

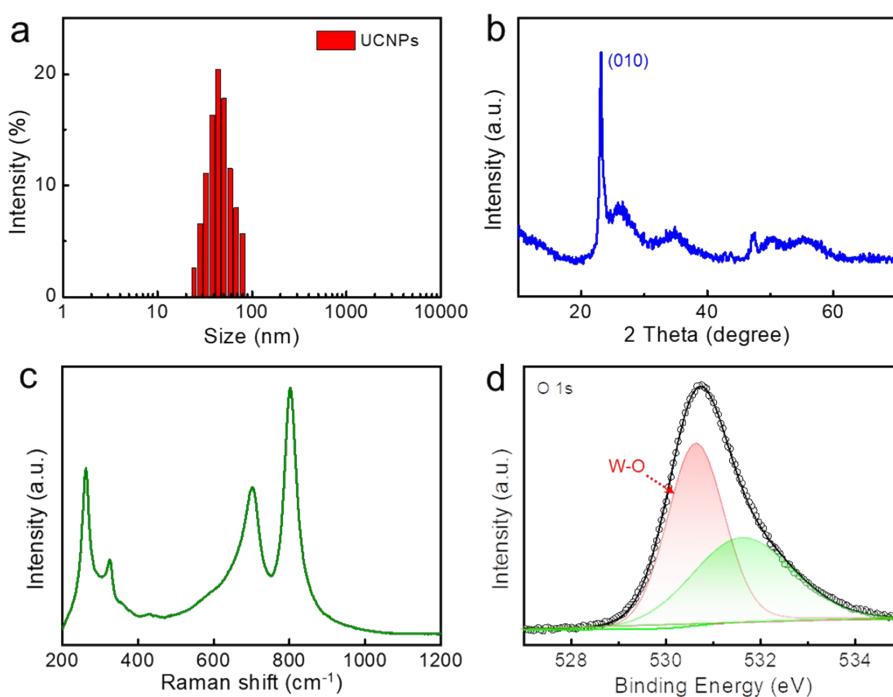
## Supporting Information

### Nonmetallic plasmon assisted upconversion fluorescence for ultrasensitive hydrogen peroxide detection from nM to $\mu$ M

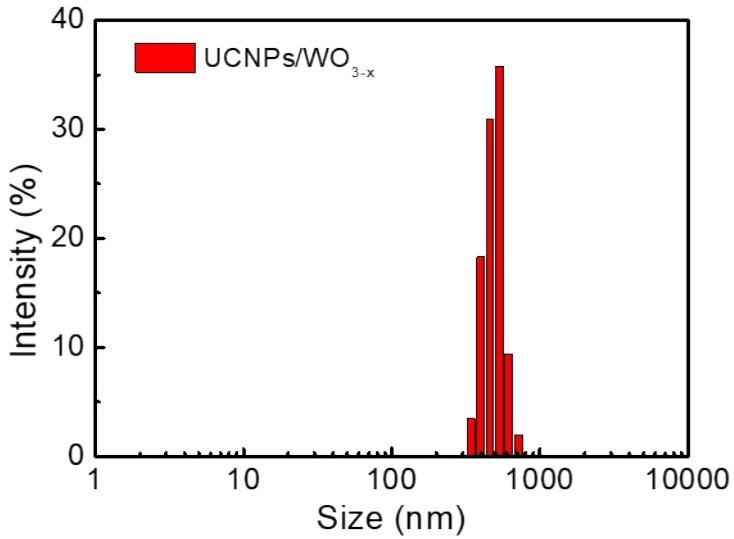
Juan Li, Xingwu Huang, Jiangyi Chen, Zaizhu Lou,\* and Baojun Li\*

Guangdong Provincial Key Laboratory of Nanophotonic Manipulation, Institute of Nanophotonics, College of Physics & Optoelectronic Engineering, Jinan University, Guangzhou, 511443, China

