

Supplementary Material

Stability improvement of the Pt/TiO₂ photocatalyst during photocatalytic pure water splitting

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S1. Supplementary Figures

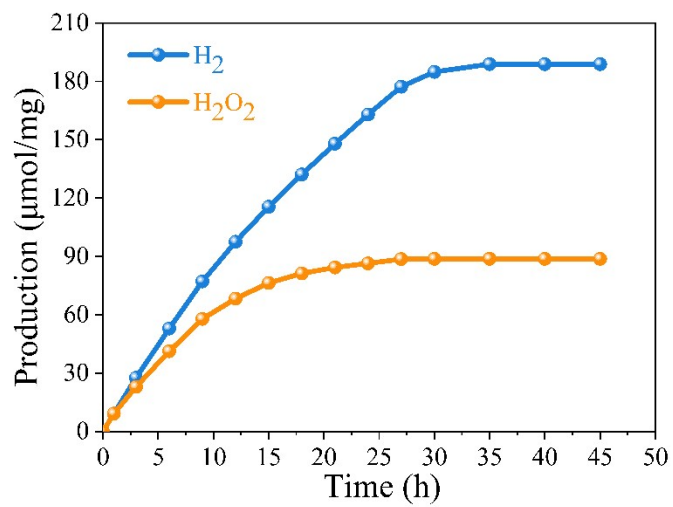


Fig. S1 The single stability of the Pt/b-TiO₂ NFs photocatalyst.

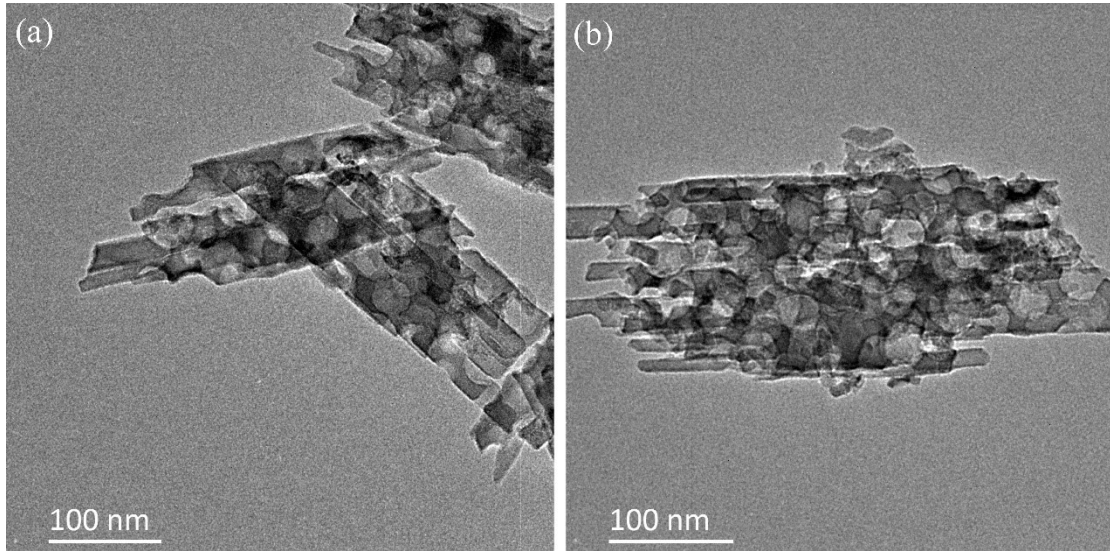


Fig. S2 TEM images of the Pt/b-TiO₂ NFs, (a) and (b) show the photocatalyst after 20 h and 40 h reaction, respectively.

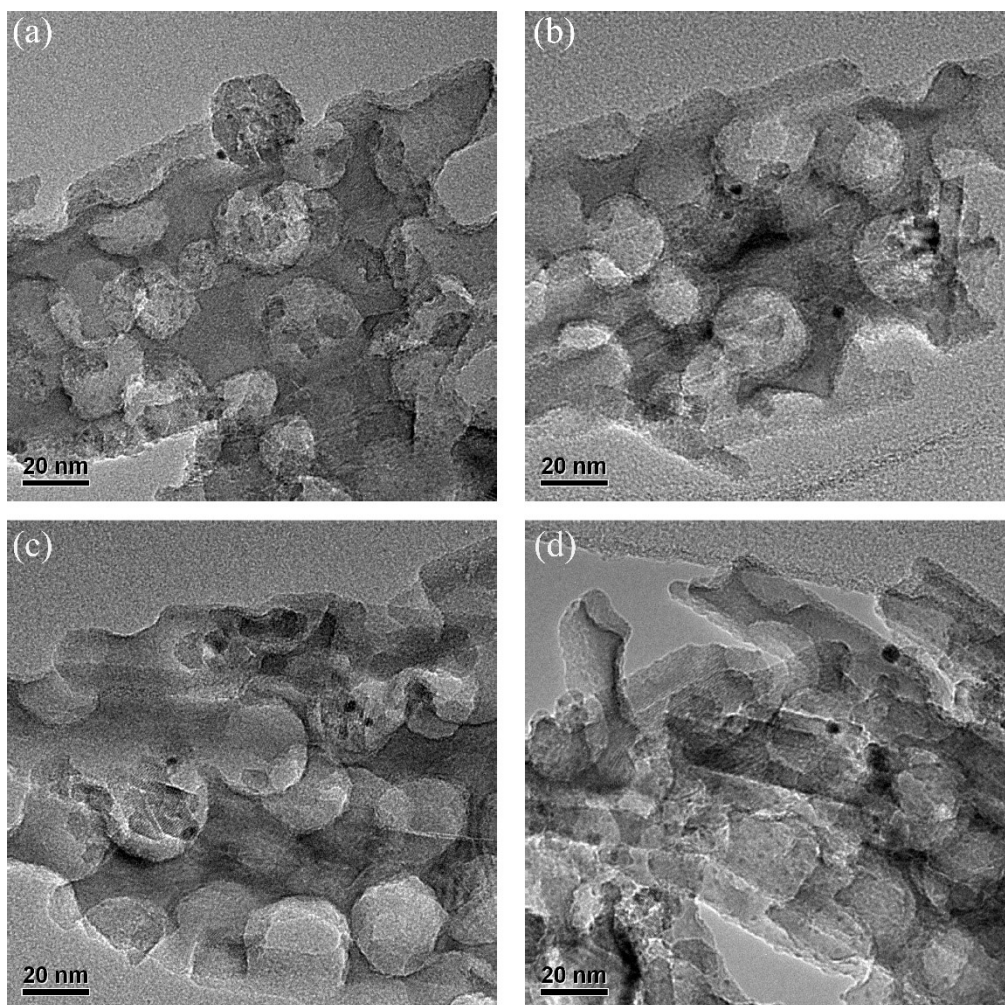


Fig. S3 (a), (b), (c), and (d) are the TEM images with higher resolution of the Pt/b-TiO₂ NFs after reaction for 0 h, 1 h, 20 h, and 40 h, respectively.

