

Electronic Supporting Information

Construction of CdS/MoS₂ Heterojunction from Core-shell

MoS₂@Cd-MOF for Efficient Photocatalytic Hydrogen Evolution

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Section S1 Experiments

Materials: Ethylsilicate (TEOS), thioacetamide (CH_3CSNH_2 , TAA), and absolute ethanol (EtOH) were received from Sinopharm Chemical Reagent Co., Ltd. $\text{NH}_3\cdot\text{H}_2\text{O}$ (28 w.t.%) was purchased from Xilong Chemical Reagent Co., Ltd. Cadmium nitrate tetrahydrate ($\text{Cd}(\text{NO}_3)_2\cdot 4\text{H}_2\text{O}$) was obtained from Aladdin Reagent Co., Ltd. 2-methylimidazole (2-MI) was provided by J&K Co., Ltd. DI-water was self-made in the laboratory. All the reagents are used without further purification.

Synthesis method of SiO_2 : The SiO_2 sphere was prepared by using Stöber's method. In a typical procedure, 9 mL $\text{NH}_3\cdot\text{H}_2\text{O}$ was added into a mixed solution of 16.25 mL EtOH and 24.75 mL H_2O under vigorous stirring, and named solution A. Solution B is composed of 4.5 mL TEOS and 45.5 mL EtOH. Solution B was poured into solution rapidly with vigorous stirring at about 1000 rpm, with little touch between solution B and the beaker wall. After 1 min, the stirring rate was set at 360 rpm, the beaker was sealed and reacted for 2 h at RT (30 °C).

Synthesis method of CdS: The CdS was synthesized by two steps of solvothermal method. Firstly, 574 mg (7 mmol) 2-MI was dissolved by ultrasound with 20 mL methanol. Then 539 mg (1.75 mmol) $\text{Cd}(\text{NO}_3)_2\cdot 4\text{H}_2\text{O}$ and 973 μL TEA were dissolved into 20 mL methanol. The Cd^{2+} -MeOH solution was added dropwise into the solution of 2-MI and stirred for 20 min. After aging for 24 h, the precipitates were collected and washed. Secondly, 40mg (0.35 mmol) of as-synthesized Cd-MOF was dispersed with 15 mL ethanol. 187 mg (2.5 mmol) TAA was added and then heated at 70 °C for 2 h under stirring. The precipitates were collected by centrifugation and washed with ethanol. The orange CdS product was dried at 60 °C in an oven.

Section S2 Tables & Figures

Table S1 ICP-OES results of series of CdS/MoS₂ heterojunction.

sample	Mass ratio (Cd:Mo)	mole ratio (Cd:Mo)
CdS/MoS ₂ -0.15	0.15/1	0.13/1
CdS/MoS ₂ -0.4	0.44/1	0.38/1
CdS/MoS ₂ -0.5	0.57/1	0.49/1
CdS/MoS ₂ -0.6	0.71/1	0.61/1
CdS/MoS ₂ -0.7	0.83/1	0.71/1

Table S2 Comparison of average photocatalytic H₂ evolution rate of CdS/MoS₂ heterojunction photocatalyst under the UV-visible and the visible light irradiation.

	Average H₂ evolution rate (μmol h⁻¹ g⁻¹)				
	CdS/MoS₂-0.15	CdS/MoS₂-0.4	CdS/MoS₂-0.5	CdS/MoS₂-0.6	CdS/MoS₂-0.7
UV-visible light	925.6	3125.6	3922.1	5587.1	5003.5
visible light	528.0	939.5	1124.7	1870.0	1722.2

Fig. S1 SEM images of CdS/MoS₂-DP composite.

