

Electronic Supporting Information for

**Enhancement of photocatalytic H₂ production by metal complex
electrostatic adsorption on TiO₂ (B) nanosheets**

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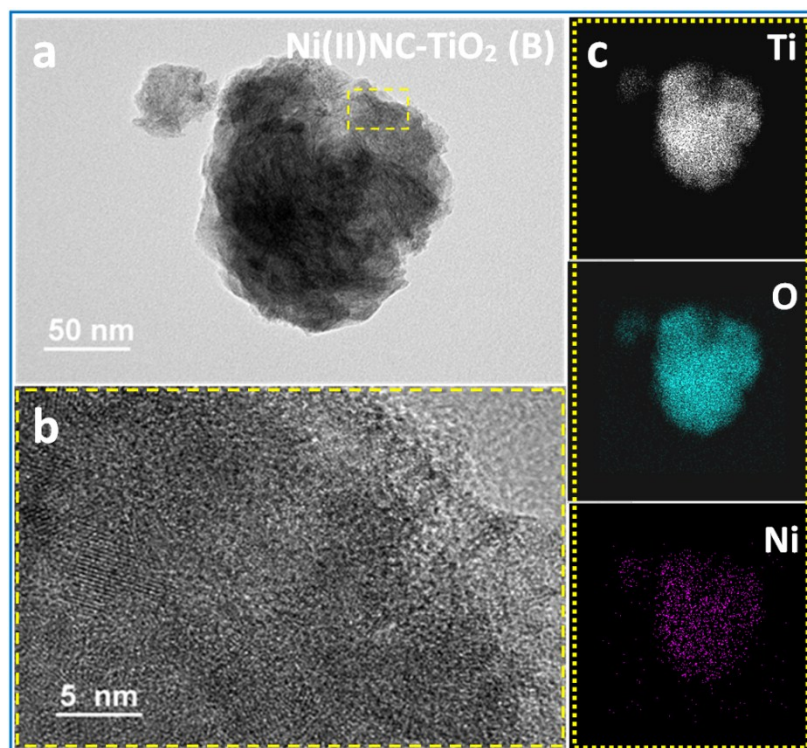


Figure S1. Structure characterizations of as-synthesized Ni(II)NC-TiO₂ (B) nanosheets. (a-b) TEM, HRTEM images of Ni(II)NC-TiO₂ (B); (c) the overlay mapping elements of Ti, O and Ni.

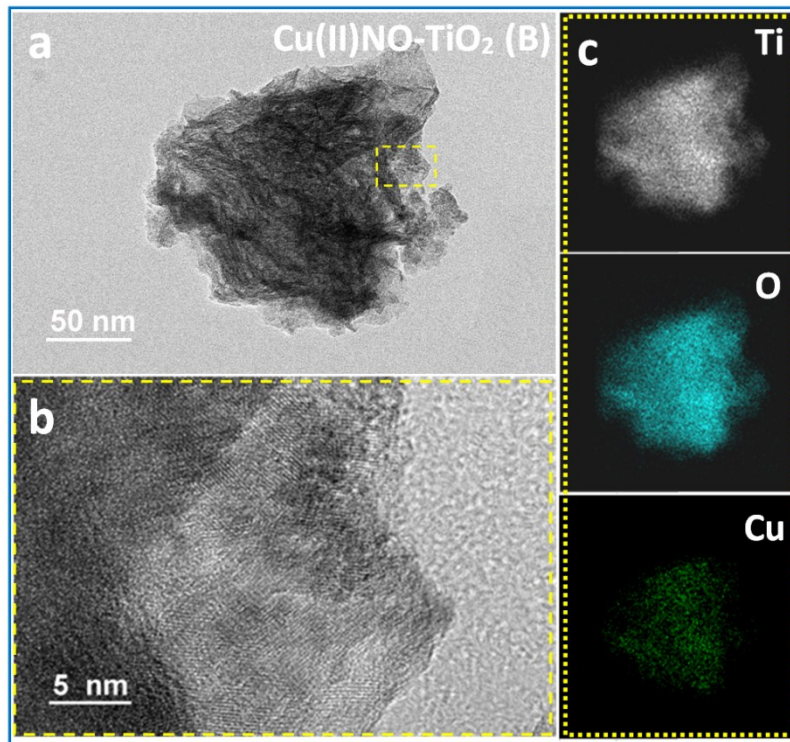


Figure S2. Structure characterizations of as-synthesized Cu(II)NO-TiO₂ (B) nanosheets. (a-b) TEM, HRTEM images of Cu(II)NO-TiO₂ (B); (c) the overlay mapping elements of Ti, O and Cu.