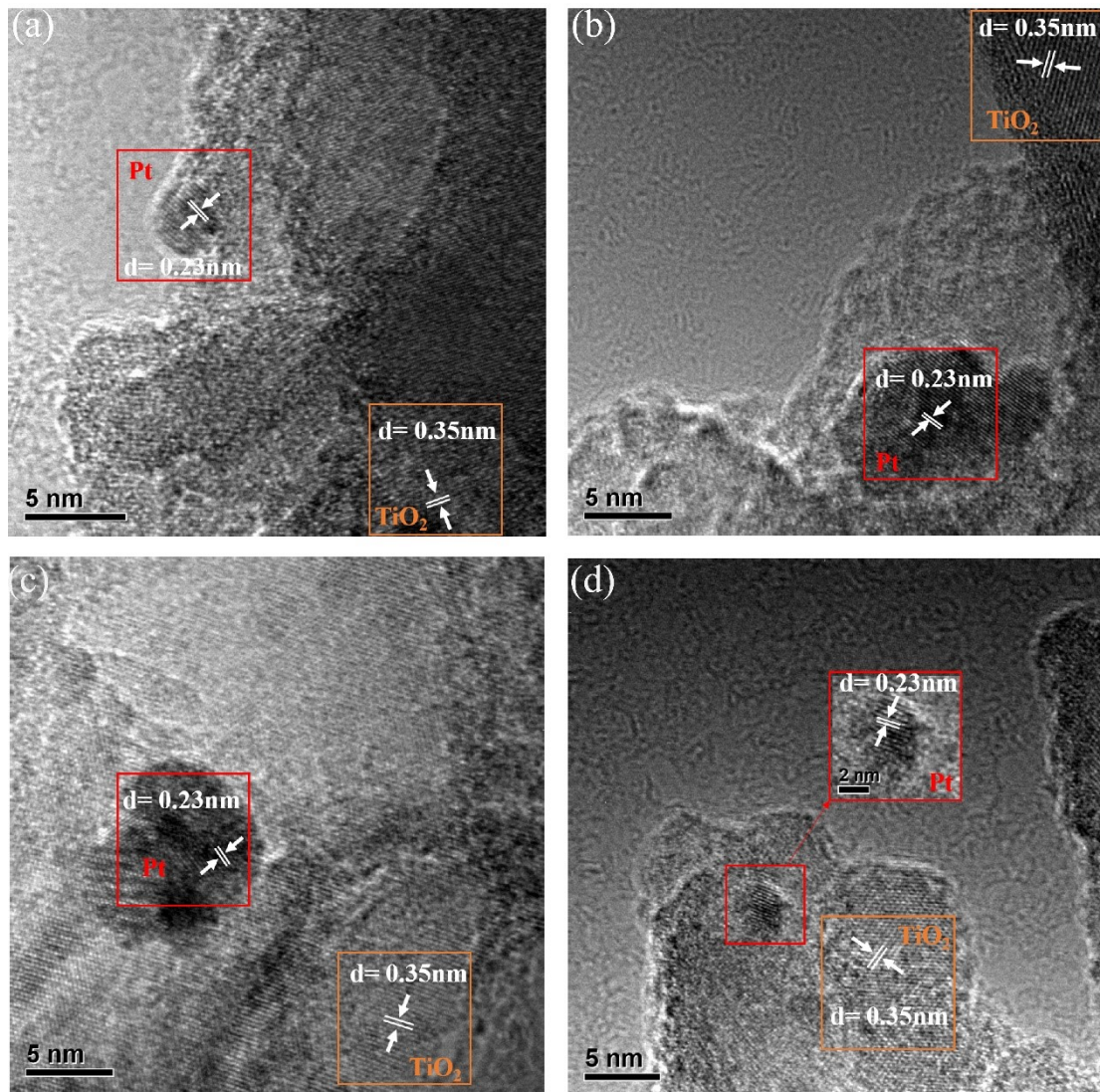


Fig. S4 (a), (b), (c), and (d) are the HRTEM images of the Pt/b-TiO₂ NFs after reaction for 0 h, 1 h, 20 h, and 40 h, respectively.

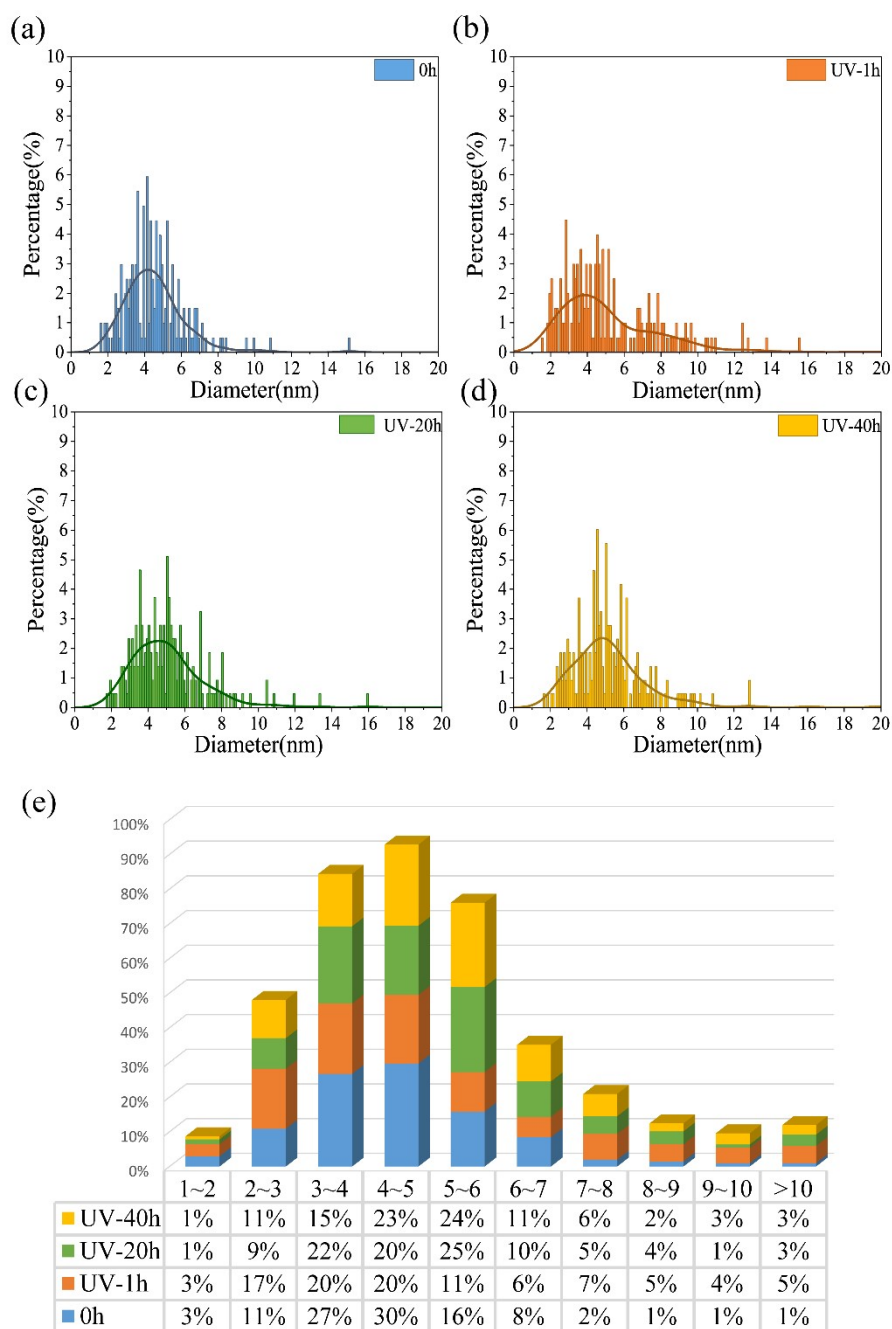


Fig. S5 (a), (b), (c), (d) are the statistical histogram of Pt nanoparticles size distribution after reaction for 0 h, 1 h, 20 h, and 40 h, respectively. (e) Statistical chart of Pt particle size percentage after the reaction for 0 h, 1 h, 20 h, and 40h.

