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Supporting Information

Controlled Synthesis of 2D-2D Conductive Metal-Organic Frameworks/g-C_3N_4 Heterojunctions for Efficient Photocatalytic Hydrogen Evolution

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1. Experimental details

1.1 Photoelectrochemical and electrochemical measurements

The Mott-Schottky plot (M-S), transient photocurrent spectra (I-T) and electrochemical impedance spectra (EIS) were recorded on the CHI660E electrochemical workstation with a standard three-electrode system with the photocatalyst-coated ITO as the working electrode, Pt plate as the counter electrode, and a saturated calomel electrode as a reference electrode. The test samples (2 mg) were added to 1ml ethanol and 10 μL Nation, and then sonicated for a while. The working electrodes were prepared by dropping the suspension (200 μL) onto an ITO glass substrate electrode surface and dried at room temperature. The electrochemical test process selects 0.25 M Na_2SO_4 solution as the electrolyte solution and A 300 W Xenon lamp with a 420 nm cut-off filter as the light source.

1.2 Photocatalytic performance measurements

A sealed system that is composed of a quartz tube and a sealed system was charged with Ni-CAT-1/g-C_3N_4 nanosheet (10 mg), 100 μL H_2PtCl_6 solution (3 wt%) as a co-catalyst, 100 mg of sodium ascorbate (SA) as the holes sacrificial agent and 50 mL PBS buffer solution and then mixture was sonicated for 5 min. The whole photocatalytic test is controlled at 10°C and irradiated with a 300 W Xe lamp (>420 nm) to achieve photocatalytic hydrogen production. The hydrogen evolution is detected by a gas chromatograph (GC7920, thermal conductivity detector (TCD), Ar carrier gas).

2. Supplementary Figures and Tables

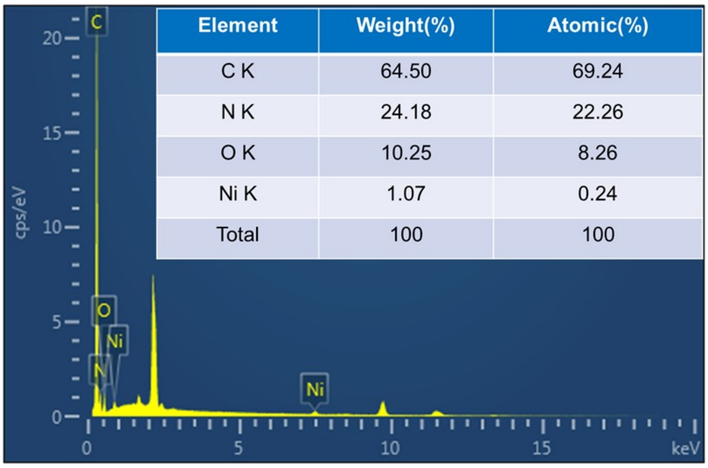

Figure S1 EDS spectra and element content of Ni-CAT-1/g-C_3N_4 composites.

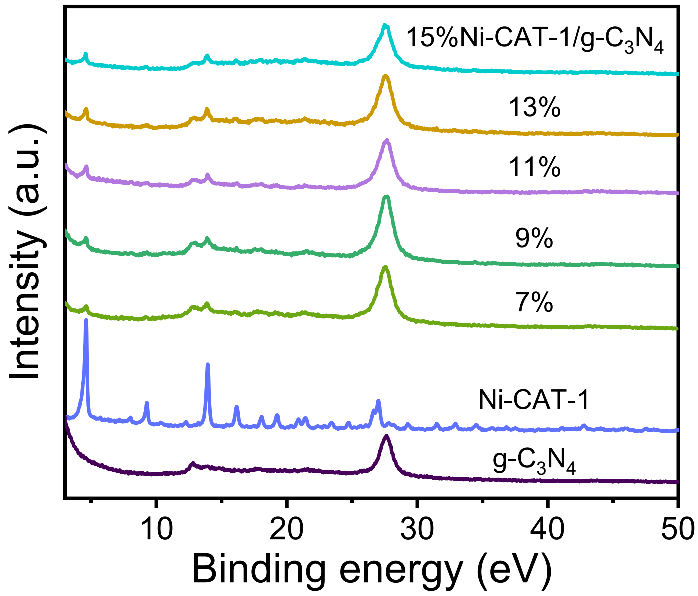

Figure S2 The PXRD patterns of a series of Ni-CAT-1/g-C_3N_4 composites.