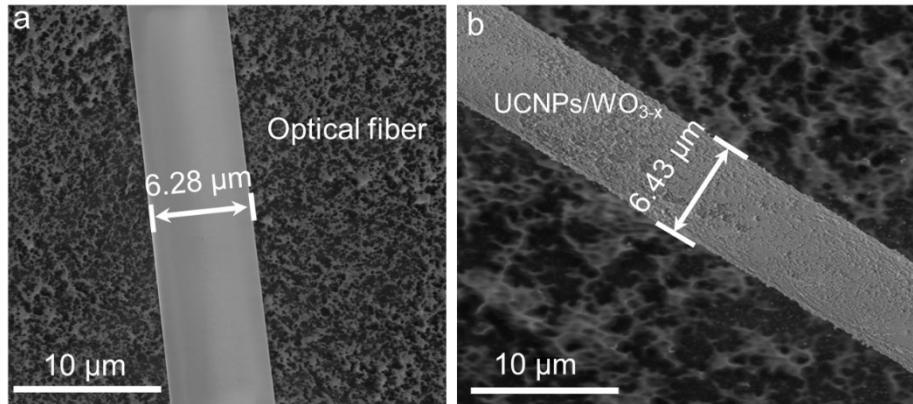
Corresponding Authors: [zzlou@jnu.edu.cn](mailto:zzlou@jnu.edu.cn); [baojunli@jnu.edu.cn](mailto:baojunli@jnu.edu.cn);



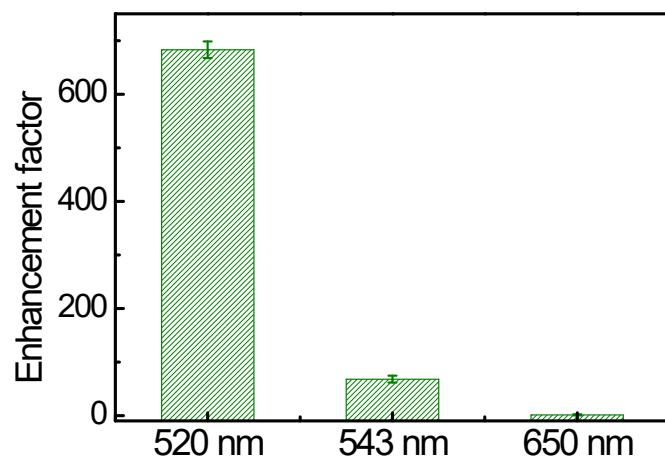
**Figure S1** DLS size distribution (a) of UCNPs. XRD pattern (b), Raman spectrum (c), O 1s XPS spectrum (d) of plasmonic  $\text{WO}_{3-x}$ .



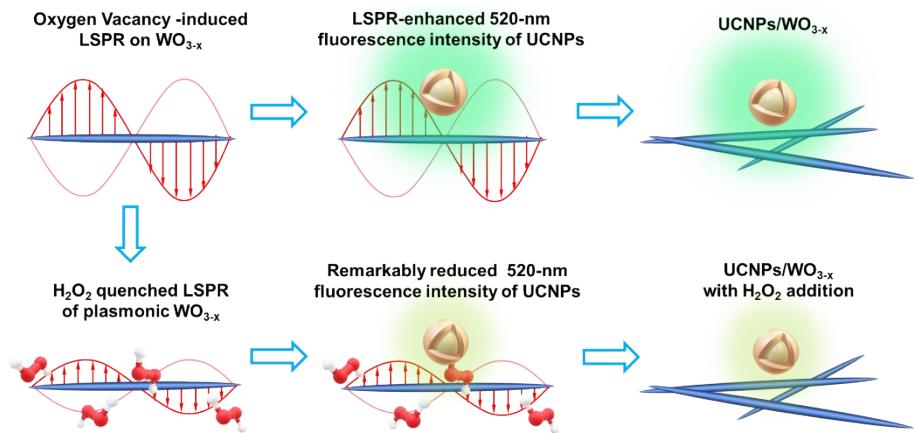
**Figure S2** DLS data of the UCNPs/WO<sub>3-x</sub> hybrid.