The average dimensions (a) of 5.0 ± 1.7 nm (Mean of $N = 200$ measurements, \pm SD) are Pt before the reaction (0h). And 5.2 ± 2.7 nm (b), 5.1 ± 2.3 nm (c), and 5.3 ± 2.3 nm (d) are average dimensions of Pt nanoparticles after the reaction for 1 h, 20 h, and 40 h, respectively. Particle sizes are all concentrated at 4~6 nm (Fig. S3e), and there is no obvious change before and after the reaction.

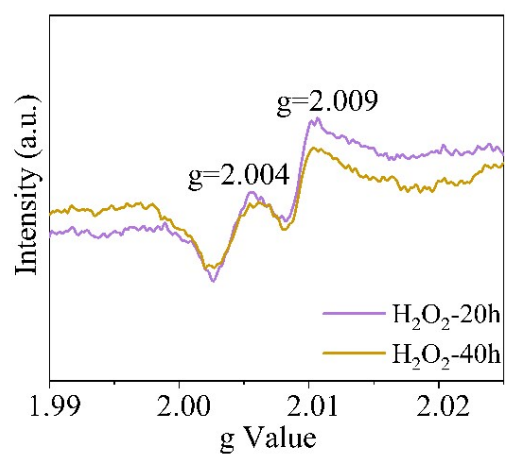


Fig. S6 EPR spectra of the Pt/b-TiO₂ NFs soaked in H₂O₂ for 20 h and 40 h, respectively.

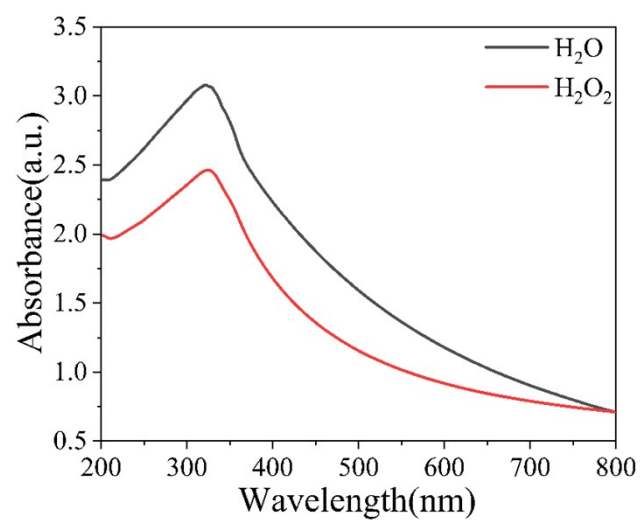


Fig. S7 UV-Vis absorption spectra of the Pt/b-TiO₂ NFs immersed in H₂O and H₂O₂. The H₂O₂ concentration here is the same as the H₂O₂ concentration under reaction conditions.

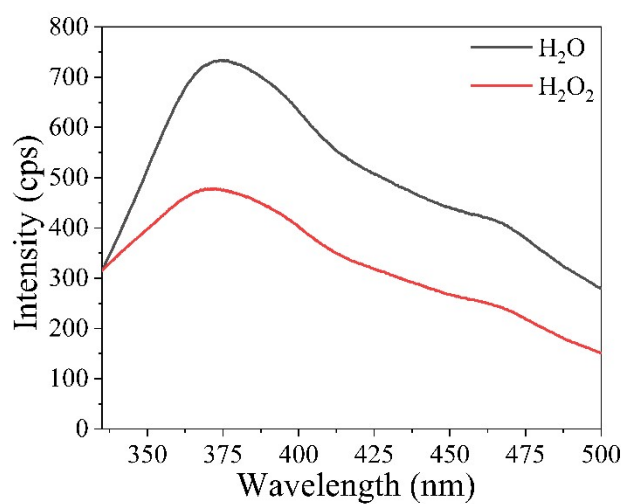


Fig. S8 Photoluminescence emission spectra of the Pt/b-TiO₂ NFs immersed in H₂O and H₂O₂. The H₂O₂ concentration here is the same as the H₂O₂ concentration under reaction conditions.

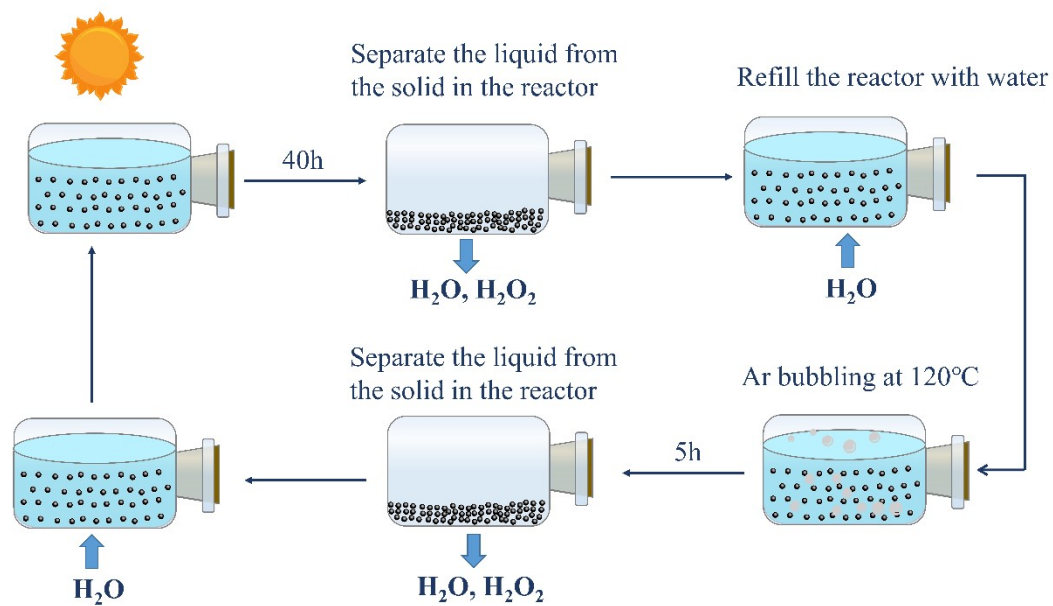


Fig. S9 Simulation diagram of in-situ photocatalyst regeneration strategy.

