# Electronic Supplementary Information (ESI):

## Facile synthesis of single-crystalline hollow α-$Fe_2O_3$ nanospheres with gas sensing properties

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# 1. Experimental

## 1.1 Materials

Zinc acetate dihydrate, thiourea, cyclohexylamine (CHA), ethanol and the reference gas sensing reagents were purchased from Beijing Chemical Factory. $FeCl_3 \cdot 6H_2O$ was purchased from Tianjin Huadong Chemical Industry. All of the reagents were of analytic grade and used as received without further purification. Deionized water was used throughout.

## 1.2 General characterization

X-ray diffractometer (XRD) with Cu Kα radiation (λ = 1.5418 Å) was assessed to determine crystalline structure of the samples Rigaku D/Max 2550. The morphology of the samples is obtained by scanning electron microscope (SEM) of JEOL JSM 6700F. Transmission electron microscope (TEM), high-resolution transmission electron microscope (HRTEM) and selected area electron diffraction (SAED) were determined by FEI Tecnai G2S-Twin. XPS spectra were recorded on an ESCALAB 250 X-ray photoelectron spectrometer, using a monochromated X-ray source (Al Kα h = 1486.6 eV). Raman spectra were charaterized with Horiba Jobin Yvon LabRAM ARAMIS with 633 nm He-Ne laser excitation. Nitrogen absorption and desorption isotherms was performed on Micromeritics ASAP 2020M system.

## 1.3 Preparation of ZnS-CHA Nanohybrid

The ZnS-CHA nanohybrid material was prepared by following the previously-reported solvothermal procedures with slight variations.[1] Zinc acetate dihydrate (0.32 g, 1.5 mmol) as the zinc source and thiourea (0.255 g, 3 mmol) as the sulfur source were added to CHA (40 mL) and stirred vigorously. The mixture was sealed and heated at 120 $^oC$ for 20 h in a 50 mL PTFE-lined stainless steel autoclave. The white ZnS-CHA precipitate was obtained after cooling down to room temperature, which was washed several times with ethanol and dried at 60 $^oC$ for 6 h.

## 1.4 Synthesis of single-crystalline hollow α-$Fe_2O_3$ nanospheres

$FeCl_3 \cdot 6H_2O$ (0.16 g) with the as prepared ZnS-CHA nanocomposite (0.03 g) were stirred in 20 mL deionized water for 20 min, and then the mixture was sealed in a 30 mL PTFE-lined stainless steel autoclave and heated at 160 $^oC$ for 24 h. After cooling down to room temperature, the red α-$Fe_2O_3$ precipitate was harvest and washed several times with ethanol and dried at 60 $^oC$ 6 h for further use.