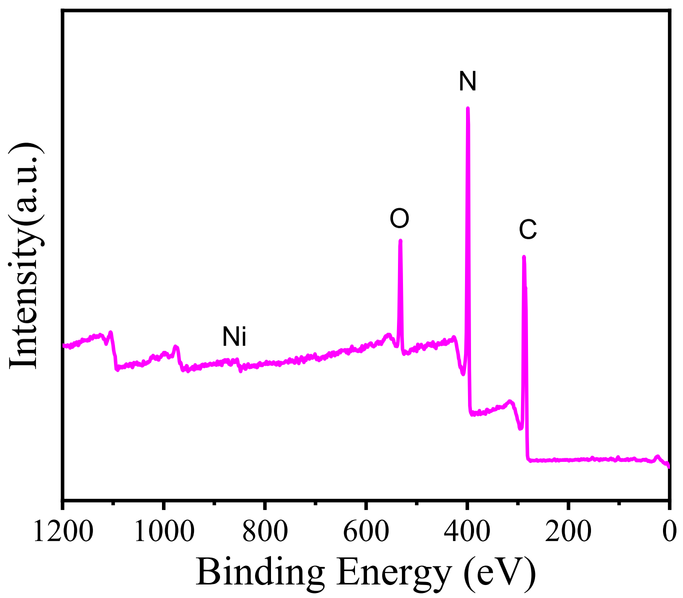

Figure S3 XPS survey spectrum of Ni-CAT-1/g-C_3N_4 composites.

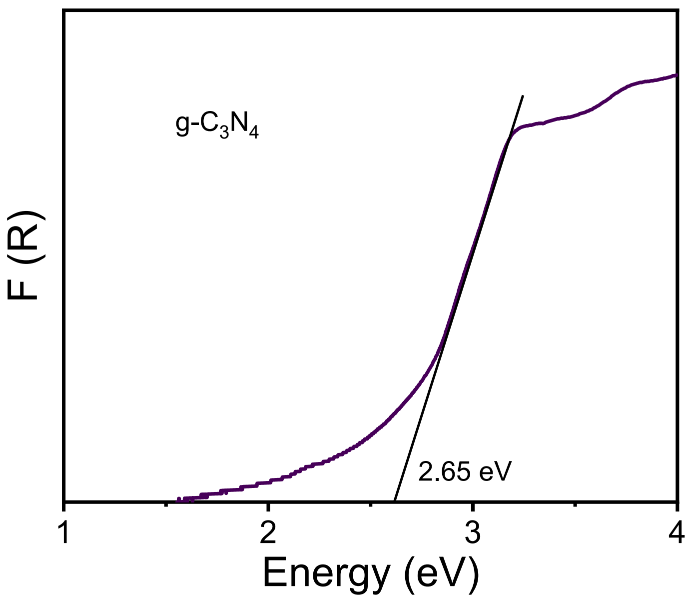

Figure S4 Tauc plots of g-C_3N_4.

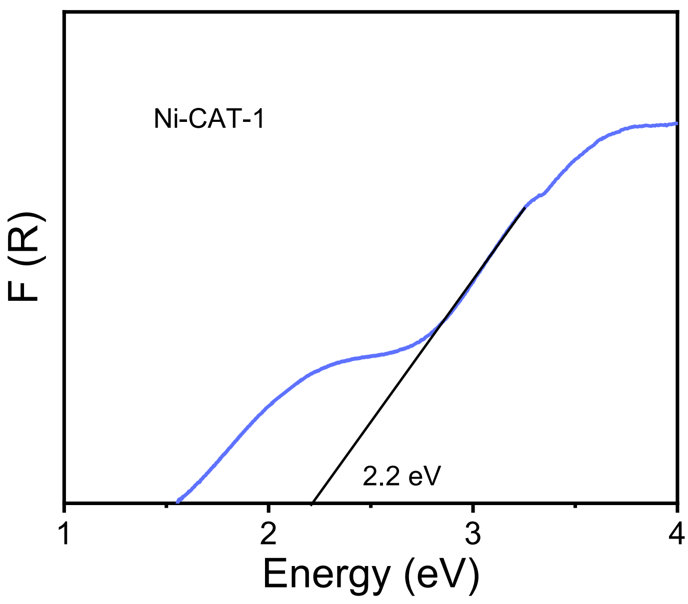

Figure S5 Tauc’s plots of Ni-CAT-1.

