



Visible light responsive photocatalytic hydrogen evolution using MoS₂ incorporated ZnO

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Abstract

In this article, we prepared efficient ZnO@MoS₂ composites through hydrothermal and solvothermal method for photocatalytic hydrogen evolution. The structural, morphological, surface area and optical properties were investigated using transmission electron microscopy (HR-TEM), X-ray diffractometer (XRD), Brunauer Emmett Teller (BET), UV–visible (UV-vis) absorption and Photoluminescence (PL) emission spectroscopy. The incorporating effect of MoS₂ on the photocatalytic performance of ZnO photocatalyst has been studied. The PL emission spectra of prepared composites elucidate that recombination of electron/hole pairs is greatly suppressed owing to the incorporation of MoS₂ sheet-like nanostructures. The composite sample (3wt % of MoS₂ in ZnO) showed the excellent photocatalytic efficiency when compared to pure photocatalyst. The considerable increase in the efficiency of nanocomposites may be accredited to extended absorption region, favorable band structure, and effective separation of charge carriers, large surface area and the reactive active sites provided by layered structure of MoS₂. This study demonstrates that prepared composites could be promising and efficient photocatalysts for the evolution of hydrogen through water-splitting under visible light illumination.

Keywords ZnO@MoS₂ composite · H₂ evolution · Photocatalytic activity · Photocatalyst

Introduction

Clean and renewable energy is probably the most important challenge facing mankind in the twenty-first century. It is estimated that global energy demand doubled in the middle of this century, and by 2100, global energy demand will be tripled. One of the most important goals of our modern

society is to build a sustainable environment (Gertler et al. 2019). The growing environmental problems associated with the widespread use of unsustainable fossil fuels (oil, natural gas and coal) and the growing demand for energy will sooner or later force humans to use clean and sustainable energy. Over the years, the scarcity of fossil fuels from biological remains of dead animals and plants for hundreds of millions of years ago and the environmental problems caused by their combustion have prompted research into the development of novel renewable energy production technique. Several methods have been proposed so far (Papadimitriou 2019; Tronchin et al. 2018). A combination of photocatalysts and solar energy has been identified by investigators as an associated source of clean and abundant energy (Christoforidis and Fornasiero 2017). The sun produces about 3×10^{24} J of energy each year, about 12,000 times greater than current energy requirement (Shaner 2016). Therefore, solar energy can be used as an alternative energy source. So far, water-splitting converts solar energy into hydrogen and considered an effective hydrogen preparation method to solve the energy problem. Research on photocatalysis has been carried out since last century and make a significant

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contribution to renewable energy and environmental treatment process (cleaning of emissions and purification of water). In the past decades, the number of applications based on photocatalysis has greatly increased. Although various material systems have been developed, the production of H_2 from water is one of the most promising ways to fulfil the current environmentally friendly energy demand (Sattler 2017; Hosseini and Wahid 2019; Rathod et al. 2016) because this technology is based on the energy of photons (or solar energy), which is a source of clean and permanent energy, mainly water, a renewable resource. It is an environmental protection technology, free of harmful by-products and pollutants. Using photocatalytic process, conversion of solar energy to hydrogen is a noble solution for energy and environmental problems (Chen et al. 2017). But the biggest challenge in using this technology is to develop high-quality and efficient photocatalyst which must have properties, such as higher electron–hole pair separation rate and higher surface-to-volume ratio for maximum interaction (Di 2016; Tan 2017; Sreethawong et al. 2008; Wang 2019). A huge effort has been made recently to build efficient photocatalytic systems, based on semiconductor materials photocatalyst, such as metal oxides (Kakuta and Abe 2009; Alkaim 2013), organic polymers (Jun 2013; Schwab 2010), sulfides (Xie et al. 2014), phosphates (Yi 2010), oxy-nitrides (Maeda et al. 2013), etc. Among all of these semiconductors, photocatalyst ZnO is a strong candidate for efficient photocatalyst because of its low cost, excellent stability, availability, and wide band gap and non-toxic properties. However, for H_2 evolution, pure ZnO shows weak photocatalytic activity. Rapid electron–hole pair recombination before migrating to the surface through the reaction is one of the reasons for non-effective photocatalyst for H_2 evolution (Pan and Zhang 2012; Kumar and Rao 2015). Loading noble metal on the surface of ZnO is an effective method to form ZnO metal hetero-structure to eliminate electron–hole recombination and increase photocatalysis ZnO efficiency. Many successful systems have been established to combine ZnO and various precious metals (such as Ag, Pt, and Au) to evolve H_2 (Gao et al. 2013; Chung 2019; He 2014). Due to the high cost of these precious noble metals, these are not suitable commercially as a photocatalyst. So, it is necessary for making highly efficient ZnO-based photocatalyst to explore co-catalysts which are easily available and low cost. MoS_2 is an exceptional photocatalyst for H_2 evolution in photocatalytic activity and also shows extensive applications toward a number of semiconductors, such as C_3N_4 (Hou 2013) CdSe (Frame and Osterloh 2010), $ZnIn_2S_4$ (Wei 2014) and TiO_2 (Zhou et al. 2013). Experimental and theoretical results also show that the active site of MoS_2 for the H_2 production reaction of unsaturated sulfur atoms terminates at the end of Mo edge (Hinnemann 2005; Sabbah 2007). A large number of studies have shown that the activity of MoS_2 is higher than