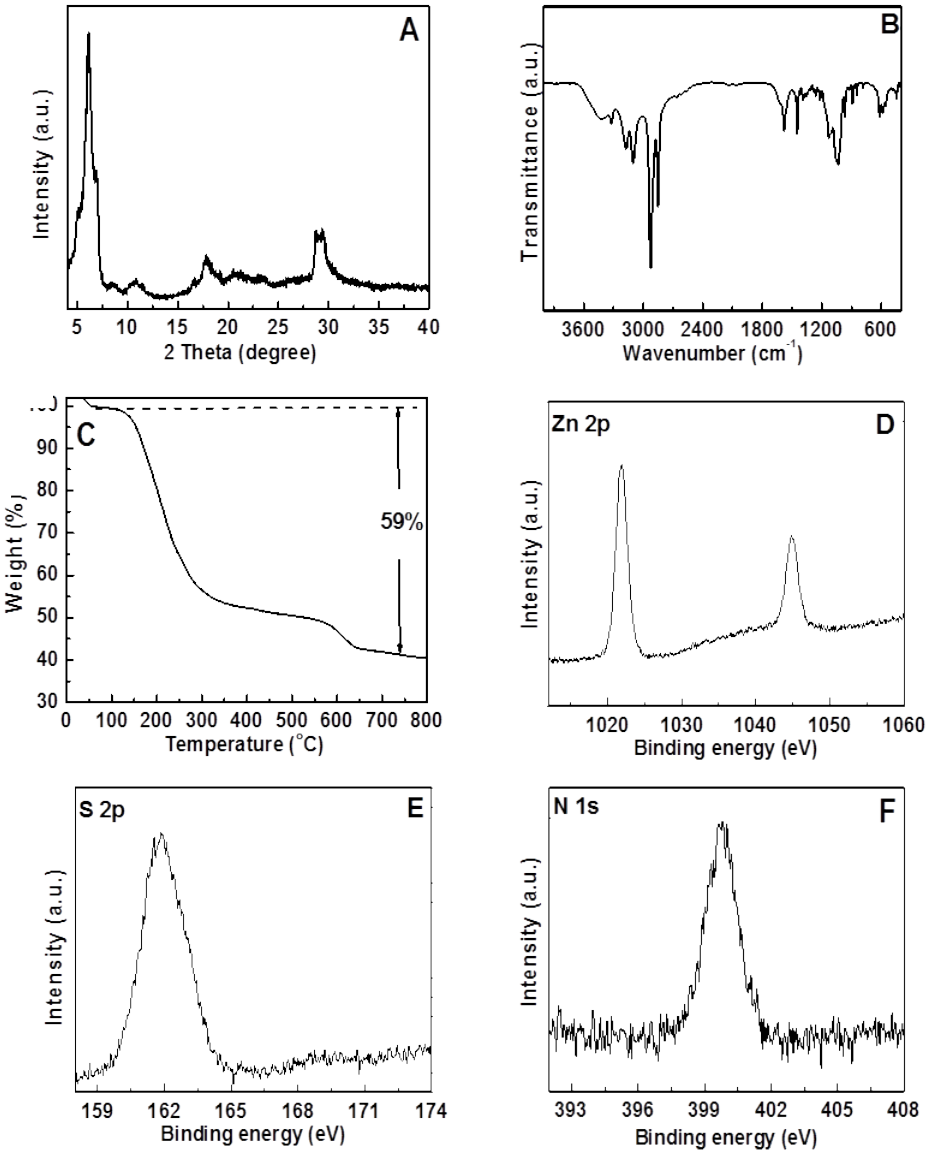



**Fig. S1** (A) XRD pattern; (B) IR sepectrum; (C) TG analysis; high resolution XPS spectra of (D) Zn 2p, (E) S 2p and (F) N 1s for the ZnS-CHA nanocomposite.

