

Supporting Information

***In situ* ion exchange synthesis of MoS₂/g-C₃N₄ heterojunction for highly efficient hydrogen production**

Min Wang,^{a, b, 1} Peng Ju,^{d, 1} Yun Zhao,^{a, b} Jiajia Li,^{a, b} Xiuxun Han^{a, c, *} and Zhaomin Hao^e

^a Laboratory of Clean Energy Chemistry and Materials, Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences, 18 Tianshui Middle Road, Lanzhou 730000, PR China

^b University of Chinese Academy of Sciences, 19 (Jia) Yuquan Road, Beijing 100039, PR China

^c Qingdao Center of Resource Chemistry & New Materials, 36 Jinshui Road, Qingdao 266100, PR China

^d Marine Ecology Center, The First Institute of Oceanography, State Oceanic Administration, 6 Xianxialing Road, Qingdao 266061, PR China

^e Henan University, 85 Minglun Road, Kaifeng 410200, PR China

* Corresponding author

Tel.: +86-931-4968054; Fax: +86-931-4968054

E-mail: xxhan@licp.cas.cn (X. Han)

¹ These two authors contributed equally to this work.

Table S1 MoS₂ loading amount in MoS₂/CNNS samples determined by ICP-AES.

	Composite (g L ⁻¹)	Mo (mg L ⁻¹)	S (mg L ⁻¹)	MoS ₂ /composite (wt%)
MoS ₂ /CNNS(15)	1.0	12.2	8.2	2.0
MoS ₂ /CNNS(20)	1.0	23.9	15.5	3.9
MoS ₂ /CNNS(30)	1.0	34.7	22.3	5.7
MoS ₂ /CNNS(50)	1.0	34.9	22.5	5.7

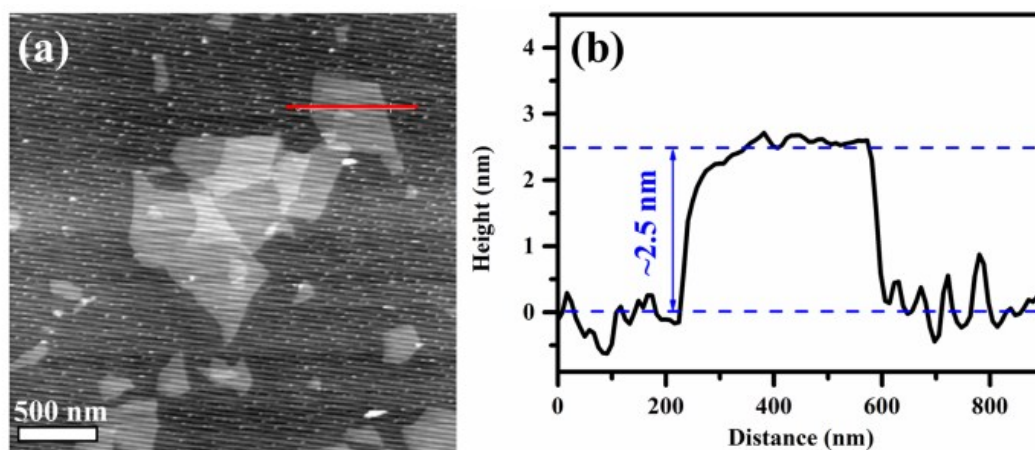


Fig. S1 (a) AFM image of CNNS deposited on the silicon wafer substrate, (b) The corresponding height curve determined along the red line in (a).