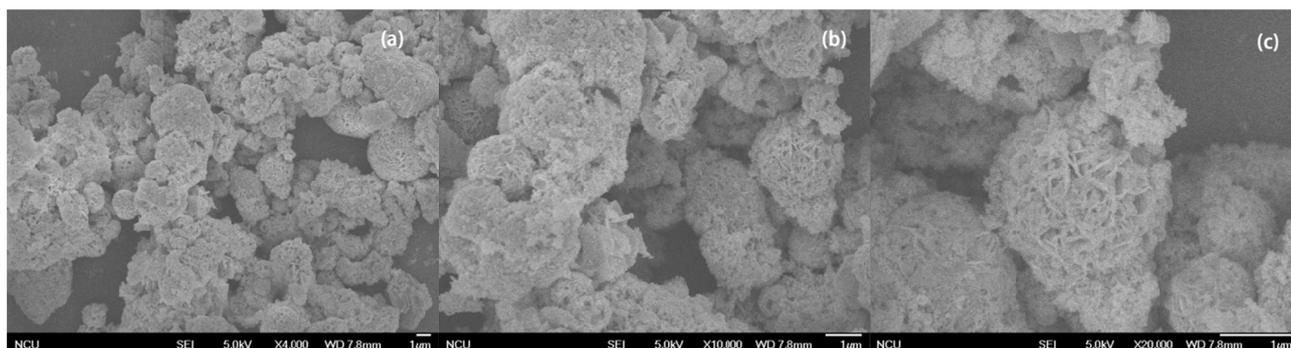


Fig. S2 TEM element Mapping results of CdS/MoS₂-DP that synthesized by a conventional deposition-precipitation method: TEM images (a-c); element mapping image of Mo (d), Cd (e), and S (f); EDS images (g), and element mapping image of Mo plus Cd (h).

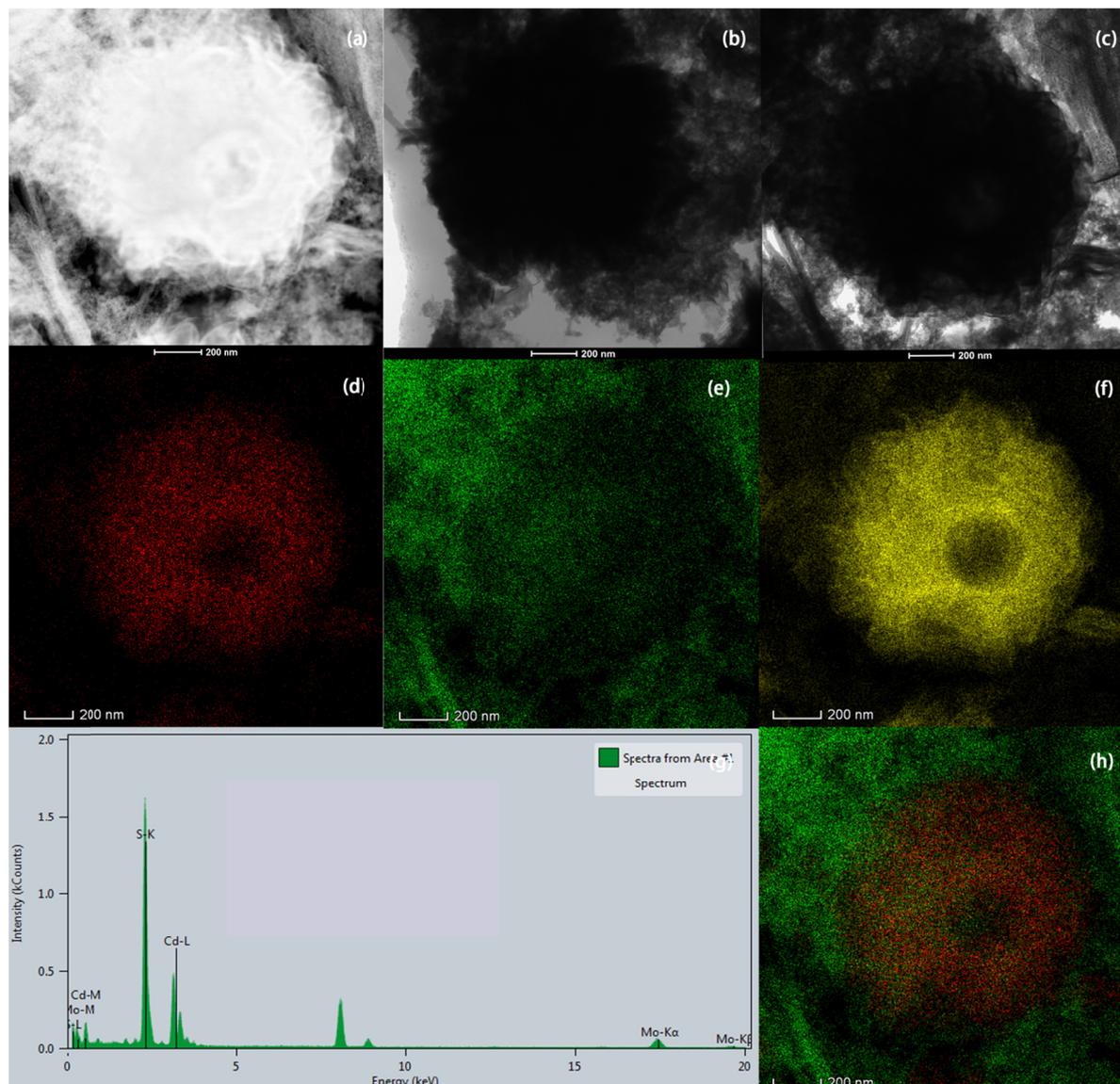


Fig. S3 Comparison of TEM images of CdS/MoS₂ heterojunction freshly synthesized and after photoirradiated (a, f), the element Mapping images of Mo (b, g), Cd (c, h), S (d, i), and Mo plus Cd (e, j), and the corresponding TEM-EDX images of CdS/MoS₂ heterojunction derived from the core-shell structure of MoS₂@Cd-MOF before and after photoreaction (k, l).

