

Supplementary Information

A novel type-II NiCo-LDH/CeO₂ heterojunction for highly efficient photocatalytic H₂ production

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1. Detail of material characterization and performance measurements

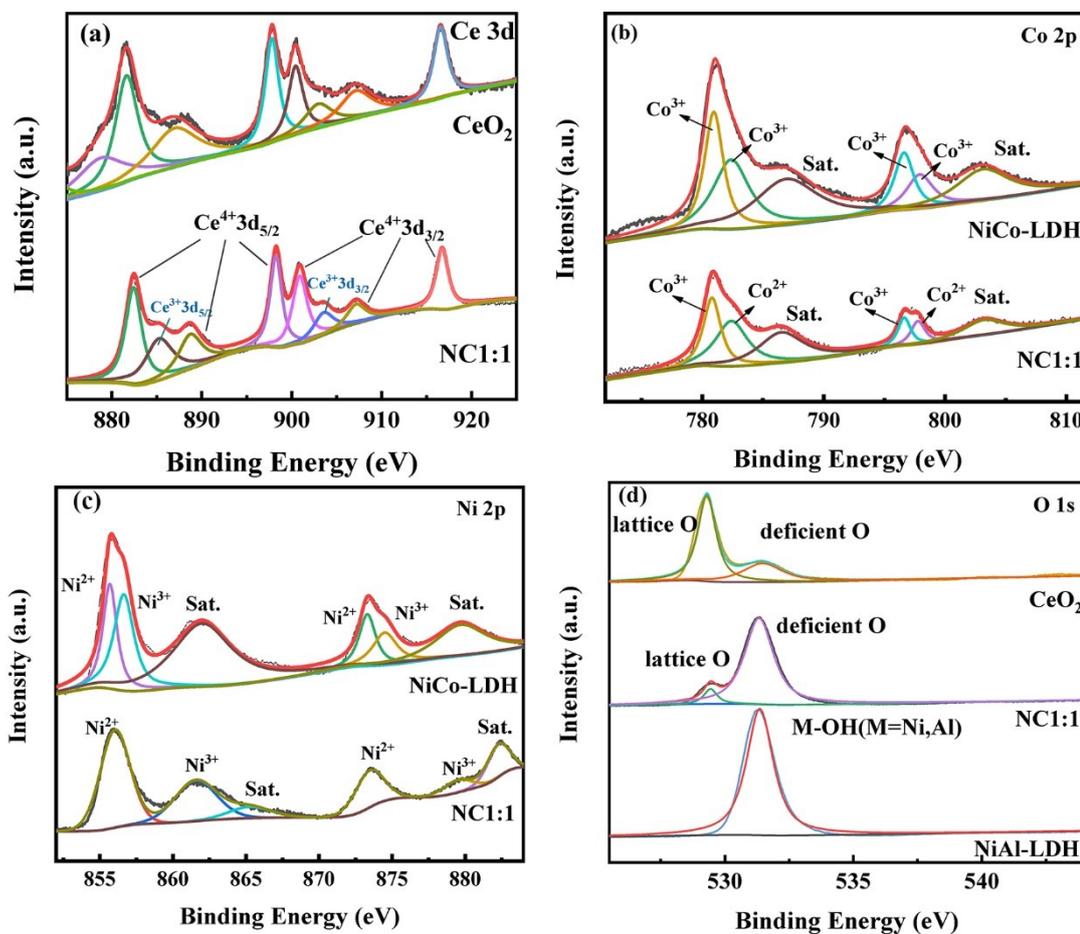
1.1 Material characterization

The crystal structure of photocatalyst was recorded by X-ray diffractometer. The structure and morphology of the photocatalyst were observed by scanning electron microscope (SEM) and transmission electron microscope (TEM). The chemical elements and valence states of the samples were studied by X-ray photoelectron spectroscopy (XPS). The UV-Vis diffuse reflectivity spectra (UV-vis DRS) of the photocatalyst were recorded using BaSO₄ as the back and back of the UV visible spectrometer. At 77 K, the BET specific surface area of the samples was determined by nitrogen adsorption desorption method. The photoluminescence (PL) and time-resolved photoluminescence spectra (TRPL) of the samples were studied by fluorescence spectrophotometry. The electrochemical activity of the samples was determined by electrochemical workstation CHI660E. In 0.2 mol·L Na₂SO₄ saturated aqueous solution, Ag/AgCl electrode and Pt electrode were used as reference electrode and counter electrode respectively. The working electrode was ITO glass coated with different sample powders with an effective area of 1 cm² (1cm×2cm). The photocurrent response curves (I-T) of different samples were measured by intermittent switching on and off xenon lamp with 300 W light source. Electrochemical impedance spectroscopy (EIS) of different samples was measured at a bias of -0.2 V.

