Supporting Information for:

Light-regulated Electrochemical Reaction Assisted Core-shell Heterostructure for Detecting Specific Volatile Marker with Controllable Sensitivity and Selectivity

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Supporting Information Available: The following files are available free of charge.

I. Experimental details of the breath analysis:

All breath samples were collected in the polyvinyl fluoride (PVF) sampling bag (Adolf Tedlar, China). Before collection, all the PVF sampling bags were washed with high purity N₂ for 30 min and kept in oven at 60°C overnight to remove any impurities. Breath samples exhaled from 10 health volunteers were collected. All breath samples were collected from the the volunteer on an empty stomach for 12 hours with teeth brushed and deep in halation for 10 min or sitting for half an hour. After deep breath and 30s of breath-holding, exhaled gas samples were collected in the PVF sampling bag. The first 1/3 breath sample will not be collected and the rest of 2/3 breath sample were collected for the test. 1L/bag breath sample will be collected for each volunteer. All the breath samples were stored in the vehicle refrigerator at 4°C during transportation and tested with the developed sensor after sampling within 24 h. Note that all volunteers were signed the informed consent to get the permission of analyzing their breath samples. Besides, volunteers with unclear medical history or diseases that may result in the changes in body VOCs (e.g. diabetes, laryngitis, asthma, etc.) shall be excluded. In addition, active smokers and volunteers who consumed alcohol in the 24 hours prior to sampling will also be excluded due to the same reason. In this study, all volunteers are interns or physician in Shanghai NO. 6 People's Hospital. Table 1S lists the personal information for each volunteer.

Volunteer	А	В	С	D	E	F	G	Н	I	J
Agenda	Female	Female	Female	Female	Female	Male	Male	Female	Female	Female
Age	26	28	37	25	31	47	26	28	25	21

 Table S-1. Personal details for all the volunteers

II. Figures for Supporting Information

- Illustration of the sensing performance for the electrochemical sensor using photoactive sensing materials, operated at (a) light off; (b) light on (Figure S-1);
- Conversion rate of various volatile markers at 425°C, catalyzed by various metal oxides or noble metal (Figure S-2);
- 3. XRD patterns of the as-synthesized ZnO, Fe₂O₃ and Fe₂O₃@ZnO (derived from different amount of zinc acetate precursor) (Figure S-3);
- FESEM images of (a) shuttle-like Fe₂O₃; (b) ZnO; Fe₂O₃@ZnO derived from (c) 0.05 mol/L; (d) 0.15 mol/L;(e) 0.25 mol/L and (f) 0.35 mol/L zinc acetate precursor (Figure S-4);
- EDX analysis of Fe₂O₃@ZnO core-shell heterostructure that derived from (a) 0.5 mol/L; (b) 0.15 mol/L; (c) 0.25 mol/L and (d) 0.35 mol/L zinc acetate precursor (Figure S-5);
- 6. Photograph of (a) the details in collecting breath samples; (b) breath samples in commercial sampling bags. (Figure S-6).



Figure S-1. Illustration of the sensing performance for the electrochemical sensor using photoactive sensing materials, operated at (a) light off; (b) light on.



Figure S-2. Conversion rate of various volatile markers at 425°C, catalyzed by various metal oxides or noble metal.



Figure S-3. XRD patterns of the as-synthesized ZnO, Fe_2O_3 and Fe_2O_3 @ZnO (derived from different amount of zinc acetate precursor).



Figure S-4. HRTEM images of (a) shuttle-like Fe_2O_3 ; (b) ZnO; Fe_2O_3 @ZnO derived from (c) 0.05 mol/L; (d) 0.15 mol/L;(e) 0.25 mol/L and (f) 0.35 mol/L zinc acetate precursor.



Figure S-5. EDX analysis of Fe_2O_3 @ZnO core-shell heterostructure that derived from (a) 0.5 mol/L; (b) 0.15 mol/L; (c) 0.25 mol/L and (d) 0.35 mol/L zinc acetate precursor.



Figure S-6. photograph of (a) the details in collecting breath samples; (b) breath samples in commercial sampling bags.