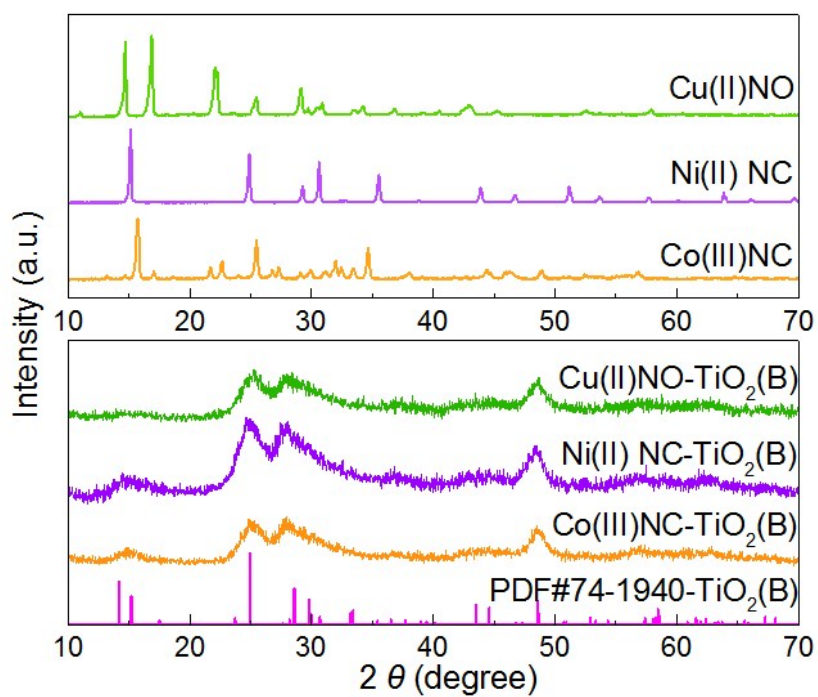


Figure S3. XRD patterns of the Co(III)NC, Ni(II)NC and Cu(II)NO metal complexes, and Co(III)NC/Ni(II)NC/Cu(II)NO-TiO₂(B) nanosheets by electrostatic adsorption.

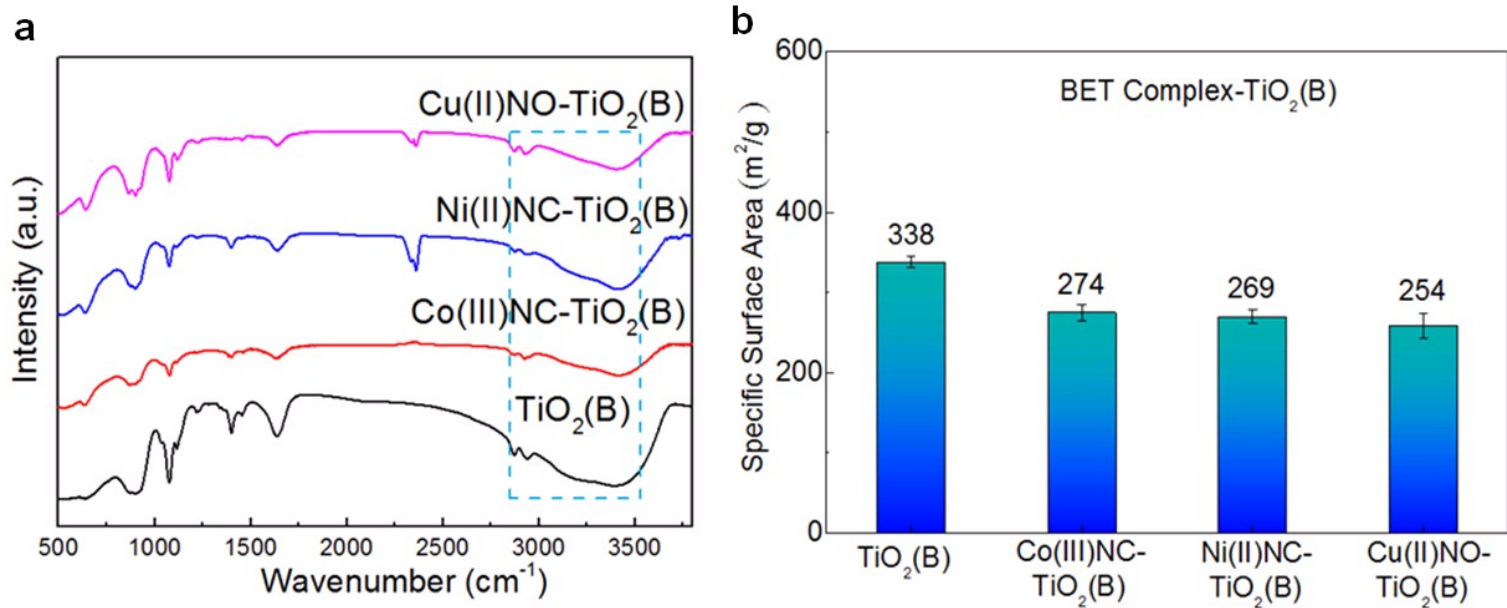


Figure S4.(a) FTIR and (b) BET of the pristine TiO₂ (B) and Co(III)NC/Ni(II)NC/Cu(II)NO-TiO₂ (B) nanosheets by electrostatic adsorption.