from mostly used noble metal, such as Pb, Pt, Rh, Au and Pd (Sabbah 2007; Zong 2008). MoS_2 is not only suitable for (Hou 2013; Hinnemann 2005) electron–hole separation rate, but also provides favorable proton reduction sites in response to highly H_2 evolution reactions. Therefore, MoS_2 is considered the best co-catalyst for H_2 evolution and suitable alternative for noble metal because it is of less cost, outstanding photo-stability, easily available and non-toxic. ZnO is expected to hybridize with the MoS_2 layer, reduce electron–hole pair recombination rate and increase photocatalytic activity. Current research work focus on fabrication of ZnO– MoS_2 photocatalyst for hydrogen evolution. First, we synthesized pure ZnO and MoS_2 via hydrothermal and solvothermal method, respectively, and then combine both of these via hydrothermal method to study the effect of different concentration of MoS_2 in ZnO. Four composite samples of ZnO– MoS_2 are fabricated by varying MoS_2 (1–4%) concentration in pure ZnO.

Materials and methods

Fabrication of ZnO

To fabricate ZnO, 2 g of zinc acetate was dissolve in 80 ml ethanol. Then, NaOH solution was prepared in water separately and added drop-wise under constant stirring in zinc acetate solution until pH of the solution changed from 9 to 11 and maintained. Then, 80 ml solution was placed in sealed 100 ml Teflon autoclave and heated at 150°C for 12 h. After reaction was complete, autoclaves were allowed to cool at room temperature. ZnO nanoparticles in white color were collected after filtering, and then washed with ethanol three to four times. To get good crystallinity, calcination of the sample was done at 500 °C for 2 h.

Fabrication of MoS_2

To fabricate MoS_2 , as a starting material and source CH_4N_2S thioure, citric acid ($C_6H_6O_7$) and hepta-molybdate tetra-hydrate ($(NH_4)_6Mo_7O_{24} \cdot 4H_2O$) were used. Two solutions were prepared; in the first solution, 1.5 g of $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ with 0.5 g of $C_6H_6O_7$ was dissolved in distilled water under constant stirring at 85 °C for 30 min and during stirring, ammonia was added drop-wise until 4 pH of the solution was maintained. In the second solution, 1.30 g of CH_4N_2S was added drop-wise in distilled water under constant stirring on hot plate for 10 min. Then, both solutions were transferred to 100 ml Teflon autoclave and heated at 180 °C for 12 h. After reaction was complete, autoclaves were allowed to cool at room temperature. Black color precipitates were collected, then filtered and washed

with ethanol three to four times. To get good crystallinity, calcination of the sample was done at 400 °C for 2 h.

