

Electronic Supplementary Materials

Three-dimensional graphitic carbon nitride belt network for enhanced visible light photocatalytic hydrogen evolution

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Table S1 Atomic percentage obtained from XPS spectra of bulk g-C₃N₄ and g-C₃N₄ NB.

Samples	C (%)	N (%)	O (%)	C/N
bulk g-C ₃ N ₄	41.59	56.99	1.42	0.73
bulk g-C ₃ N ₄	42.32	53.08	4.60	0.80

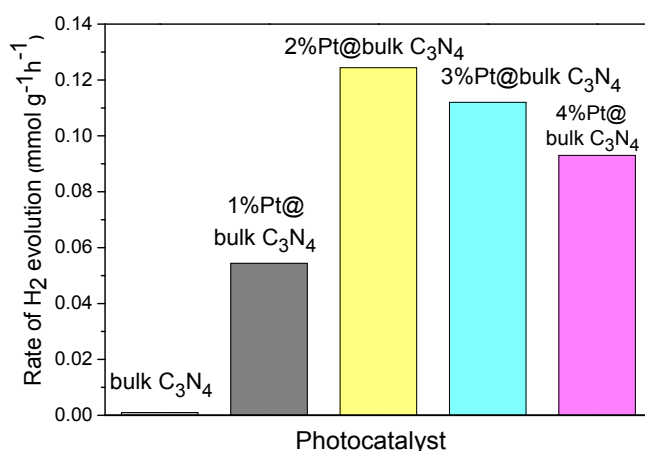


Fig. S1 HER of bulk g-C₃N₄ loaded with various content Pt under visible light ($\lambda > 420$ nm) using 10% TEOA as sacrificial agent.

Determination of apparent quantum efficiency

The apparent quantum efficiency (AQE) for 3wt% Pt@g-C₃N₄ NB was measured under the same photocatalytic reaction condition and the incident light was supplied by a 300W Xe lamp with specific band-pass filters to get the desired incident wavelength (420 nm). The catalyst solution was irradiated for 10 hours. The average intensity of irradiation was determined to be 3.58 mW·cm⁻² and the irradiation area was 5.96 cm². The amount of H₂ molecules generated in 10 hours was 158.1 μ mol. The quantum efficiency was calculated by Equation 1. The number of evolved H₂ molecules was

calculated by Equation 2. The number of incident photons was 1.606×10^{21} as calculated by Equation 3.

$$\begin{aligned} \text{AQE}[\%] &= \frac{\text{Number of reacted electrons}}{\text{Number of incident photons}} \times 100 \\ &= \frac{\text{Number of H}_2 \text{ molecules} \times 2}{\text{Number of incident photos}} \times 100 \quad (1) \end{aligned}$$

$$\text{Number of H}_2 \text{ molecules} = 6.02 \times 10^{23} \times 158.1 \times 10^{-6} = 7.89 \times 10^{19} \quad (2)$$

$$\begin{aligned} \text{Number of incident photons} &= E\lambda/hc = (3.58 \times 10^{-3} \times 5.96 \times 3600 \times 10 \times 420 \times 10^{-9}) / \\ &(6.626 \times 10^{-34} \times 3 \times 10^8) = 1.62 \times 10^{21} \quad (3) \end{aligned}$$

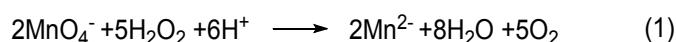
The calculated AQE at 420 nm is 12%.

Table S2. Comparison g-C₃N₄ based composites for visible-light-driven photocatalytic HER using sacrificial agent as electron donor.

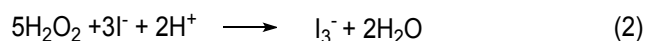
Photocatalysts	Light source Xe lamp (nm)	Sacrificial agent (vol %)	Activity (mmol/g/h)	AQE (%)
Pt/g-C ₃ N ₄ ^{S1}	300W $\lambda \geq 420$	10 % TEOA	0.375	-
Pt/g-C ₃ N ₄ ^{S2}	300W $\lambda \geq 420$	10 % TEOA	1.150	-
Pt/g-C ₃ N ₄ ^{S3}	300W $\lambda \geq 400$	10 % TEOA	0.640	-
Pt/g-C ₃ N ₄ ^{S4}	300W $\lambda \geq 420$	10 % TEOA	0.230	-
Pt/g-C ₃ N ₄ ^{S5}	125W $\lambda \geq 400$	10 % TEOA	0.240	-
Pt/g-C ₃ N ₄ ^{S6}	300W $\lambda \geq 420$	10 % TEOA	0.098	0.58% (440 nm)
NiS/g-C ₃ N ₄ ^{S7}	300W $\lambda \geq 420$	15 % TEOA	0.482	4.4% (440nm)
Pt/BiPO ₄ /g-C ₃ N ₄ ^{S8}	300W $\lambda \geq 420$	10%methanol	1.110	-
Pt/g-PAN/g-C ₃ N ₄ ^{S9}	300W $\lambda \geq 400$	10 % TEOA	0.370	-
Ag ₂ O/g-C ₃ N ₄ ^{S10}	300W $\lambda \geq 420$	10 % TEOA	0.330	-
Pt/g-C ₃ N ₄ ^{S11}	300W $\lambda \geq 400$	10 % TEOA	0.604	-
Pt/g-C ₃ N ₄ ^{S12}	300W $\lambda \geq 400$	11 % TEOA	1.510	5.7%
Pt/g-C ₃ N ₄ (our work)	300W $\lambda \geq 420$	10 % TEOA	1.360	12%