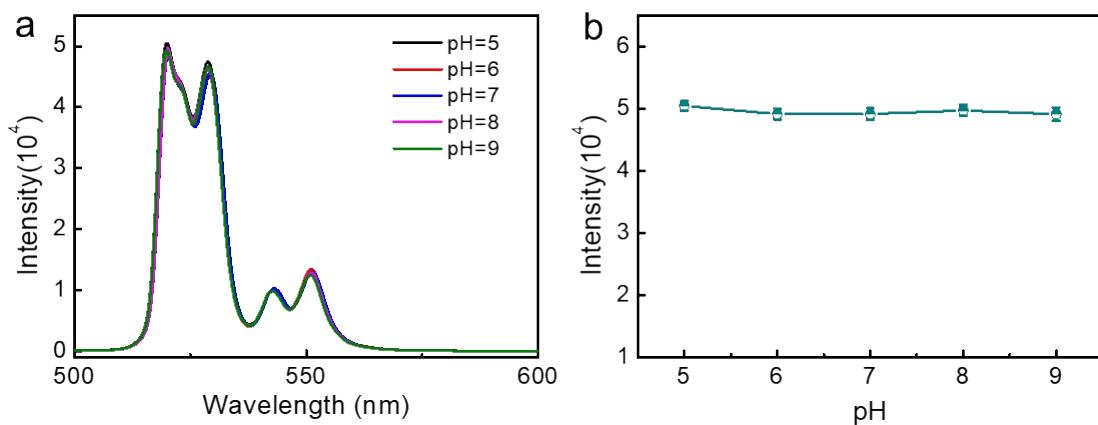
**Figure S3** Typical SEM images of optical fiber (a) and optical fiber coated with UCNPs/WO<sub>3-x</sub> (b).



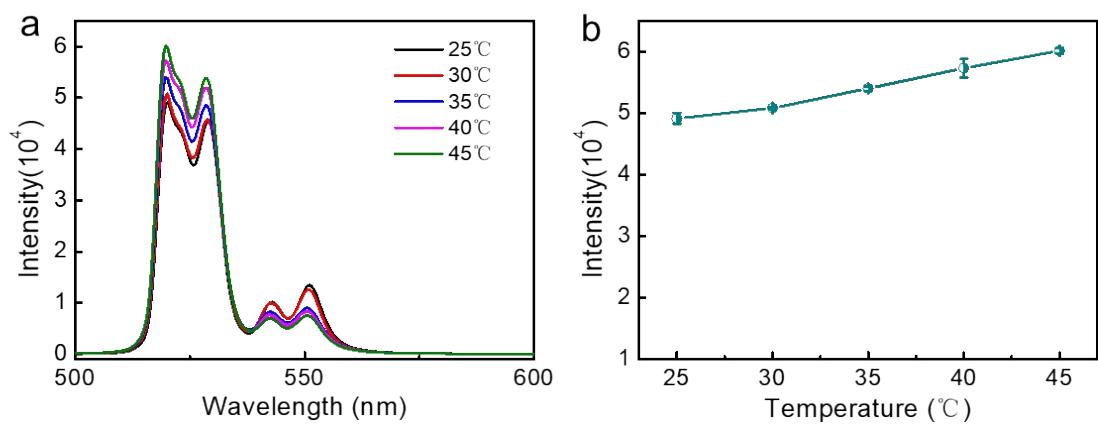
**Figure S4** The enhancement factors diagram of UCNPs/WO<sub>3-x</sub> for 520, 543, and 650-nm emission bands.



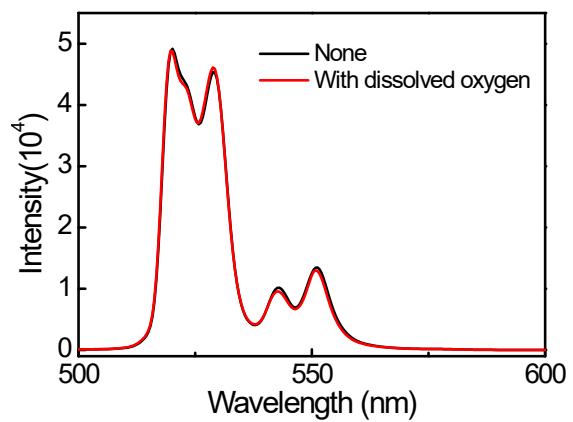
**Figure S5** The illustrated principle diagram of UCNPs/WO<sub>3-x</sub> probe for H<sub>2</sub>O<sub>2</sub> detection.



