

Supporting Information for

**Preparation, characterization of 0D Au NPs@3D BiOI  
nanoflowers/2D NiO nanosheet arrays heterostructure and  
application for a self-powered photoelectrochemical biosensing  
platform**

Qingzhi Han, Hanyu Wang, Yanting Qi, Dan wu\*, Qin wei

Key Laboratory of Interfacial Reaction & Sensing Analysis in Universities of

Shandong, University of Jinan, Jinan 250022, PR China

\*Corresponding author:

Email: wudan791108@163.com (D. Wu)

Tel: + 86-531-82767872

Chemicals and apparatus; synthesis of NiO, BiOI/NiO, and Au@BiOI/NiO; SEM of excess BiOI CBD repeating cycles; simulation parameters of the equivalent circuit components; comparison of various methods for the determination glucose; determination of glucose in diluted glucose injections have been supplied in this Supporting Information.

### **. Reagents and apparatus**

Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O was obtained from Guangcheng chemical reagent factory. Potassium persulfate and H<sub>2</sub>AuCl<sub>4</sub>·4H<sub>2</sub>O were purchased from Sinopharm Chemical Reagent Co., Ltd (Shanghai, China). Aqueous ammonia (25-28%) was obtained from Tian In Fuyu Fine Chemical Co., Ltd (Tianjin, China). Bi(NO<sub>3</sub>)<sub>3</sub>·5H<sub>2</sub>O was purchased from Macklin Biochemical Co., Ltd (Shanghai, China). Ethylene glycol was obtained from Tianjin Damao chemical reagent factory. Poly-(diallyldimethylammonium chloride) (PDDA) was purchased from Aladdin Industrial Corporation (Shanghai, China). The Indium-Tin Oxides (ITO) was purchased from China Southern Glass Holding Co., Ltd (Shenzhen, China). The glucose injections were obtained from local hospital. All the reactants were analytically pure, and used as received.

X-ray photoelectron spectroscopy (XPS) analysis was performed on Thermo ESCALAB 250XI spectrometer (Thermo Fisher Scientific, USA). X-ray power diffraction (XRD) was obtained using a D8 advance X-ray diffractometer (Bruker AXS, Germany) with CuK $\alpha$  radiation ( $\lambda = 1.5406 \text{ \AA}$ ), in the range of 10 - 80° (2 $\theta$ ). The scanning electron microscope (SEM) and energy dispersive spectrometer (EDS) images were obtained from a field emission SEM (FEI Quanta 250, USA). The high resolution transmission electron microscopy (HRTEM) was performed with a JEM-2100 electron microscope (JEOL, Japan). Diffuse reflectance ultraviolet-visible light spectra were measured on a UV-vis spectrophotometer (TU-1901, Beijing Purkinje General Instrument Co Ltd.), in the range of 200 ~ 800 nm. The PEC measurement was performed on an electrochemical workstation (IVIUM Vertex One, Netherlands). The electrochemical impedance spectroscopy (EIS) was measured on a Zahner Zennium electrochemical workstation (PP211, Germany) with a three electrodes

system: a modified ITO electrode with an active area of 1 cm<sup>2</sup> (A square of one centimeter on each side) as the working electrode, a Pt wire as the counter electrode, and a saturated calomel electrode (SCE) as the reference electrode. A 420 ~ 430 nm purple-light (PEC-10 W, Tianjin Brillante Technology Limited, China) was used as the light source (optical output power: 5400 mW). All the photocurrent measurements were performed at a bias voltage of 0 V (versus SCE). The Tris-HCl solution (0.1 M, pH = 7.0) was used as the electrolyte for photocurrent measurements. Ultrapure water (UW) (18.25 MΩ·cm<sup>-2</sup>) was obtained from Youpu water purification system (UPR-11-10T, Chengdu Youpu Equipment Co Ltd., China) and was used throughout the experiment.

### **Synthesis of 2D NiO nanosheets (NSs) arrays**

Before use, the ITO glasses (2.0 × 4.5 cm<sup>2</sup>) were cleaned by sonication in a detergent aqueous solution, acetone, ultrapure water (UW), and ethanol for 20 min each. To grow a single-crystalline Ni(OH)<sub>2</sub> platelet nanoarrays, the precursor solution was prepared by dissolving 1.236 g Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O and 0.2 g potassium persulfate in 35 mL UW, followed by addition of 5 mL condensed aqueous ammonia. After stirring for 15 min, the mixture solution was transferred into a Teflon-lined steel autoclave (50 mL). A piece of indium-tin oxides (ITO) was placed at an inclined angle with the conductive surface facing down, and kept at 150 °C for 600 min. This process led to the growth of a green Ni(OH)<sub>2</sub> array film on the ITO side. The film then was annealed at 400 °C for 120 min under argon atmosphere, giving a dark NiO film.

