

Supporting Information

Photodeposited Nickel loaded ZnCdS nanoparticles for photocatalytic water splitting to hydrogen production

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1 Experimental section

1.1 Characterization

The crystal structures of the samples were analyzed by a Rigaku D/max-ga X-ray diffractometer (XRD) with a scan rate of 6° min^{-1} and a 2θ range of 10° to 80° using Cu K α radiation ($\lambda = 1.54178 \text{ \AA}$). X-ray photoelectron spectroscopy (XPS) measurements were performed on a Thermo Fisher Scientific XPS ESCALAB 250 Xi instrument which uses an Al K α (1486.8 eV) X-ray source to determine the valence states of all elements. Field emission scanning electron microscopy (FESEM) was performed by a Hitachi SU8010 at an accelerating voltage of 5 kV in order to observe the morphology of the prepared samples. High resolution transmission electron microscopy (HRTEM) analysis was performed on a Hitachi 600TEM at an acceleration voltage of 200 kV to analyze the microstructure of the samples. UV-Vis measurements were performed on a Unico UV-4802S with barium sulfate as a reference.

1.2 Photocatalytic H₂ production

Photocatalyst (10mg) was added into a 20 mL photoreactor with circulating water. At the same time, 10 mL of Na₂S (0.1 mol/L) and Na₂SO₃ (0.1 mol/L) solution was added into the bottle and sealed. The air inside the bottle was replaced with argon for 10 min, and place under a xenon lamp with magnetic stirring. The light source was a 300 W xenon lamp with cutoff wavelength >420 nm. 100 μL of gas in the bottle was taken every hour to measure hydrogen by gas chromatography. Gas chromatography (Japan Shimadzu Corporation GC-14B) used a thermal conductivity detector (TCD) with a column of 5 \AA molecular sieves (4 mm \times 2 m). Argon was used as the carrier gas.

1.3 Electrochemical testing

All the electrochemical measurements were performed on a CHI660D electrochemical workstation (Shanghai Chenhua Instrument, Ltd. Shanghai, China) with standard three electrode system. The following methods was used to prepare working electrodes: 5 mg sample was put into a sample tube, and 980 μL of absolute ethanol and 20 μL of Nafion solution were added. The samples were dispersed by ultrasonic for 30 min and then dropped on the prepared $1 \times 1 \text{ cm}^2$ FTO

(fluorine-doped tin oxide) glass. Mott-Schottky analysis was performed at potentials from -1 to 1 V. All tests were performed with Hg/Hg₂Cl₂ (saturated KCl) electrode as reference electrode, platinum sheet as counter electrode and 0.5 mol·L⁻¹ Na₂SO₄ as electrolytes at room temperature.