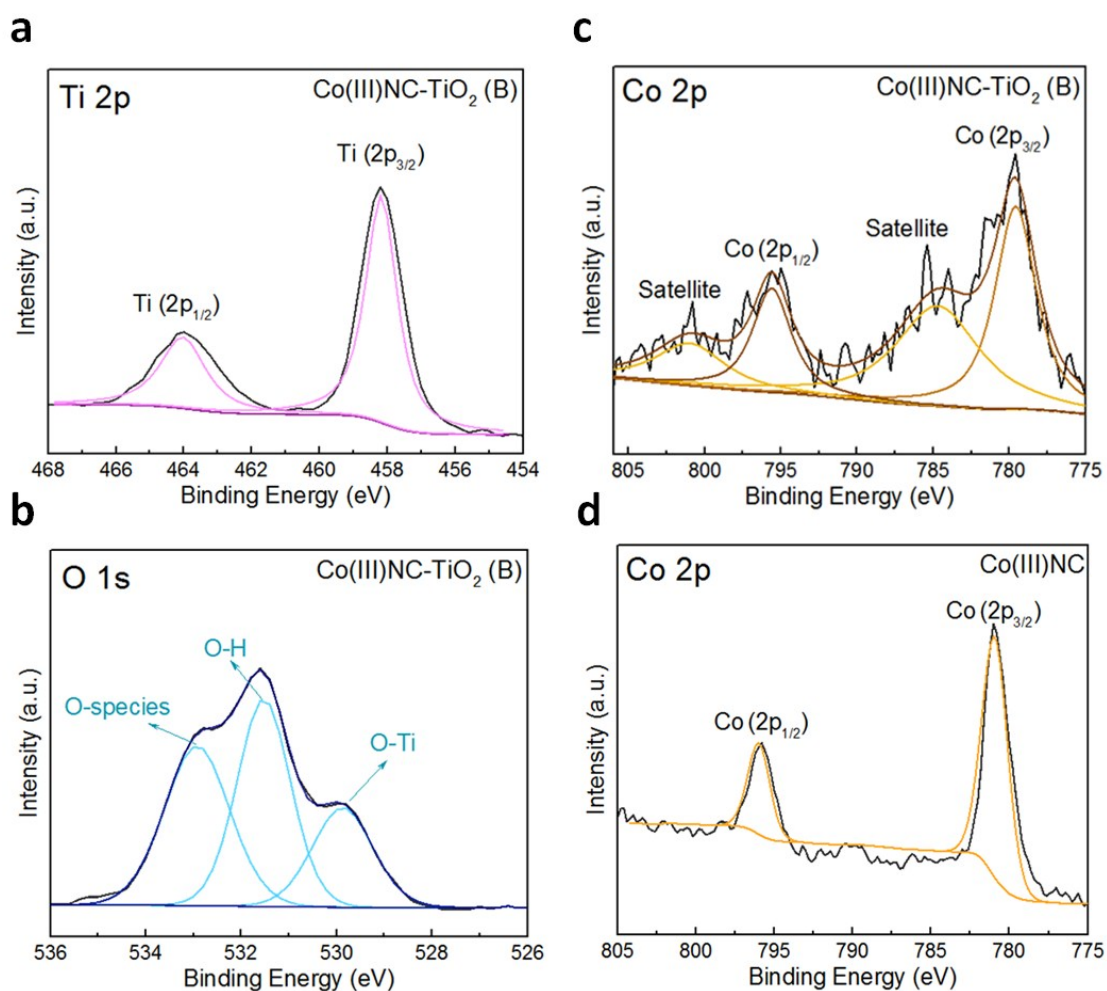


Figure S5. XPS spectra of (a) Ti 2p, (b) O 1s and (c) Co 2p region of Co(II)NC-TiO₂ (B); (d) Co 2p spectra of pristine Co(II)NC.

