## **Electronic Supplementary Information for**

## Sea urchin-like Ag/α-Fe<sub>2</sub>O<sub>3</sub> Nanocomposite Microspheres: Synthesis and Gas Sensing Application

Xijun Liu, Zheng Chang, Liang Luo, Xiaodong Lei, Junfeng Liu\* and Xiaoming Sun

State Key Laboratory of Chemical Resource Engineering, Beijing University of Chemical

Technology, Beijing, 100029, P. R. China

\* Corresponding author. *E-mail*: ljf@mail.buct.edu.cn.



Fig. S1 SEM images of (A) a single  $\alpha$ -FeOOH microsphere and (B), (C) magnified view of parts of the microsphere from the different visual direction.



Fig. S2 TEM images of Ag-loaded  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> architectures, corresponding to the 5.0 wt% (A), 10.0 wt% (B), and 15.0 wt% (C) Ag/ $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>.



**Fig. S3** Typical SEM images of the as-prepared samples at 120 °C for different reaction times: (A) 1.5 h, (B) 3 h, (C) 4.5 h, and corresponding XRD patterns (D).



Fig. S4 Schematic illustration for the formation process of the sea urchin-like  $\alpha$ -FeOOH microspheres.

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Fig. S5 Typical response curve and variations of the sensitivity of 3D sea urchin-like 4.0 wt% Ag/ $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> sensors exposed to methanol, ethanol, and acetone.



**Fig. S6** Typical response curve and variations of the sensitivity of nanocubes (a) and 3D sea urchin-like  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> (b) sensors exposed to acetone at concentration of 200 ppm measured at 350 °C.

$Ag/Fe_2O_3$ (wt%)	0	0.5	1.0	2.0	4.0	5.0	10.0	15.0	
Specific surface area (m <sup>2</sup> /g)	37.6	44.7	55.1	62.3	70.5	50.8	29.9	25.9	
Pore size (nm)	5.51	5.47	5.49	5.50	5.52	5.56	5.53	5.54	

Table S1. Brunauer–Emmett–Teller surface area and pore size distribution of

Ag/Fe<sub>2</sub>O<sub>3</sub> samples.

Table S2. Elemental analysis of Ag/Fe<sub>2</sub>O<sub>3</sub> samples.

	Ultimate analysis (wt%)				
Ag/Fe <sub>2</sub> O <sub>3</sub> loaded with different Ag contents (wt%)	С	Н	S		
ng contents (wt/o)	0.008	0.283	1 325		
0.5	0.018	0.230	1.636		
1.0	0.003	0.207	1.636		
2.0	0.016	0.198	1.789		
4.0	0.002	0.134	1.982		
5.0	0.004	0.151	1.891		
15.0	0.008	0.203	1.785		
15.0	0.002	0.179	1.0/2		