

Electronic Supplementary Information

**Facile synthesis of mesoporous g-C₃N₄ with highly enhanced
photocatalytic H₂ evolution performance**

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Experimental Section

1. Materials

Melamine (99%), triethanolamine (99%) and chloroplatinic acid hexahydrate were purchased from Aladdin. Sucrose was purchased from the Shanghai Chemical Company. Triethanolamine (TEOA) was purchased from Tianjin Kermel Chemical Reagent Co., Ltd. All chemicals were used as received without further treatment.

2. Preparation of photocatalysts.

Pure g-C₃N₄ was obtained by directly heating melamine at 600 °C for 2 h. The mesoporous g-C₃N₄ was prepared as follows: Firstly, a certain amount of sucrose was dissolved in 50 mL 50 vol% ethanol aqueous solution, and then 4 g melamine was added into above mixed solution and stirred in 80 °C water bath until it turns dry completely. Finally, the dry mixture was sintered at 600 °C for 2 h using a crucible. The mass ratios of sucrose: melamine was 0.5, 1, and 2.5 wt %, respectively.

3. Characterization.

X-ray diffraction (XRD) patterns were measured on a Rigaku D/max-2000 diffractometer with Cu K α radiation ($\lambda=0.15406$ nm) at a scan rate of $2\theta=4^\circ \text{ min}^{-1}$. X-ray photoelectron spectroscopy (XPS) spectra were obtained on a PHI5700ESCA instrument with an Al-K α (1486.6 eV) monochromatic X-ray radiation (operated at 200 W) from a twin anode in the constant analyzer energy mode with an energy of 30 eV. All binding energies were referenced to the C 1s peak (284.6 eV) arising from carbon impurities. Raman spectra were recorded on a micro-Raman spectrometer (Renishaw InVia) with an excitation source of 532 nm Ar⁺ laser at room temperature.

Elemental analysis (EA) was measured on a vario MICRO CUBE elemental. The microstructures of photocatalysts were obtained on a FEI, Tecnai G2 S-Twin TEM at an accelerating voltage of 300 kV. Nitrogen adsorption–desorption isotherms were recorded on an AUTOSORB-1-MP surface analyzer at 77 K. Nitrogen adsorption–desorption isotherms were recorded on an AUTOSORB-1-MP surface analyzer at 77 K. All the samples were degassed at 150 °C before nitrogen adsorption measurements. The BET surface area was determined using adsorption data in a relative pressure (P/P_0) range of 0.05–0.3, and the pore-size distributions calculated with the Barret–Joyner–Halender (BJH) method from the desorption branches of the nitrogen isotherms. Thermogravimetric-differential scanning calorimetry analysis (TG-DSC) was conducted on a SDT Q600 TG-DSC instrument with a heating rate of 10 °C min⁻¹. The UV–vis absorption spectra were carried out on a UH4150 spectrophotometer using BaSO₄ as the reflectance standard. The photoluminescence (PL) spectra were obtained on FluoroMax-4 Fluorescence spectrometer using 375 nm as the excitation wavelength at room temperature.

4. Photoelectrochemical Measurements.

Electrochemical measurements were carried out on a CHI 660 C electrochemical instrument in a standard three-electrode system. The Pt flake and Ag/AgCl (saturated KCl) act as counter electrode and reference electrode respectively, and using prepared electrodes as working electrodes. A 300 W Xe arc lamp with a UV-cut off filter ($\lambda > 400$ nm) used as light source with the electrolyte of 0.5 mol L⁻¹ Na₂SO₄ aqueous solution. Electrochemical impedance spectroscopy (EIS) were obtained at an AC

voltage magnitude of 5 mV with the frequency range of 10^5 to 10^{-1} Hz. Working electrodes were prepared as follows: FTO glass was cleaned with distilled water, acetone and ethanol by sonication for 20 min respectively. Then, 0.02 g of g-C₃N₄ or sucrose-mediated g-C₃N₄ was ground with 1 mL of terpineol to obtain slurry, and then the slurry was coated onto 1 cm×2 cm FTO glass electrode by drip coating. After drying at 80 °C for 12 h, the electrodes were sintered at 250 °C for 1 h to improve adhesion.

5. Photocatalytic H₂ evolution Reactions.

The photocatalytic H₂ evolution reactions were performed on a gas-tight circulation system with a side window. 0.1 g of photocatalyst was suspended in 300 mL 10 vol % triethanolamine (TEOA) aqueous solution with 3 wt % Pt loading under a 300 W Xe lamp with a cutoff filter ($\lambda > 400$ nm) irradiation. Prior to irradiation, suspensions were dispersed by sonication for 5 min and the reaction system was bubbling with N₂ for 25 min to remove O₂. The amount of H₂ was determined on an online Agilent 7890 gas chromatography (TCD), using Ar as the carrier gas.