Assembling of ZnO–MoS₂ composite

Four samples (1–4) of composite ZnO–MoS₂ were prepared by varying the mass fraction of MoS₂ 1%, 2%, 3% and 4% in pure ZnO sample by hydrothermal method. First, sample was prepared by taking 99wt % of ZnO mixed with 1wt % of MoS₂ for gelatinization and dispersion and added in deionized water. Then, sample was centrifuged at 3000 rpm for 40 min and dried at 200 °C on magnetic stirrer. Similarly, second, third and fourth samples were prepared by varying 2wt %, 3wt % and 4wt % of MoS₂ in pure ZnO particles.

Characterization

Morphology and composition of the synthesized samples were determined by TEM (JEM-2100). Optical and electronic properties of fabricated samples were investigated by UV–visible spectroscopy (UV-1700, Shimadzu) and PL (FP-8200, JASCO), where the BET surface area of fabricated particles were determined using nitrogen (N₂) absorption device Micrometer TriStarII-3020 (Fig. 1).

Photocatalytic hydrogen production measurements

Green approach of H₂ production as a fuel is carried out in closed quartz reactor, and the reaction chamber was totally sealed so that no other gas exchange takes place in it. In the reaction chamber, 60 mg photocatalyst was used in 100 ml solution containing deionized water with 0.1 M Na₂S and 0.05 Na₂SO₃. 300-W Xeon Lamp was used as a light source for photocatalytic reaction with wavelength $\lambda \geq 400$ nm. Initially, reaction chamber was placed in the dark with constant

stirring for 50 min and then to make homogenous solution treated ultrasonically for 10 min, after this, N₂ gas was used to exhaust O₂ from the reactor. Finally, reactor was exposed to light for H₂ evaluation. In reaction chamber, H₂ evaluation was examined by GC-7890II chromatograph (Ar carrier, MS – 5 Å column, Beifen-Ruili, TCD and SP-2100).

Results and discussion

Photoluminescence (PL) spectrum is used to examine migration, electron–hole transfer efficiency and trapping in the semiconductors. PL spectra of pure ZnO and composite samples 1–4 were shown in (Fig. 2). PL spectra consist of two emission regions, one is UV region consisting of band-gap peaks that range 350–400 nm and the second one is general broad-band spectrum region inferring about the structural defect in fabricated samples that range 400–700 nm (Peng 2008; Park 2003). First, peaks of all samples observed in UV region almost at the same point show that because of minute doping variation in band-gap being very small. Other peaks are in visible region from 400 to 470 nm range and show the lattice defect in fabricated samples. Figure 2 depicts PL spectra, intensity peak falls down as compared to pure ZnO in composite samples, maximum intensity peak falls in sample three, high-intensity peak shows high electron–hole pair recombination, fall-down intensity peak representing electron–hole pair recombination decreases and suitable for photocatalytic reaction (Dong 2019; Li 2018). However, in sample 4, intensity peak once again rises by increasing the concentration of MoS₂ representing that further doping MoS₂ is not suitable as a photocatalyst.

UV–visible absorption spectra of pure ZnO and composite samples one and two are shown in Fig. 3.