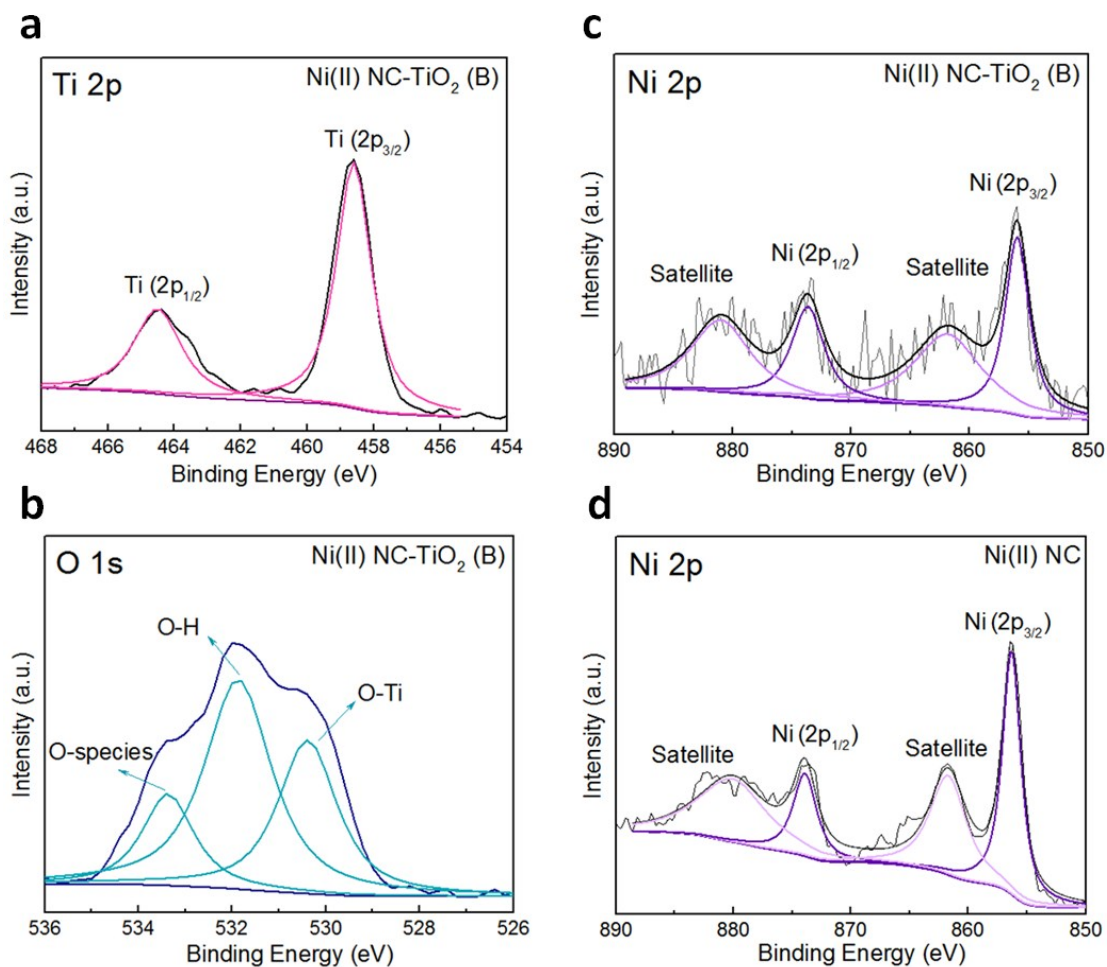


Figure S6. XPS spectra of (a) Ti 2p, (b) O 1s and (c) Ni 2p region of Ni(II)NC-TiO₂ (B); (d) Ni 2p spectra of pristine Ni(II)NC.

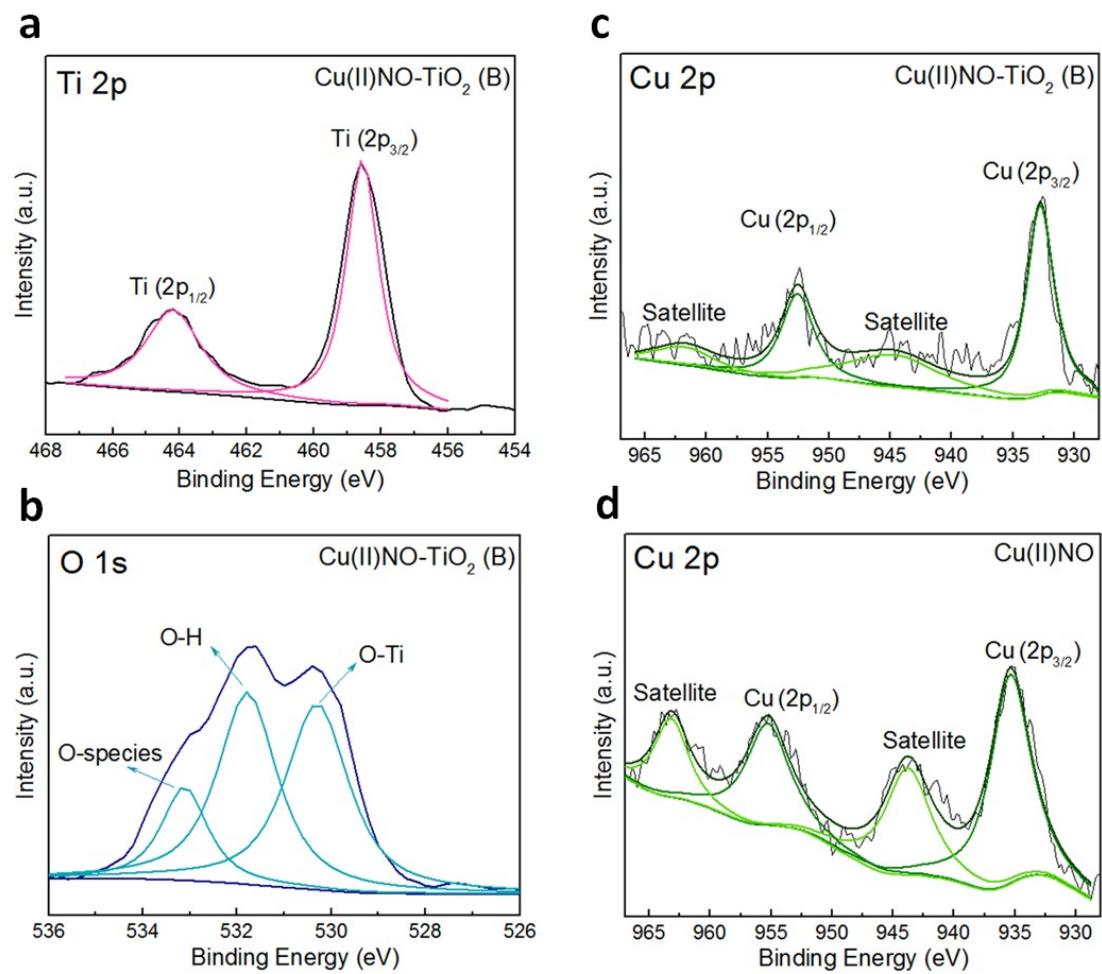


Figure S7. XPS spectra of (a) Ti 2p, (b) O 1s and (c) Cu 2p region of Cu(II)NO-TiO₂ (B); (d) Cu 2p spectra of pristine Cu(II)NO.