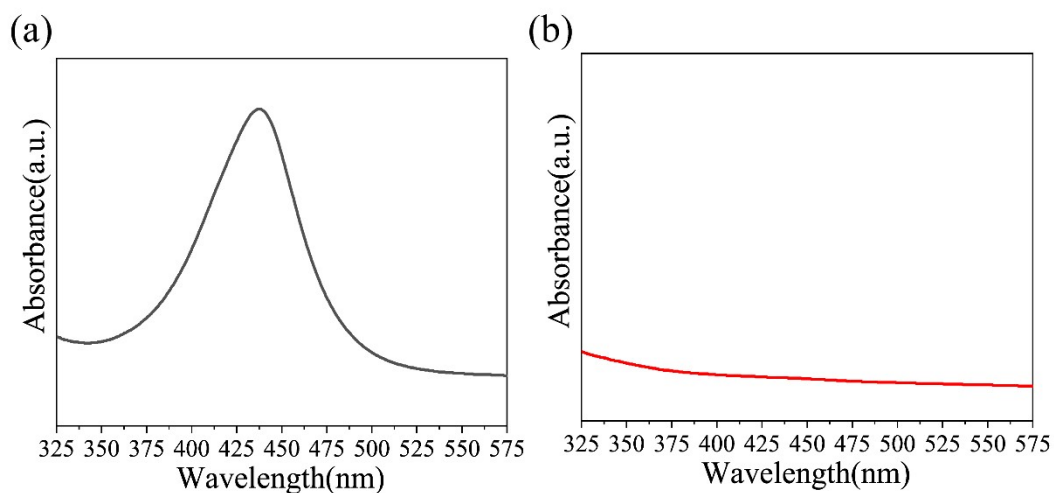


Fig. S10 The UV spectra for *o*-tolidine oxidation test of detecting peroxides. (a) the Pt/b-TiO₂ NFs photocatalyst after 40 h reaction, (b) the regenerated Pt/b-TiO₂ NFs photocatalyst. The Pt/b-TiO₂ NFs photocatalyst after 40 h reaction has strong characteristic absorption at 438 nm, indicating that H₂O₂ is adsorbed on the photocatalyst surface. The regenerated sample does not have this characteristic absorption peak, indicating that there is no H₂O₂ adsorbed on the surface of the regenerated photocatalyst.

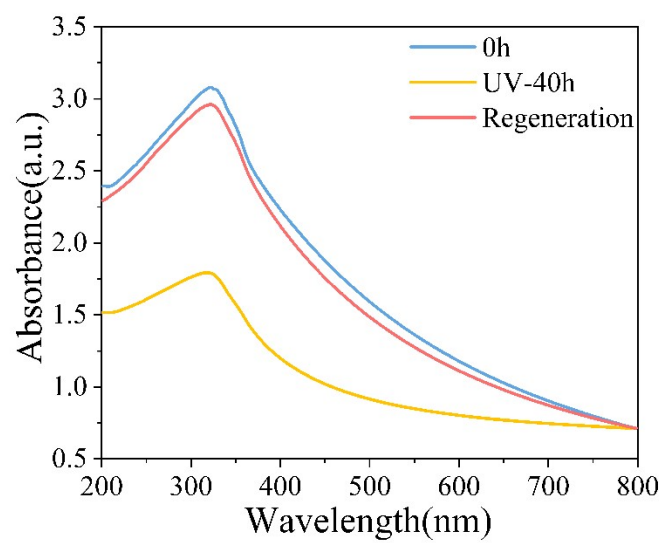


Fig. S11 UV-Vis absorption spectra of the fresh, reacted for 40 h, and regenerated Pt/b-TiO₂ NFs photocatalyst.

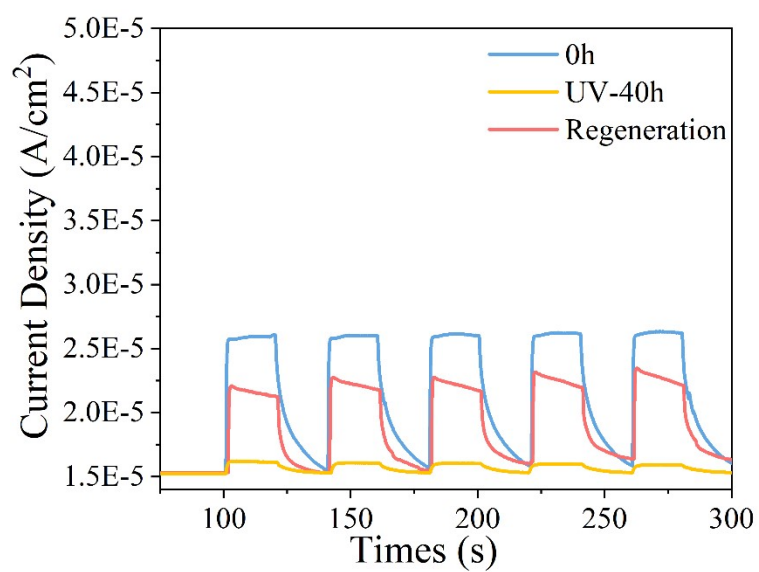


Fig. S12 Photocurrent I-t curves of the fresh, reacted for 40 h, and regenerated Pt/b-TiO₂ NFs photocatalyst.

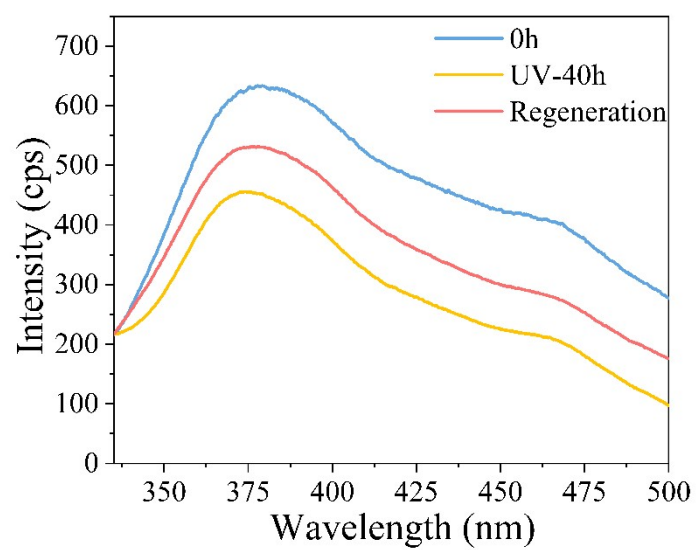


Fig. S13 Photoluminescence emission spectra of the fresh, reacted for 40 h, and regenerated Pt/b-TiO₂ NFs photocatalyst.

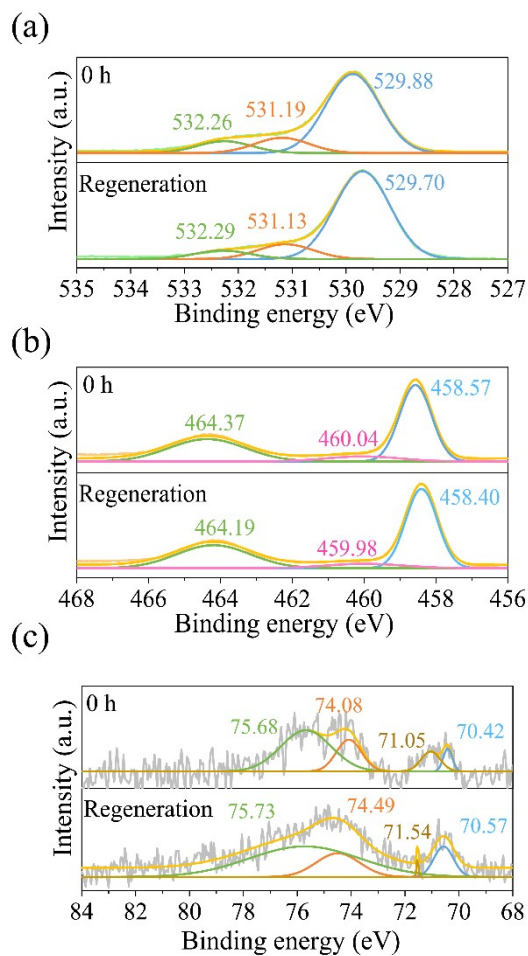


Fig. S14 XPS spectra of (a) O 1s, (b) Ti 2p and (c) Pt 4f of the fresh, reacted for 40 h, and regenerated Pt/b-TiO₂ NFs photocatalyst.