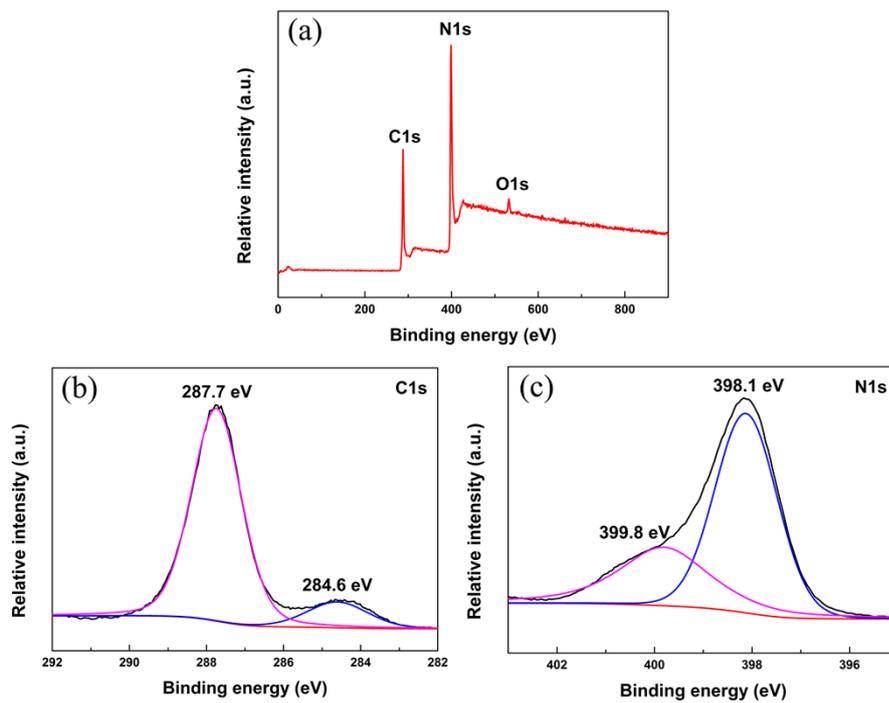


Fig. S1 (a) XPS survey spectra, high-resolution XPS spectra of (b) C 1s, and (c) N 1s for 1 wt% sucrose-mediated g-C₃N₄.

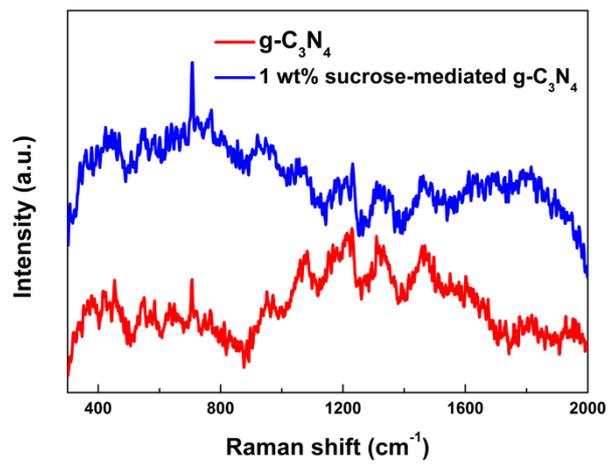


Fig. S2 Raman spectra of pure g-C₃N₄ and 1 wt% sucrose-mediated g-C₃N₄.

Table S1. Effects of sucrose content on the textural properties of the sucrose-mediated g-C₃N₄

catalyst	S _{BET} (m ² ·g ⁻¹)	pore volume (cm ³ g ⁻¹)	pore size (nm)
pure g-C ₃ N ₄	18.6	0.084	18.5
0.5 wt% sucrose-mediated g-C ₃ N ₄	76.8	0.268	17.7
1 wt% sucrose-mediated g-C ₃ N ₄	121	0.355	13.2
2.5 wt% sucrose-mediated g-C ₃ N ₄	128	0.428	17.5

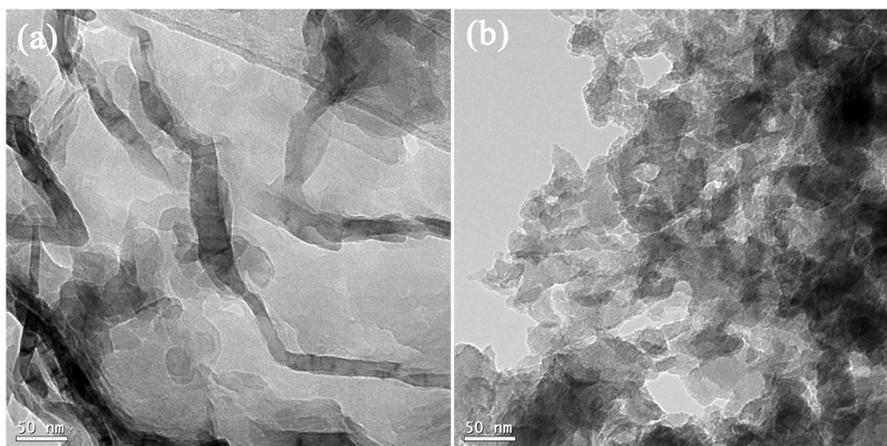


Fig. S3 TEM image of (a) pure g-C₃N₄ and (b) 1 wt% sucrose-mediated g-C₃N₄.