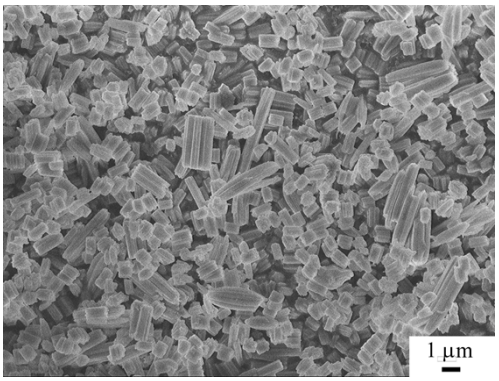



**Fig. S2** SEM image of the ZnS-CHA nanohybrid.

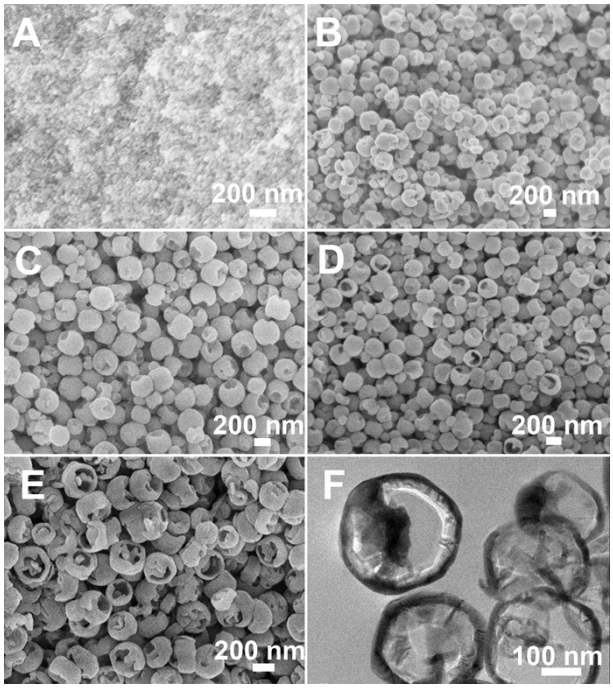
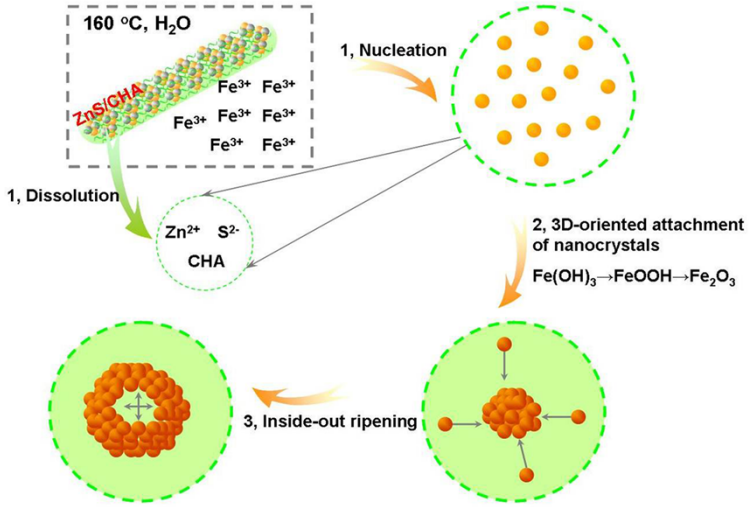



**Fig. S3** SEM images of the of the products obtained at (A) 30 min, (B) 1 h, (C) 6 h, (D) 8 h (E) 24 h, and (F) low magnification TEM for 24 h.




