

## Supporting Information

# Electrochemical Synthesis of $\text{CoFe}_2\text{O}_4$ Porous Nanosheets for Visible Light Driven Photoelectrochemical Applications

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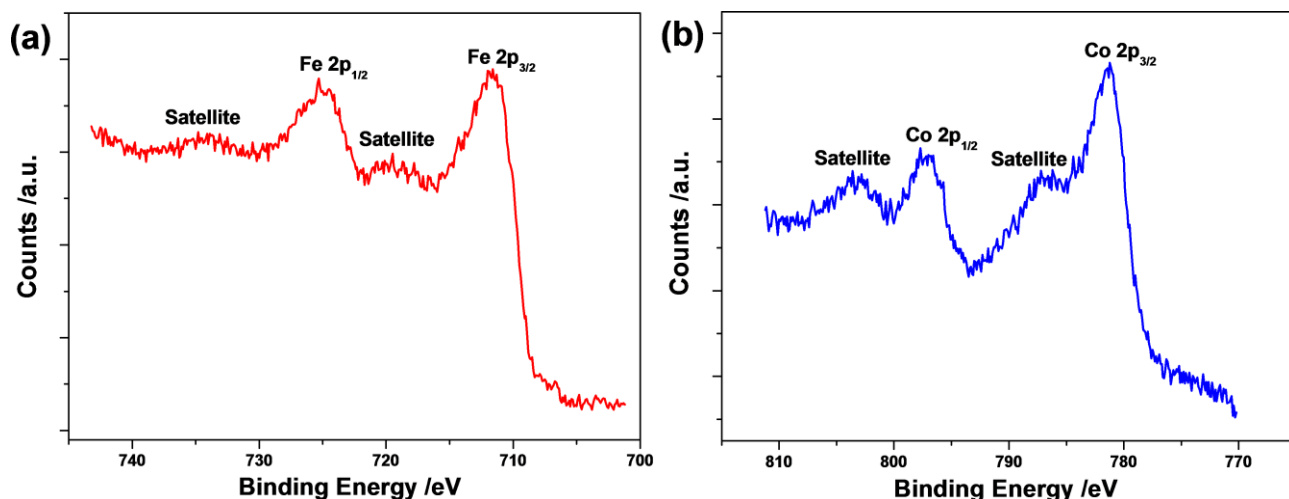
### Experimental Section

**Synthesis of  $\text{CoFe}_2\text{O}_4$  nanostructures:** All chemical reagents were of analytical grade and were used directly without any purification. Electrodeposition was carried out in a simple three-electrode cell. A saturated calomel electrode (SCE) electrode was used as reference and a graphite electrode was used as counter electrode, respectively. The working electrode was the FTO glass with a sheet resistance of  $14 \Omega/\square$ . The FTO glass was cleaned ultrasonically in distilled water, ethanol and finally rinsed in distilled water again before electrodeposition. The cathodic electrodeposition of CFOPNSs was performed at a constant current density of  $4 \text{ mA cm}^{-2}$  for 120 min at  $40 \text{ }^\circ\text{C}$  in aqueous solution of  $0.01 \text{ M Co(NO}_3)_2 + 0.02 \text{ M Fe(NO}_3)_3 + 0.1 \text{ M urea}$ . The urea is employed as electrochemical additive in the electrodeposition.<sup>1</sup> The as-deposited films were further annealed at  $600 \text{ }^\circ\text{C}$  for 10 h in air after heating at a rate of  $2 \text{ }^\circ\text{C/min}$ .

**Characterizations:** The phase of the samples were characterized by X-ray diffractometry (Bruker, D8 ADVANCE) with  $\text{Cu K}\alpha$  ( $\lambda = 1.541 \text{ \AA}$ ) radiation. The morphologies of the samples were analyzed by

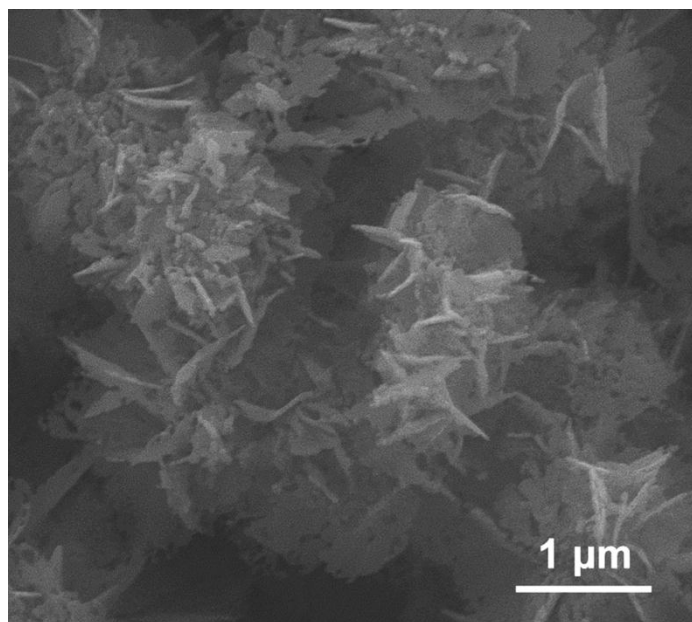
SEM (Quanta 400). The structure of the samples was investigated by using TEM (JEOL JEM2010-HR, accelerating voltage: 200 kV). For the TEM measurement, the prepared films were scratched and ground into powders. The scratched specimens were dispersed into ethanol via ultrasound, and then were collected with carbon copper grids. Raman spectroscopy were measured with a laser micro-Raman spectrometer (Renishaw inVia) using a visible laser ( $\lambda = 514.5$  nm) at room temperature. The XPS analysis was carried out in an ESCA Lab 250 (Thermo VG) with a 200 W Al  $K\alpha$  radiation. The pass energy constant was 20 eV for high resolution scans, and the analysis chamber pressure was about  $7.5 \times 10^{-6}$  Pa. The binding energies were calibrated by placing the C1s peak at 284.8 eV from adventitious carbon. Nitrogen adsorption–desorption isotherms at 77 K were performed on an ASAP 2020M instrument. All samples were outgassed for 300 min at 100 °C under flowing nitrogen before measurements. The specific surface area calculated by the BET method. The poresize distributions were calculated by Barrett-Joyner-Halenda (BJH) method. The optical absorption properties of the samples were measured with a UV-vis-NIR spectrophotometer (Varian Cary 5000). The prepared samples were mechanically exfoliated from the substrate and ground into powders for the optical absorption measurement.

