



Enhanced photocatalytic hydrogen production from aqueous sulfide/sulfite solution by $\text{ZnO}_{0.6}\text{S}_{0.4}$ with simultaneous dye degradation under visible-light irradiation



Ka Him Chu^a, Liqun Ye^{a, b}, Wei Wang^{c, **}, Dan Wu^a, Donald Ka Long Chan^d,
Cuiping Zeng^a, Ho Yin Yip^a, Jimmy C. Yu^d, Po Keung Wong^{a, *}

^a School of Life Sciences, The Chinese University of Hong Kong, Shatin, NT, Hong Kong, China

^b College of Chemistry and Pharmaceutical Engineering, Nanyang Normal University, Nanyang, 473061, China

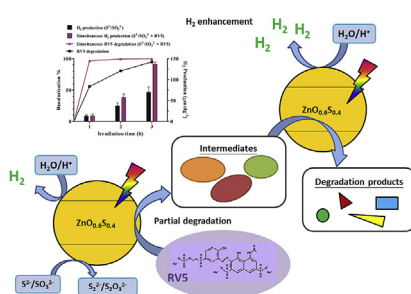
^c College of Materials Science and Engineering, Huazhong University of Science and Technology, Wuhan, 430074, China

^d Department of Chemistry and Shenzhen Research Institute, The Chinese University of Hong Kong, Shatin, NT, Hong Kong, China

HIGHLIGHTS

- $\text{ZnO}_{0.6}\text{S}_{0.4}$ was synthesized by simple co-precipitation and calcination method.
- Photocatalytic H_2 production with simultaneous pollutant degradation was achieved.
- The maximum of 110% H_2 promotion was obtained with optimal concentration of RV5.
- Degradation intermediates of RV5 were effective to enhance H_2 production.

GRAPHICAL ABSTRACT



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ABSTRACT

Photocatalytic hydrogen (H_2) production was performed by visible-light-driven (VLD) ternary photocatalyst, zinc oxysulfide ($\text{ZnO}_{0.6}\text{S}_{0.4}$) in the presence of sulfide/sulfite ($\text{S}_2^{2-}/\text{SO}_3^{2-}$) sacrificing system, with simultaneous azo-dye Reactive Violet 5 (RV5) degradation. Enhancement in both RV5 degradation and H_2 production was achieved, with the promotion of H_2 production after decolorization of RV5. The effect of initial concentration of RV5 was found to be influential on the enhancement of H_2 during the simultaneous processes, with a maximum of 110% increase of H_2 produced. The mechanism of the simultaneous system was investigated by scavenger study and intermediate analysis, including Fourier transform-infrared (FTIR) spectroscopy and total organic carbon (TOC) analysis. It was confirmed that the partial degradation of RV5 and presence of dynamic organic intermediates contributed to the enhancement in H_2 production. The present study revealed the feasibility of developing VLD photocatalysis as a sustainable and environmentally friendly technology for concurrent organic pollutant degradation with energy generation.

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* Corresponding author.

** Corresponding author.

E-mail addresses: weiwang@hust.edu.cn (W. Wang), pkwong@cuhk.edu.hk (P.K. Wong).

1. Introduction

Facing the global energy depletion and pollutions caused by fossil fuels, hydrogen (H_2) is considered as one of the potential

alternative energy carrier and fuel in the future with high energy capacity and nearly-zero emission of pollutants (Nowotny et al., 2005; Ni et al., 2007; Chen et al., 2010; Lior, 2010; Yuan et al., 2017). However, recently 95% of H₂ generation relies heavily on fossil fuels conversion such as natural gas reformation (Ni et al., 2007), which would even deteriorate the environmental problems of fossil fuels. Therefore, to develop a promising and environmentally friendly technology for yielding H₂ as clean energy source, there has been increasing interests in photocatalytic H₂ production since the first study of water splitting by Fujishima and Honda in 1972 (Fujishima and Honda, 1972; Ni et al., 2007; Li et al., 2016). Concurrently, high attention has been paid on the huge amount of dye-containing wastewater generated by textile industry for decades. Apart from negative aesthetic impact, the discharge of the colored effluents to water resources causes serious environmental and ecological pollutions, and threatens the health of natural lives and humans (Solís et al., 2012; Gupta et al., 2015). Moreover, those aromatic dyes with complex structures are highly resistant to conventional physicochemical and biological degradation (Solís et al., 2012; Gupta et al., 2015). Photocatalysis is one of the developing potential alternatives for treating wastewater containing organic dyes and pollutants (Herrmann et al., 2007; Fernández et al., 2010; Ye et al., 2014).

Many previous studies have been investigated on enhancing photocatalytic H₂ production efficiency by using visible-light-driven (VLD) photocatalysts for better utilization of the whole solar spectrum over other photocatalysts with wide band-gap (Li et al., 2016). Addition of sacrificing agents also allows improvement on the photocatalytic H₂ production efficiency by consuming the photo-induced h⁺ for better e⁻–h⁺ separation (Li et al., 2016). For example, one of the common inorganic sacrificing agents is sulfide/sulfite (S²⁻/SO₃²⁻) system which provides additional protection on S-based photocatalyst from anodic photo-corrosion (Bao et al., 2008; Li et al., 2009; Chen et al., 2010; Daskalaki et al., 2010; Gomathisankar et al., 2013a; Markovskaya et al., 2015; Zhao et al., 2015; Li et al., 2016). However, it was not practical to add sacrificing agent intentionally as they might be actually another sources of energy, such as methanol, and even more expensive than H₂ (Cho et al., 2016).