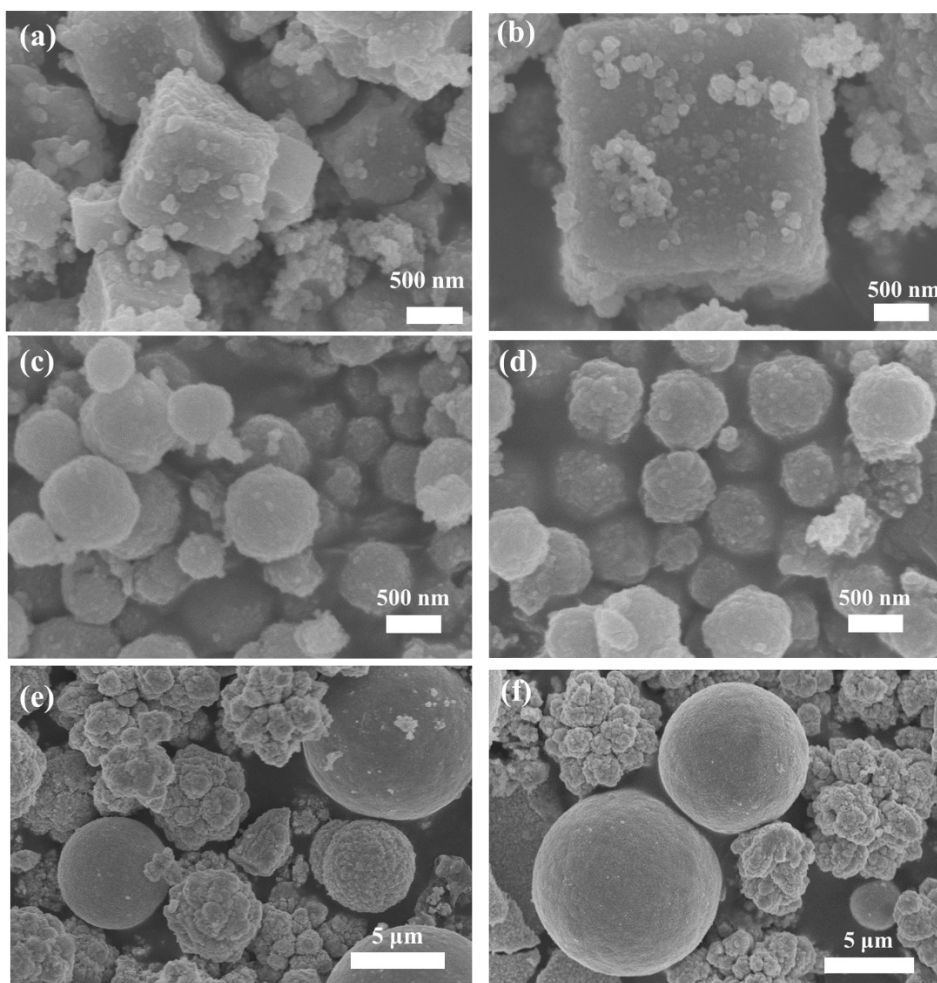


Fig. S1 SEM images of CdS-cube (a), CdS/Ni-cube (b), ZnCdS(TS) (c), ZnCdS(TS)/Ni-0.5H (d) and ZnCdS(OS) (e) and ZnCdS(OS)/Ni (f).

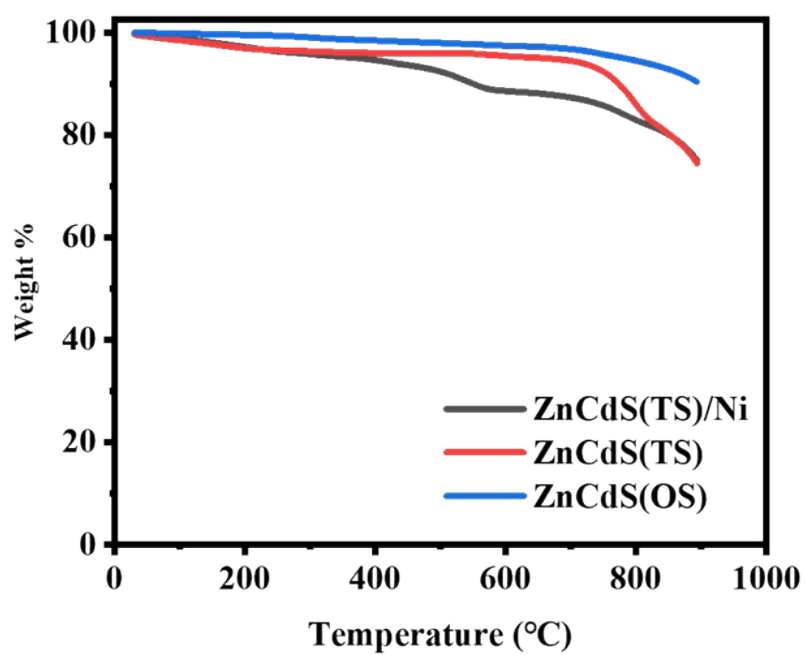


Fig. S2 TGA text of different catalysts.

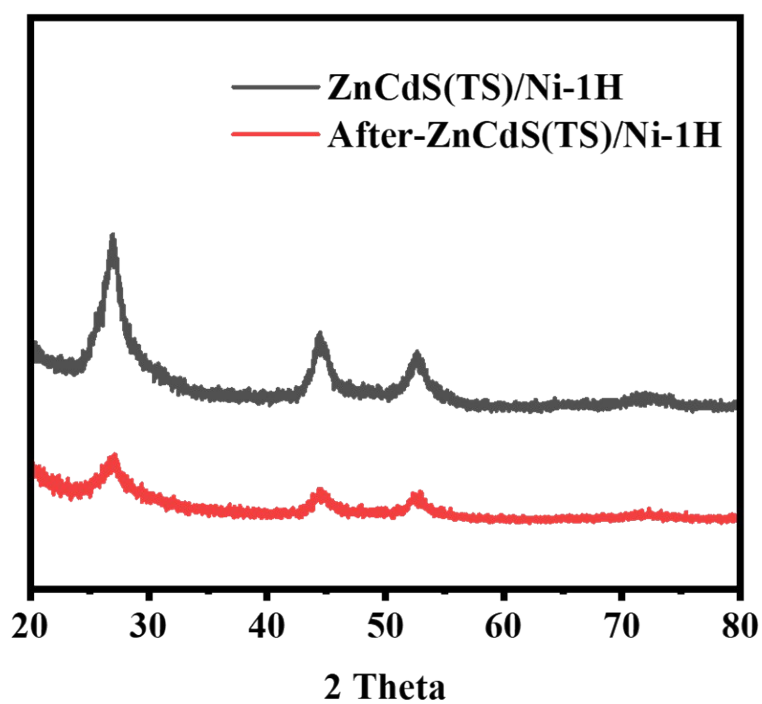


Fig. S3 XRD pattern of ZnCdS(TS)/Ni-1H after cycle experiment.