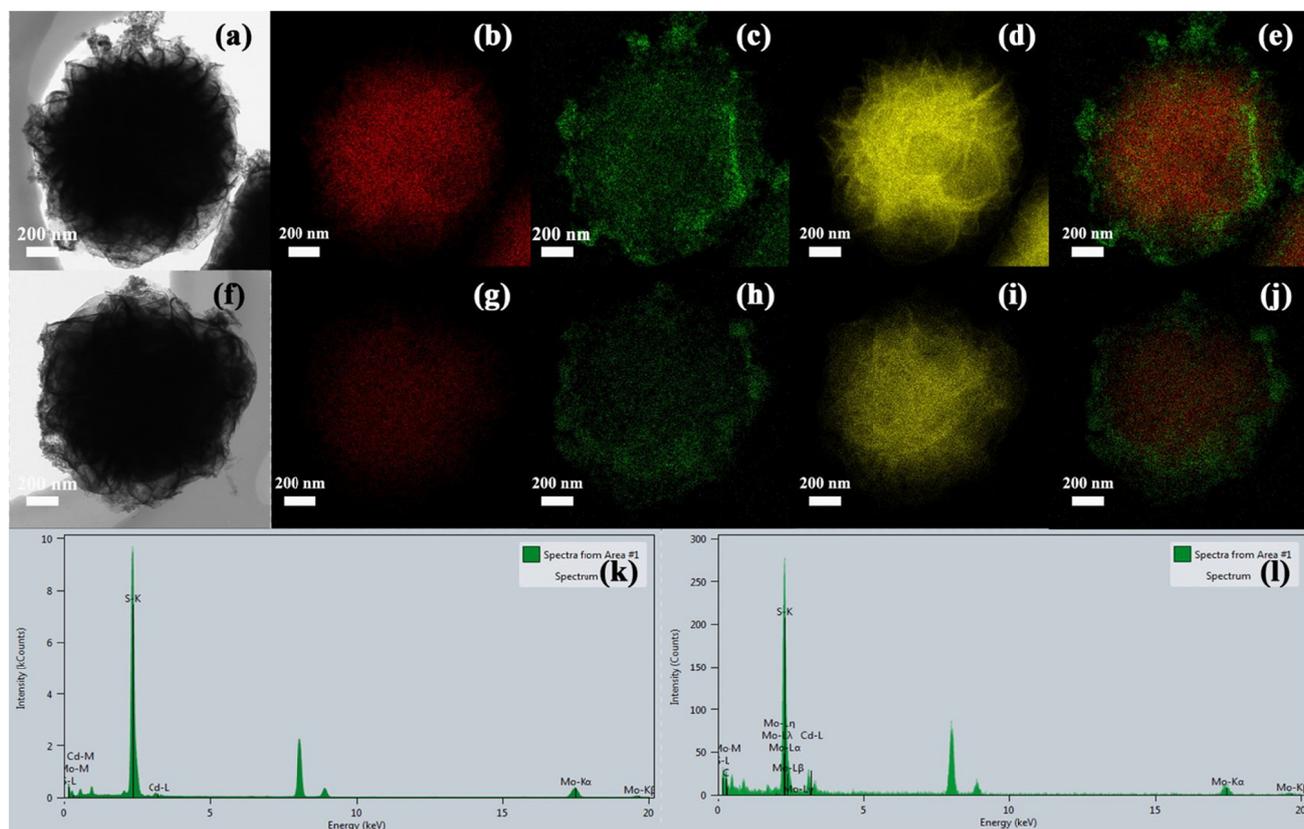


Fig. S4 Cycle measurement of hydrogen evolution performance with CdS/MoS₂ heterojunction photocatalyst under the irradiation of a 300 W Xe lamp.

