RGO is evaluated. In addition, the mechanism of electron transfer in the potassium niobate microspheres/RGO is also investigated.

Experimental section

Preparation of RGO loaded potassium niobate microspheres

RGO was prepared according to the published procedure [22]. The potassium niobate microspheres were synthesized using hydrothermal method [14]. All chemicals were used without further purification.

Without other pre-modifications to potassium niobate microspheres and RGO, the RGO loaded potassium niobate microspheres was prepared only by simple mixing a certain amount of potassium niobate microspheres and RGO. In a typical procedure, 100 mg of potassium niobate microsphere and 5.3 mg of RGO were dispersed into 100 mL of deionized water, and then slowly stirred for 24 h. The solid obtained by centrifugation was thoroughly washed with water, and followed by drying at 60 °C overnight.

Preparation of working electrodes

Indium–tin oxide (ITO) glasses were cleaned by sonication in ethanol, acetone, chloroform, and deionized water for 15 min, respectively, and then dried in the air. Various working electrodes were prepared via impregnation and subsequent calcination method. In brief, 10 mg of sample (potassium niobate microspheres or potassium niobate microspheres/RGO) was added into 2 mL of alcohol, and the obtained mixture was sonicated for 30 s. After that, the ITO glass ($1 \times 1.5 \text{ cm}^2$) was soaked into the slurries for 2 min, and heat-treated at 100 °C for 1 h.

Photoelectrochemical behavior measurement

The photoelectrochemical measurements were carried out at room temperature using Chenhua CHI 660E computer-controlled electrochemical analyzer with a standard three-electrode system (Chenhua Instruments Co., Shanghai, China). A platinum wire electrode was used as the counter electrode and Ag/AgCl electrode was used as the reference electrode. The electrolyte was 1 mol L^{-1} of NaCl aqueous solution. The scan rate was 50 mV s^{-1} . Electrochemical impedance spectra (EIS) were tested in the potentiostatic mode with the frequency range of 1 to 1 MHz, and the bias potential was 0.5 V. The amplitude was 5 mV. The light source was a 150 W Xe lamp. The distance between light source and the working electrode was 7 cm. The air in the solution was removed by purging nitrogen for 15 min.

Photocatalytic experiment

The photocatalytic water reduction for hydrogen evolution was performed using a CEL-SP2N water splitting system with an outer-irradiation type quartz cell. In a typical process,

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