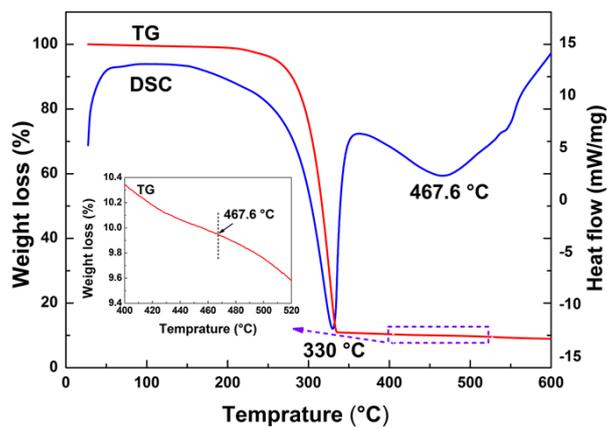


Fig. S4 TG-DSC thermograms for heating the mixture of 1 wt% sucrose and melamine.

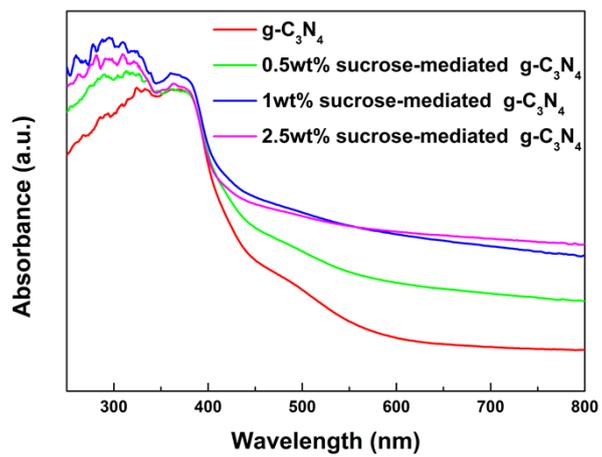


Fig. S5 UV-vis absorption spectra of pure g-C₃N₄ and sucrose-mediated g-C₃N₄ with different sucrose contents.

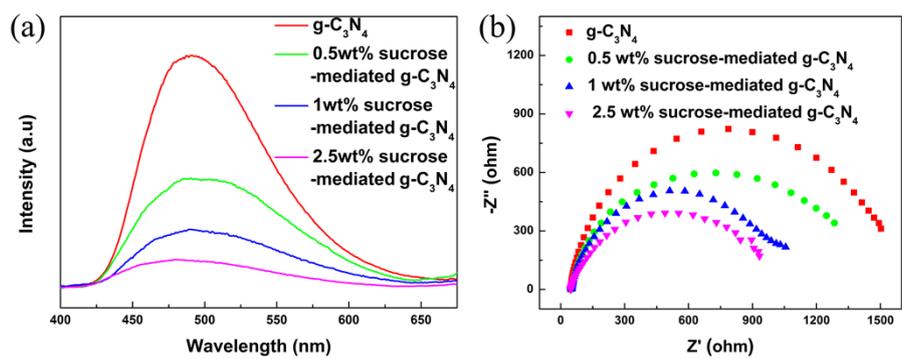


Fig. S6 (a) Photoluminescence spectra under 375 nm excitation at room temperature; (b) EIS Nyquist plots obtained at an AC voltage with amplitude of 5 mV over the frequency range of 10^5 to 10^{-1} Hz in $0.5 \text{ mol L}^{-1} \text{ Na}_2\text{SO}_4$ aqueous solution for pure $g-C_3N_4$ and sucrose-mediated $g-C_3N_4$ with different sucrose contents.

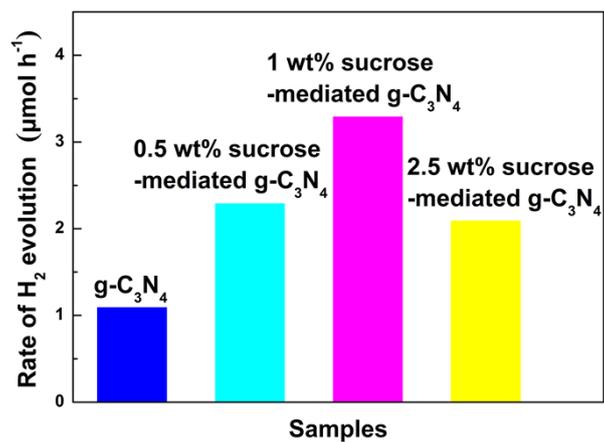


Fig. S7 Photocatalytic H₂ evolution rate of pure g-C₃N₄ and sucrose-mediated g-C₃N₄ with different sucrose contents in the absence of Pt in 10 vol% TEOA aqueous solution.