

# Self-Powered Photoelectrochemical Assay for Hg<sup>2+</sup> Detection Based on g-C<sub>3</sub>N<sub>4</sub>-CdS-CuO Composites and Redox Cycle Signal Amplification Strategy

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## 1. Synthesis of the g-C<sub>3</sub>N<sub>4</sub> and CdS

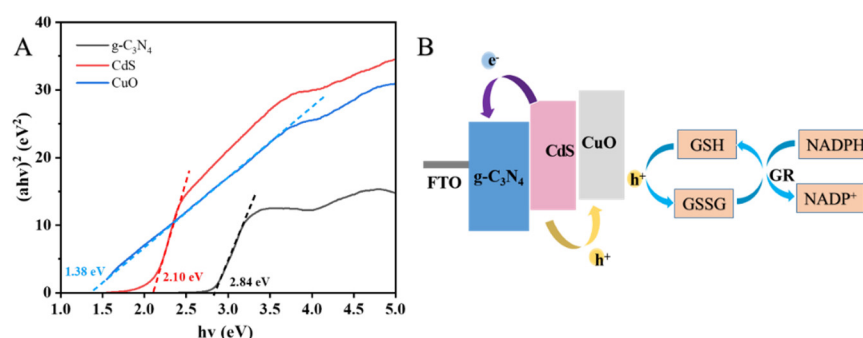
The water-dispersing g-C<sub>3</sub>N<sub>4</sub> was prepared according to the literature [1] as follows: 5 g of melamine in a crucible was annealed at 550 °C for 4 h. Then, 1 g of the as-prepared bulk-C<sub>3</sub>N<sub>4</sub> powder was refluxed in 100 mL of 5 M HNO<sub>3</sub> at 125 °C for 12 h and kept overnight. The formed light yellow precipitates were isolated by centrifugation and washed with water several times. Afterward, the final product was dried at 60 °C for further use.

Preparation of CdS QDs [2], firstly, 0.1623 g CdCl<sub>2</sub>·2.5H<sub>2</sub>O was mixed with 30 mL of ultrapure water and heated to 70 °C. Next, a freshly prepared Na<sub>2</sub>S (30 mL, 0.0627 g/mL) solution was slowly injected into the mixture solution and kept at 70 °C for 3 h with continuous reflux. The color of the reaction solution immediately turned orange-yellow. The collected precipitation was centrifuged and washed twice with absolute ethanol and ultrapure water, respectively. Finally, the CdS QDs were re-dispersed in ultrapure water and stored at 4 °C in the dark for further use.

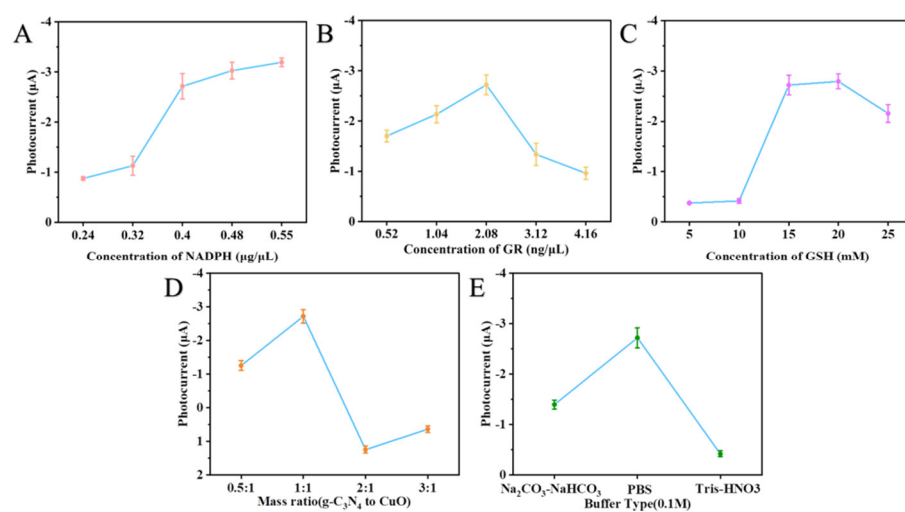
## 2. Setting parameters of the EIS and Cyclic Voltammetry(CV)

The electrochemical workstation was used to record the EIS, and the parameter was set for Init E (V) = 0.315, High Frequency (Hz) = 1 MHz, Low Frequency (Hz) = 0.1, Amplitude (V) = 0.005, Quiet Time (sec) = 2, Cycles (.1-1Hz) = 1.

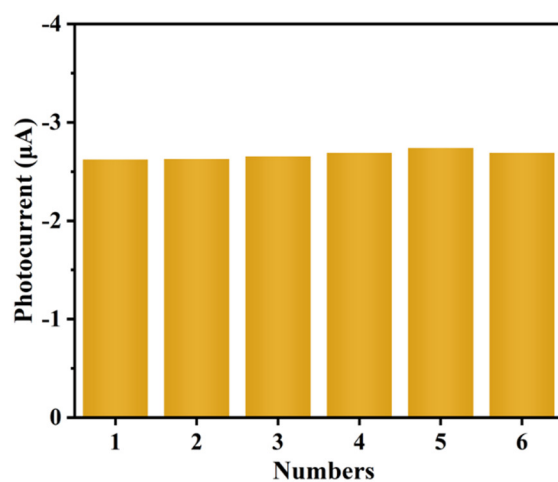
CV uses an electrochemical workstation, Set the parameters to Init E (V) = -0.2, High E (V) = 1, Low E (V) = -0.2, Init P/N = P, Scan Rate (V/s) = 0.02, Segment = 2, Sample Interval (V) = 0.001, Quiet Time (SEC) = 2, Sensitivity (A/V) = 0.001.



**Figure S1.** The band gaps of g-C<sub>3</sub>N<sub>4</sub>, CdS and CuO were measured by the UV-vis diffuse reflection spectra (DRS) (A), the picture shows the direction of electrons in the photoelectric sensor under light conditions (B).



**Figure S2.** (A) Concentration of NADPH; (B) Concentration of GR; (C) Concentration of GSH; (D) Mass ratio of g-C<sub>3</sub>N<sub>4</sub> to CuO; (E) Different types of buffer solutions (0.1M).



**Figure S3.** Reproducibility of g-C<sub>3</sub>N<sub>4</sub>-CdS-CuO at 0.5 nM Hg<sup>2+</sup>.

**Table S1.** Comparison of other analysis strategies for Hg<sup>2+</sup> detection.

Methods	Materials	Linear Ranges	LODs	Refs
Fluorescence	CDs-AgNPs	100–160 μM	22.2 nM	[3]
Colorimetric	Hg-MB	0–400 μM	19.5 nM	[4]
Electrochemical	Cu-MOF	0.1–50 nM	0.063 nM	[5]
Electrochemical	S-rGO	0.125–6 μM	1.6 nM	[6]
Electrochemical	N,S-doped C@Pd	0.00054–5.0 μM	0.18 nM	[7]
Photoelectrochemical	g-C <sub>3</sub> N <sub>4</sub> @CdS	20–550 nM	12 nM	[8]
Photoelectrochemical	Bi <sub>2</sub> MoO <sub>6</sub> /CuS-6%	0.5–230 nM	0.23 nM	[9]
Photoelectrochemical	g-C <sub>3</sub> N <sub>4</sub> -CdS-CuO	0.005–100 nM	0.84 pM	this work

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