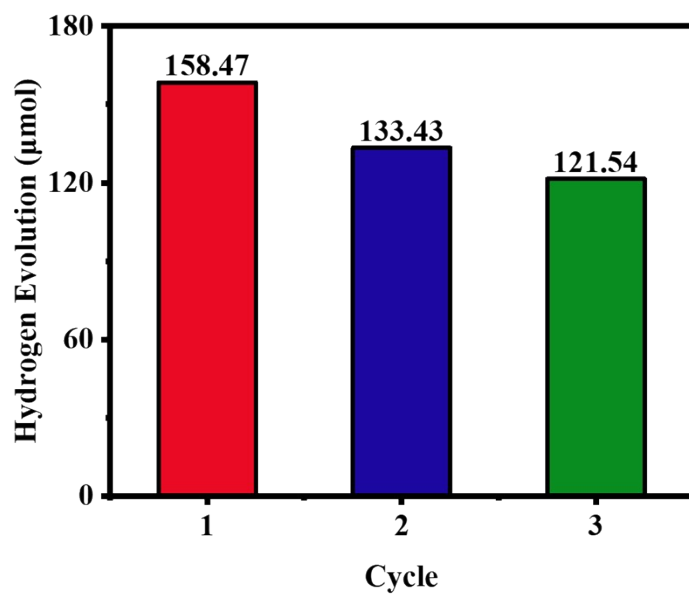


Fig. S4 The recyclability study of ZnCdS(TS)/Ni-1H

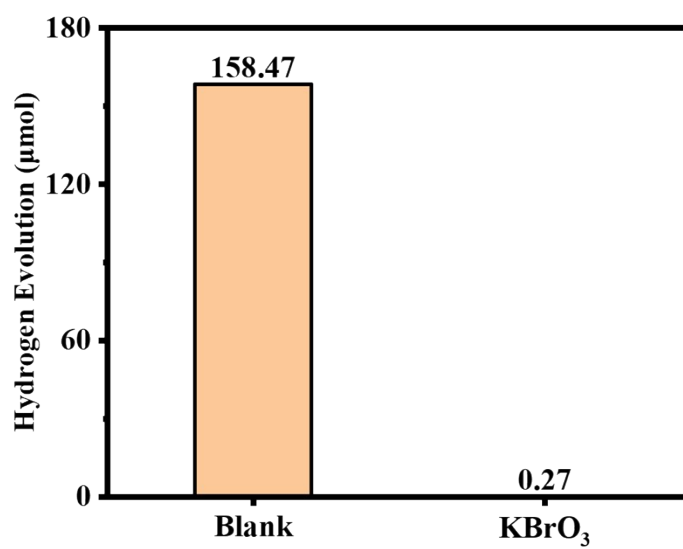


Fig. S5 Free radical capture experiment.