**Scheme S1**. Scheme for synthesis of the hollow single-crystalline α-$Fe_2O_3$ nanospheres.

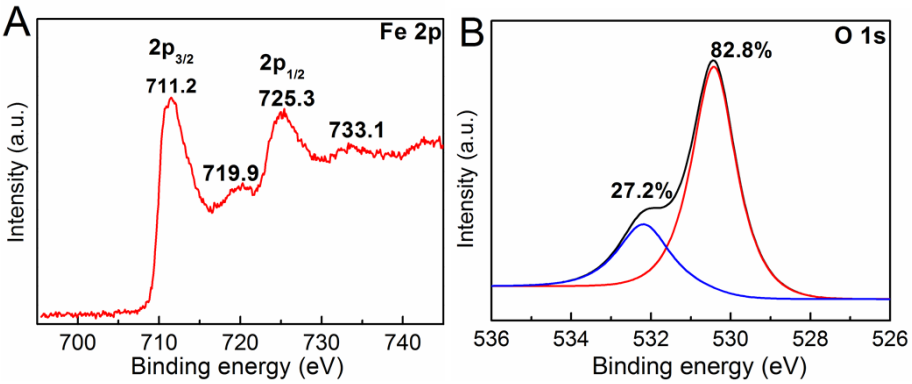



**Fig. S4** High resolution XPS spectra (A) Fe 2p and (B) O 1s of the single-crystalline hollow $Fe_2O_3$.

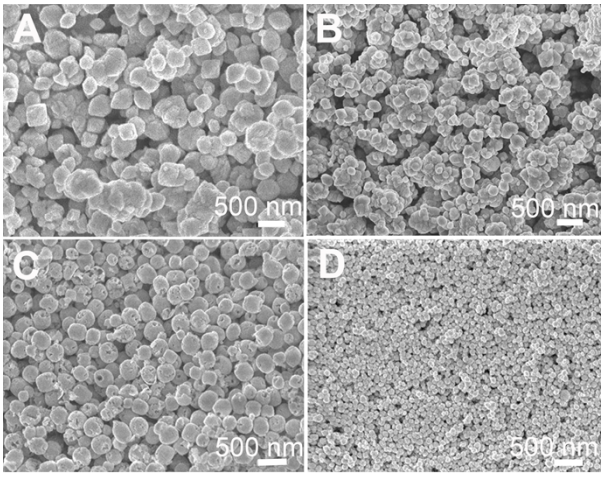



**Fig. S5** Contrast SEM images of the obtained $Fe_2O_3$ with different reaction conditions named (A) $Fe_2O_3$-1; (B) $Fe_2O_3$-2; (C) $Fe_2O_3$-3; (D) $Fe_2O_3$-4, respectively.

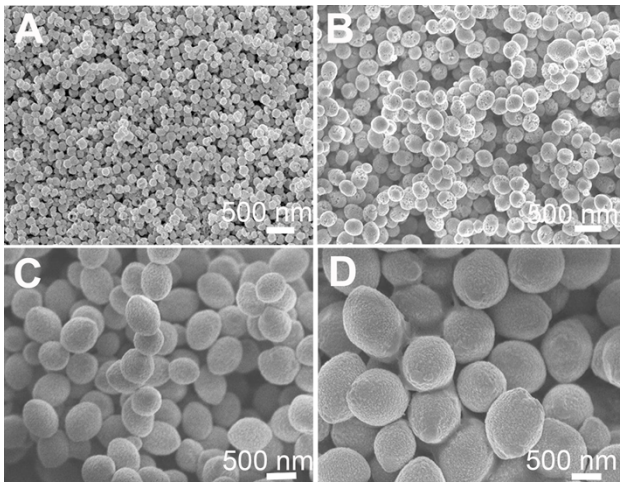



**Fig. S6** SEM images of the different size $Fe_2O_3$ nanospheres with multiplied $Fe^{3+}$ source (A) 0.16 g; (B) 0.32 g; (C) 0.48 g and (D) 0.64 g $FeCl_3{\cdot}6H_2O$.

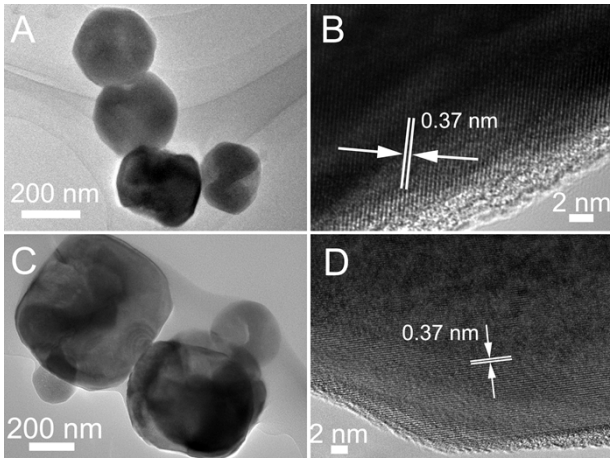



**Fig. S7** (A) TEM and (B) HRTEM images of the $Fe_2O_3$ obtained at 6 h; (C) TEM and (D) HRTEM images of the $Fe_2O_3$-1 (In control experiment section (1)).

