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Molybdenum carbide microcrystals: Efficient and stable catalyst for photocatalytic H_2 evolution from water in the presence of dye sensitizer

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Abstract

Rod-like molybdenum carbide (Mo_2C) microcrystals were obtained from the pyrolysis of Mo-containing organic-inorganic hybrid composite. We investigated the photocatalytic H_2 evolution activity of Mo_2C by constructing a Mo_2C-dye sensitizer photocatalyst system. A high quantum efficiency of 29.7% was obtained at 480 nm. Moreover, Mo_2C catalyst can be easily recycled by simple filtration.

Keywords: Water splitting; Hydrogen production; Molybdenum carbide; Dye; Visible light

Solar energy has been considered as one of the alternatives to replace fossil fuels [1]. Solar light-induced H_2 evolution from water splitting reaction is an attractive approach to store solar energy in the form of H_2 fuel. Tremendous research efforts have been devoted to the development of novel photocatalyst systems with enhanced response to the more abundant visible light. Besides the well-studied heterogeneous photocatalyst system involving semiconductor particulates [2–6], dye-sensitized photocatalyst system composed of H_2 evolution center and photosensitizer (PS) is also attractive due to the wide light absorption ability of the sensitizer [7–12]. Noble metal (Pt, Rh, Ru, etc.) and their complexes have been well studied as H_2 evolution catalysts or PS [7–9,11,12]. However, from the economic point of view, noble-metal free photocatalyst systems are highly desirable. Transition metal (Co, Ni, Fe, etc.) based complexes and inorganic materials (oxide, sulfide, etc.) have shown promising H_2 evolution activity from aqueous solutions containing PS [13–19]. In recent years, molybdenum-based materials, especially molybdenum sulfides, have drawn much research interest for H_2 evolution reaction [20–25]. However, most of the studies focused on electrocatalytic water splitting [20,22–24]. Recently, commercially available molybdenum carbide (Mo_2C), which was commonly used in desulfurization [26], water gas shift [27], and hydrogenation reactions [28] was applied as an electrocatalyst for H_2 evolution [29–31]. Mo_2C was also combined with other semiconductors to construct photocatalyst system for water splitting [32]. The promising H_2 evolution activity of molybdenum carbide compared with Pt catalyst in water electrolysis and photocatalysis inspired us to construct a photocatalyst system composed of molybdenum carbide as the H_2 evolution catalyst, a low-cost xanthene dye of Erythrosin B (ErB) as the photosensitizer, and triethanolamine (TEOA) as the sacrificial reagent. Under visible light irradiation ($\lambda > 420$ nm), a promising H_2 evolution rate of 524.9 $\mu mol\ h^{-1}$ was obtained over the optimized sample and the quantum efficiency achieved 29.7% at 480 nm.

Typically, molybdenum carbide was prepared *via* a temperature programmed reduction (TPR) method which

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employed flammable and costly carbonaceous gas (H_2, CH_4, C_2H_6, etc.) as the carbon source [27,33–35]. In this work, molybdenum carbide was synthesized from the pyrolysis of a Mo-containing organic-inorganic hybrid composite. The composite can be formed between ammonium molybdate ($(NH_4)_6Mo_7O_{24}\cdot 4H_2O$) and 2-picolylamine (PA, $C_6H_8N_2$). After calcination of the Mo-PA complex under N_2 flow, rod-like molybdenum carbide microcrystals were obtained. The organic compound in Mo-PA complex served as carbon source for the formation of molybdenum carbide, which avoided the use of high cost carbonaceous gases.