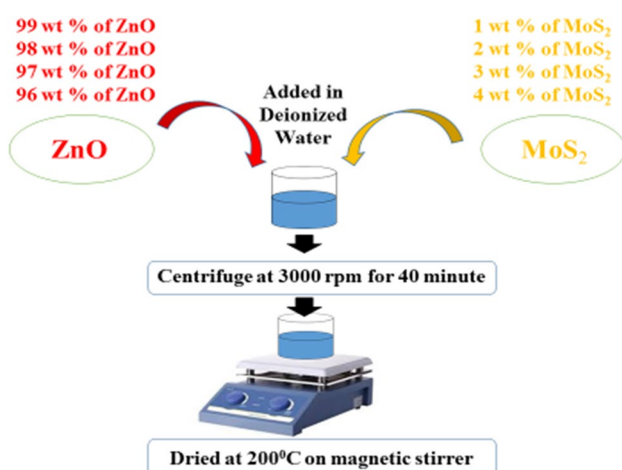


Fig. 1 Assembling of ZnO–MoS₂ composite

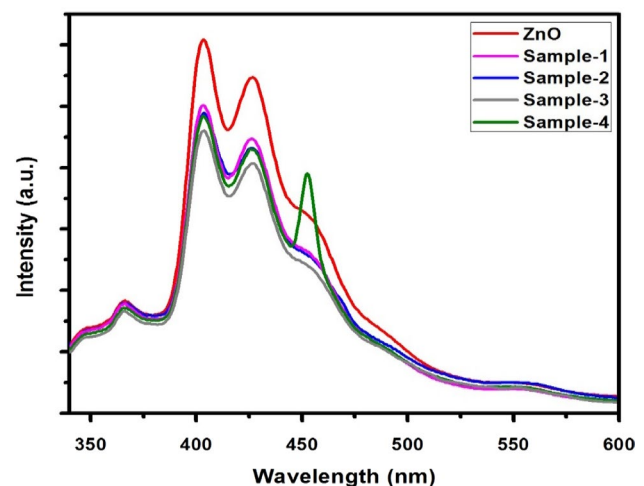


Fig. 2 Photoluminescence (PL) spectra of pure-ZnO composites (ZnO–MoS₂) samples

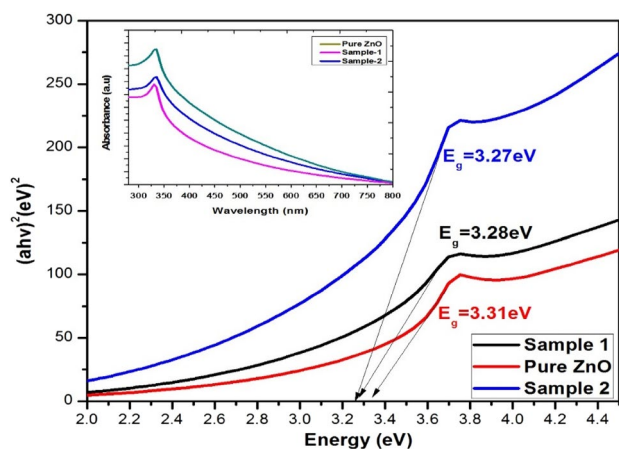


Fig. 3 UV-visible absorption spectra of pure-ZnO composites (ZnO-MoS₂) samples

Absorption peak of all three samples are observed in almost same range from 340 to 370 nm, which confirms, from doping of MoS₂ in the very minute of variation in band gap, that the same trend is observed in above PL spectra. Using Tauc plot relation, band gap of pure ZnO and composites sample were determine using relation describe in Eq. 1 (Saravanan 2016). In (Fig. 3), graph was plotted between energy versus α hv.

$$\alpha h\nu = K(h\nu - E_g)^n \quad (1)$$

Band gap of pure ZnO and composite samples are 3.31, 3.28 and 3.27, respectively. By varying the concentration of MoS₂, the band decreases toward the visible region because of very small doping minute variation observed like PL spectra but variation toward the visible region suitable for photocatalytic reaction (Nayak et al. 2015; Takanaabe and Domen 2011).

BET surface area of pure ZnO and composite (ZnO-MoS₂) samples 1, 2, 3 and 4 are investigated via nitrogen absorption-desorption spectra shown in (Fig. 4). Results show that pure ZnO particles have surface area 33.19 m²/g less than the ZnO-MoS₂ composite sample, as graphs in (Fig. 4) indicate, by increasing the concentration of MoS₂ in pure ZnO particle, surface area increased 39.43 m²/g, 76.23 m²/g and 129.79 m²/g, respectively, suitable for effective photocatalyst (Tahir 2018). In the last sample, by further increasing the concentration 4wt % of MoS₂ in pure ZnO, surface area decreased 116.02 m²/g as compared to sample 3. Same trend was also observed in TEM characterization pictures shown in (Fig. 5). By increasing the concentration in composite samples, particle size decreases as shown in (Fig. 5). 2-D clear nano-sheet morphology was observed in sample 3 that by further increasing the concentration of MoS₂ in sample 4, size increased and morphology scattered.

