

## **Ni<sub>2</sub>P-NiS 双助剂促进 g-C<sub>3</sub>N<sub>4</sub> 光催化产氢动力学**

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## **Kinetics Enhancement of Photocatalytic H<sub>2</sub> Evolution over Dual Cocatalyst Ni<sub>2</sub>P-NiS Decorated g-C<sub>3</sub>N<sub>4</sub> Heterojunctions**

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## Experimental

### Materials

Urea, nickel chloride hexahydrate ( $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ ), Thioacetamide ( $\text{C}_2\text{H}_5\text{NS}$ ), Sodium hydroxide ( $\text{NaOH}$ ) were received from by Shanghai Aladdin Biochemical Technology Co. Ltd. Triethanolamine (TEOA) was obtained from Fuyu Fine Chemical Co. Ltd.

### **Purification of commercial red phosphorus**

Typically, 3 g commercial red phosphorus (RP) was dispersed in 60 mL deionized water. The suspension was put into a 100 mL Teflon-lined stainless autoclave and maintained at 200 °C for 12 h. Then, the product was washed with water and ethanol several times. Subsequently, the sample was collected by centrifugation and dried at 60 °C.

### Material characterization

The elemental composition, chemical state and morphology of the samples were characterized by X-ray photoelectron spectroscopy (Kratos AXIS NOVA), SEM (JEOLJSM-6390 system, Scanning electron microscope with X-ray energy dispersive spectrometer, EDX) and TEM (Tecnai G2F20S-TWIN, Transmission electron microscope). UV/Vis spectrophotometry (Shimadzu UV-3600) was performed to analyze the photoresponse properties of the catalysts. The photoluminescence spectra were obtained on a Hitachi F-7000. Fourier transform infrared spectra (FT-IR) was examined by PerkinElmer Frontier. The specific surface area of the samples was detected by  $\text{N}_2$  adsorption-desorption (Quantachrome NOVA 2000e).

### Photochemical test

The photoelectric test of the samples was evaluated on an electrochemical workstation (CHI660E, ChenHua in Shanghai) with a standard three-electrode system in  $0.5 \text{ mol} \cdot \text{L}^{-1} \text{ Na}_2\text{SO}_4$  electrolyte. Among them, a platinum wire and a standard calomel electrode (SCE) are used as the counter electrode and the reference electrode, respectively. The working electrode is prepared as follows: 3 mg of samples was dispersed in 2 mL of deionized water, 1 mL of ethyl alcohol and 30  $\mu\text{L}$  naphthol, after ultrasonic treatment 2 h. 1 mL of homogeneous solution was dropped on  $2 \text{ cm} \times 3 \text{ cm}$  FTO glass and dried at room temperature naturally. The transient photocurrent response ( $I-t$  curve, at  $-0.5 \text{ V}$  potential vs. SCE), electrochemical impedance analysis (EIS, frequency ranging from  $10^{-2}$  to  $10^5 \text{ Hz}$ ), MS (Mott-Schottky curves, ranging from  $-0.6 \text{ V}$  to  $0.8 \text{ V}$  vs. SCE) and LSV curve analysis (linear sweep voltammetry curves, ranging from  $-2.0$  to  $2 \text{ V}$  vs. SCE) were measured in this work.

### Photocatalytic activity test

The activity of the samples was carried out in the on-line photocatalytic analysis system using Microsolar 300 W Xe-lamp as the light sources (Labsolar-III AG, Beijing Perfectlight Technology Co. Ltd., China). 10 mg catalyst was added into 100 mL mixture containing 80 mL deionized water and 20 mL of TEOA (20 vol%) as the sacrificial agent, then the suspension was placed into a reactor followed by magnetic stirring. The reaction was performed for several hours under vacuum condition and light irradiation (Full spectrum). The gas is injected into the GC7900 gas chromatograph (TCD channel) through a six-way valve every hour.