1.2 Hydrogen production condition

The photocatalytic production hydrogen was carried out in an airtight quartz bottle, which

could be degassed and sampled. Usually, 10mg of catalytic material, 20mg of Eosin Y (EY), and 30 mL of TEOA solution containing 10% were weighed and added into a quartz flask. Before the solution was illuminated by visible light, the reactant system was degassed by vacuuming to remove excess gas from the solution. The amount of hydrogen evolution was analyzed by online gas chromatograph (SP 2100, N₂ as carrier).



S1. electron transfer contrast

Table 1. Comparison of the H₂ production activity of NiCo-LDH/CeO₂ photocatalyst and the recently reported photocatalyst.

Photocatalyst	Scavenger	Light source	H ₂ evolution rate ($\mu\text{mol g}^{-1} \text{h}^{-1}$)	Ref
NiCo-LDH/CeO ₂	TEOA	5 W LED lights	2700	本文
NiAl-LDH/MoS ₂	TEOA	5 W LED lights	4590	[1]
CoO/NiCo-LDH	0.2 M Na ₂ SO ₃ and 0.2 M Na ₂ S	100 mW cm ⁻² Xe lamp	1500	[2]
NiAl-LDH/Ti ₃ C ₂ /g- C ₃ N ₄	TEOA	35 W Ballast CAR HID Lamp	180	[3]
Ag-pCN/TiO ₂	glycerol-water mixture.	35 W HID Xe lamp	3160	[4]

g-C ₃ N ₄ /CoAl-LDH/	TEOA	300 W Xe lamp	680.13	[5]
CeO ₂ /WO ₃	10 vol% ethanol	300 W lamp	410	[6]
Pt-CeO ₂ /g-C ₃ N ₄	TEOA	300 W Xe-lamp	860	[7]
CeO ₂ /CoS ₂	TEOA	5 W LED	5172	[8]

References

- [1] X. Liu, J. Xu, L. Ma, Y. Liu, L. Hu, Nano-flower S-scheme heterojunction NiAl-LDH/MoS₂ for enhancing photocatalytic hydrogen production, *New Journal of Chemistry*, 46 (2021) 228-238.
- [2] Y. Wang, S. Guo, X. Xin, Y. Zhang, B. Wang, S. Tang, X. Li, Effective interface contact on the hierarchical 1D/2D CoO/NiCo-LDH heterojunction for boosting photocatalytic hydrogen evolution, *Applied Surface Science*, 549 (2021).
- [3] W. Almusattar, M. Tahir, M. Madi, B. Tahir, Fabricating Ti₃C₂ MXene cocatalyst supported NiAl-LDH/g-C₃N₄ ternary nanocomposite for stimulating solar photocatalytic H₂ production, *Journal of Environmental Chemical Engineering*, 10 (2022) 108010.
- [4] N. Fajrina, M. Tahir, Engineering approach in stimulating photocatalytic H₂ production in a slurry and monolithic photoreactor systems using Ag-bridged Z-scheme pCN/TiO₂ nanocomposite, *Chemical Engineering Journal*, 374 (2019) 1076-1095.
- [5] J. Zhang, Q. Zhu, L. Wang, M. Nasir, S.-H. Cho, J. Zhang, g-C₃N₄/CoAl-LDH 2D/2D hybrid heterojunction for boosting photocatalytic hydrogen evolution, *International Journal of Hydrogen Energy*, 45 (2020) 21331-21340.
- [6] R. Fiorenza, S.A. Balsamo, M. Condorelli, L. D'Urso, G. Compagnini, S. Scire, Solar photocatalytic H₂ production over CeO₂-based catalysts: Influence of chemical and structural modifications, *Catalysis Today*, 380 (2021) 187-198.
- [7] W. Zou, Y. Shao, Y. Pu, Y. Luo, J. Sun, K. Ma, C. Tang, F. Gao, L. Dong, Enhanced visible light photocatalytic hydrogen evolution via cubic CeO₂ hybridized g-C₃N₄ composite, *Applied Catalysis B-Environmental*, 218 (2017) 51-59.
- [8] L. Ma, J. Xu, J. Zhang, Z. Liu, X. Liu, Rare earth material CeO₂ modified CoS₂ nanospheres for efficient photocatalytic hydrogen evolution, *New Journal of Chemistry*, 45 (2021) 21795-21806.