Most of the regenerated Pt could not return to the initial state, because the content of Pt²⁺ gradually increased with the progress of the photocatalytic pure water splitting reaction, and the bubbling regeneration method could not reduce the content of oxidized Pt.

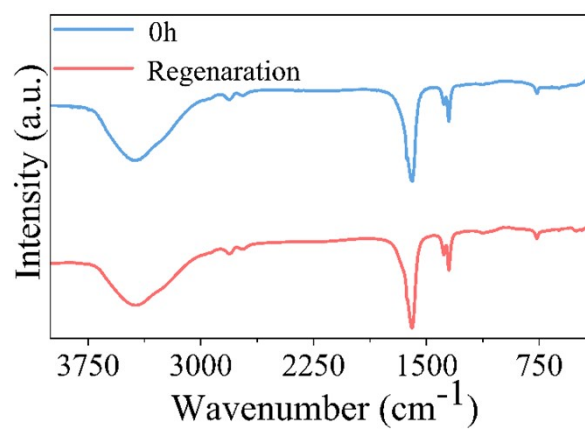


Fig. S15 FTIR spectra of the fresh, reacted for 40 h, and regenerated Pt/b-TiO₂ NFs photocatalyst.

S2. Supplementary Tables

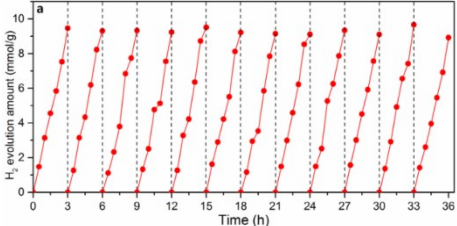
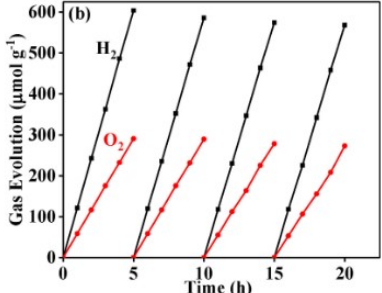
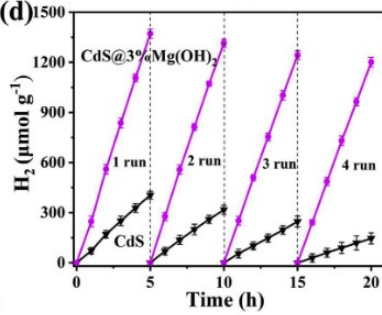
Table. S1 Quantitative analysis of Pt/b-TiO₂ NFs after 0 h, 1 h, 20 h, and 40 h of UV light irradiation.

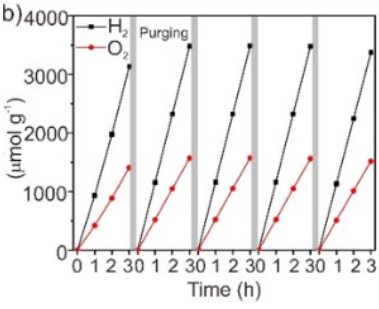
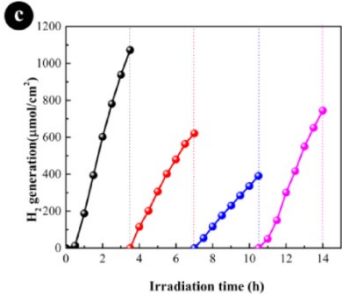
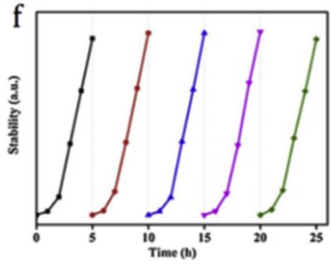
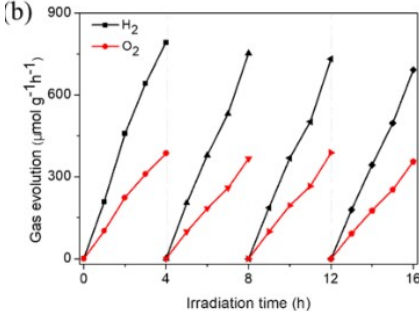
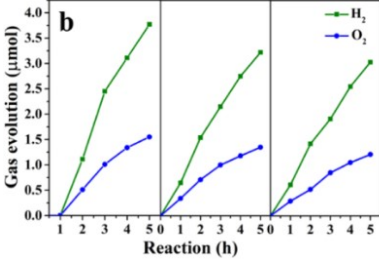
	Lattice oxygen (Ti-O)	Surface Hydroxyl (Ti-OH)	Physisorbed Water
0h	74.47%	14.36%	11.17%
UV-1h	63.14%	20.85%	16.01%
UV-20h	64.17%	18.37%	17.46%
UV-40h	73.15%	15.77%	11.09%
Regeneration	79.11%	13.30%	7.59%

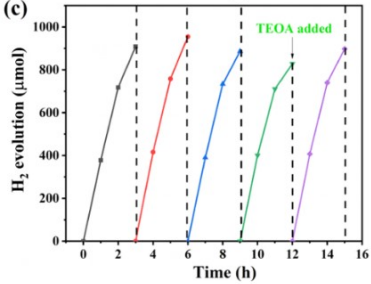
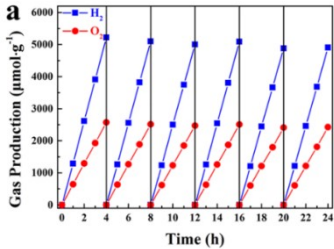
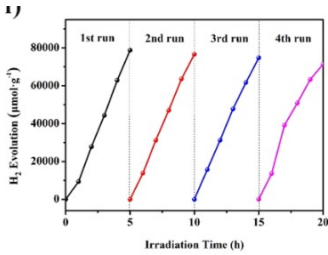
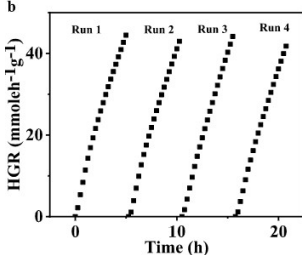
	Ti⁴⁺ 2p_{5/2}	Ti³⁺ 2p_{5/2}	Ti⁴⁺ 2p_{3/2}
0h	53.87%	8.48%	37.65%
UV-1h	68.00%	6.75%	25.25%
UV-20h	68.90%	7.95%	23.15%
UV-40h	69.56%	5.28%	25.15%
Regeneration	55.07%	7.60%	37.33%

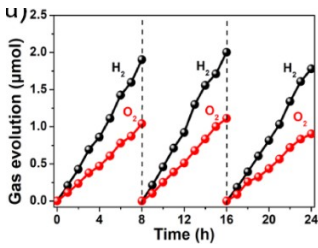
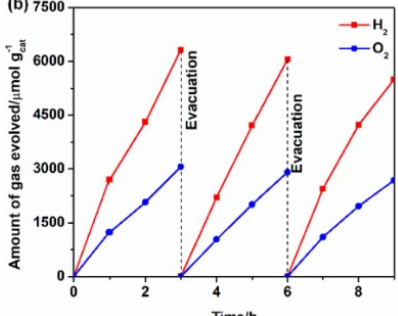
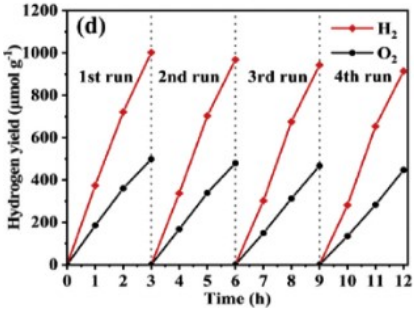
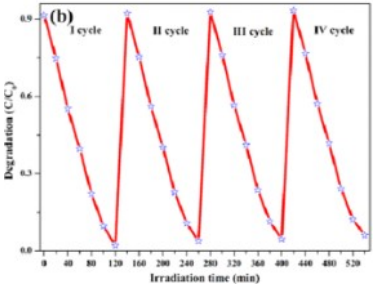
	Pt⁰ 4f_{7/2}	Pt⁰ 4f_{5/2}	Pt²⁺ 4f_{7/2}	Pt²⁺ 4f_{5/2}
0h	5.90%	23.13%	11.07%	59.90%
UV-1h	17.44%	46.36%	0.95%	35.25%
UV-20h	13.02%	19.67%	4.06%	63.25%
UV-40h	13.02%	19.66%	4.05%	63.27%
Regeneration	11.95%	20.39%	0.71%	67.05%