Fig. 1 shows the FESEM and TEM images of Mo-PA complex and molybdenum carbide microcrystals obtained at 650 °C (entry 2 in Table S1). Mo-PA complex exhibited a microrod-like structure with a length of 1–5 μm and a diameter of hundreds of nanometers (Fig. 1a and b). After calcination at 650 °C for 2 h under N_2 flow, the rod-like structure was preserved for molybdenum carbide crystals with some shrinkage of both length and diameter (Fig. 1c and d). Due to the removal of organic compound in Mo-PA complex under high temperature, more rods with smaller sizes appeared. The HRTEM image shown in Fig. S1 further reveals that molybdenum carbide microrods are composed of nanoparticles with the diameter of 3–5 nm. The crystal phase of as-obtained microrods was identified by powder X-ray diffraction (XRD) measurement and the results are shown in Fig. 2A. The influence of calcination temperature on the formation of molybdenum carbide microcrystals was firstly investigated. The XRD pattern of Mo_2C sample obtained from commercial source (Sigma–Aldrich, Fig. 2A–f) was also included in Fig. 2A for comparison. At a relative low calcination temperature of 600 °C (Fig. 2A–a), only MoC crystals (MoC, cubic phase, JPCDS No: 65-0280) with broad and weak diffraction peaks were obtained. FTIR (Fig. S2) and elemental analysis (Table S1) results also confirmed that the organic compound was not totally removed in this sample. With the increasing of calcination temperature, Mo_2C crystals (α-Mo_2C, hexagonal phase, JCPDS No: 35-0787) can be obtained at 650 °C. Besides the strong diffraction peaks belonging to Mo_2C phase, a broad shoulder peak located at 2θ of 36.7° was

