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# Hollow $NiFe_2O_4$ hexagonal biyramids for high-performance n-propanol sensing at low temperature

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**Text S1**: Experimental Details.

Materials.

Preparation of hollow $NiFe_2O_4$ hexagonal biyramids.

Materials Characterization.

Sensor Fabrication and Gas Sensing Measurement.

**Text 1: Experimental Details**

**Materials**.

All starting chemical reagents, including $Ni(NO_3)_2 \cdot 6H_2O$, $Fe(NO_3)_3 \cdot 9H_2O$, fumaric acid, polyvinyl pyrrolidone (PVP, K30), dimethylacetamide (DMAc) and EtOH in this experiment were analytical grade and were used without further purification.

**Preparation of hollow $NiFe_2O_4$ hexagonal biyramids**.

In a typical procedure of Fe and Ni containing bimetallic MOF precursors, $Ni(NO_3)_2 \cdot 6H_2O$ (72.7 mg), $Fe(NO_3)_3 \cdot 9H_2O$ (176.6 mg), fumaric acid (928.5 mg) and PVP (600 mg) were added into DMAc (10 mL) under ultrasonication to obtain a homogenous solution. Then the resulting solution was transferred to a 15 mL Teflon-lined stainless-steel autoclave and reacted at 100 °C for 8 h. After it was cooled to room temperature, the products were collected by centrifugation and further washed with DMAc and ethanol for several times, and then dried at room temperature for 12 h in air.

The as-synthesized bimetallic MOF precursors were placed in a muffle furnace and then annealed in air at 450 °C for 2 h with a heating rate of 1 °C·min$^{-1}$. The final products of hollow $NiFe_2O_4$ hexagonal biyramids were taken out after cooling to room temperature.

**Materials Characterization**.

The powder X-ray diffraction (PXRD) patterns were performed on a SHIMADZU XRD-7000S diffractometer equipped with Cu K$\alpha_1$ radiation ($\lambda$ = 1.5406 Å) at a scanning speed of 5°·min$^{-1}$ to investigate the crystallographic phases of the as-prepared samples. The morphologies and microstructures were observed using field-emission scanning electron microscope (FEI Nova NanoSEM 450) with an accelerating voltage of 15.00 kV, transmission electron microscopy (TEM) operating on FEI Tecnai-G$^2$F30 transmission electron microscope at 200 kV. And the energy dispersive X-ray spectrometry (EDS) was obtained by attachment on the TEM microscope. X-ray Photoelectron Spectroscopy (XPS) was performed using a

ThermoFisher ESCALAB$^{TM}$ 250Xi equipment with Al K$\alpha$ X-ray radiation as the X-ray source for excitation. The specific surface area was estimated by the Brunauer−Emmett−Teller (BET) equation based on the nitrogen adsorption isotherm at 77 K, which was measured with a Micromeritics ASAP2020 surface area analyzer after prior degassing of the product under vacuum at 200 °C overnight.

**Sensor Fabrication and Gas Sensing Measurement**.

The detailed fabrication procedure of sensor devices could be described as follows: First, an appropriate amount of as-synthesized sensing material was mixed with deionized water to form homogeneous slurry, and was subsequently coated on a ceramic tube (about 4 mm in length, 1.2 mm in external diameter, and 0.8 mm in internal diameter) by using a small brush. The ceramic tube is equipped with two Au electrodes and four Pt wires on both end of the tube. After dried in air at room temperature, a Ni-Cr alloy coil was inserted into the ceramic tube as heater to control the working temperature by adjusting the heating current. The ceramic tube with sensing material and heater was installed on the socket to the completed sensor device, which was then put in a closed chamber. A given amount of the target sensing gas was introduced into the testing chamber using microsyringe. Gas sensing measurements by a static process were carried out on a commercial CGS-8 Intelligent Gas Sensing Analysis System (Beijing Elite Tech Co., Ltd, China), using air as the dilution and reference gas. The gas response ($S$) is defined as the ratio between $R_a$ and $R_g$ ($S = R_a/R_g$), where $R_a$ and $R_g$ are the resistances when the sensor device was exposed in air and test gas atmosphere, respectively.