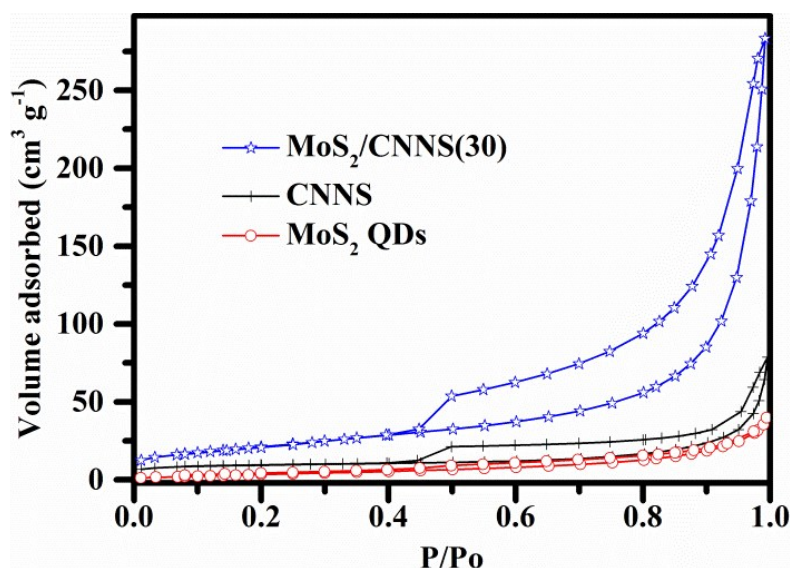


Fig. S2 N₂ adsorption/desorption isotherm curves of MoS₂ QDs, CNNS, and MoS₂/CNNS(30).

The calculations of BET specific surface area were based on N₂ adsorption/desorption isotherm curves of MoS₂ QDs, CNNS and the MoS₂/CNNS(30) composite, as shown in Fig. S2. MoS₂ QDs possess a small surface area (29.15 m² g⁻¹) due to aggregation, while the MoS₂/CNNS(30) composite shows the larger specific surface area (78.95 m² g⁻¹).

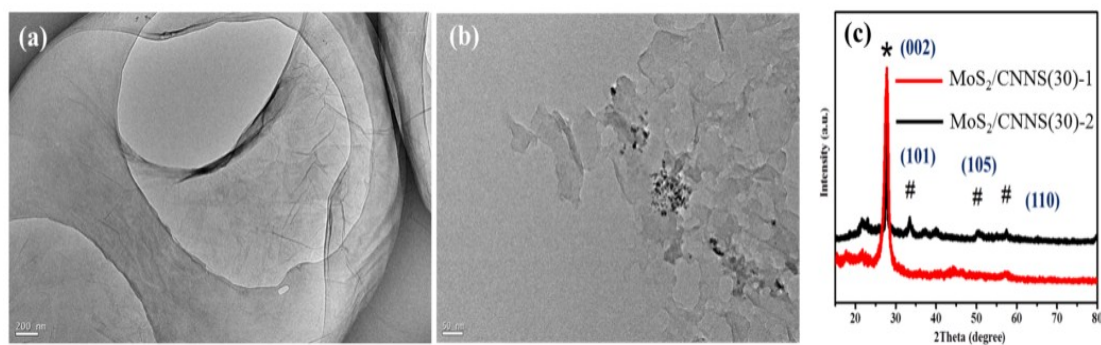


Fig. S3 TEM images of (a) MoS₂/CNNS(30)-1 and (b) MoS₂/CNNS(30)-2; (c) XRD patterns of MoS₂/CNNS(30)-1 and MoS₂/CNNS(30)-2.

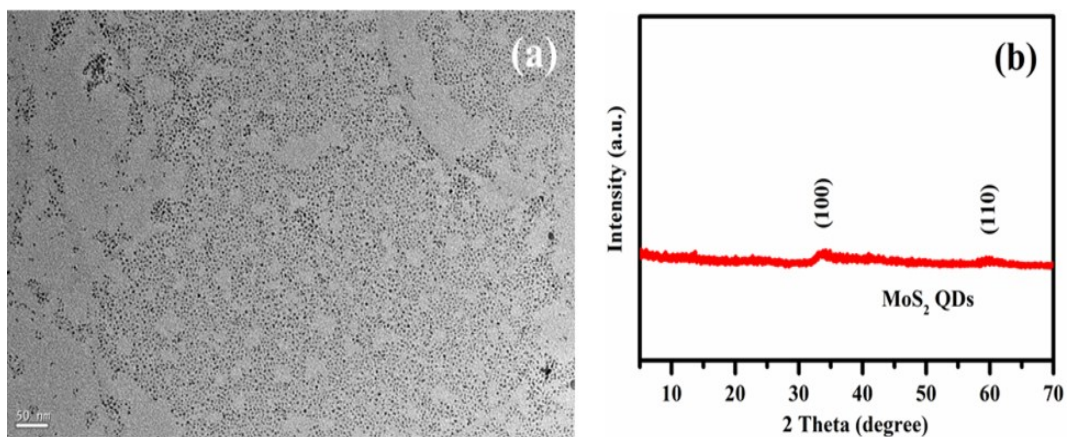


Fig. S4 (a) TEM image and (b) XRD pattern of MoS₂ QDs.