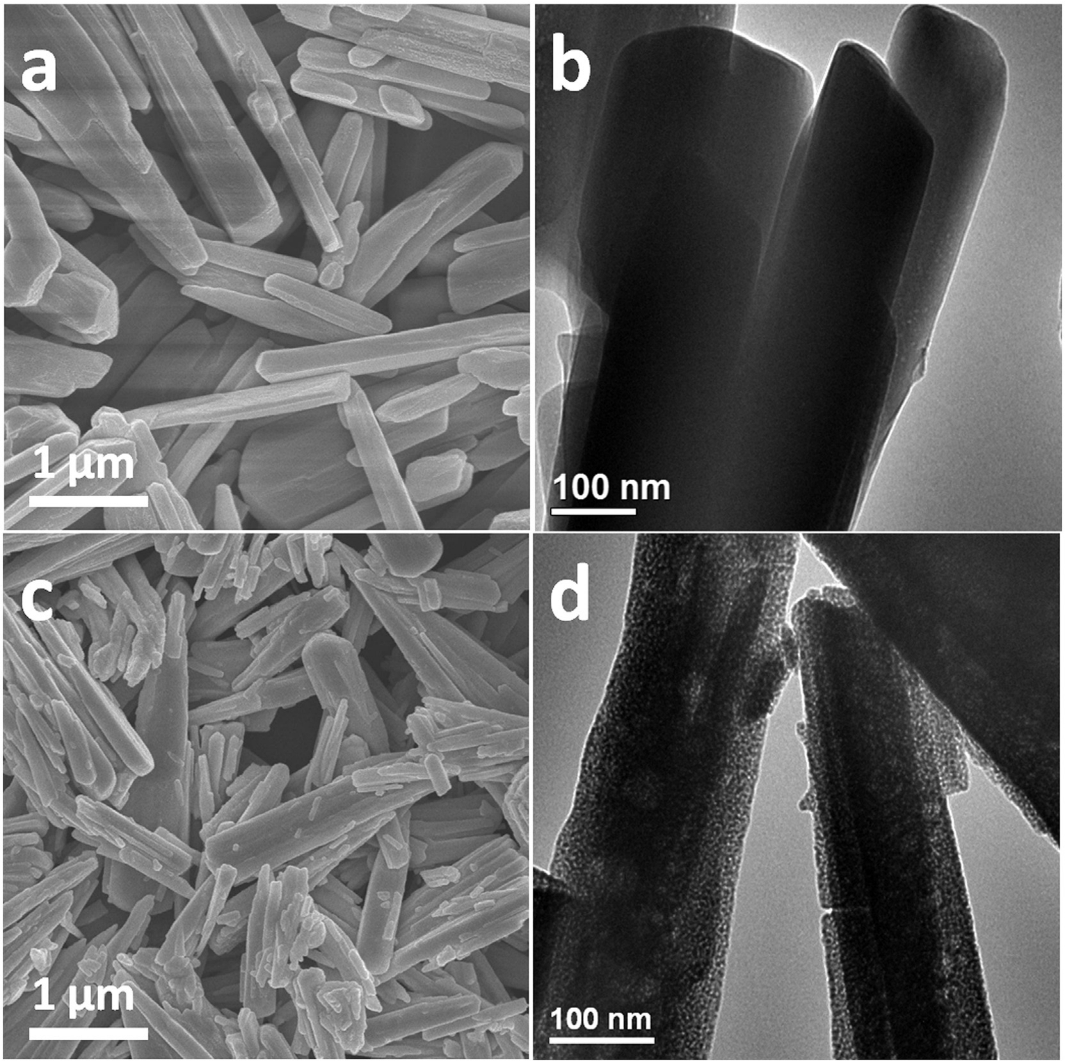

Fig. 1. FESEM and TEM images of Mo-PA complex (a, b) and molybdenum carbide microcrystals obtained at 650 °C (c, d).

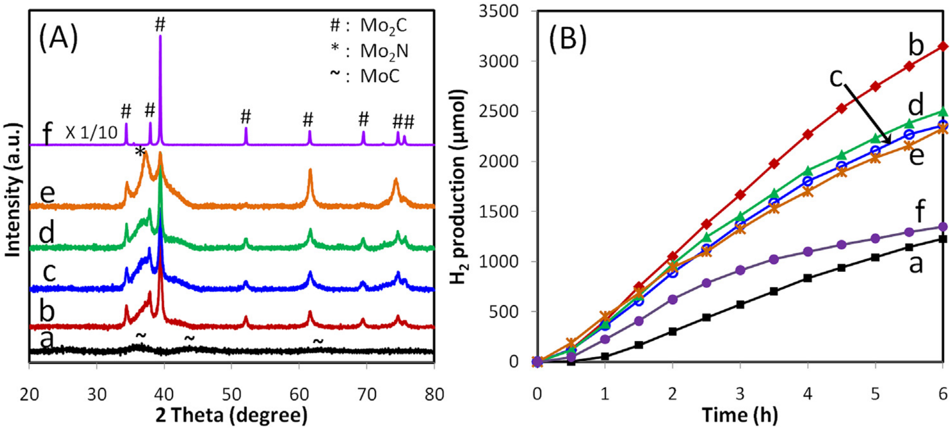

Fig. 2. XRD patterns (A) and H_2 evolution activities (B) of molybdenum carbide samples obtained at different calcination temperature and a constant ramping rate of 5 °C min^{-1} and calcination time of 2 h. (a) 600 °C; (b) 650 °C; (c) 700 °C; (d) 800 °C; (e) 900 °C; (f) Mo_2C-Sigma. Photocatalytic reaction conditions: 0.1 g catalyst, 0.2 g ErB, 100 mL 5% v/v TEOA aqueous solution (pH = 9, adjusted by HNO_3), 300 W Xe lamp with a 420 nm cut-off filter.

observed, which should be assigned to MoC phase. The formation of Mo_2C (major) and MoC (minor) mixed phase in the sample obtained at 650 °C was further confirmed by XPS measurement. As shown in Fig. S3, three molybdenum species were found after deconvolution. The Mo $3d_{5/2}$ XPS peaks with binding energies of 228.5 eV, 230.0 eV and 232.5 eV can be assigned to Mo^{2+}, Mo^{4+} and Mo^{6+} respectively, indicating the existence of Mo_2C and MoC phases [36,37]. The Mo^{6+} species detected by XPS should be attributed to surface oxides [37]. Further increasing the temperature led to the formation of molybdenum nitride (Mo_2N) phase as evidenced by the appearance of XRD peak at 2θ of 37.2°, especially at high temperature of 900 °C. Due to the existence of nitrogen element in PA molecule ($C_6H_8N_2$), the formation of molybdenum nitride is not surprising. Actually, according to elemental analysis results listed in Table S1, 1–2 wt% N element existed in almost all molybdenum carbide samples obtained over 650 °C, which may come from Mo_2N. It was also found that the carbon contents in molybdenum carbide samples were much higher than the theoretical values (5.89 wt% in Mo_2C and 11.13 wt% in MoC). Due to the high content of carbon (66.67 wt%) in organic compound of PA molecule ($C_6H_8N_2$), it is possible that amorphous carbon can be deposited in molybdenum carbide crystals during the pyrolysis procedure, leading to the high carbon content in the final products. The disadvantage of the deposited carbon is that it may block the mesopores in molybdenum carbide crystals, which led to the unexpected low specific surface area as listed in Table S1.