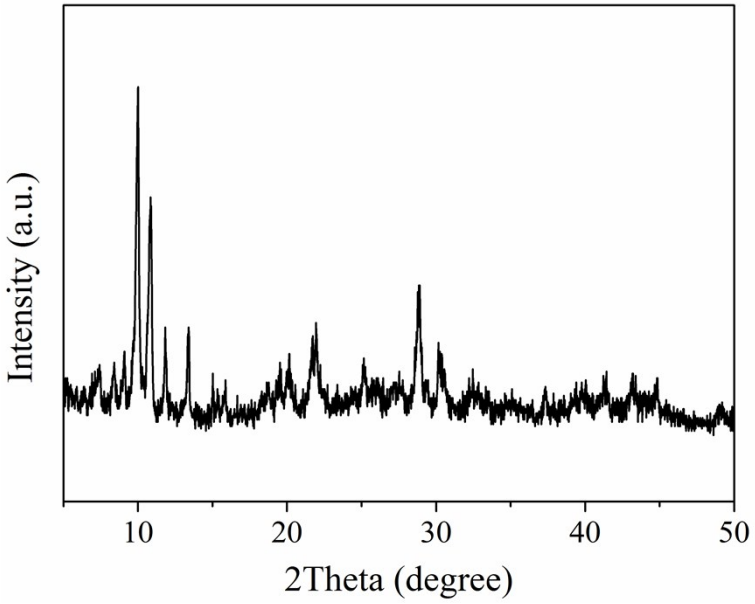


Fig S1. Ni/Fe-fumaric acid bimetallic MOF precursors.

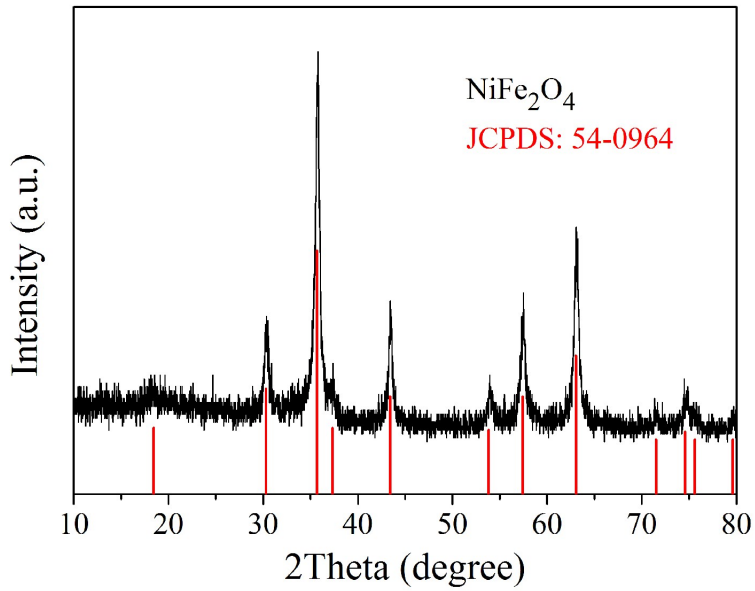


Fig S2. Powder XRD pattern of hollow $NiFe_2O_4$ hexagonal biyramids.

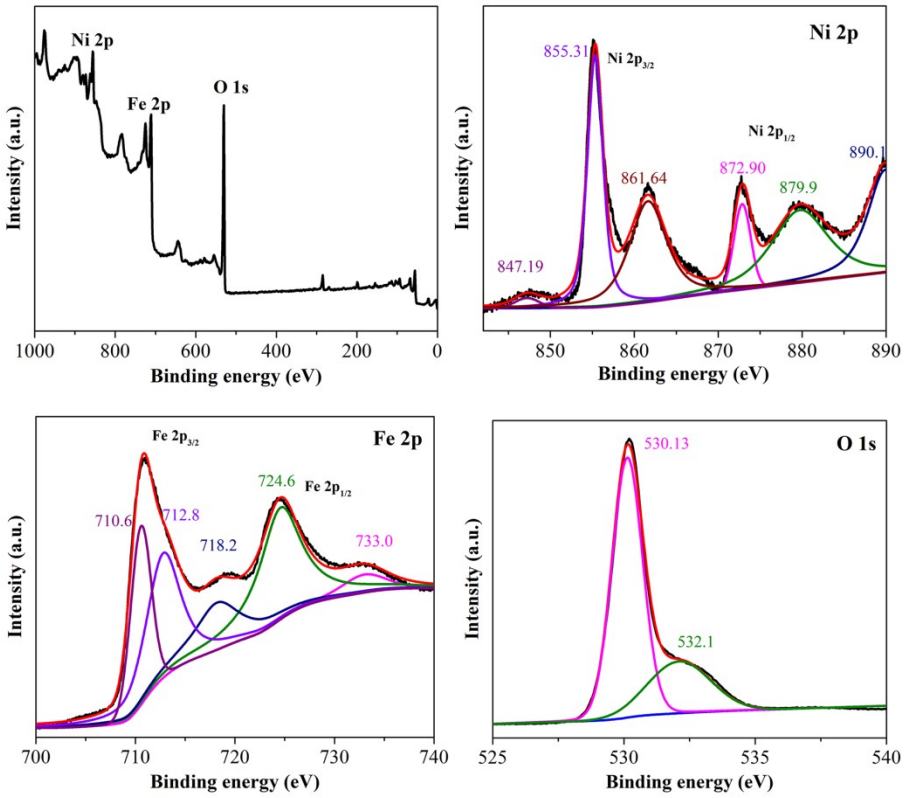


Fig. S3. XPS spectra and fitted data of hollow $NiFe_2O_4$ hexagonal biyramids: survey spectrum, and Ni 2p, Fe 2p, O 1s.