Recently, the VLD photocatalyst zinc oxysulfide (ZnOS) has been reported to have adjustable band-gap with satisfactory performances in individual photocatalytic dye degradation and H₂ production (Kim et al., 2007; Li et al., 2009; Chen et al., 2010; Lehr et al., 2012; Pandey et al., 2013). In particular, it was reported that ZnOS with O:S molar ratio of 0.6:0.4 possessed a minimum band-gap of 2.7 eV (Pandey et al., 2013). In our previous study, ZnO_{0.6}S_{0.4} nanoparticles were synthesized with satisfactory photocatalytic bacterial inactivation activity (Wu et al., 2016). However, to the best of our knowledge, there were limited studies on multi-component system for photocatalytic H₂ production with simultaneous pollutant degradation by ZnOS in the S²⁻/SO₃²⁻ solution.

In the present study, the main objective is to demonstrate the effective VLD photocatalytic dye degradation and simultaneous H₂ production by synthesized ZnO_{0.6}S_{0.4} in the presence of S²⁻/SO₃²⁻ solution as the sacrificing agent. Reactive Violet 5 (RV5), a mono-azo dye, was used as the model industrial dye for the study. As S²⁻ and SO₃²⁻ ions are abundant by-products generated in industries after processes such as flue-gas desulfurization and fossil fuel extractions (Bao et al., 2008; Gomathisankar et al., 2013a, 2013b; Li et al., 2016), it would be a more environmentally friendly and sustainable approach to make use of these undesirable industrial dye and S wastes while generating beneficial H₂ energy in a single system. The possible mechanism of the system will be discussed based on the relevant experimental results.

2. Experimental

2.1. Materials

All reagents were used as received. Zinc nitrate 6-hydrate (Zn(NO₃)₂·6H₂O) was purchased from Uni Chem Incorporation. Tri-sodium citrate dihydrate (Na₃C₆H₅O₇·2H₂O) was obtained from Scharlau. Sodium hydroxide (NaOH), anhydrous sodium sulfite (Na₂SO₃) and sodium sulfide nonahydrate (Na₂S·9H₂O) were purchased from International Laboratory. Sodium oxalate (Na₂C₂O₄) was purchased from Peking Chemical Works. Isopropanol and potassium dichromate (K₂Cr₂O₇) were obtained from Merck Chemical Company. Nafion solution was purchased from Sigma-Aldrich Corporation. Reactive violet 5 (RV5, Fig. S1, Supplementary Information) was obtained from DyStar Singapore Private Limited.

2.2. Synthesis of zinc oxysulfide

Nanoparticles of zinc oxysulfide (ZnOS) were prepared by coprecipitation followed by calcination (Wu et al., 2016). Preliminary optimizations had been conducted by performing photocatalytic degradation of RV5 using ZnOS with different O:S ratios and calcination temperatures (Fig. S2, Supplementary Information). O:S ratio and calcination temperature for ZnOS synthesis were optimized to be 0.6:0.4 and 400 °C respectively. Briefly, an aqueous zinc ion solution was prepared by dissolving 2.98 g of Zn(NO₃)₂·6H₂O and 0.082 g of Na₃C₆H₅O₇·2H₂O into 50 mL of distilled water. Another aqueous solution containing 0.48 g of NaOH and 0.96 g of Na₂S·9H₂O was added dropwise into the zinc ion solution with continuous stirring for 2 h at room temperature (ca. 25 °C). The precipitates were washed with distilled water and ethanol for several times, respectively, and dried at 50 °C. Finally, the precipitates were calcined at 400 °C for 2 h in argon (Ar) atmosphere to obtain ZnOS nanoparticles. For XRD comparison, pure ZnO and ZnS were prepared using only 0.8 g of NaOH and only 2.4 g of Na₂S·9H₂O respectively in the precipitation step while all other procedure remained unchanged.

2.3. Characterization

The general morphologies of the prepared samples were examined by a Quanta 400 FEG scanning electron microscope (FESEM, FEI Company, Oregon, USA). The high resolution transmission electron microscopy (HRTEM) and energy dispersive X-ray (EDX) analysis were conducted by a Tecnai F20 scanning transmission electron microscope (STEM, FEI Company, Oregon, USA) operated at 200 kV. The nitrogen adsorption-desorption isotherm was measured by a Quadrasorb SI analyzer (Quantachrome Instruments, Florida, USA). The specific surface area and porous properties of the prepared sample were determined by the Brunauer-Emmett-Teller (BET) and Barrett-Joyner-Halenda (BJH) methods. X-ray diffraction (XRD) patterns were recorded by a Rigaku SmartLab X-ray diffractometer (Rigaku Corporation, Tokyo, Japan) using high intensity Cu Kα1 irradiation (λ = 1.5406 Å), operating at 40 kV and 40 mA as the accelerating voltage and applied current respectively. UV–vis diffuse reflectance spectrum (UV–vis DRS) of the photocatalyst was measured by a Cary 100 UV–vis spectrophotometer (Agilent Technologies Inc., Santa Clara, CA, USA) equipped with a Labsphere diffuse reflectance accessory and BaSO₄ was used as the reflectance standard. X-ray photoelectron spectroscopy (XPS) spectra were measured by a Thermo ESCALAB 250XI X-ray photoelectron spectrometer (Thermo Scientific, Waltham, MA, USA) (Al Ka, 150 W) with calibration using C1s = 284.8 eV. Photocurrent measurements of the samples were performed by a CHI 760E electrochemical workstation (Shanghai

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