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Copper-doped flower-like molybdenum disulfide/bismuth sulfide photocatalysts for enhanced solar water splitting

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ABSTRACT

A facile synthesis approach to fabricate Cu-doped MoS₂/Bi₂S₃ (Cu-MoS₂/Bi₂S₃) photocatalysts is reported. The photocatalyst samples with varying amounts of Cu are applied in the photocatalytic splitting of water to produce H₂ under the irradiation of simulated solar light. The Cu-MoS₂/Bi₂S₃ photocatalysts with an optimum Cu loading of 20 mol% exhibited high photocatalytic performance, achieving a total H₂ yield of 32.4 μmol/h after 6 h of reaction. The photoactivity of the Cu-doped sample was shown to have risen more than 40% than that of pure MoS₂/Bi₂S₃. The improved performance is attributed to the impurity states generated within Cu-doped MoS₂, which serve as trapping sites for photogenerated electrons. The effective charge transfer mechanism achieved was evidenced by photoelectrochemical measurements. Based on the experimental results obtained, a plausible mechanism for the photocatalytic process associated with Cu-MoS₂/Bi₂S₃ was proposed.

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Introduction

With fossil fuels making up approximately 85% of the global demand for energy [1], there is an urgent need for alternative energy sources that are both sustainable and environmentally friendly. Solar energy is widely regarded as a free, abundant and endlessly renewable source of clean energy, which could tackle the global energy supply and related environmental issues [2]. Utilizing fuels generated from solar-chemical

reactions, such as hydrogen (H₂) from photocatalytic water splitting, could substitute fossil fuels with little change to the existing energy infrastructure and without the unwanted CO₂ emissions. Since the pioneering work of Fujishima and Honda in 1972 [3], photocatalytic water splitting into H₂ and O₂ using semiconductor photocatalysts has been accepted to be a promising strategy for converting solar energy into clean and carbon-neutral H₂ fuel [4].

The input energy of the overall water splitting reaction (H₂O → H₂ + ½ O₂) corresponds to a thermodynamic voltage

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requirement of 1.23 V [2]. In the absence of bias, the conduction band (CB) minimum of the semiconductor photocatalyst should be more negative than the reduction potential of H^+/H_2 ; while the valence band (VB) maximum should be more positive than the oxidation potential of O_2/H_2O . Despite the tremendous effort directed towards the development of efficient photocatalysts, the high recombination rate between photoinduced charge carriers, the rapid backward reaction between H_2 and O_2 in water, and the limited light harvesting ability of the photocatalysts restrict their practical applications. The present technology has yet to be successful in meeting the required goal of 30% quantum yield at 600 nm, which correspond to 5% solar energy conversion [5].

Various strategies have been studied to improve photocatalytic efficiencies, including co-catalyst impregnation, noble-metal loading, plasmonic sensitization and so on [6–10]. Platinum (Pt) and its alloys have been widely reported as the most efficient hydrogen evolution reaction (HER) catalysts, but commercialization of the photocatalytic system is greatly hindered owing to the high cost and scarcity of these materials. Hence, finding alternative and inexpensive earth-abundant materials while simultaneously attaining high catalytic activities remains a crucial task. Recently, transition metal dichalcogenides have emerged as an attractive non-noble metal catalyst with high catalytic efficiency based on theoretical and experimental studies [11–19]. In particular molybdenum disulphide (MoS_2), with the structure of S-Mo-S, has been reported to be highly efficient co-catalyst for photocatalytic H_2 production [20–22]. This is due to strong affinity of S atom at the edge of MoS_2 towards H^+ adsorption before reducing the H^+ ion to H_2 [23]. Despite its large availability of catalytically active edge sites, MoS_2 suffers from shortcomings such as high charge carrier recombination, unstable sulfide state, and formation of Mo-S-O links during photocatalytic reactions [24]. Furthermore, Yu et al. [25] reported that one of the major factors influencing MoS_2 as an electrocatalyst for H_2 evolution was its thickness, i.e. the number of MoS_2 layers. Numerous strategies have been applied to overcome the limitations of MoS_2 , including sulfur enrichment of Mo-edge sites, doping and forming heterojunctions with other semiconductors and exfoliation [26–28]. Exfoliation, where bulk MoS_2 is separated into few-layers through the aid of sonication, exposes more active sites for reaction. However, these sheets of MoS_2 tend to agglomerate over time hence reducing its photoreactivity [29]. Bismuth sulfide (Bi_2S_3), a well-known lamellar-like structure with a narrow band gap of 1.3–1.7 eV, has also garnered incessant research interest owing to its large absorption coefficient and reasonable incident photon to electron conversion efficiency [30,31]. In our previous report, Bi_2S_3 -modified MoS_2 hybrid composite was shown to exhibit a high H_2 production rate under simulated solar light [32]. Nevertheless, there is still room to increase the photocatalytic activity of the composites by further suppressing the charge recombination rate through the incorporation of other active components into the binary composites [33].

