

Supplementary Material

Artificial photosynthesis of H₂O₂ over self-assembly two-dimensional g-C₃N₄ film

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Characterizations

Morphological analysis was conducted using field emission scanning electron microscopy (FESEM, FEI, QUANTA FEG 250). X-ray diffraction patterns were obtained using a Cu-K α radiation XRD diffractometer ($\lambda = 0.154056$ nm, Bruker, D8 Advance). Atomic force microscopy image analysis was performed with an AFM microscope (Bruker, Dimension ICON). UV-Vis diffuse reflectance spectra were measured at room temperature with BaSO₄ as the reference. X-ray photoelectron spectroscopy was conducted with using an Al K α XPS electron spectrometer ($h\nu = 1486.6$ eV, Thermo Fisher, Escalab xi+). Photoluminescence spectra analysis was conducted on a Hitachi F-4600 fluorescence spectrometer with a 350 nm excitation wavelength and a 150 W Xe lamp as the light source. The transient photocurrent response, Mott-Schottky curve and Electrochemical Impedance Spectroscopy were taken using an electrochemical system (CHI-660E, China) with a standard three-electrode system and Ag/AgCl electrode as the reference electrode in a 0.1 M Na₂SO₄ electrolyte solution and a 300 W Xe lamp as the light source. The EPR measurements were conducted on the Bruker EMXplus-6/1 model spectrometer in Germany. The time-resolved fluorescence decay spectra were recorded by the Edinburgh FLS1000 spectrometer, with a step size of 1nm and an integral time of 0.1s. The O₂-TPD measurements were carried out on the Micromeritics AutoChem 2950 HP in the United States. Electrochemical performance tests were performed at CHI 660e MSR with a rotating disk electrode unit (PINE) AFMSRCE.

Electrochemical measurement of oxygen reduction reaction. The number of transferred electrons (n) and H₂O₂ selectivity of the sample in the ORR reaction were determined through the rotating ring-disk electrode (RRDE). A three-electrode system was employed for electrochemical measurement, in which RRDE was served as a working electrode, a Hg/HgO as the reference electrode, and a Pt/C as the counter electrode. A 0.1M saturated KOH solution was utilized as the electrolyte. The RRDE speed was set to 1600 rpm and the potential range was set to 0 ~ 1.0V vs. RHE. N is the collection efficiency ($N=0.35$).

DMPO spin-trapping EPR measurement. With 5, 5-dimethyl-1-pyrroline N-oxide (DMPO) serving as a spin trap, the reactive $\cdot\text{O}_2^-$ species were detected under light irradiation. The $30\ \mu\text{L}$ catalyst (1g mL^{-1}) was dispersed in a solvent and mixed with certain amount of DMPO solution. Subsequently, a certain amount of the mixed liquid was absorbed into a capillary tube under light irradiation (5 min) for the detection of photogenerated $\cdot\text{O}_2^-$ species.

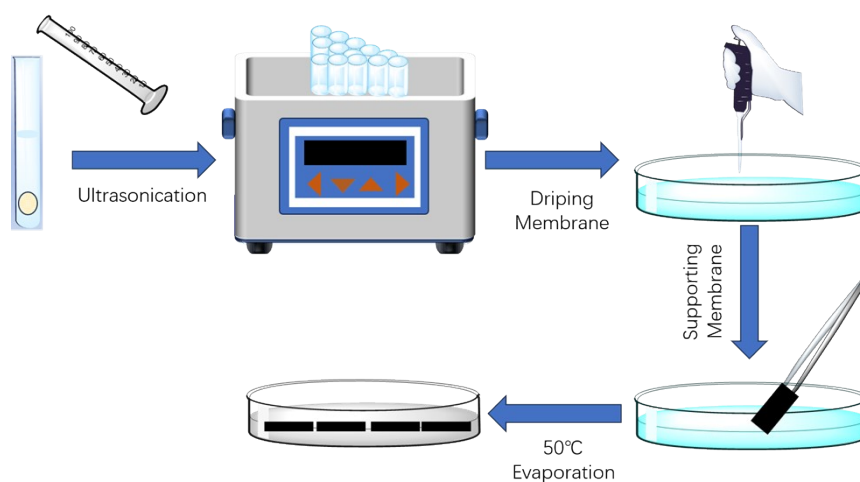


Figure S1 Schematic illustration for the preparation process of $\text{g-C}_3\text{N}_4$ film

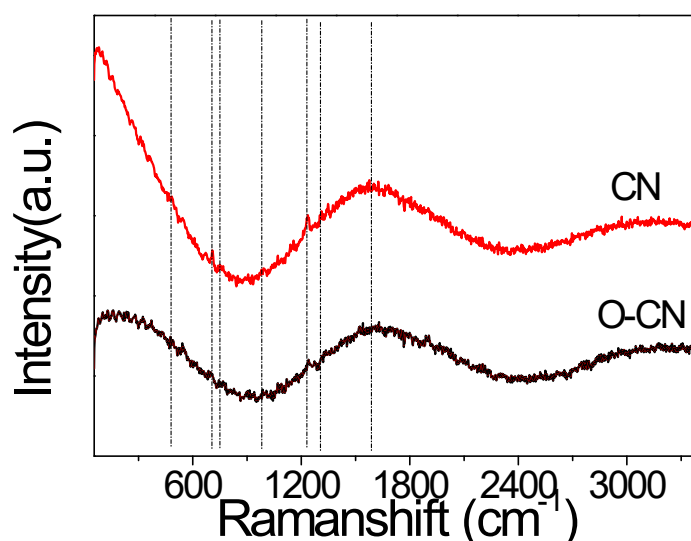


Figure S2 Raman spectra of $\text{g-C}_3\text{N}_4$ samples