H₂O₂ determination principle by the I₃⁻ method

H₂O₂ determination was realized according to I₃⁻ strategy.^{S13} In detail, standard K₂MnO₄ solution (0.02M) was used to precisely titrate concentration of H₂O₂ according to equation (1):



Then prepare a suite of gradient concentration standard H₂O₂ solution. After added I⁻ anion completely reduces H₂O₂ molecule into H₂O in equation (2), and oxidation product I₃⁻ anion was quantitatively measured by UV-Vis spectra.



In the end, calculated the oxidation product H_2O_2 content derived from photocatalytic overall water splitting after centrifugation to remove photocatalyst.

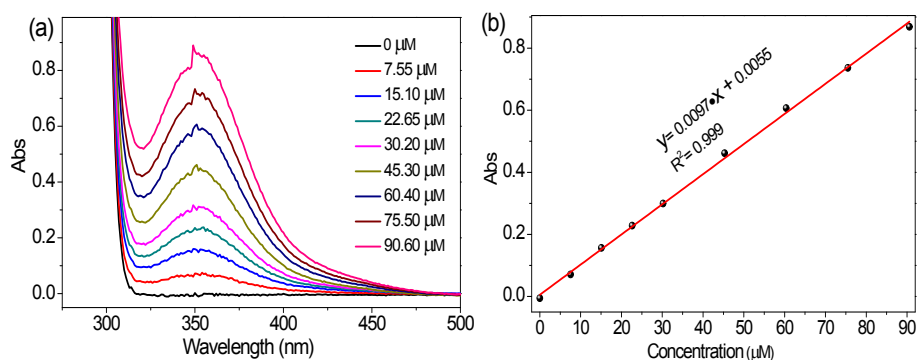


Fig. S2 (a) UV-Vis spectra of I_3^- by H_2O_2 oxidation and (b) linear fitting curve.

Accurate analysis of aqueous trace H_2O_2 concentration as low as μM can be done by I_3^- method, based on the spectrophotometric determination of I_3^- formed when H_2O_2 added to a mixed concentrated solution of I^- and potassium acid phthalate. In the UV-Vis spectrum in Fig. S2a, maximum absorption peak at ca. 351 nm was unique characteristic peak of I_3^- , thus in accordance with a series of gradient concentration I_3^- solution acquires Abs function I_3^- concentration in Fig. S2b.

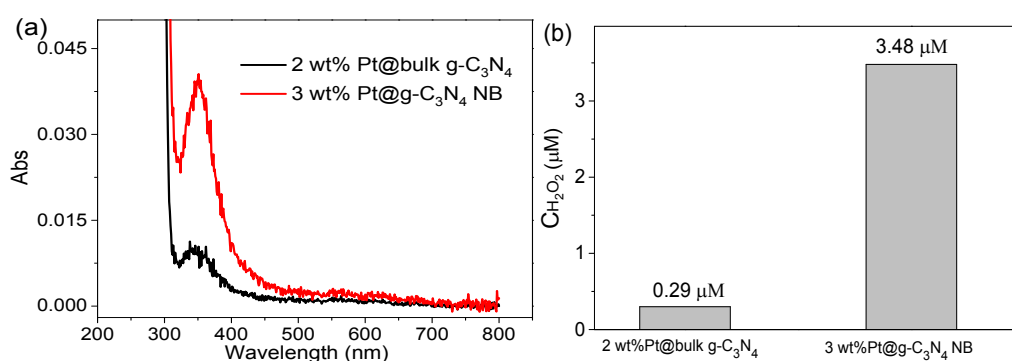


Fig. S3 (a) UV-Vis spectra of I_3^- by H_2O_2 oxidation from photocatalytic overall water splitting of 3 wt%Pt@g- C_3N_4 NB under visible light illumination ($\lambda > 420$ nm) and (b) corresponding H_2O_2 concentration. (Conditions: 20 mg photocatalyst, reaction hour: 1 hour)

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