MoS₂ QDs were characterized by means of TEM and XRD. A typical TEM image of the MoS₂ QDs (Fig. S4a) shows highly uniform and monodisperse nanocrystals in narrow distribution of 2.15 ± 0.34 nm in diameter (the size is statistically calculated from more than 100 QDs in the TEM images). It can be seen in Fig. S4b that two major diffraction peaks can be clearly observed in the XRD pattern of MoS₂ QDs, which can be attributed to the (100) and (110) planes of MoS₂, respectively. Moreover, it can be seen that the peak intensity is weak, implying the poor crystalline of MoS₂ QDs. These results are in good agreement with the morphology and phase structure of MoS₂ QDs in MoS₂/CNNS composites.

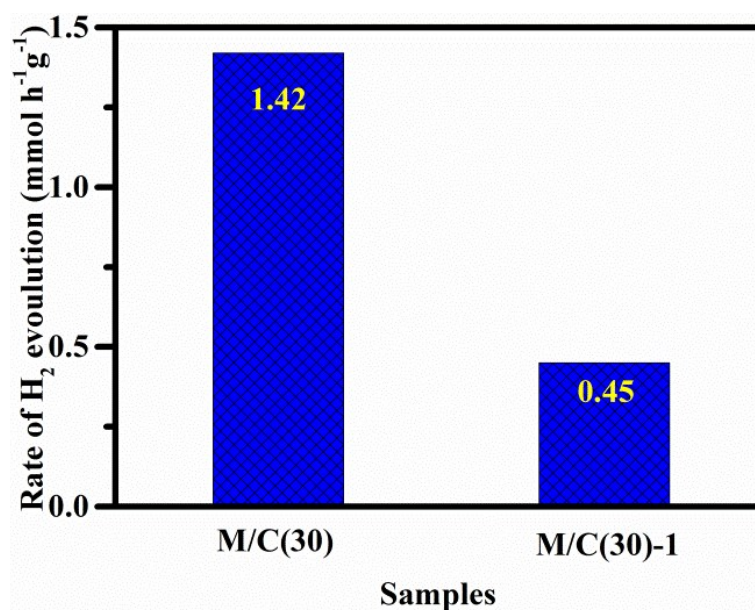


Fig. S5 Photocatalytic H₂ production rates of MoS₂/CNNS (30) and MoS₂/CNNS (30)-1.

Table S2 Comparison of the photocatalytic performance of MoS₂/CNNS prepared by ion exchange process and MoS₂/C₃N₄ photocatalysts reported previously.

Photocatalyst	Light source	Concentration of photocatalysts (g L ⁻¹)	H ₂ evolution (μmol h ⁻¹ g ⁻¹)	Ref.
0.2 wt% MoS ₂ /mpg-CN (+1 wt% Pt)	300 W Xe lamp (λ > 420 nm)	0.2	1250	1
0.5 wt% MoS ₂ /g-CN (+5 wt% Pt)	300 W Xe lamp (λ > 400 nm)	0.8 g	231	2
2.89 wt% MoS ₂ /g-C ₃ N ₄ (+5 wt% Pt)	300 W Xe lamp (λ > 400 nm)	1.0 g	252	3
1 wt% MoS ₂ QDs/CN (+1 wt% Pt)	300 W Xe lamp (λ > 420 nm)	0.5 g	393	4
0.5 wt% MoS ₂ /HCNS (+1 wt% Pt)	300 W Xe lamp (λ > 420 nm)	0.2 g	1340	5
5.7 wt% MoS ₂ /CNNS (without Pt)	Simulated Sunlight (AM1.5G)	0.5 g	1420	This work

