



Palladium nanoparticles and rGO co-modified BiVO₄ with greatly improved visible light-induced photocatalytic activity

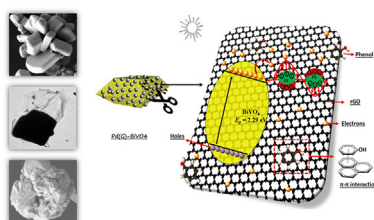
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HIGHLIGHTS

- A plasmonic ternary composite was fabricated with greatly improved activity.
- Mechanisms to the enhancement were explored and discussed.
- Increased separation of charge carriers boost the photocatalytic reaction.
- Pathways of phenol removal were studied and proposed.

GRAPHICAL ABSTRACT



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ABSTRACT

A ternary composite, Pd-rGO-BiVO₄, was fabricated with reduced graphene oxide (rGO) and palladium nanoparticles decorated on the surface of BiVO₄. As-prepared samples were tested for the photocatalytic degradation of phenol under visible light irradiation. Enhancement was observed for the ternary structure, merits of which may be as follows: 1) rGO wrapped BiVO₄ facilitated the photogenerated electrons transfer, 2) palladium nanoparticles served as electron acceptors, 3) palladium nanoparticles on the surface were capable of absorbing visible light photons. The uptake of photogenerated charge carriers would improve their separation and more oxidative species may be produced that can participate in the degradation of organics. Due to the SPR effect of palladium nanoparticles on the surface, the harvesting capacity of the photocatalyst to absorb visible light photons was increased, and thus its photocatalytic activity was improved. It should be noted that phenol was more easily adsorbed by rGO due to the π - π interaction between rGO and phenol, which also contributed to the enhancement in the photocatalytic activity. This work provides new evidence to confirm the advances of ternary structures applied in the photocatalytic removal of phenolic compounds in water under visible light irradiation.

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1. Introduction

BiVO₄ as a ferroelastic and ion conductive material has attracted extensive attentions (Tokunaga et al., 2001; Madhusudan et al., 2011; Huang et al., 2014; Martinez Suarez et al., 2015). The properties of BiVO₄ significantly depend on the crystal form. There are

three primary crystal forms: zircon structure with tetragonal system (ZT), scheelite structure with monoclinic system (SM), and scheelite structure with tetragonal (ST) system. Generally, it was reported that BiVO₄ with monoclinic crystal structure exhibited higher photocatalytic activity than that with tetragonal crystal structure (Zhang et al., 2006, 2007). This phenomenon was probably attributed to the narrower band gap (ca. 2.4 eV) for monoclinic BiVO₄ compared with that (ca. 2.9 eV) for tetragonal BiVO₄ (Obregon et al., 2014). As a result, monoclinic BiVO₄ with the suitable band gap has been comprehensively studied and

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implemented in visible light-driven photocatalysis.

One of the hindrances for photocatalysis with BiVO_4 , to date, is the low photoconversion efficiency, mainly due to the high recombination rate of photogenerated charge carriers. Aiming to deal with this hindrance for BiVO_4 based systems, various approaches were employed, mainly including metal/nonmetal doping (Zhang et al., 2009; Zhang and Zhang, 2010; Meng and Zhang, 2016a, 2017b; Regmi et al., 2017) and the formation of heterojunctions (Long et al., 2006; Madhusudan et al., 2011; Zhao et al., 2012; Zhang et al., 2014a; Meng et al., 2015, 2017a; Meng and Zhang, 2015b, a; Singh et al., 2017). Li et al. deposited Au, Pt and Ag nanoparticles on the surface of BiVO_4 polyhedrons (Li et al., 2013). It was found metals and metal oxides were facet-selectively photo-deposited on the surface of BiVO_4 . This phenomenon may occur because the photogenerated electrons tend to transfer to the {010} facets and holes accumulate on the {110} facets. This mechanism can be applied in designing efficient photocatalysts. Moreover, well-dispersed metallic nanoparticles on the surface are capable of facilitating the separation of photogenerated charge carriers and absorbing visible light photons via the surface plasmon resonance (SPR) effect (Hou and Cronin, 2013). Ag, Au and Pt nanoparticles loaded on BiVO_4 have been reported with enhanced visible light-driven photocatalytic activity in the oxidation of organics in water (Kohtani et al., 2005; Ge, 2008; Cao et al., 2012). Recently, we reported palladium nanoparticles can also be photoreduced onto a support to improve its photocatalytic activity (Meng and Zhang, 2016c; Meng et al., 2017b; Meng et al., 2018). The SPR peaks for palladium nanoparticles can be tuned from 410 to 870 nm (Xiong et al., 2005a, 2005b).

Graphene, a single layer of sp^2 -bonded carbon atoms tightly packed into a two-dimensional honeycomb structure, has been extensively studied and implemented in various areas including photocatalysis. The excellent conductivity of graphene suggests it may be a promising candidate in facilitating the separation of photogenerated charge carriers. Graphene wrapped BiVO_4 were reported with greatly improved photocatalytic activity in the organics degradation (Fu et al., 2011). It should also be noted that graphene may be prepared by reducing graphene oxide (GO), which is an effective and inexpensive approach. Ng et al. reported on oxygen-functional groups on GO may be reduced through hydrothermal treatment (Ng et al., 2010). Since rGO can be regarded as an electron acceptor, a rGO layer may be introduced between the metallic nanoparticles and BiVO_4 to further facilitate the separation of photogenerated charge carriers (Xiang et al., 2012). We recently successfully prepared ternary structures of Ag/Pd and rGO co-modified Bi_2MoO_6 with synergetic effects in enhancing the photocatalytic activity of the support (Meng and Zhang, 2016d, 2017a).