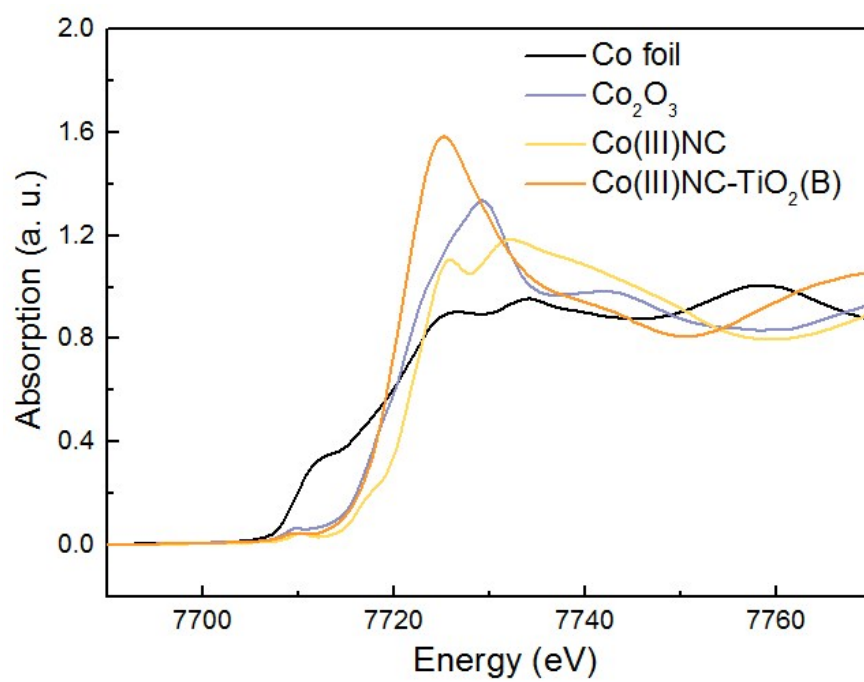
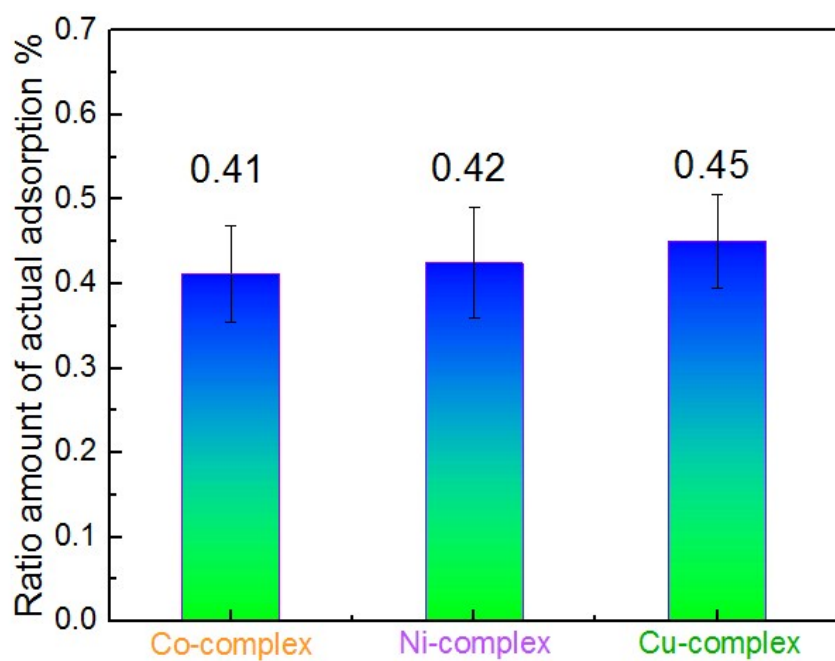


Figure S8. The overall profile of Co K-edge XANES spectra of Co(III)NC and Co(III)NC-TiO₂(B) indicated the presence of trivalent Co.



The variety of element under electrostatic adsorption

Figure S9. The actual adsorption ratio amounts of Co, Ni and Cu on TiO₂ (B) tested by ICP-OES.