In recent years, transition metals dopants have been widely studied to improve the photocatalyst activity [34,35]. In addition, transition metals have shown to enhance the electronic properties [36] and increase the active sites of MoS_2 [20,37]. The use of metallic copper (Cu) and its oxides as co-

catalysts has been widely studied owing to their virtual non-toxicity, abundance as well as high conductivity and stability [38]. Cu-doped semiconductors have been demonstrated to be inexpensive, active and easily-producible nanocomposite photocatalysts [39,40]. Besides that, Cu-based photocatalyst such as Cu_2O nanowire has been shown to have remarkable photocatalytic activity towards H_2 production through water splitting [41]. Dong et al. [42] reported that in comparison to bare ZnS, Cu-doped ZnS composite was capable of suppressing electron-hole recombination due to its high photoinduced hole to electron ratio. Cu dopants were reported to have improved conductivity of the base photocatalyst, hence reducing the charge transfer resistance within the photocatalyst system [43,44].

Many researches involving use of non-metal dopants to increase the photoactivity of MoS_2 have been reported [45,46]. However, there are limited studies on the effect of transition metal on MoS_2/Bi_2S_3 for photocatalytic H^+ production. Herein, the effect of interstitial doping of Cu into MoS_2/Bi_2S_3 photocatalysts has been reported. The as-prepared Cu- MoS_2/Bi_2S_3 photocatalysts were evaluated for photocatalytic H_2 production under simulated solar light irradiation. As a proof of concept, we demonstrated that the Cu-doped MoS_2/Bi_2S_3 photocatalyst showed an improved activity towards the photocatalytic HER as compared to the undoped MoS_2/Bi_2S_3 . The photocatalytic enhancement can be attributed to the trap state formed within Cu-doped MoS_2 , which reduced the recombination of photogenerated charge carriers.

Materials and methods

Synthesis of Cu- MoS_2/Bi_2S_3

The hybrid composite of MoS_2/Bi_2S_3 was prepared according to our previous report [32] with slight modifications. In brief, the Bi_2WO_6 was firstly synthesized by dissolving 0.33 g of $Na_2WO_4 \cdot 5H_2O$ in 100 mL of deionized (DI) water, after which 0.97 g of $Bi(NO_3)_3 \cdot 5H_2O$ was slowly added. The mixture was then stirred for 1 h at ambient condition before transferred to a Teflon lined autoclave and subsequently heated at 160 °C for 15 h. After cooling to room temperature, the white precipitate was collected and washed with DI water and ethanol. The synthesized Bi_2WO_6 was then suspended in DI water with the aid of sonication for 1 h. Next, a pre-determined amount of sodium molybdate ($Na_2MoO_4 \cdot 2H_2O$) and thioacetamide (C_2H_5NS) were added into the suspension. In a separate beaker, copper acetate hydrate ($Cu(CO_2CH_3) \cdot xH_2O$) was dissolved in 10 mL of DI water. The Cu-containing solution was then added dropwise into the Bi_2WO_6 suspension under constant stirring. The mixture was placed in a Teflon lined autoclave for 24 h at 240 °C. After cooling to room temperature, the as-synthesized Cu- MoS_2/Bi_2S_3 were removed and washed thoroughly with DI water and ethanol. Finally, the samples were allowed to dry overnight in an oven. As a control, Bi_2S_3/MoS_2 was prepared following the same procedure but without adding the Cu precursor. The samples were designated as xCu- MoS_2/Bi_2S_3 , where “x” represents the percentage of Cu with respect to Mo content and set at 0, 5, 10, 20, 30 and 40 mol%.

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