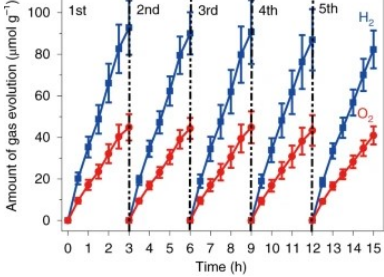
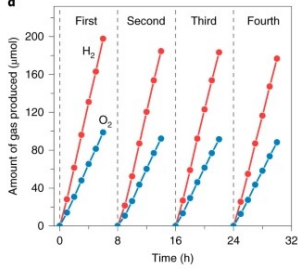
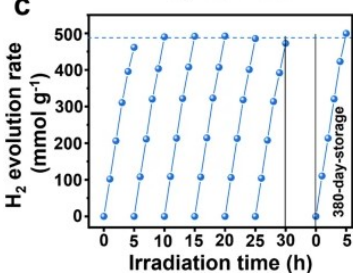
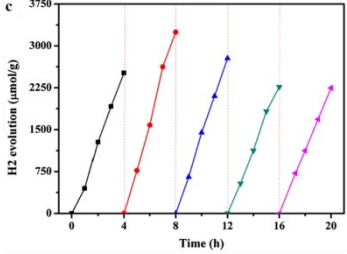
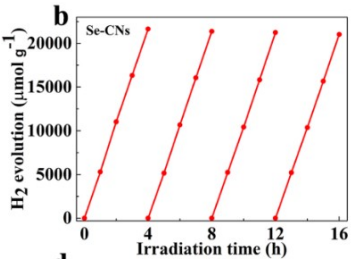
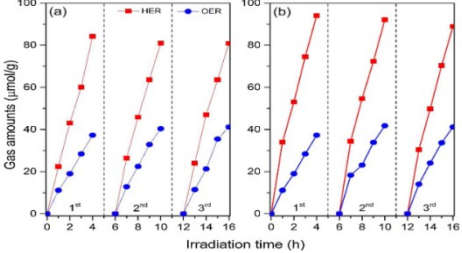
Table. S2 Comparison of data on the stability of photocatalysts for photocatalytic overall water splitting. In the past three years, the highest stability time was 216 h, and 95% of them were stable for 20~30 h.

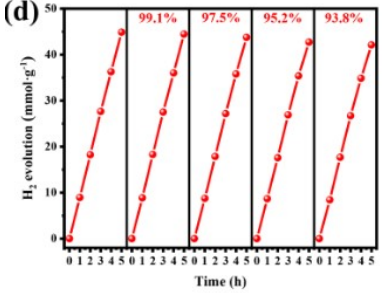
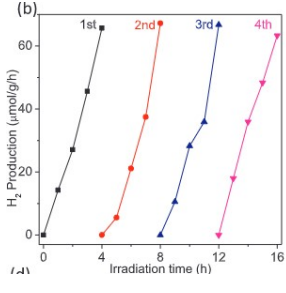
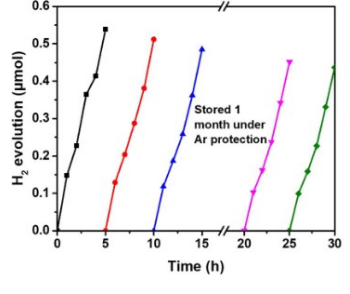
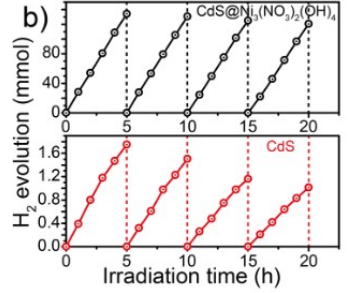
Reference	Stability	Image
<p>Self-assembly photocatalytic reduction synthesis of graphene-encapsulated LaNiO_3 nanoreactor with high efficiency and stability for photocatalytic water splitting to hydrogen, <i>Chemical Engineering Journal</i>, 2018, 356, 580-591.</p>	36 h	
<p>Highly efficient photocatalytic overall water splitting on plasmonic Cu_6Sn_5/polyaniline nanocomposites, <i>Journal of Colloid and Interface Science</i>, 2021, 609, 785-793.</p>	20 h	
<p>$\text{CdS}@\text{Mg}(\text{OH})_2$ core/shell composite photocatalyst for efficient visible-light photocatalytic overall water splitting, <i>International Journal of Hydrogen Energy</i>, 2022, 47(14), 8729-8738.</p>	20 h	

<p>Switching from two-electron to four-electron photocatalytic pure water splitting via band bending engineering with boosted activity, <i>Applied Catalysis B: Environmental</i>, 2022, 305, 121054-121054.</p>	<p>15 h</p>	 <p>Graph b) shows the evolution of H₂ (black squares) and O₂ (red circles) in μmol g⁻¹ over time (h). The y-axis ranges from 0 to 4000. The x-axis shows a series of 3-hour intervals, each followed by a 'Purging' period. H₂ evolution reaches approximately 3500 μmol g⁻¹ after each 3-hour interval, while O₂ evolution reaches approximately 1500 μmol g⁻¹.</p>
<p>Enhanced photoelectrochemical performances in photocatalytic pollutant degradation and water splitting by direct Z-scheme Bi₂Sn₂O₇/TiO₂ NTAs, <i>Ceramics International</i>, 2022, 48(3), 3941-3953.</p>	<p>14 h</p>	 <p>Graph c) shows H₂ generation (μmol/cm²) versus irradiation time (h). The y-axis ranges from 0 to 1200. The x-axis ranges from 0 to 14 hours. The data points are shown in black, red, blue, and pink, indicating a linear increase in H₂ generation over time, reaching approximately 1000 μmol/cm² at 14 hours.</p>
<p>Layered g-C₃N₄/TiO₂ nanocomposites for efficient photocatalytic water splitting and CO₂ reduction: a review, <i>Materials Today Energy</i>, 2022, 23, 100904-100904.</p>	<p>25 h</p>	 <p>Graph f) shows stability (a.u.) versus time (h). The y-axis ranges from 0 to 1.0. The x-axis ranges from 0 to 25 hours. The data points are shown in black, red, blue, purple, and green, indicating a linear increase in stability over time, reaching approximately 1.0 at 25 hours.</p>
<p>Fabrication of 0D/2D TiO₂ Nanodots/g-C₃N₄ S-scheme heterojunction photocatalyst for efficient photocatalytic overall water splitting, <i>Applied Surface Science</i>, 2022, 571, 151287-151287.</p>	<p>16 h</p>	 <p>Graph (b) shows gas evolution (μmol g⁻¹h⁻¹) versus irradiation time (h). The y-axis ranges from 0 to 900. The x-axis ranges from 0 to 16 hours. The data points are shown in black (H₂) and red (O₂), indicating a linear increase in gas evolution over time, reaching approximately 750 μmol g⁻¹h⁻¹ for H₂ and 350 μmol g⁻¹h⁻¹ for O₂ at 16 hours.</p>
<p>Band structure-controlled P-C₃N₄ for photocatalytic water splitting via appropriately decreasing oxidation capacity, <i>Journal of Alloys and Compounds</i>, 2022, 895, 162513-162513.</p>	<p>15 h</p>	 <p>Graph b) shows gas evolution (μmol) versus reaction time (h). The y-axis ranges from 0.0 to 4.0. The x-axis ranges from 0 to 5 hours. The data points are shown in green (H₂) and blue (O₂), indicating a linear increase in gas evolution over time, reaching approximately 3.8 μmol for H₂ and 1.5 μmol for O₂ at 5 hours.</p>