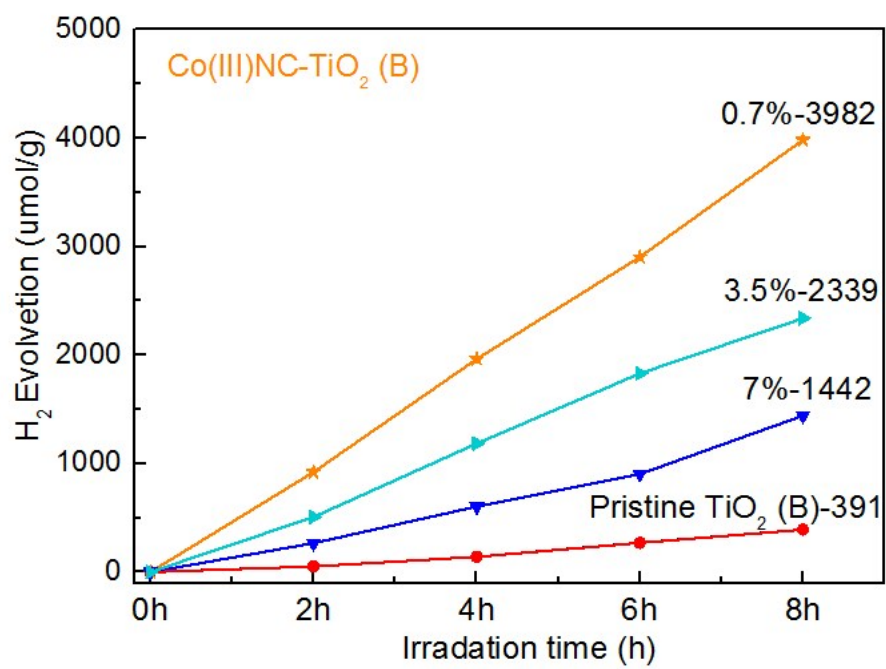


Figure S10. Photocatalytic H₂ evolution activities of Co(III)NC-TiO₂ (B) with different loading contents.

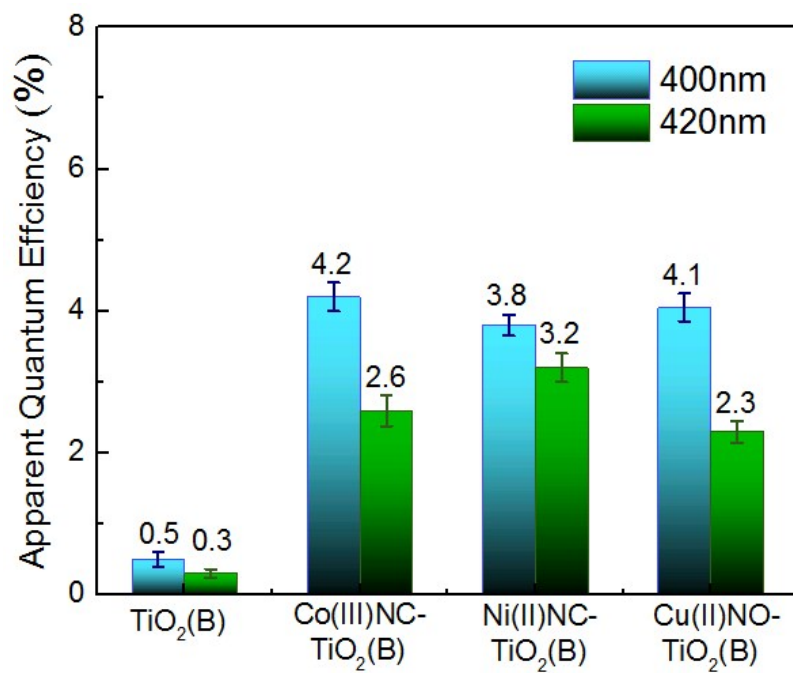


Figure S11. AQE of pristine TiO₂(B), Co(III)NC-TiO₂(B), Ni(II)NC-TiO₂(B) and Cu(II)NO-TiO₂(B) nanosheets at 400nm and 420nm.

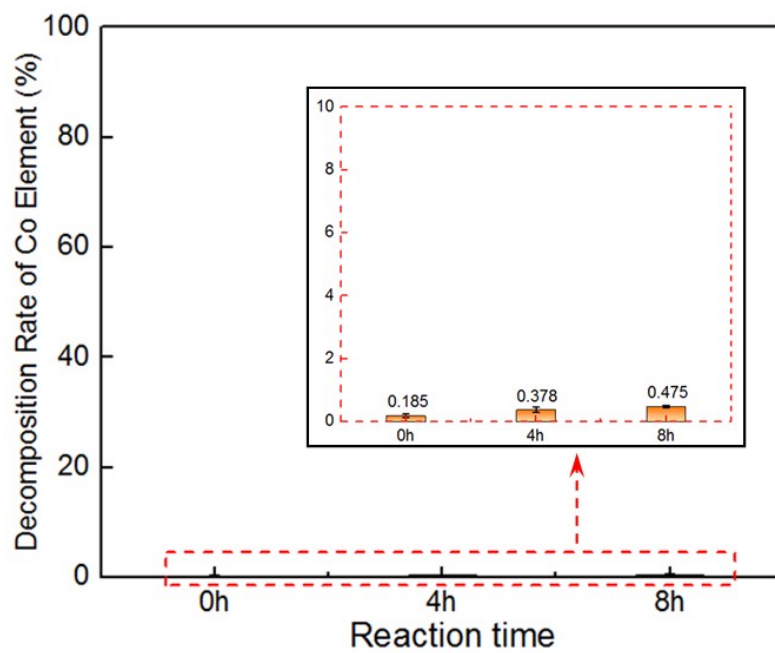


Figure S12. the decomposition rate of Co element with different photocatalytic reaction time