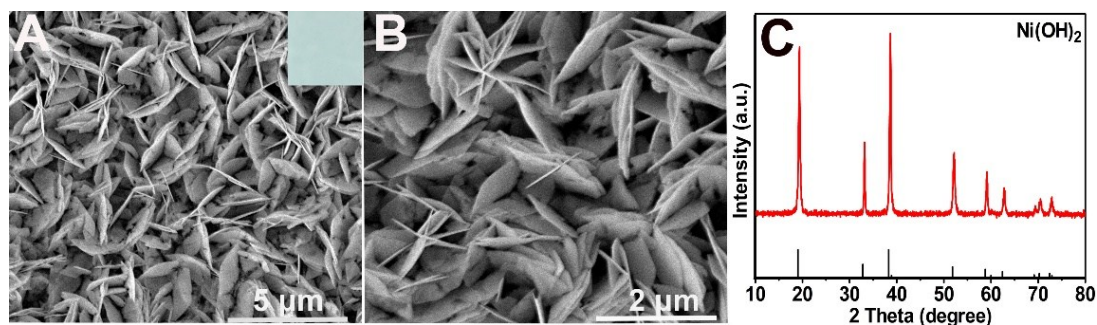
### **Preparation of three-dimensional BiOI nanoflowers (NFs)/two-dimensional NiO (NSs) arrays**

The ITO/NiO glass (1.0 × 2 cm<sup>2</sup>) with an effective area (NiO array) of 1.0 cm<sup>2</sup> modified with BiOI by a previously reported chemical bath deposition (CBD) process. In detail, the ITO/NiO was dipped in Bi(OCH<sub>2</sub>CH<sub>2</sub>OH)I solution for 120 s, rinsed with ethanol absolute, dried in room temperature (natural drying), and then putted into UW for 60 s, rinsing and drying as well. The twostep forms one cycle, and the incorporated amount of BiOI NFs could be adjusted by different repeating cycles. The whole process was conducted in the room temperature of 24 °C.

### Preparation of zero-dimensional Au NPs@three-dimensional BiOI NFs/two-dimensional NiO NSs hybrid nanostructure

**Preparation of Au nanoparticles (Au NPs).** Au nanoparticles were prepared according to a previously reported method. Briefly, an aqueous solution of  $\text{HAuCl}_4 \cdot 4\text{H}_2\text{O}$  (1 mL, 1wt%) was diluted to 100 mL, under vigorous stirring the diluted solution was heated to boiling. Subsequently, a sodium citrate aqueous solution (2.5 mL, 1 wt%) was added to the above solution. Then the solution was kept boiling for 5 mins, following a claret-red suspension of Au was obtained. After the solution cooling down to room temperature, a PVP aqueous solution (0.40 mL, 25.5 mg/mL in  $\text{H}_2\text{O}$ ) was added to the as-prepared Au solution (20 mL) under continuous stirring, and the mixture was further stirred for 1 day at room temperature. Then, a deep-red PVP-stabilized Au solution was obtained. The Au/BiOI/NiO nanoplates arrays were prepared according to a previously report.<sup>1</sup> The ITO substrate with the BiOI/NiO was immersed into the above prepared Au solution for 300s, and then, the substrate was taken out of the solution and dried in a hot panel at 50 °C for 5 min. This whole process (immersion, withdrawing and drying) was repeated for 4 times. To make the Au NPs to stick onto the BiOI/NiO surface tightly, the obtained Au@BiOI/NiO NSs arrays were placed in a sealed container purged with  $\text{N}_2$  for 15 min, and then the container was slowly heated to 85 °C for 30 min.

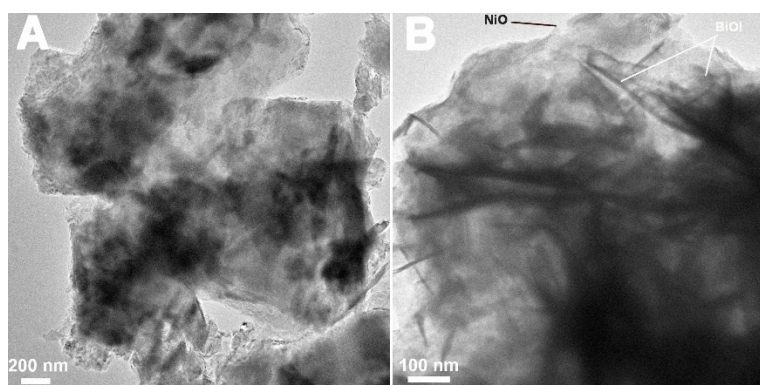
**Figure S1.** (A) SEM image of  $\text{Ni}(\text{OH})_2$  NSAs. Inset shows the  $\text{Ni}(\text{OH})_2$  coated ITO. (B) High-magnification SEM image of  $\text{Ni}(\text{OH})_2$  NSAs. (C) XRD pattern of the as-fabricated  $\text{Ni}(\text{OH})_2$  NSAs.