Although the obtained molybdenum carbide microcrystals contained a small amount of impurity phase of MoC, Mo_2N and amorphous carbon, they still gave promising H_2 evolution activities when combining with a photosensitizer, ErB dye. Control experiments showed that in the absence of ErB or molybdenum carbide, no H_2 gas could be detected under the same reaction conditions. The time course of H_2 evolution over molybdenum carbide samples is displayed in Fig. 2B. Sample obtained at 600 °C showed low H_2 evolution activity due to its low crystallinity as shown in Fig. 2A-a. With the increasing of calcination temperature to 650 °C, well crystallized Mo_2C sample was obtained, leading to its high H_2 evolution activity of 524.9 μmol h^{-1} in the first 6 h time duration. Mo_2C samples obtained at higher calcination temperature (e.g. 700 °C and above) did not give higher H_2 evolution activities, which may result from their slightly decreased specific surface area as well as the increased amorphous carbon content (Table S1). The activity of Mo_2C obtained from commercial source (Sigma–Aldrich) was also evaluated and shown in Fig. 2B-f. Although the commercial sample possesses much better phase purity and crystallinity as indicated by its much stronger XRD peaks in Fig. 2A-f, it showed low activity for H_2 evolution (Fig. 2B-f), which should be ascribed to its much smaller specific surface area of 0.80 m^2 g^{-1} (Table S1).

Other synthesis conditions including the ramping rate and calcination time for Mo_2C preparation were also investigated. It was found that the calcination condition did not affect the overall morphology of molybdenum carbide samples as shown in Fig. S4. H_2 evolution activities of as-obtained molybdenum carbide samples were also evaluated and the results are shown in Fig. S5 and Fig. S6. If the ramping rate was slow (e.g. 1 °C min^{-1}), only MoC crystals were obtained. And it showed relatively low activity for H_2 evolution. Increasing the ramping rate to 2.5 °C min^{-1} led to the formation of molybdenum carbide with a mixed phase of MoC and Mo_2C. With a higher ramping rate of 5 °C min^{-1}, more Mo_2C crystals appeared as evidenced by the strong diffraction peaks belonging to Mo_2C (Fig. S5A). And further increasing ramping rate to 10 or 20 °C min^{-1} did not affect the crystal phase of molybdenum carbide. Experimental and computational studies showed that the Mo edge is the active site in Mo-based materials for electrocatalytic water splitting reaction [20,38]. Since more Mo-edge existed in Mo_2C than that in MoC crystal, a higher H_2 evolution activity was expected over Mo_2C than that over MoC, which was also confirmed by the photocatalytic H_2 evolution results shown in Fig. S5. Mo_2C samples obtained with fast ramping rate (e.g. 5, 10, 20 °C min^{-1}) showed similar higher H_2 evolution activities than sample obtained with slow ramping rate (e.g. 1, 2.5 °C min^{-1}). When considering the effect of calcination time duration, it was found that a long time of 6 or 12 h did not affect the crystal phase or crystallinity of Mo_2C samples (Fig. S6A), compared with a short calcination time of 2 h. Thus, only negligible difference of H_2 evolution activity was observed between Mo_2C samples obtained with different calcination time (Fig. S6B).