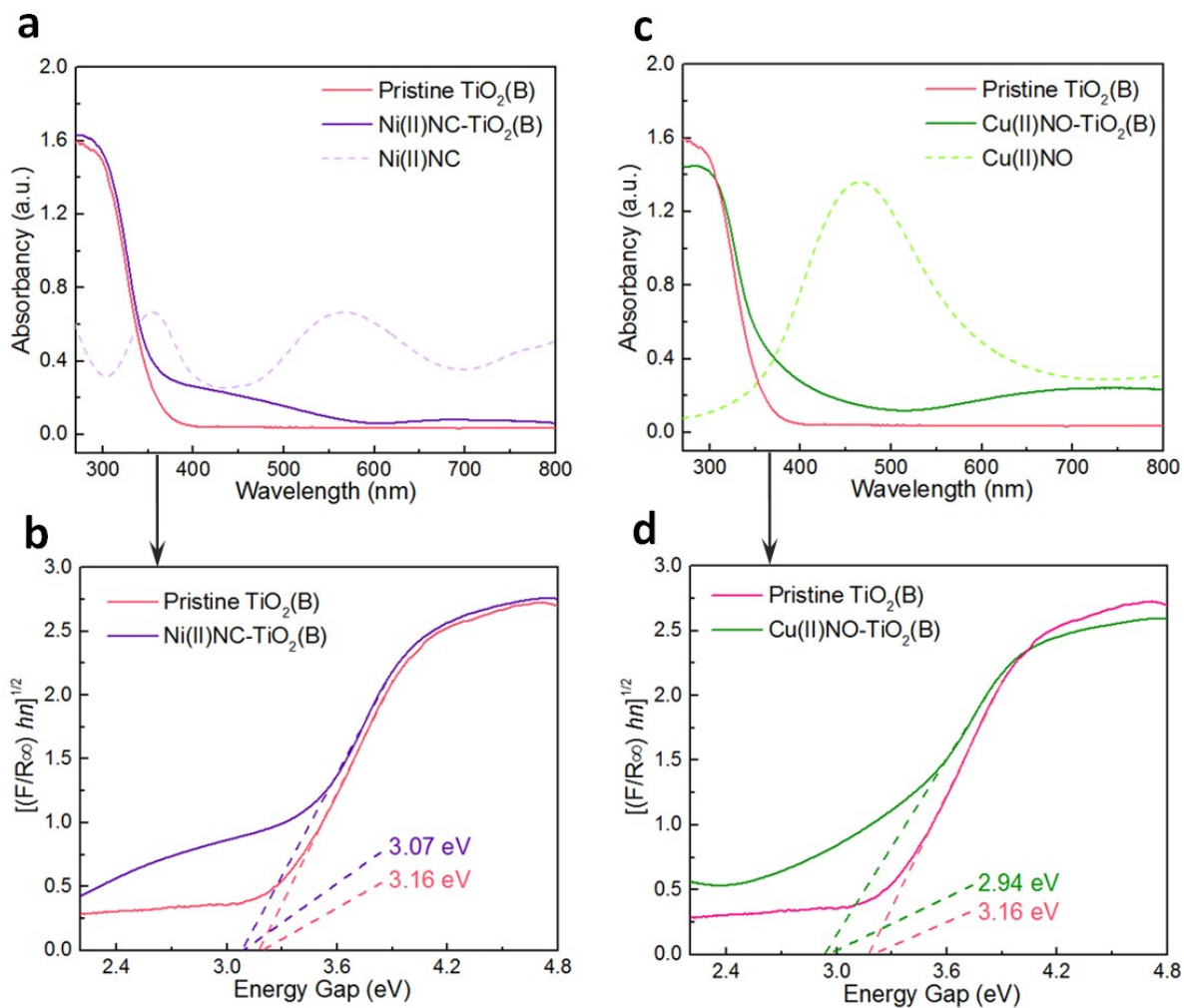


Figure S13. (a) UV-Vis diffuse reflectance spectra of pure $\text{TiO}_2(\text{B})$, $\text{Ni(II)NC-TiO}_2(\text{B})$ and Ni(II)NC and (b) transform of Kubelka-Munk function versus the energy of photon of $\text{TiO}_2(\text{B})$, $\text{Ni(II)NC-TiO}_2(\text{B})$; (c) UV-Vis diffuse reflectance spectra of pure $\text{TiO}_2(\text{B})$, $\text{Cu(II)NO-TiO}_2(\text{B})$ and Cu(II)NO and (d) transform of Kubelka-Munk function versus the energy of photon of $\text{TiO}_2(\text{B})$, $\text{Cu(II)NO-TiO}_2(\text{B})$.