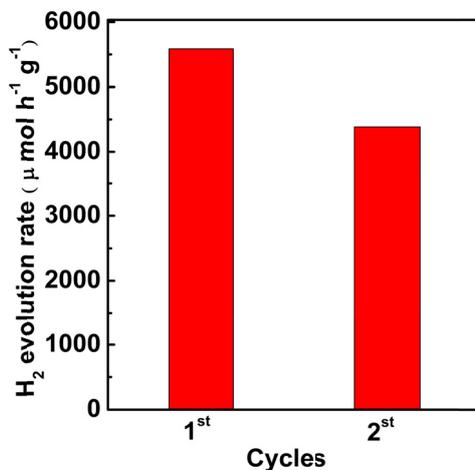


Fig. S5 XPS spectra of (a) Mo 3d, (b) Cd 3d, and (c) S 2p of CdS/MoS₂ heterojunction before and after photoreaction.

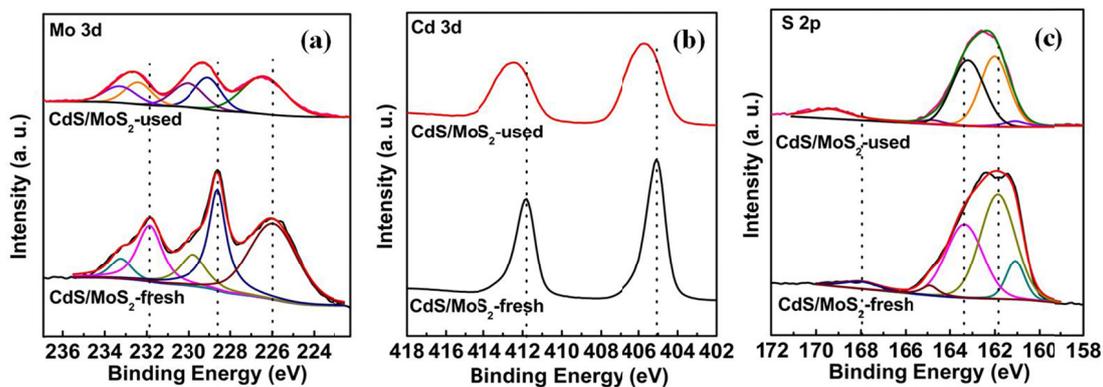


Fig. S6 Plots of transformed Kubelka–Munk function $(F(R)h\nu)^2$ vs light energy $(h\nu)$.

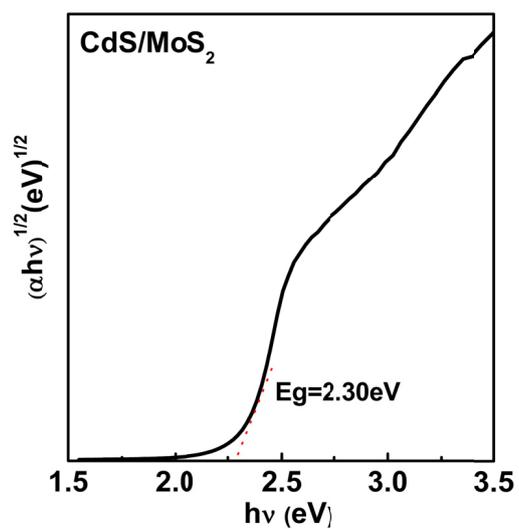


Fig. S7 Optical photos of MoS₂ (a), MoS₂@Cd-MOF (b), CdS/MoS₂ heterojunction (c), and CdS (d).