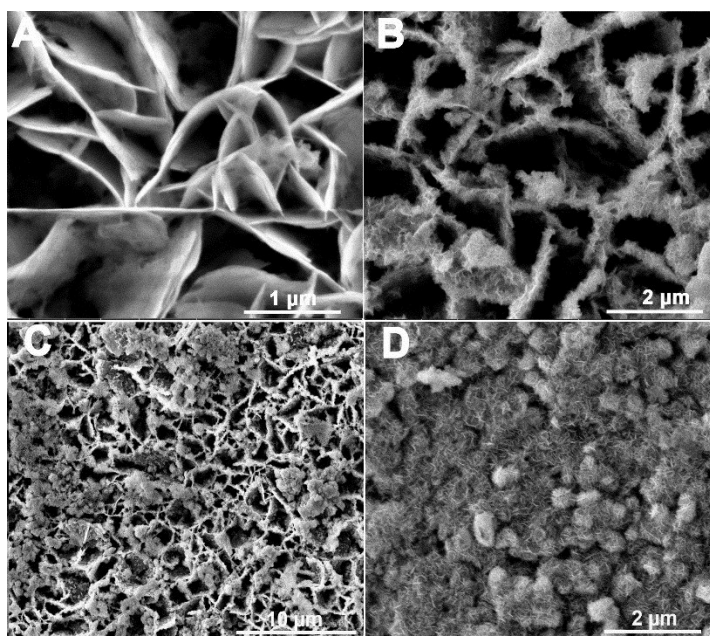
The scanning electron microscopic (SEM) images of  $\text{Ni}(\text{OH})_2$  with different

magnification are displayed in Figure S1A and S1B. It can be seen that the  $\text{Ni}(\text{OH})_2$  NSAs were grown vertically and uniformly on the indium-tin oxide (ITO) glass with an average length about 1.8  $\mu\text{m}$ . Figure S1C showed the XRD pattern of  $\text{Ni}(\text{OH})_2$ , which was corresponding to theophrastrite (JCPDS PDF#14-0117), indicating the successful synthesis of  $\text{Ni}(\text{OH})_2$ .

**Figure S2.** TEM of (A) NiO and (B) BiOI/NiO



**Figure S3.** SEM of BiOI CBD repeating cycles of (A) 1, (B) 4, (C) 5 and (D) 8



**Table S1.** Simulation parameters of the equivalent circuit components

Electrode	$R_s (\Omega)$	$R_{et} (\Omega)$	$C_{dl} (F)$	$Z_w$
NiO/ITO	43.57	4.26	$1.74 \times 10^{-5}$	0.0181
BiOI/NiO/ITO	51.65	12.29	$8.255 \times 10^{-6}$	0.01655
Au@BiOI/NiO/ITO	50.54	9.30	$6.542 \times 10^{-6}$	0.01823
GOD-Au@BiOI/NiO/ITO	49.32	20.60	$9.146 \times 10^{-6}$	0.009651

**Table S2.** Comparison of various methods for the determination glucose

Method	Linear range	LOD	Ref
PEC	$1.0 \times 10^{-6} \sim 1.0 \times 10^{-2} \text{ M}$	$3.0 \times 10^{-7} \text{ M}$	2
Electrochemical	$2.0 \times 10^{-7} \sim 0.4 \text{ M}$	$1.7 \times 10^{-7} \text{ M}$	3
PEC	$5.0 \times 10^{-6} \sim 1.0 \times 10^{-2} \text{ M}$	$1.6 \times 10^{-6} \text{ M}$	4
Fluorescent	$0 \sim 6.0 \times 10^{-5} \text{ M}$	$3.0 \times 10^{-7} \text{ M}$	5
Chemiluminescence	$1.0 \times 10^{-6} \sim 1 \times 10^{-3} \text{ M}$	$4.0 \times 10^{-7} \text{ M}$	6
Electrochemical	$1.0 \times 10^{-3} \sim 8.0 \times 10^{-3} \text{ M}$	$2.37 \times 10^{-4} \text{ M}$	7
Colorimetric	$0 \sim 1.0 \times 10^{-2} \text{ M}$	$5.0 \times 10^{-6} \text{ M}$	8
PEC	$1 \times 10^{-7} \sim 5.0 \times 10^{-2} \text{ M}$	$8.71 \times 10^{-8} \text{ M}$	This work

**Table S3.** Determination of glucose in diluted glucose injections

Sample	Added (mM)	Found (mM)	RSD, n = 5 (%)	Recovery (%)
1	0.0	1.32	4.6	96.0
	2.0	3.24		
2	0.0	3.22	4.1	100.2
	6.0	9.23		
3	0.0	5.31	4.6	103.9
	15	20.89		

## References

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