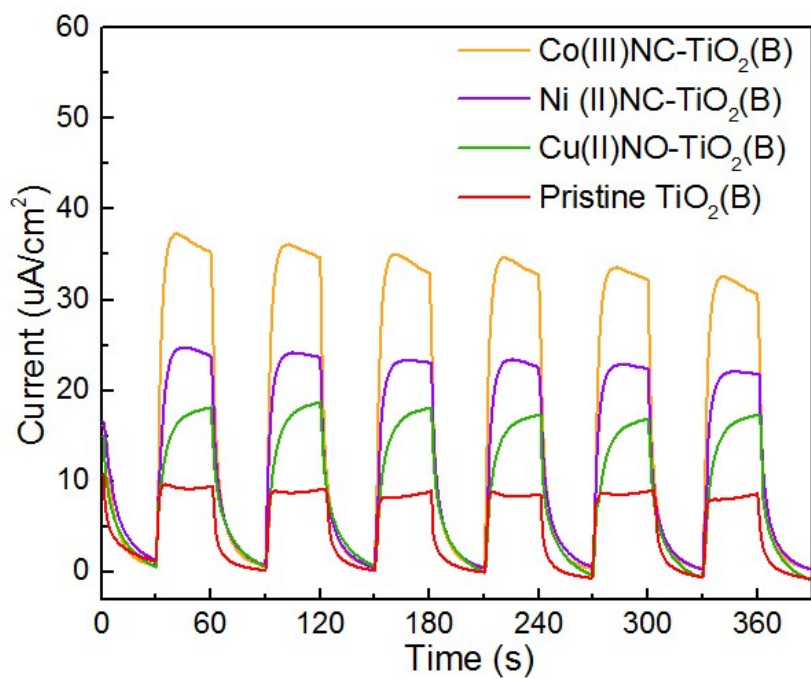


Figure S14. Transient photocurrent responses of Co(III)NC/Ni(II)NC/Cu(II)NO-TiO₂(B) nanosheets using a bias potential of 0.6 V (vs. Ag/AgCl) under on-off cycling irradiation.

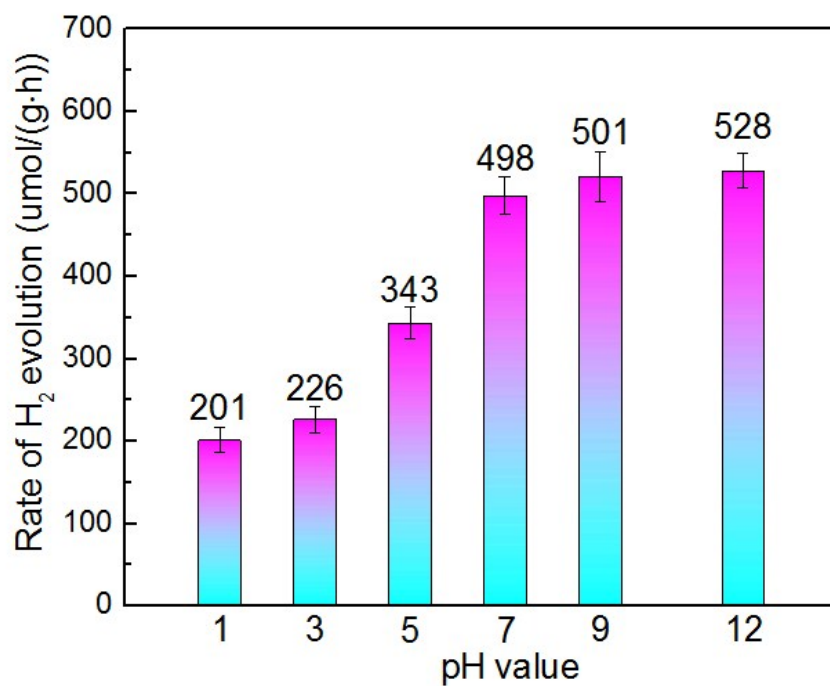


Figure S15. Photocatalytic H₂ evolution activities of Co(III)NC-TiO₂ (B) in aqueous methanol solution with different pH adjusted by dropping NH₃·H₂O solution.

Table S1 Photocatalytic H₂ evolution performance of TiO₂ materials

Semiconductor	Modification method	Surface specific area	H ₂ evolution	Published time	references
Fe ₂ O ₃ /TiO ₂	Polycrystal mixed growth	28 m ² /g	217.6 μmol/(g•h)	2014	1
Ag/TiO ₂	Photodeposition method	15 m ² /g	220 μmol/(g•h)	2015	2
Core–Shell TiO ₂	Hydrothermal treatment with hydrofluoric acid	1.6 m ² /g	268.3 μmol/(g•h)	2016	3
ZnTiO ₃ /TiO ₂	Hydrothermal treatment of zeolitic imidazolate framework	132 m ² /g	192.5 μmol/(g•h)	2017	4
Branched TiO ₂	Alkali-hydrothermal method	205.5 m ² /g	410 μmol/(g•h)	2017	5
Au/TiO ₂	Sol immobilization method	---	65 μmol/(g•h)	2018	6
RGO/ TiO ₂	Sol–gel electrospinning method	55 m ² /g	149 μmol/(g•h)	2018	7
Plasma TiO ₂ (B)	Plasma engraving	515 m ² /g	155 μmol/(g•h)	2018	8
TiO ₂ (B)-ZnO	Reflux method	23.3 m ² /g	193 μmol/(g•h)	2018	9
Au/TiO ₂ –gC ₃ N ₄	Sol–gel and chemical reduction	75 m ² /g	350 μmol/(g•h)	2018	10
Pt- TiO ₂ (B)	Photodeposition method	301 m ² /g	869.4 μmol/(g•h)		This work
Co(III)NC-TiO ₂ (B)	Electrostatic adsorption	316 m ² /g	497.8 μmol/(g•h)		This work
Ni(II)NC-TiO ₂ (B)	Electrostatic adsorption	309 m ² /g	431.6 μmol/(g•h)		This work
Cu(II)NO-TiO ₂ (B)	Electrostatic adsorption	321 m ² /g	364.6 μmol/(g•h)		This work

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