**Photoelectrochemical experiments:** The photoelectrochemical experiments were performed on a CHI 660d electrochemical workstation (CHI Instruments, Shanghai). A three-electrode system in a quartz cell was used in this experiment to facilitate illumination of the photoelectrode. A Pt foil and an Ag/AgCl electrode were used as the counter and reference electrodes, respectively. The photoanode consisting of the  $\text{CoFe}_2\text{O}_4$  film with illuminated area  $0.16 \text{ cm}^2$  was immersed in 0.1 M aqueous  $\text{Na}_2\text{S}$  solution. The illumination source was a 500W Xe arc lamp with a UV filter (absorb  $\lambda < 390$  nm), which was used to cut off the UV energy. The intensity of the light at the sample position was determined to be  $30 \text{ mW/cm}^2$ .

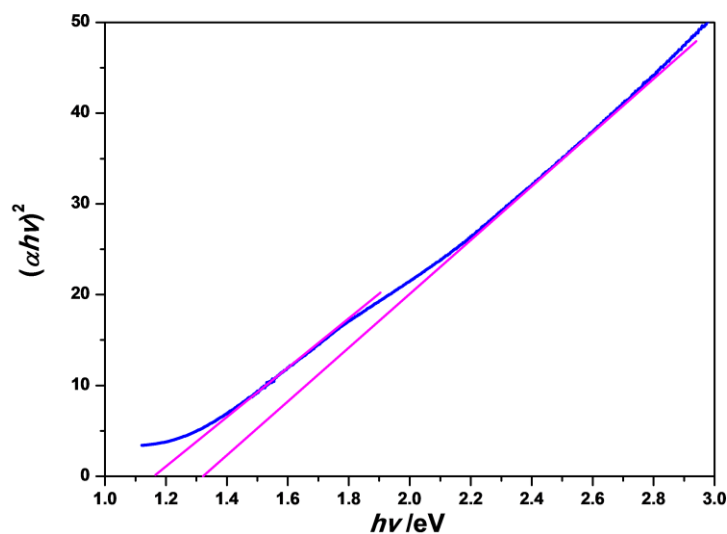


**Fig. S1.** Fe 2p and Co 2p XPS spectra of the as-deposited samples before annealing.

Fig. S1 shows the typical XPS spectrum of the as-deposited samples before annealing. The Fe 2p<sub>3/2</sub> peak shown in Fig. S1a located at 711.3 eV is assigned to Fe<sup>3+</sup> in FeO(OH),<sup>2</sup> which is consistent with the XRD result. The Fe 2p<sub>1/2</sub> peak occurs at 724.9 eV and the energy separation between the two Fe 2p<sub>3/2</sub> and Fe 2p<sub>1/2</sub> peaks is 13.6 eV. The Co 2p<sub>3/2</sub> peak in the XPS spectrum of the as-deposited samples is shown in Fig. S1b. The Co 2p<sub>3/2</sub> peak centered at 781.1 eV corresponds to Co<sup>2+</sup> in Co(OH)<sub>2</sub>.<sup>3</sup> The energy separation between Co 2p<sub>3/2</sub> and Co 2p<sub>1/2</sub> peaks is 15.7 eV. Thus, the XPS result confirms that the as-deposited products are consisted of FeO(OH) and Co(OH)<sub>2</sub>.



**Fig. S2.** (a) SEM image of the as-deposited samples before annealing.



**Fig. S3**  $(\alpha hv)^2$  vs  $hv$  curves for the CFOPNSs.

The  $E_g$  of the CFOPNSs can be calculated by using the equation of  $(\alpha hv)^2 = A (hv - E_g)$ , where  $hv$  is the photon energy,  $A$  is a material related constant and  $\alpha$  is the absorption coefficient. Fig. S3 shows two optical transitions at 1.16 and 1.32 eV. The larger optical transition at 1.32 eV is assigned to the  $E_g$  transition of valence to conduction band excitation.

## References

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