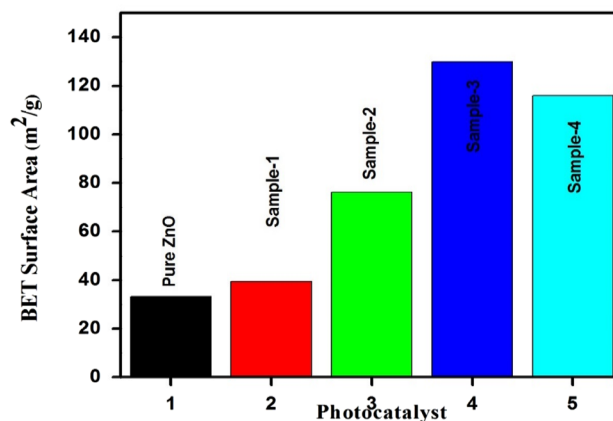


Fig. 4 BET-Surface area of pure-ZnO composites (ZnO-MoS₂) samples

Photocatalytic activity for H₂ evolution with pure ZnO, MoS₂ and with composite (ZnO-MoS₂) samples 1–4 were shown in (Fig. 5). Minimum catalytic efficiency 15 $\mu\text{molh}^{-1} \text{g}^{-1}$ was observed from pure MoS₂, then catalytic efficiency varied with pure ZnO 29 $\mu\text{molh}^{-1} \text{g}^{-1}$ but not enough as an efficient photocatalyst. H₂ evolution results from composite (ZnO-MoS₂) as compared to pure samples remarkably, maximum H₂ evolution 165 $\mu\text{molh}^{-1} \text{g}^{-1}$ obtained from sample 3 (ZnO mixed with 3wt % of MoS₂), by varying the concentration of MoS₂ in pure-ZnO 1%, 2% and 3%. H₂ evolution efficiency increased 54 $\mu\text{molh}^{-1} \text{g}^{-1}$, 117 $\mu\text{molh}^{-1} \text{g}^{-1}$ and 165 $\mu\text{molh}^{-1} \text{g}^{-1}$, respectively, but in sample 4 (ZnO mixed with 4wt % of MoS₂), further increase in concentration causes decrease in H₂ evolution 141 $\mu\text{molh}^{-1} \text{g}^{-1}$ as compared to sample 3.

Here, different factors play an important role in H₂ evolution for efficient photocatalyst surface-to-volume ratio and morphology have an important role as also observed here in this photocatalytic activity. In (Fig. 4), BET surface area graph shows that pure ZnO has the smallest surface-to-volume ratio, and sample 3 has the highest surface-to-volume ratio, and then sample 4 surface-to-volume ratio decreases. Surface-to-volume ratio directly links with particle size. Similarly, in Fig. 5, TEM images show pure MoS₂ has the maximum size 50 μm and composite sample 3 has the smallest size 10 nm. So, according to TEM and BET surface, characterization in composite sample by varying the concentration of MoS₂ in samples 1–3 size decreases and surface-to-volume ratio increases and in sample 4, size once again increases and surface-to-volume ratio decreases. Overall, H₂ evaluation results are also according to the same trend and photocatalytic efficiency also directly links with surface-to-volume ratio. When high surface-to-volume ratio and maximum photo-catalytic surface are available for reaction, better results are obtained (Fig. 6).