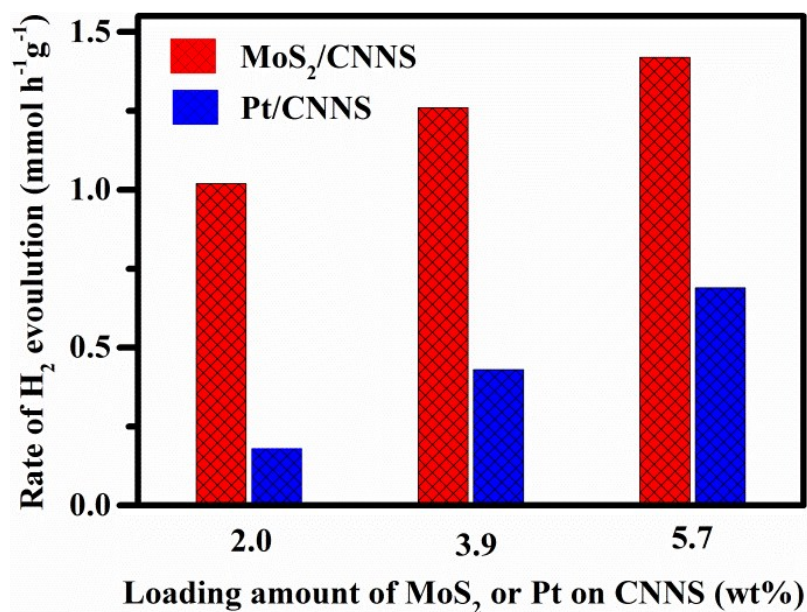


Fig. S6 The H₂ production rate of CNNS loaded with different amounts of MoS₂ or Pt.

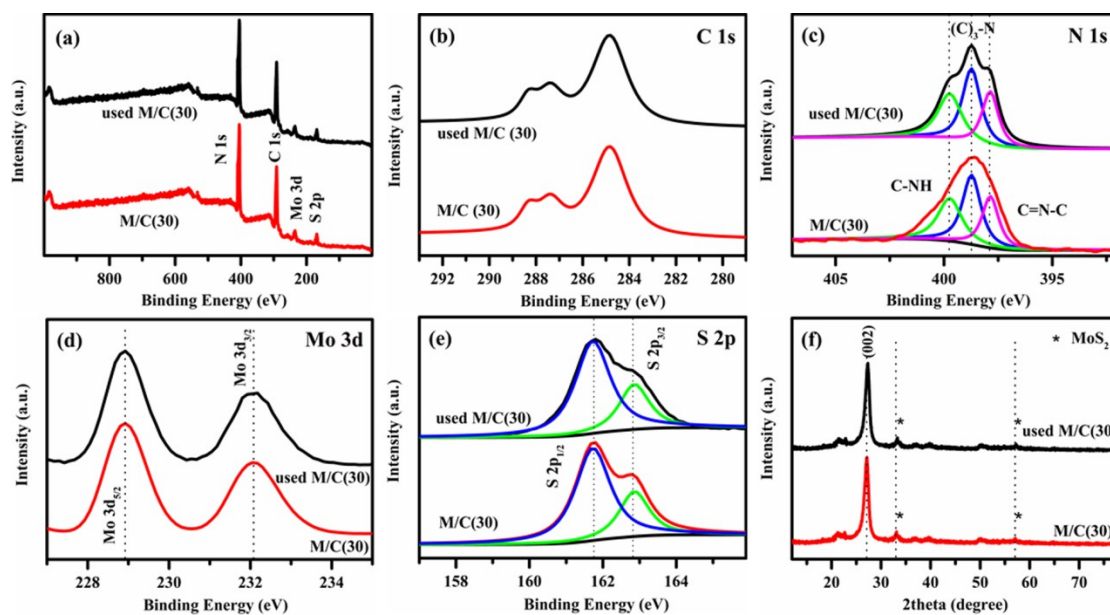


Fig. S7 (a) XPS spectrum, high resolution XPS spectra of (b) C 1s, (c) N 1s, (d) Mo 3d, and (e) S 2p, and (f) XRD patterns of MoS₂/CNNS(30) before and after reaction.