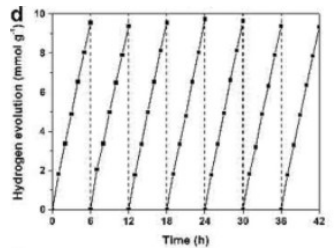
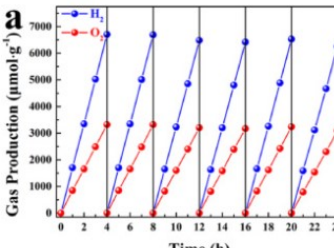
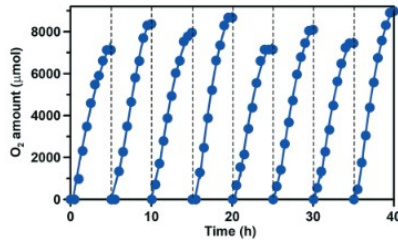
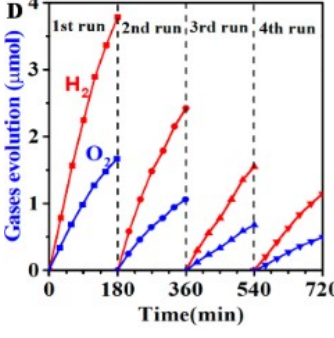
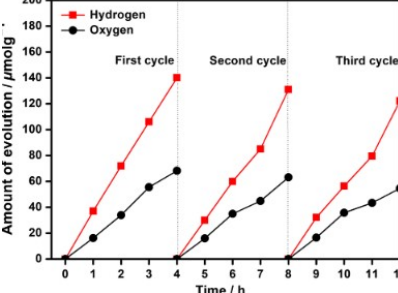
<p>Heat treatment to prepare boron doped g-C₃N₄ nanodots/carbon-rich g-C₃N₄ nanosheets heterojunction with enhanced photocatalytic performance for water splitting hydrogen evolution, <i>Journal of Alloys and Compounds</i>, 2022, 898, 162846-162846.</p>	<p>15 h</p>	
<p>The photocatalytic overall water splitting hydrogen production of g-C₃N₄/CdS hollow core-shell heterojunction via the HER/OER matching of Pt/MnO_x, <i>Chemical Engineering Journal</i>, 2021, 405, 126622-126622.</p>	<p>24 h</p>	
<p>Synthesis of CdS/CoP hollow nanocages with improved photocatalytic water splitting performance for hydrogen evolution, <i>Journal of Environmental Chemical Engineering</i>, 2021, 9(6), 106270-106270</p>	<p>20 h</p>	
<p>Photocatalytic hydrogen generation via water splitting using ZIF-67 derived Co₃O₄@C/TiO₂, <i>Journal of Environmental Chemical Engineering</i>, 2021, 9(4), 105702-105702.</p>	<p>20 h</p>	

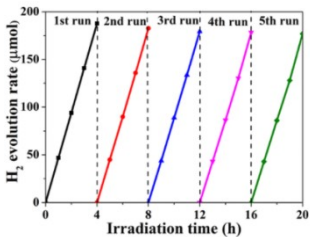
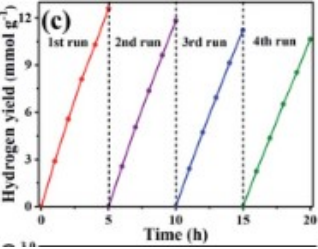
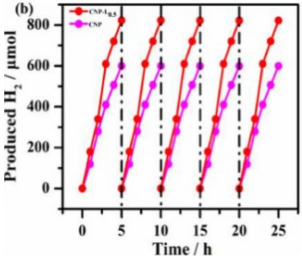
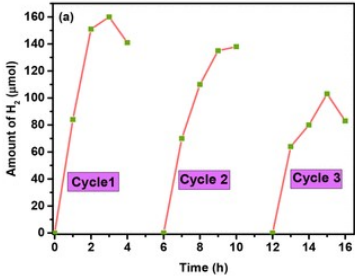
<p>2D/2D heterostructure of ultrathin BiVO₄/Ti₃C₂ nanosheets for photocatalytic overall Water splitting, Applied Catalysis B: Environmental, 2021, 285, 119855-119855.</p>	<p>24 h</p>	
<p>Photocatalytic overall water splitting without noble-metal: Decorating CoP on Al-doped SrTiO₃, Journal of Colloid and Interface Science, 2022, 606, 491-499.</p>	<p>9 h</p>	
<p>Facile synthesis of anatase/rutile TiO₂/g-C₃N₄ multi-heterostructure for efficient photocatalytic overall water splitting, International Journal of Hydrogen Energy, 2020, 45(35), 17378-17387.</p>	<p>12 h</p>	
<p>Novel Z-scheme binary zinc tungsten oxide/nickel ferrite nano hybrids for photocatalytic reduction of chromium (Cr (VI)), photoelectrochemical water splitting and degradation of toxic organic pollutants, Journal of Hazardous Materials, 2022, 423, 127044-127044.</p>	<p>~10 h</p>	