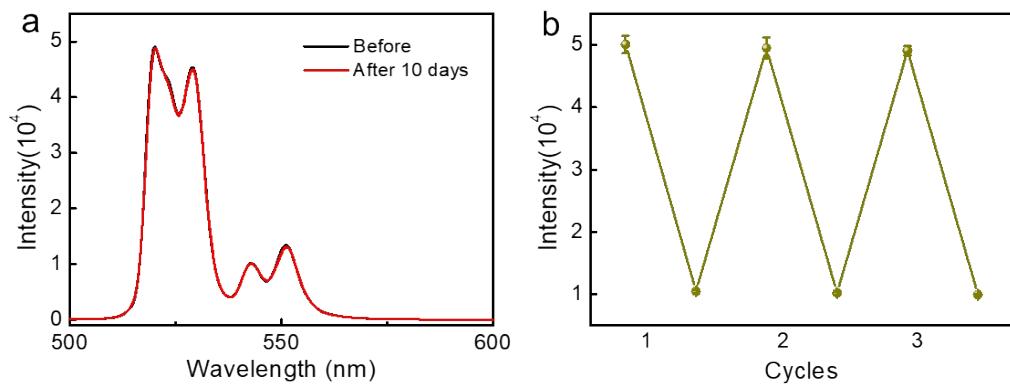
**Figure S6** The fluorescence spectra (a) of UCNPs/WO<sub>3-x</sub> probe under different PH conditions. Dependence of 520-nm fluorescence intensity of UCNPs/WO<sub>3-x</sub> probe on pH (b).



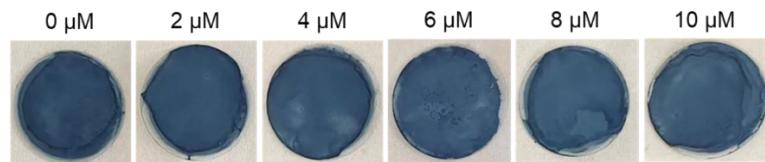
**Figure S7** The fluorescence spectra of UCNPs/WO<sub>3-x</sub> probe under different temperature (a). Dependence of 520-nm fluorescence intensity of UCNPs/WO<sub>3-x</sub> probe on the temperature (b).



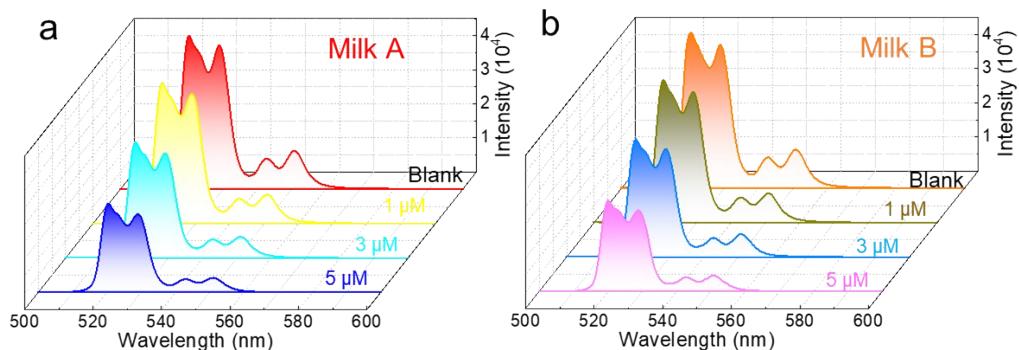
**Figure S8** The fluorescence spectra of UCNPs/WO<sub>3-x</sub> probe in fresh ultrapure water solution with dissolved oxygen.



**Figure S9** The fluorescence spectra of UCNPs/WO<sub>3-x</sub> probe before and after 10 days  
(a). Repeated H<sub>2</sub>O<sub>2</sub> detection cycles (c) from 0 to 10  $\mu$ M of UCNPs/WO<sub>3-x</sub> probe.