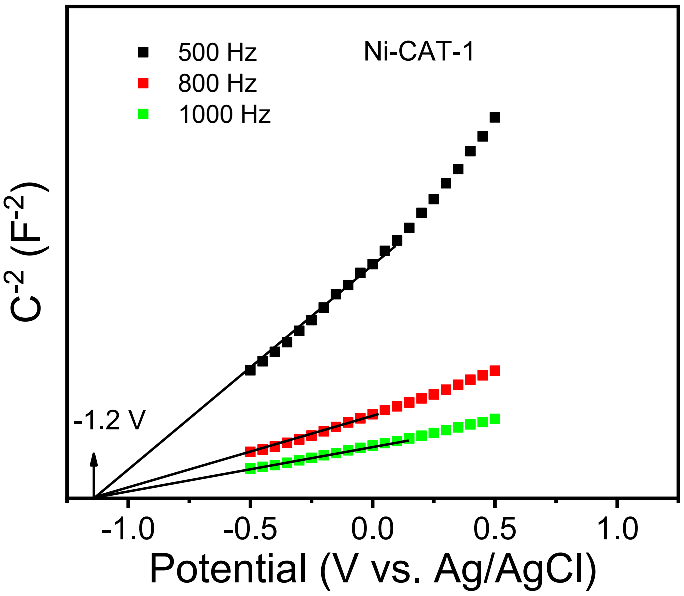

Figure S6 Mott-Schottky plot of Ni-CAT-1.

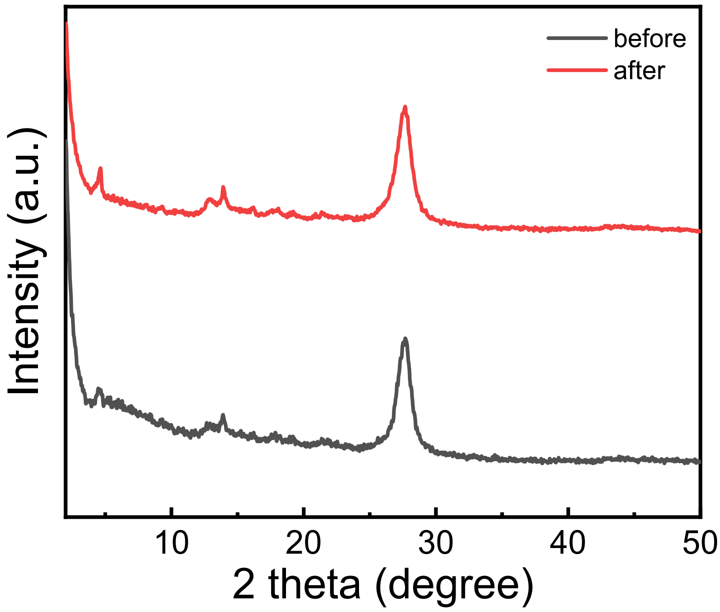

Figure S7 PXRD patterns of Ni-CAT-1/g-C_3N_4 composites before and after photocatalysis.

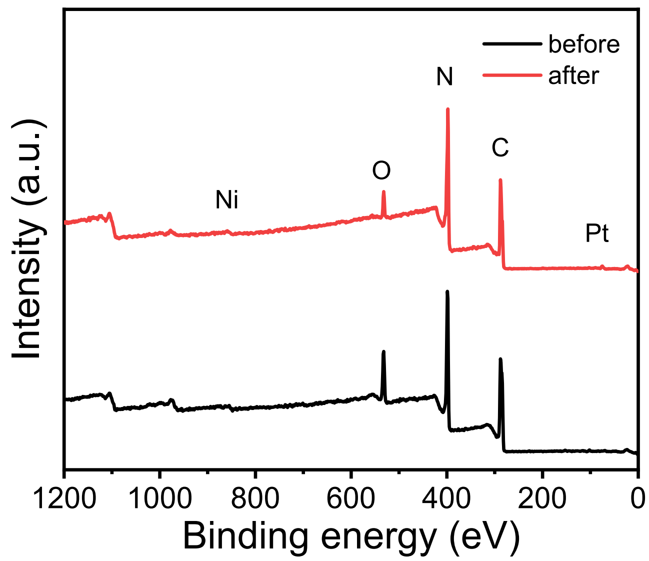

Figure S8 XPS survey spectrum of Ni-CAT-1/g-C_3N_4 composites before and after photocatalysis.