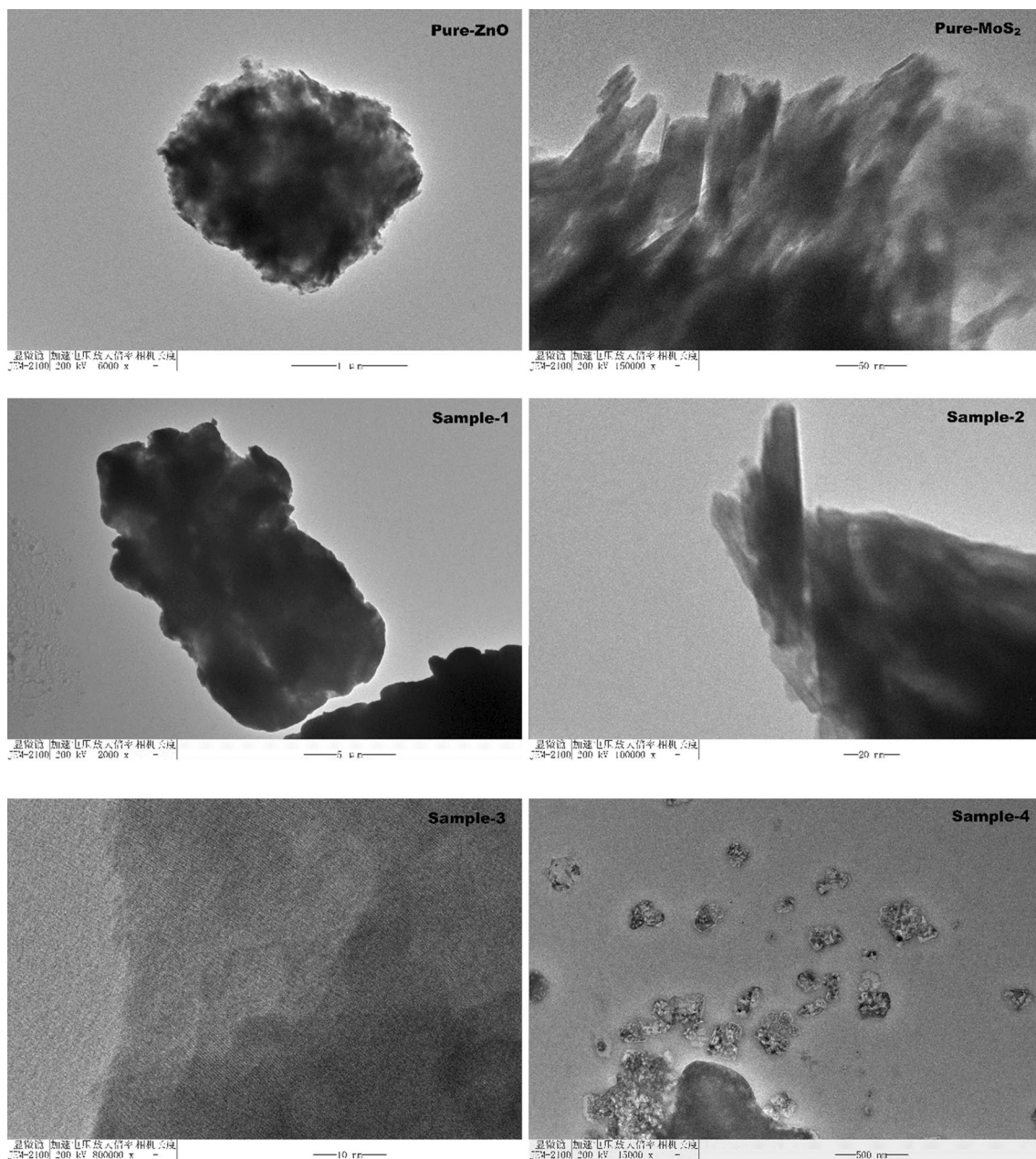


Fig. 5 TEM images of fabricated samples

Moreover, electron–hole pair recombination has an important role, weak activity of pure ZnO could be attributed to the rapid electron–hole pair recombination. MoS₂ loading on the pure ZnO surface caused a significant increase in H₂ production. Due to narrow band gap MoS₂, MoS₂ in composite ZnO–MoS₂ acts as photo-sensitizer like macro-molecule organic dye (Feng 2014). In addition, pure ZnO, after sensitization, effectively used visible light (Pawar and Lee 2014; Bu 2013). ZnO–MoS₂ intermediate contact and due to charge-carrier density at boundary, hole in MoS₂ diffuses in ZnO. Similarly, electron in pure