Compared with heterogeneous photocatalyst systems containing only semiconductor particulates, one of the advantages of dye-sensitized photocatalyst systems lies in its wide-range light absorption ability. Fig. 3A displays the time course of H_2 evolution over Mo_2C microcrystals (5 °C min^{-1}, 650 °C, 2 h) under light with different wavelength range. A H_2 evolution rate of 524.9 μmol h^{-1} was obtained when a 420 nm cut-off filter was equipped. Increasing the wavelength of cut-off filter led to the gradually decreasing of H_2 evolution activity since less visible light is available. Interestingly, it was found that even under the light irradiation with the wavelength longer than 520 nm, a total 891 μmol of H_2 gas still could be collected in 6 h. The quantum efficiency (QE) of Mo_2C–ErB photocatalyst system was also examined in the visible light range as shown in Fig. 3B. The highest QE of 29.7% was achieved at 480 nm and it still remained 18.0% at 520 nm. Such features are highly desirable for the utilization of visible light. It was also noticed that at 420 nm and 440 nm, although light absorption was not high, the QE still reached 20.8% and 28.8%, which should be ascribed to the high energy of light with short wavelength.

When using inorganic material as H_2 evolution centers, the recyclability of solid catalyst is another advantage. Owing to the relative large particle size in micrometer range, Mo_2C microcrystals can be easily collected by simple filtration after photocatalytic reaction. Upon re-dispersion in fresh TEOA aqueous solution containing ErB as the PS, the H_2 evolution activity can be totally resumed, even after 6 runs (each run for 10 h, Fig. 3C), indicating the good stability and recyclability