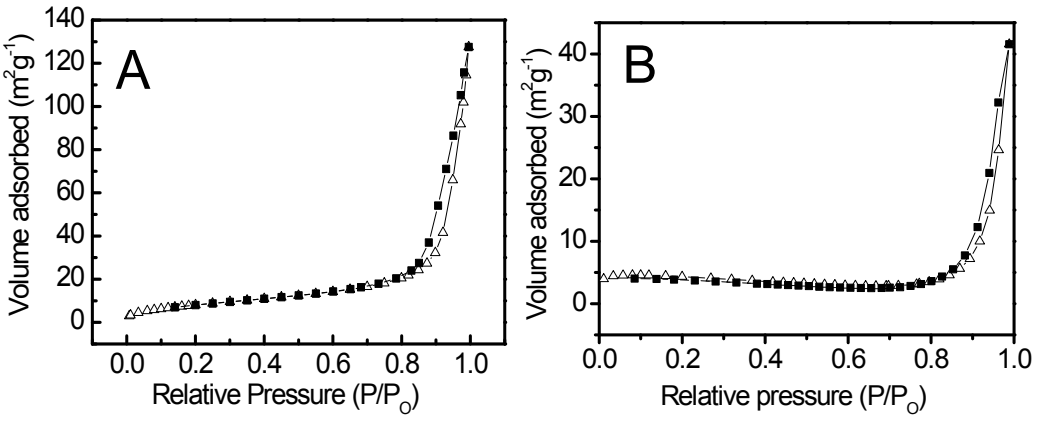



**Fig. S8** Nitrogen adsorption-desorption isotherm of (A) the single-crystalline hollow $Fe_2O_3$ nanospheres and (B) $Fe_2O_3$-1.

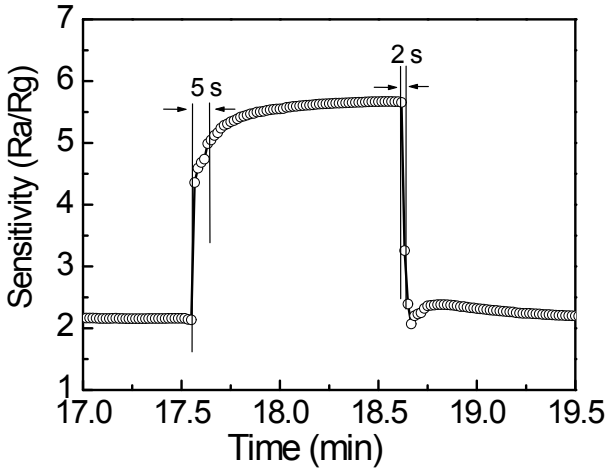



**Fig. S9** Magnification curve of the single-crystalline hollow $Fe_2O_3$ nanospheres towards 500 ppm ethanol.

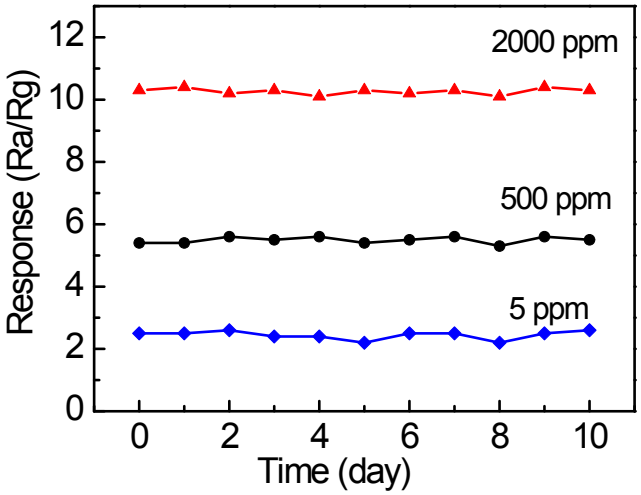



**Fig. S10** Stability sensing measurement to ethanol of the single-crystalline hollow $Fe_2O_3$ nanospheres.

Reference

1. X. X. Zou, G. D. Li, J. Zhao, P. P. Wang, Y. N. Wang, L. J. Zhou, J. Su, L. Li and J. S. Chen, *Inorg. chem*. 2011, **50**, 9106-9113.