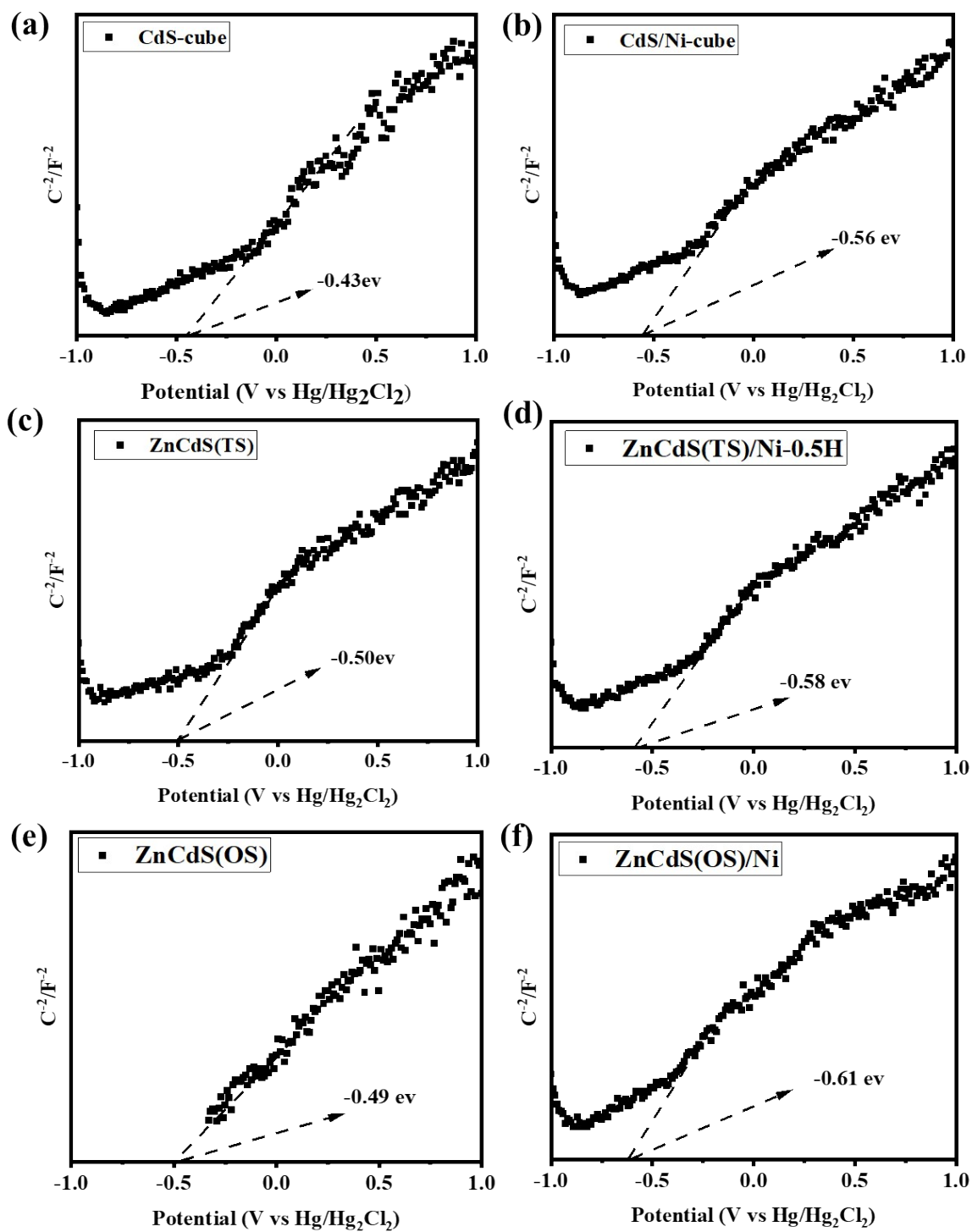


Fig. S6 Mott-Schottky plots of CdS-cube (a), CdS/Ni-cube (b), ZnCdS(TS) (c), ZnCdS(TS)/Ni-0.5H (d), ZnCdS(OS)/Ni (e) and ZnCdS(OS)/Ni (f).

Table S1 Comparison of photocatalytic hydrogen production performance with other catalysts

Photocatalysts	Cocatalyst	Reactant solution (Sacrificial Agent)	Light source	HER activity	Refs.
ZnCdS/Ni		0.1 M Na ₂ SO ₃ and 0.1 M Na ₂ S	300 W Xenon lamp with 420 nm cutoff filter	2641 $\mu\text{mol}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$	This work
NiO _x /B- TiO ₂ @CdS		0.1 M Na ₂ SO ₃ and 0.1 M Na ₂ S	300 W Xenon lamp with standard AM 1.5G filter	3.04 $\text{mmol}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$	[1]
TiO ₂	NiO	20 vol% Methanol	300 W Xenon lamp with standard AM 1.5G filter	228 $\mu\text{mol}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$	[2]
ZnWO ₄ /CuO/T CPP		40 mL H ₂ O + 20 mL TEOA	500 W Mercury lamp	1.84 $\text{mmol}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$	[3]
g-C ₃ N ₄	5 % wt Nb ₂ O ₅	10 vol% aq. TEOA	300 W Xenon lamp with 420 nm cutoff filter	2.07 ± 0.03 $\text{mmol}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$	[4]
Co ₃ O ₄ /gC ₃ N ₄ /C oTiO ₃		15 vol% aq. TEOA	5 W LED Lamp	1971.7 $\mu\text{mol}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$	[5]
Hollow OvCeO ₂ /CdS nanocage cubes		0.75 mol/L Na ₂ S and 1.05 mol/L Na ₂ SO ₃	300 W Xenon lamp with 420 nm cutoff filter	~12.6 $\text{mmol}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$	[6]
ZnP-Pz-PEO- COF	[Mo ₃ S ₁₃] ²⁻	50 mM Ascorbic Acid and 15 vol% Lactic Acid	300 W Xenon lamp with 420 nm cutoff filter	1.38 $\text{mmol}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$	[7]
Ag-AgMOM		TEOA	300 W Xenon lamp without any cutoff filter	3153 $\mu\text{mol}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$	[8]
Ag/CN	1% wt. Pt	20 vol% aq. TEOA	300 W Xenon lamp with 400 nm cutoff filter	1688.9 $\text{mmol}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$	[9]

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