Table S1 Comparison of Photocatalytic Hydrogen Evolution Performance with Literature Reports

Catalyst	Sacrificial agent	Co-catalyst	Light source	HER (mmol g^{-1} h^{-1})	Refs
PAN/g-C_3N_4	TEOA	Pt	300 W Xe lamp (λ > 400 nm)	0.37	1
g-C_3N_4/PDI	AA	Pt	300 W Xe lamp (λ ≥ 420 nm)	1.65	2
5N-PTEtOH/g-C_3N_4	TEOA	Pt	300 W Xe lamp (λ ≥ 420 nm)	2.424	3
PCzF/g-C_3N_4	TEOA	Pt	300 W Xe lamp (λ ≥ 420 nm)	0.628	4
C_3N_4-PEDOT	TEA	Pt	high energy Xe lamp (λ > 400 nm)	0.327	5
1NP-3Mg-CN	TEOA	Pt	300 W Xe lamp (λ ≥ 420 nm)	1.496	6
BP/A-CN	TEOA	Pt	300 W Xe lamp (λ ≥ 420 nm)	0.86	7
Zr-MOF/g-C3N4	AA	Pt	300 W Xe lamp (λ ≥ 420 nm)	1.252	8
TAPT/CN	TEOA	Pt	300 W Xe lamp (λ ≥ 420 nm)	1.98	9
CCN	TEOA	Pt	300 W Xe lamp (λ ≥ 400 nm)	1.224	10
UCNs	TEOA	Pt	300 W Xe lamp (λ ≥ 400 nm)	2.59	11
$W_{18}O_{49}$/g-C_3N_4	TEOA	Pt	300 W Xe lamp (λ ≥ 420 nm)	0.912	12
5PPFBT/CN-OH	TEOA	Pt	300 W Xe lamp (λ ≥ 420 nm)	2.662	13
Ni-CAT-1/g-C_3N_4	SA	Pt	300 W Xe lamp (λ ≥ 420 nm)	2.76	This work

References:

1 F. He, G. Chen, Y. Yu, S. Hao, Y. Zhou and Y. Zheng, *ACS Appl. Mater. Interfaces*, 2014, **6**, 7171–7179.

2 H. Miao, J. Yang, Y. Sheng, W. Li and Y. Zhu, *Solar RRL*, 2021, **5**, 2000453.

3 Q. Zhao, Y. Li, K. Hu, X. Guo, Y. Qu, Z. Li, F. Yang, H. Liu, C. Qin and L. Jing, *ACS Sustainable Chem. Eng.*, 2021, **9**, 7306–7317.

4 J. Chen, C.-L. Dong, D. Zhao, Y.-C. Huang, X. Wang, L. Samad, L. Dang, M. Shearer, S. Shen and L. Guo, *Advanced Materials*, 2017, **29**, 1606198.

5 Z. Xing, Z. Chen, X. Zong and L. Wang, *Chem. Commun.*, 2014, **50**, 6762–6764.
6 X. Guo, K. Hu, M. Chu, Y. Li, J. Bian, Y. Qu, X. Chu, F. Yang, Q. Zhao, C. Qin and L. Jing, *ChemSusChem*, 2020, **13**, 3707–3717.
7 M. H. Suhag, H. Katsumata, I. Tateishi, M. Furukawa and S. Kaneco, *Langmuir*, 2023, **39**, 13121–13131.
8 H. Xing, J. Shi, W. Yang, Y. Li, R. Wu and J. Wu, *New J. Chem.*, 2023, **47**, 21685–21691.
9 C.-C. Li, I. Ullah, G. Wang and A.-W. Xu, *Catal. Sci. Technol.*, 2023, **13**, 5456–5461.
10 E. Liu, X. Lin, Y. Hong, L. Yang, B. Luo, W. Shi and J. Shi, *Renewable Energy*, 2021, **178**, 757–765.
11 L. Wang, Y. Hong, E. Liu, X. Duan, X. Lin and J. Shi, *Carbon*, 2020, **163**, 234–243.
12 A. Li, Z. Peng and X. Fu, *Solid State Sciences*, 2020, **106**, 106298.
13 X. Pang, Y. Li, X. Wu, B. Zhang, M. Hao, Y. Zhu, Y. Zhang, C. Qin, H. Zhan and C. Qin, *Journal of Colloid and Interface Science*, 2023, **652**, 1405–1416.