**Figure S10** The photographs of WO<sub>3-x</sub> samples with different H<sub>2</sub>O<sub>2</sub> concentration addition.



**Figure S11** The fluorescence spectra of UCNPs/WO<sub>3-x</sub> probe with addition of different milk samples.

**Table S1** Comparison of detection performance of various methods

Method	Materials	Detection range ( $\mu\text{M}$ )	Detection limit ( $\mu\text{M}$ )	Emission	Response time	Application	Ref.
<b>Colorimetric assay</b>	NiFe-LDHNS	10–500	4.4	/	10 min	Glucose and H <sub>2</sub> O <sub>2</sub>	[1] 2018
	$\alpha$ -AgVO <sub>3</sub> rods	60–200	2	/	2 min	Commercial antiseptic sample	[2] 2017
<b>Electrochemical assay</b>	CoS/RGO nanohybrids	0.1–2542.4	0.042	/	3 s	Biological samples	[3] 2018
	Pt-NPs/EC	0.05–2220	0.01	/	/	Non-enzymatic determination	[4] 2017
	PB/PEDOT	0.5–46	0.17	/	/	H <sub>2</sub> O <sub>2</sub> Upon NADH Treatment	[5] 2023
<b>Chemiluminescent</b>	Co(II)-monoethanolamine	1.0–154	0.2	/	12.5s		[6] 2024
<b>Fluorometric assay</b>	C-dots	0.1–40	0.05	/	10 min	Choline and acetylcholine	[7] 2014
	AlFgen TPE-M-L	0.01–1.0	0.01	460 nm	30 min	Human serum samples	[8] 2018
	MIL-101(Fe)	0.1–130	0.0011	430 nm	90 min	Choline and acetylcholine	[9] 2020
	Sulfur dope CDs	0–100	0.05	830 nm	200 s	Cell Mitosis and Mouse Brain	[10] 2018
	Coumarin pyran+galactose	1.0–50	0.33	706 nm	15 min	Zebrafish	[11] 2020
	MoO <sub>3-x</sub> QDs	0–1	0.003	770 nm	/		[12] 2021
	BTMB+ESIPT	0–20	0.109	542 nm	0–45 min	Ferroptosis process	[13] 2021
	Dicyanoisophorone	0–30	0.35	657 nm	15–20 min	Milk samples and in vivo	[14] 2023
	UCNPs	0.25–5	0.1	800 nm	/	Choline and H <sub>2</sub> O <sub>2</sub>	[15] 2019
	UCNPs/B-R-COOH	0–70	0.00434	478 nm	/	Gold pigment	[16] 2024
<b>This work</b>	UCNPs-DCM	0–10	0.168	660 nm	/	Hydrogen Peroxide in Vivo	[17] 2019
	UCNPs/WO <sub>3-x</sub>	<b>0–50</b>	<b>0.001</b>	<b>520 nm</b>	/	<b>Human serum and milk</b>	

## References

1. Zhan, T.; Kang, J.; Li, X.; Pan, L.; Li, G.; Hou, W., NiFe layered double hydroxide nanosheets as an efficiently mimic enzyme for colorimetric determination of glucose and H<sub>2</sub>O<sub>2</sub>. *Sensors and Actuators B: Chemical* **2018**, *255*, 2635–2642.
2. Wang, Y.; Zhang, D.; Wang, J., Metastable  $\alpha$ -AgVO<sub>3</sub> microrods as peroxidase mimetics for colorimetric determination of H<sub>2</sub>O<sub>2</sub>. *Microchimica Acta* **2017**, *185*.
3. Kubendhiran, S.; Thirumalraj, B.; Chen, S. M.; Karuppiah, C., Electrochemical co-preparation of cobalt sulfide/reduced graphene oxide composite for electrocatalytic activity and determination of H<sub>2</sub>O<sub>2</sub> in biological samples. *Journal of Colloid and Interface Science* **2018**, *509*, 153–162.
4. Karthik, R.; Karikalan, N.; Chen, S. M., Rapid synthesis of ethyl cellulose supported platinum nanoparticles for the non-enzymatic determination of H<sub>2</sub>O<sub>2</sub>. *Carbohydrate Polymers* **2017**, *164*, 102–108.
5. Zhang, S.; Qin, H.; Cheng, S.; Zhang, Y.; Gao, N.; Zhang, M., An electrochemical