ZnO diffuses to the MoS₂ and form positive- and negative-charged regions. These charged regions are the source of intermediate electrostatic field and band bending shown in (Fig. 7). Due to electrostatic field, photo-excited electron in MoS₂ conduction band transfers to ZnO and hole in ZnO transfers to the MoS₂. In this way, in composite ZnO–MoS₂, electron–hole pair separation rate increases and causes maximum H₂ evolution (Tan 2014; Tajima 1990). H₂ evolution activity of the photocatalyst loaded with a relatively high amount of MoS₂ (4wt % of MoS₂ in pure ZnO) can be attributed to the shading effect of MoS₂

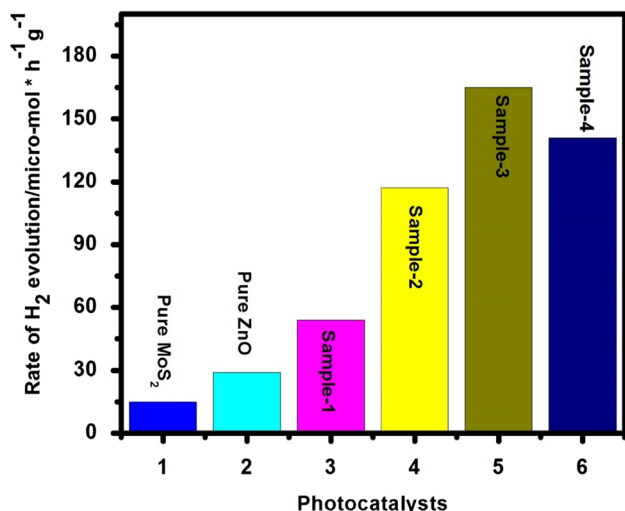


Fig. 6 Photocatalytic activity for H₂ evolution

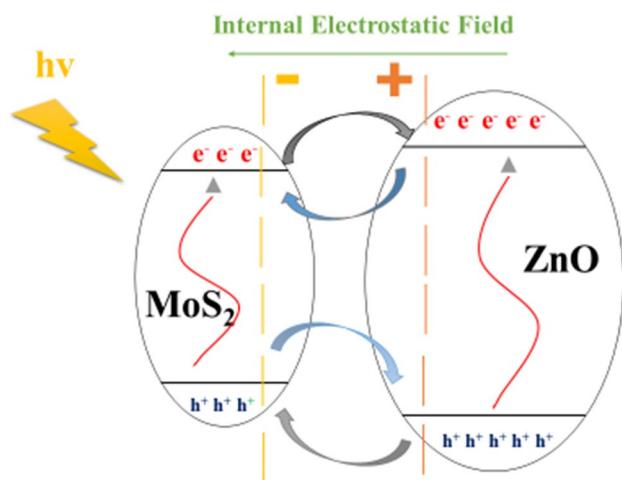


Fig. 7 Schematic diagram of electron hole pair separation due to internal electrostatic field

(Yuan 2015), which suppresses the easy absorption of light in ZnO component.

Conclusion

ZnO and MoS₂ samples were successfully fabricated via hydrothermal and solvothermal method, respectively, and ZnO-MoS₂ composite with different concentration of MoS₂ combined via hydrothermal method. For H₂ evolution, concentration of MoS₂ in composite samples (ZnO-MoS₂) plays an important role. In composite samples, varying concentration of MoS₂ directly affect the composite size, surface-to-volume ratio, electron-hole pair recombination rate and H₂ evolution activity. Maximum photocatalytic activity for

H₂ evolution was observed with composite (3wt % of MoS₂ in pure ZnO). Further increase in concentration may cause to lower the photocatalytic performance owing to surface-to-volume ratio and electron-hole pair recombination rate.

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Compliance with ethical standards

Conflict of interest The authors declare that there is no conflict of interest.

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