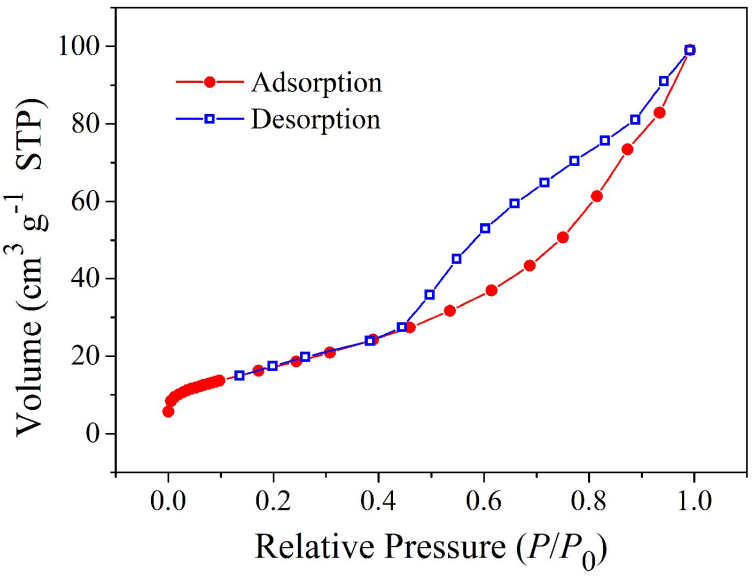


Fig S4. Nitrogen adsorption−desorption isotherm of hollow $NiFe_2O_4$ hexagonal biyramids.

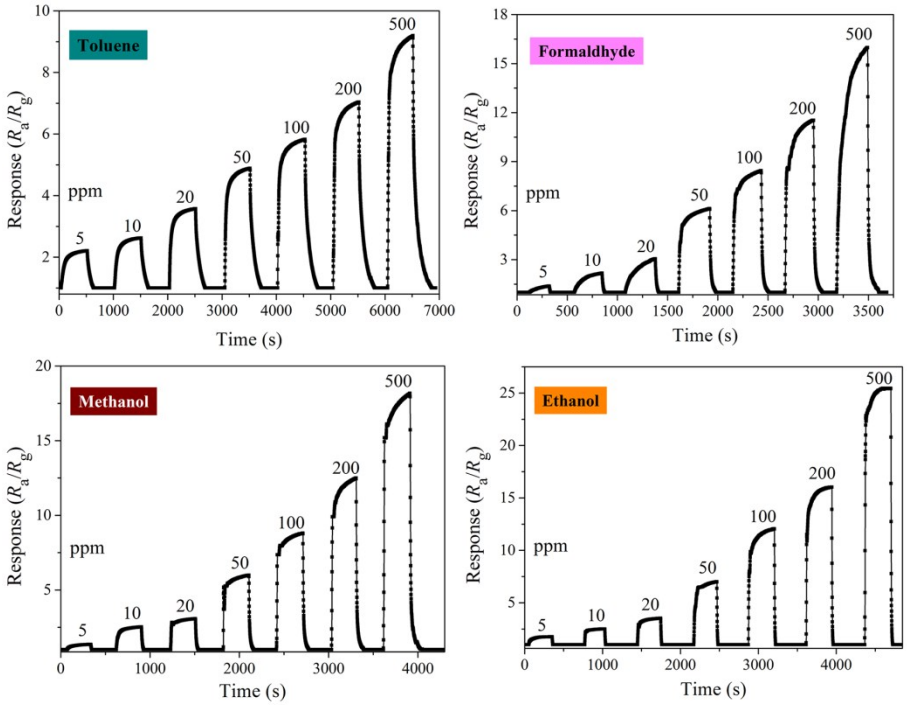


Fig. S5. The dynamic response-recovery transients of the sensor to different gases at different concentrations.

**Table S1**. Comparisons of n-Propanol sensing performances of various gas sensors.

| Material and Morphology | Working Temperature (°C) | n-propanol concentration (ppm) | Response | Ref. |
|---|---|---|---|---|
| hollow $NiFe_2O_4$ hexagonal biyramids | 120 | 100 | 20.01 | our work |
| CuO nanowires | 190 | 100 | 6.2 | *Sens. Actuators B: Chem.* **2017**, *252*, 1-8 |
| Mesoporous C-doped $ZrO_2$ film | Room temperature | 500 | 3.4 | *Sens. Actuators B: Chem.* **2017**, *242*, 202-214 |
| Au nanoparticles decorated ZnS hollow spheres | 260 | 100 | 1.9 | *Sens. Actuators B: Chem.* **2017**, *245*, 112-121 |
| Hollow CuO fibers | 200 | 100 | 4.66 | *Sens. Actuators B: Chem.* **2016**, *230*, 1-8 |
| $TeO_2$ nanowires | 50 | 1000 | 4.45 | *RSC Adv.* **2015**, *5*, 29126-29130 |