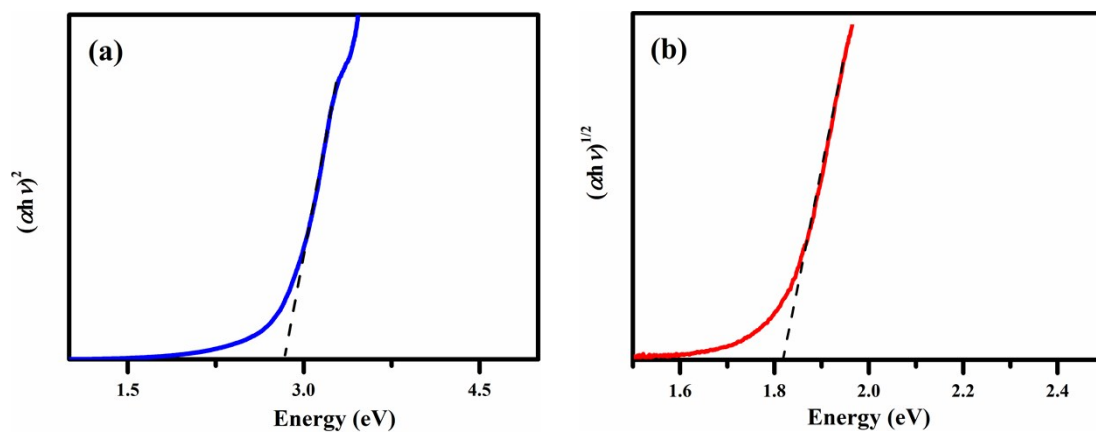


Fig. S8 Tauc plots of (a) CNNS and (b) MoS₂ QDs.

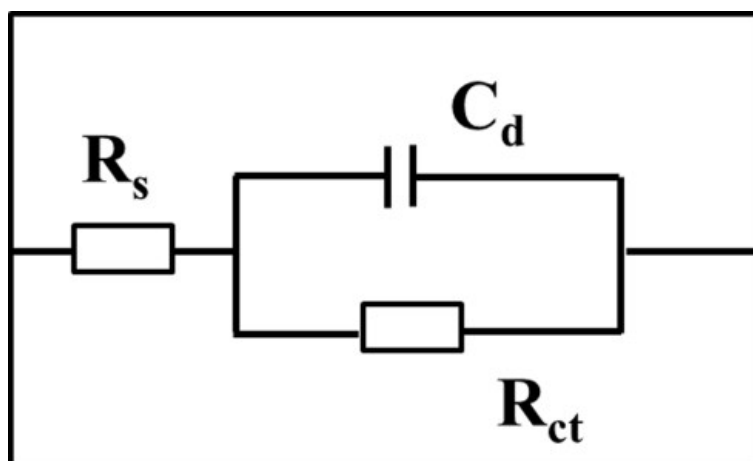


Fig. S9 The equivalent electric circuit used for fitting the EIS experiment results in Fig. 5b.

Table S3 Parameters used to fit the experimental impedance data in Fig. S8.

	R_s (Ω)	C_d (F)	R_{ct} (Ω)
CNNS	16.83	16	17 585
MoS ₂ QDs	16.83	12	12 698
MoS ₂ /CNNS(30)	16.83	46	2 395

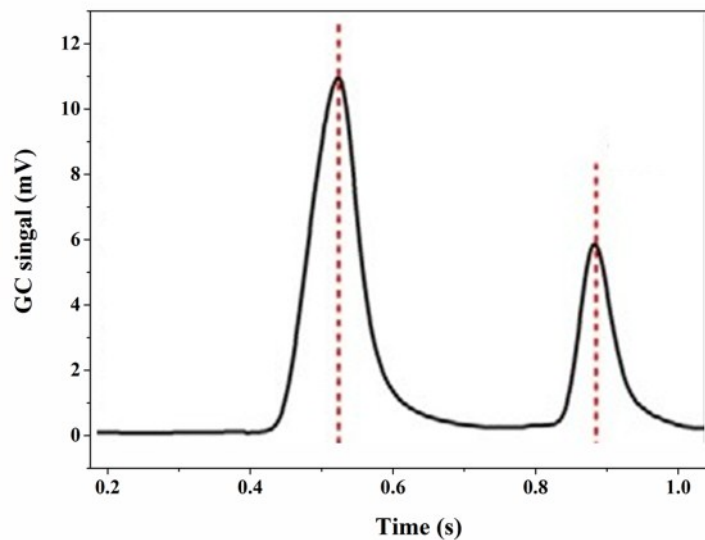


Fig. S10 The typical signal detected in the photocatalytic test.

As shown in Fig. S10, H₂ ($t_1 = 0.523$ s) and O₂ ($t_2 = 0.890$ s) can be detected in our photocatalytic test simultaneously.

References

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