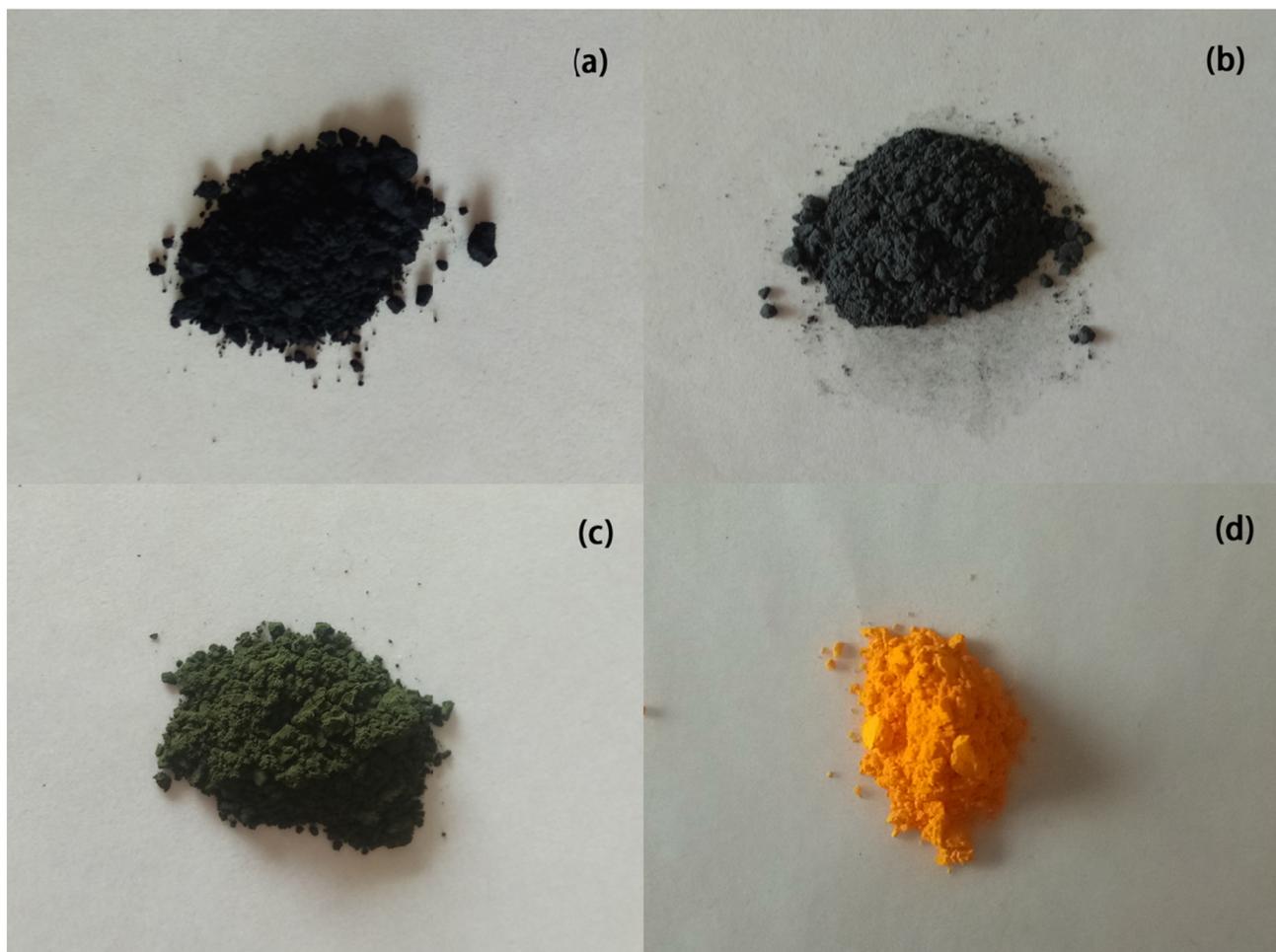
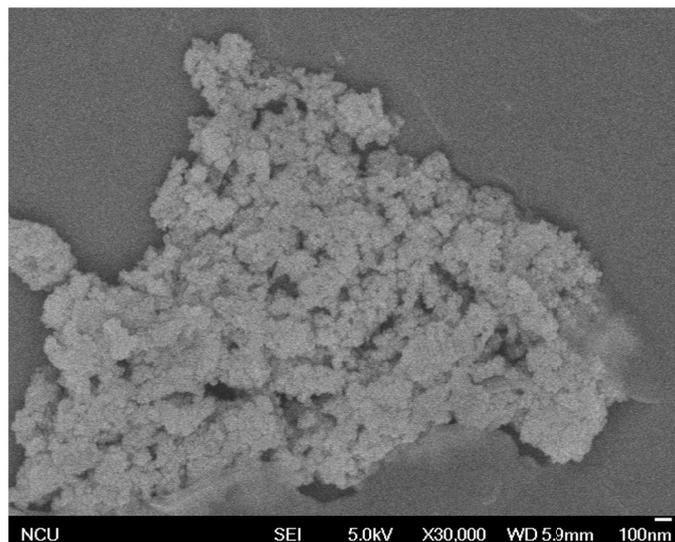


Fig. S8 SEM images of CdS nanocrystals derived from the Cd-MOF precursor.



Reference:

- 1.K. Fan, Z. L. Jin, G. R. Wang, H. Yang, D. D. Liu, H. Y. Hu, G. X. Lu and Y. P. Bi, *Catalysis Science & Technology* 2018, 8,2352;
2. M. Wang, S. B. Naghadeh, C. H. Li, L. Ying, A. L. Allen and J. Z. Zhang, *Science China Materials* 2018, 61 (6), 839-850.