In this work, GO wrapped BiVO_4 polyhedrons would be fabricated and further hydrothermally reduced to rGO- BiVO_4 composites. Palladium nanoparticles would then be photo-dispersed on rGO- BiVO_4 composites to fabricate the ternary structure. As-prepared composites were applied in photocatalytic degradation of a colorless organics model, phenol, which is widely found in wastewater from many industrial processes and is generally difficult to decompose (Gogate, 2008; Oller et al., 2011). This work would shed a light on the possibility of using ternary photocatalysts for treating phenolic compounds in water under visible light irradiation.

2. Experimental

2.1. Preparation of BiVO_4 , rGO- BiVO_4 and Pd-rGO- BiVO_4

BiVO_4 polyhedrons were prepared using a hydrothermal method. Typically, 36 mmol NH_4VO_3 (Fisher Scientific, Certified

ACS) and 36 mmol of $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ (Fisher Scientific, Certified ACS) were first dissolved in 300 mL of nitric acid solution (2.0 M). The pH was then adjusted to 2 using an ammonia solution. Orange precipitates were immediately formed and the suspension was then magnetically stirred for 30 min. After a 2 h aging period, the orange precipitates at the bottom were transferred to a 100-mL Teflon-lined autoclave (Parr Instrument Company, USA) and heated up to 200 °C for 24 h. After the autoclave naturally cooled down to room temperature, the yellow precipitates were filtered out and washed twice with distilled deionized water (DDW) and one time with absolute ethanol. The precipitates were then dried at 60 °C for 12 h before getting collected for further use.

The rGO- BiVO_4 composites were synthesized through an evaporation-induced self-assembly process to form GO- BiVO_4 composites followed with a photo-reduction process (Wang et al., 2014). Firstly, 1.0 g of as-prepared BiVO_4 polyhedrons dispersed in 50 mL ethanol and 3.75 mL of graphene oxide (GO, 4 mg/mL, Sigma-Aldrich Canada) dispersed in 50 mL DDW, respectively, were ultrasonicated for 1 h. Then, GO precursor suspension was dropwise added into the BiVO_4 suspension and magnetically stirred for 8 h without sealing. After that, the mixture was heated up to 60 °C and maintained using a water bath, and continuously magnetically stirred until the solvent evaporated completely. The formed precipitates were dried at 60 °C for 12 h before they were collected and labelled as GO-Bi for further use. The formed GO-Bi samples were further reduced using a photo-reduction method. Specifically, 1.0 g of GO-Bi were dispersed in a solution containing 100 mL DDW and 20 mL ethanol. After magnetically stirring for 30 min, the suspension was purged using pure N_2 for 30 min. After that, the suspension was illuminated for 3 h using a 300-W halogen tungsten projector lamp (Ushio, USA). Prepared particles in the suspension were filtered out, dried at 60 °C for 12 h and labelled as rGO-Bi.

Palladium nanoparticles are dispersed on the rGO-Bi composites through a photo-reduction process (Meng and Zhang, 2016c, 2017a). Typically, 0.30 g of rGO-Bi composites were dispersed in 40 mL DDW under ultrasonication for 30 min. A designate amount of PdCl_2 (Fisher Scientific Canada, ACS certified) suspension was then added into the suspension with magnetic stirring for 10 min. After that, the suspension was illuminated under a 300-W halogen tungsten projector lamp (Ushio, USA) for 1 h. The resultant products were filtered out, washed twice with DDW and dried at 60 °C for 12 h. Composites of 0.5, 1.0 and 2.0 wt% Pd-rGO- BiVO_4 were prepared following the above procedures and labelled as 0.5, 1.0 and 2.0 Pd(G)-Bi. For comparison, composites of 1.0 wt% Pd- BiVO_4 were also prepared following the above procedures except with pure BiVO_4 instead of rGO-Bi, and labelled as 1.0 Pd-Bi.

2.2. Characterization

Crystal structures of prepared samples were measured using a Rigaku Ultima IV diffractometer with $\text{Cu K}\alpha$ radiation ($\lambda = 0.15418 \text{ nm}$) at 40 kV and 44 mA for the X-ray diffraction analysis (XRD). Surface composition and chemical states were studied using a XSAM-800 X-ray Photoelectron Spectroscopy (XPS). Morphologies of prepared samples are investigated using a field-emission scanning electron microscope (FE-SEM, JEOL JSM-7500 F) and a transmission electron microscope (TEM, JEM-2100 F). An Energy-dispersive X-ray spectroscopy (EDS) was also equipped in the FE-SEM for the elemental analysis of prepared samples. Optical properties were explored on an ultraviolet–visible diffuse reflectance spectroscopy (UV–Vis DRS). Electrochemical properties of prepared samples were performed on a CHI 604 E electrochemical analyzer (CH Instruments Inc., USA) with a platinum wire as a counter electrode, a calomel reference electrode and a working electrode. The working electrode was composed of indium tin oxide

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