- nanosensor for monitoring the dynamics of intracellular H<sub>2</sub>O<sub>2</sub> upon NADH treatment. *Angewandte Chemie International Edition* **2023**, *62*.
- 6. Zhang, R.; Zhong, Y.; Hu, Y.; Chen, Y.; Xia, L.; Li, G., Liquid-phase cyclic chemiluminescence for the Identification of cobalt speciation. *Analytical Chemistry* **2024**, *96*, 3933-3941.
  - 7. Wei, J. F., Ren, J., Liu, J., Meng, X. W., Ren, X. L., Chen, Z. Z., Tang, F. Q., An eco-friendly, simple, and sensitive fluorescence biosensor for the detection of choline and acetylcholine based on C-dots and the Fenton reaction. *Biosensors and Bioelectronics*, **2014**, *52*, 304-309.
  - 8. Chang, J.; Li, H.; Hou, T.; Duan, W.; Li, F., Paper-based fluorescent sensor via aggregation induced emission fluorogen for facile and sensitive visual detection of hydrogen peroxide and glucose. *Biosens Bioelectron* **2018**, *104*, 152-157.
  - 9. Guo, J.; Wu, S.; Wang, Y.; Zhao, M., A label-free fluorescence biosensor based on a bifunctional MIL-101(Fe) nanozyme for sensitive detection of choline and acetylcholine at nanomolar level. *Sensors and Actuators B: Chemical* **2020**, *312*.
  - 10. Guo, H. L.; Chen, G.; Gao, M.; Wang, R.; Liu, Y. X.; Yu, F. B., Imaging-of-endogenous-hydrogen-peroxide-during-the-process-of-cell-mitosis-and-mouse-brain-development. *Analytical Chemistry* **2019**, *91*, 1203-1210.
  - 11. Jiang, W. L.; Wang, W. X.; Liu, J.; Li, Y.; Li, C. Y., A novel hepatocyte-targeting ratiometric fluorescent probe for imaging hydrogen peroxide in zebrafish. *Sensors and Actuators B: Chemical* **2020**, *313*.
  - 12. Zhong, S.; Xing, C.; Cao, A.; Zhang, T.; Li, X.; Yu, J.; Cai, W.; Li, Y., Ultra-fast synthesis of water soluble MoO<sub>3-x</sub> quantum dots with controlled oxygen vacancies and their near infrared fluorescence sensing to detect H<sub>2</sub>O<sub>2</sub>. *Nanoscale Horizons* **2020**, *5*, 1538-1543.
  - 13. Zhang, H. C.; Tian, D. H.; Zheng, Y. L.; Dai, F.; Zhou, B., Designing an ESIPT-based fluorescent probe for imaging of hydrogen peroxide during the ferroptosis process. *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy* **2021**, *248*.
  - 14. Peng, T.; Ye, S.; Liu, R.; Qu, J., Colorimetric and fluorescent dual-signals probes for naked-eye detection of hydrogen peroxide and applications in milk samples and in vivo. *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy* **2023**, *297*.
  - 15. Zhang, L.; Yin, S.; Hou, J.; Zhang, W.; Huang, H.; Li, Y.; Yu, C., Detection of choline and hydrogen peroxide in infant formula milk powder with near infrared upconverting luminescent nanoparticles. *Food Chemistry* **2019**, *270*, 415-419.
  - 16. Feng, Y.; Lei, D.; Zu, B.; Li, J.; Li, Y.; Dou, X., A self-accelerating naphthalimide-based probe coupled with upconversion nanoparticles for ultra-accurate tri-mode visualization of hydrogen peroxide. *Advanced Science* **2024**.
  - 17. Wang, H.; Li, Y.; Yang, M.; Wang, P.; Gu, Y., FRET-based upconversion nanoprobe sensitized by Nd<sup>3+</sup> for the ratiometric detection of hydrogen Peroxide in Vivo. *ACS Applied Materials & Interfaces* **2019**, *11*, 7441-7449.