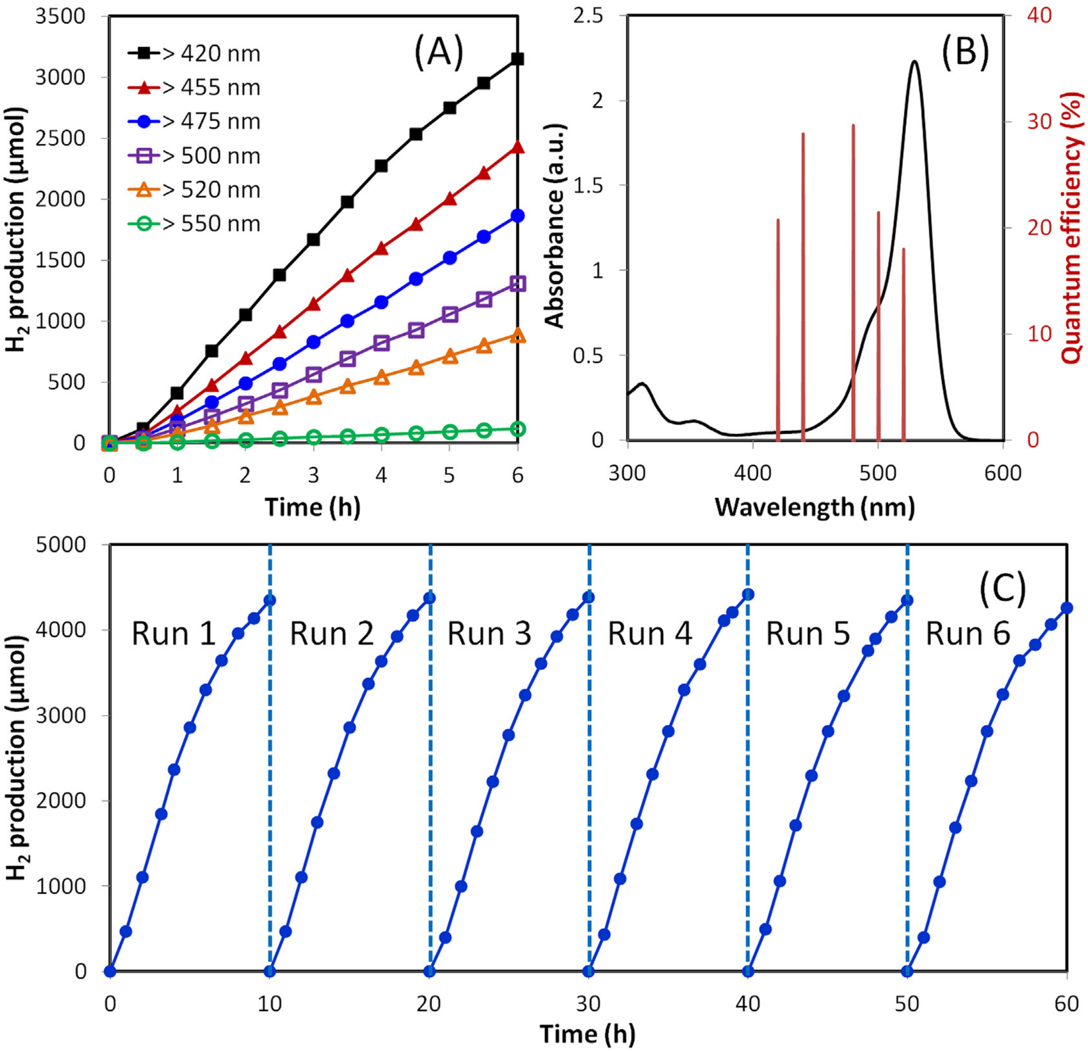

Fig. 3. (A) Time course of H_2 evolution using 0.1 g of Mo_2C sample (5 °C min^{-1}, 650 °C, 2 h) and 0.2 g of ErB with a 300 W Xe lamp equipped with different long-pass cut-off filters, (B) quantum efficiencies for photons with different wavelength over the Mo_2C sample (5 °C min^{-1}, 650 °C, 2 h), and (C) time course of H_2 evolution in the recycle study. Other photocatalytic reaction conditions are the same as those in Fig. 2.

of Mo_2C microcrystals. It was also found that H_2 production rate gradually decreased from 556 − 592 μmol h^{-1} in the first 4 h to 190−216 μmol h^{-1} in the 10th hour, which mainly be ascribed to the degradation of ErB molecules. Previous studies showed that iodide groups in ErB are gradually lost upon cleavage of the C−I bond during photoirradiation, leading to halogen-free Fluorescein (FL) molecules [16,39].

Besides Erythrosin B, other xanthene dyes including Erythrosin Yellowish (ErY), Eosin Yellowish (EY), Rose Bengal (RB) and Fluorescein (FL) were also investigated as the PS for molybdenum carbide catalyst. The molecular structures of these dyes and their corresponding sensitization effects for H_2 evolution are displayed in Figure S7. Among all the dyes investigated, ErB was found to be the best PS for molybdenum carbide catalyst. It was reported that different halogen substitution in xanthene dyes affects their ground and excited states, leading to the difference in light absorption and potential of photogenerated electrons [16]. Heavier halogen atoms substitution made the formation of long-lived triplet states of xanthene dyes more easily [40]. Thus, ErB outperformed other dyes in H_2 evolution activity.

The present work realized the photocatalytic H_2 evolution ability of molybdenum carbide from water splitting in the presence of dye sensitizer. The constructed Mo_2C-ErB photocatalyst system exhibited promising H_2 evolution activity even under light irradiation with long wavelength. This work inspires us to explore other novel transitional metal-based materials for photocatalytic H_2 evolution, besides oxides and sulfides.

Acknowledgements

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Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.jmat.2016.09.003.

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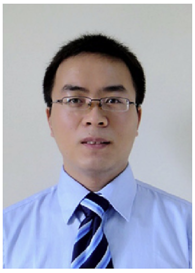

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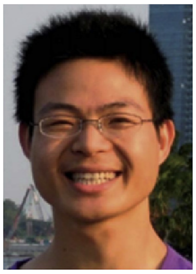

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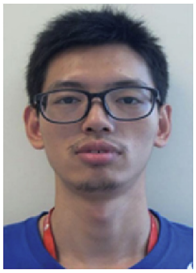

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