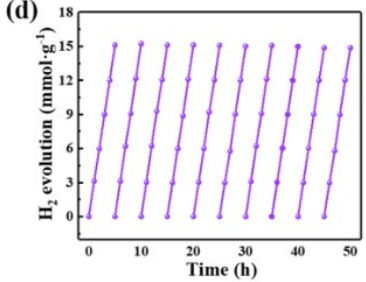
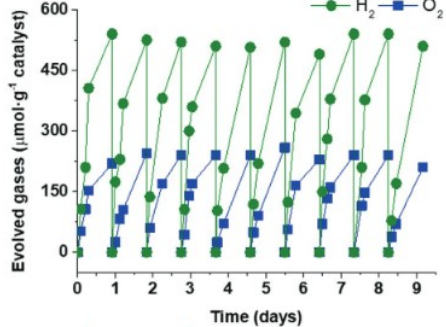
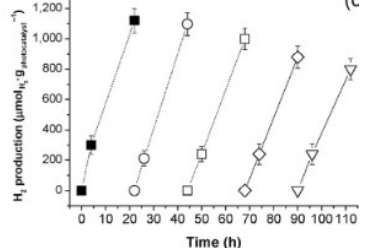
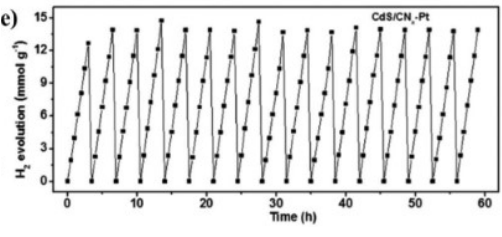
<p>Overall photocatalytic water splitting by an organolead iodide crystalline material, <i>Nature Catalysis</i>, 2020, 3(12), 1027-1033.</p>	<p>15 h</p>	
<p>Boron-doped nitrogen-deficient carbon nitride-based Z-scheme heterostructures for photocatalytic overall water splitting, <i>Nature Energy</i>, 2021, 6(4), 388-397.</p>	<p>32 h</p>	
<p>Single-atom Cu anchored catalysts for photocatalytic renewable H₂ production with a quantum efficiency of 56%, <i>Nature Communications</i>, 2022, 13(1), 1-10.</p>	<p>30 h</p>	
<p>The high photocatalytic efficiency and stability of LaNiO₃/g-C₃N₄ heterojunction nanocomposites for photocatalytic water splitting to hydrogen, <i>BMC chemistry</i>, 2022, 14(1), 1-13.</p>	<p>20 h</p>	
<p>Introducing spin polarization into atomically thin 2D carbon nitride sheets for greatly extended visible-light photocatalytic water splitting, <i>Nano Energy</i>, 2021, 83, 105783-105783.</p>	<p>16 h</p>	
<p>Z-scheme photocatalyst Pt/GaP-TiO₂-SiO₂: Rh for the separated H₂ evolution from photocatalytic seawater splitting, <i>Applied Catalysis B: Environmental</i>, 2021, 296,</p>	<p>16 h</p>	

120339-120339.		
Holey defected TiO ₂ nanosheets with oxygen vacancies for efficient photocatalytic hydrogen production from water splitting, <i>Surfaces and Interfaces</i> , 2021, 23, 100979-100979.	25 h	
Single 2D MXene precursor-derived TiO ₂ nanosheets with a uniform decoration of amorphous carbon for enhancing photocatalytic water splitting, <i>Applied Catalysis B: Environmental</i> , 2020, 270, 118885-118885.	16 h	
Z-scheme heterojunction through interface engineering for broad spectrum photocatalytic water splitting, <i>Applied Catalysis B: Environmental</i> , 2020, 267, 118661-118661.	30 h	
CdS@Ni ₃ (NO ₃) ₂ (OH) ₄ nanorods@nanosheets for boosted photocatalytic H ₂ generation rate and stability under visible light irradiation, <i>Colloids and Surfaces A: Physicochemical and Engineering Aspects</i> , 2022, 639, 128352-128352.	20 h	

<p>Overall water splitting on surface-polarized Sn₃O₄ through weakening the trap of Sn(II) to holes, Applied Catalysis B: Environmental, 2021, 299, 120689-120689.</p>	<p>15 h</p>	
<p>Surface-Polarity-Induced Spatial Charge Separation Boosts Photocatalytic Overall Water Splitting on GaN Nanorod Arrays, Angewandte Chemie, 2020, 132(2), 945-952.</p>	<p>9 h</p>	
<p>Atomically thin PdSe₃ nanosheets: a promising 2D photocatalyst produced by quaternary ammonium intercalation and exfoliation, Chemical Communications, 2020, 56(41), 5504-5507.</p>	<p>33 h</p>	
<p>La, Al-Codoped SrTiO₃ as a Photocatalyst in Overall Water Splitting: Significant Surface Engineering Effects on Defect Engineering, ACS Catalysis, 2021, 11(18), 11429-11439.</p>	<p>18 h</p>	
<p>Steering Hole Transfer from the Light Absorber to Oxygen Evolution Sites for Photocatalytic Overall Water Splitting, Advanced Materials Interfaces, 2021, 8(22), 2101158-2101158.</p>	<p>25 h</p>	

<p>Smart Assembly of Sulfide Heterojunction Photocatalysts with Well-Defined Interfaces for Direct Z-Scheme Water Splitting under Visible Light, <i>ChemSusChem</i>, 2020, 13(11), 2996-3004.</p>	<p>42 h</p>	
<p>The Pt-free 1T/2H-MoS₂/CdS/MnO_x hollow core-shell nanocomposites toward overall water splitting via HER/OER synergy of 1T-MoS₂/MnO_x, <i>Materials Today Chemistry</i>, 2021, 21, 100528-100528.</p>	<p>24 h</p>	
<p>Development of BiOI as an effective photocatalyst for oxygen evolution reaction under simulated solar irradiation, <i>Catalysis Science & Technology</i>, 2020, 10(10), 3223-3231.</p>	<p>40 h</p>	
<p>In Situ Photodeposited Construction of Pt-CdS/g-C₃N₄-MnO_x Composite Photocatalyst for Efficient Visible-Light-Driven Overall Water Splitting, <i>ACS Applied Materials & Interfaces</i>, 2020, 12(18), 20579-20588.</p>	<p>12 h</p>	
<p>Temperature effect on green-synthesized Co₃O₄nanoparticle as photocatalyst for overall water splitting, <i>Journal of Photonics for Energy</i>, 2020, 10(4), 042006-042006.</p>	<p>12 h</p>	

<p>Graphitic Carbon Nitride Decorated with Nickel(II)-(3-Pyridyl) Benzimidazole Complexes and Pt Nanoparticles as a Cocatalyst for Photocatalytic Hydrogen Production from Water Splitting, ACS Applied Nano Materials, 2020, 3(11), 10659-10667.</p>	<p>20 h</p>	
<p>FeNi intermetallic compound nanoparticles wrapped with N-doped graphitized carbon: a novel cocatalyst for boosting photocatalytic hydrogen evolution, Journal of Materials Chemistry A, 2020, 8(6), 3481-3490.</p>	<p>20 h</p>	
<p>Biomass Lignin Integrated Polymeric Carbon Nitride for Boosted Photocatalytic Hydrogen and Oxygen Evolution Reactions, Molecular Catalysis, 2022, 518, 112064-112064.</p>	<p>25 h</p>	
<p>Constructing La_xCo_yO₃ Perovskite Anchored 3D g-C₃N₄ Hollow Tube Heterojunction with Proficient Interface Charge Separation for Stimulating Photocatalytic H₂ Production, Energy & Fuels, 2021, 35(11), 9727-9746.</p>	<p>16 h</p>	

<p>Stable and efficient Ti_3C_2 MXene/MAPbI_3-HI system for visible-light-driven photocatalytic HI splitting, <i>Journal of Power Sources</i>, 2022, 522, 231006-231006.</p>	<p>50 h</p>	
<p>A Novel Porous Ti-Squarate as Efficient Photocatalyst in the Overall Water Splitting Reaction under Simulated Sunlight Irradiation, <i>Advanced Materials</i>, 2021, 33(52), 2106627-2106627.</p>	<p>216 h</p>	
<p>Nickel phosphonate MOF as efficient water splitting photocatalyst, <i>Nano Research</i>, 2021, 14(2), 450-457</p>	<p>110 h</p>	
<p>Semiconductor-based photocatalysts for photocatalytic and photoelectrochemical water splitting: will we stop with photocorrosion? <i>Journal of Materials Chemistry A</i>, 2020, 8(5), 2286-2322.</p>	<p>60 h</p>	
<p>Carbon quantum dots enriching molecular nickel polyoxometalate over CdS semiconductor for photocatalytic water splitting, <i>Applied Catalysis B: Environmental</i>, 2021, 293, 120214-120214.</p>	<p>96 h</p>	