1 **Oxygen Vacancy Engineering via Mo Doping in NiO Nanofibers for Selective**

2 **Trace Xylene Detection**

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4 Xiaowen Zhang [a, b, c, d], Qi Lei [a, b, c, d], Lixiang Wang [a, b, c, d], Qingge Feng [a, b, c, d], Qihua

5 Liang [a, b, c, d, *]

6 [a]School of Resources, Environment and Materials, Guangxi University, Nanning

7 530004, China.

8 [b]Guangxi Key Laboratory of Processing for Non-ferrous Metals and Featured

9 Materials, Guangxi University, Nanning, 530004.

10 [c]Key Laboratory of Environmental Protection (Guangxi University), Education

11 Department of Guangxi Zhuang Autonomous Region, Guangxi Nanning 530004,
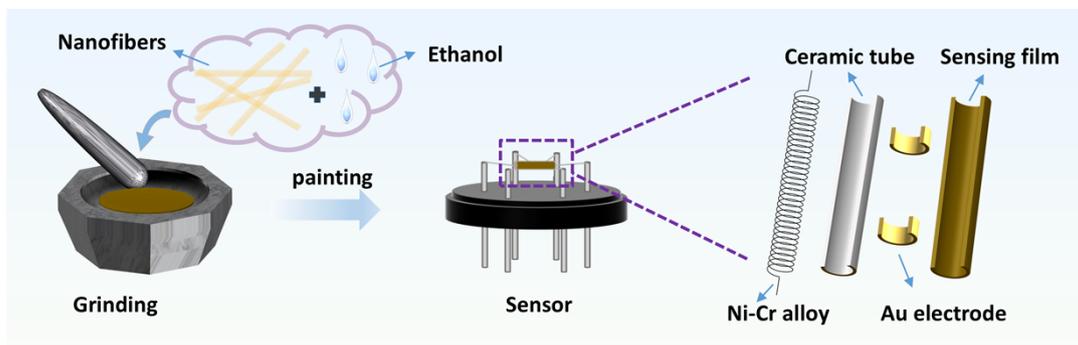
12 China.

13 [d]Guangxi Key Laboratory of Emerging Contaminants Monitoring, Early Warning and

14 Environmental Health Risk Assessment, Guangxi Nanning 530004, China.

15 ***Corresponding Author:**

16 School of Resources, Environment and Materials, Guangxi University, Nanning

17 530004, China
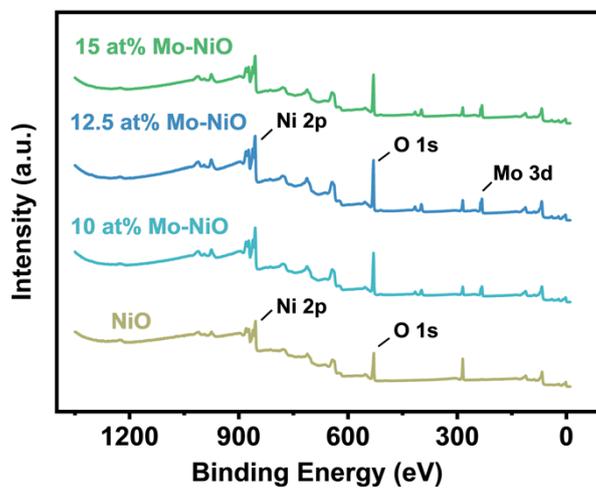
18 **E-mail address:** lqh@gxu.edu.cn

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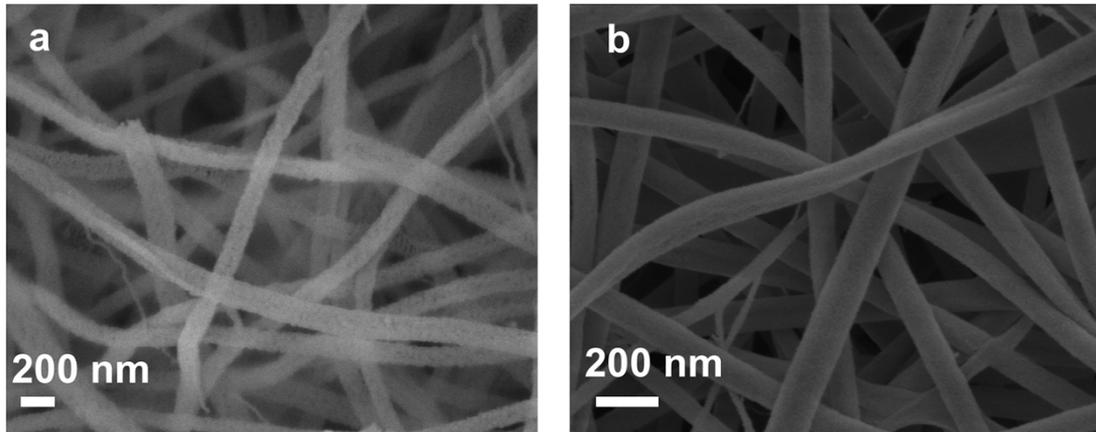21 **Fig. S1.** The painting process of sensing material and the structure of the sensor.

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25 **Fig. S2.** XPS full spectra of NiO and x at% Mo-NiO (x = 10, 12.5, and 15).

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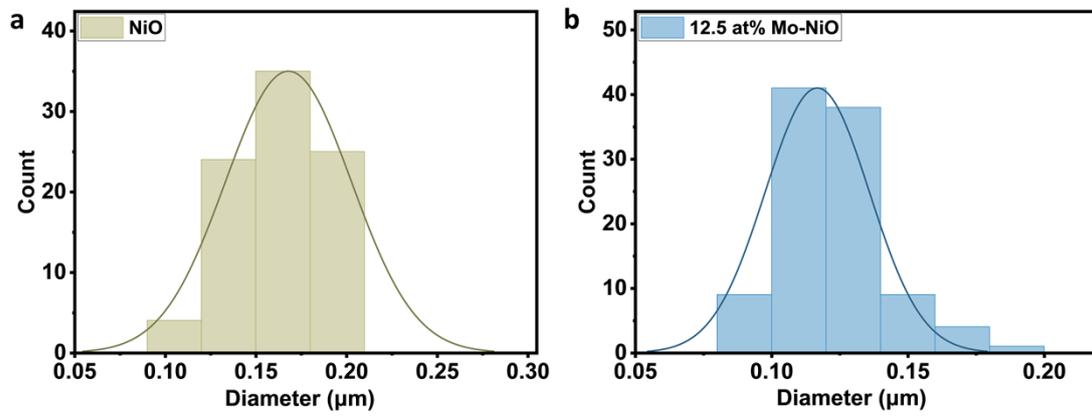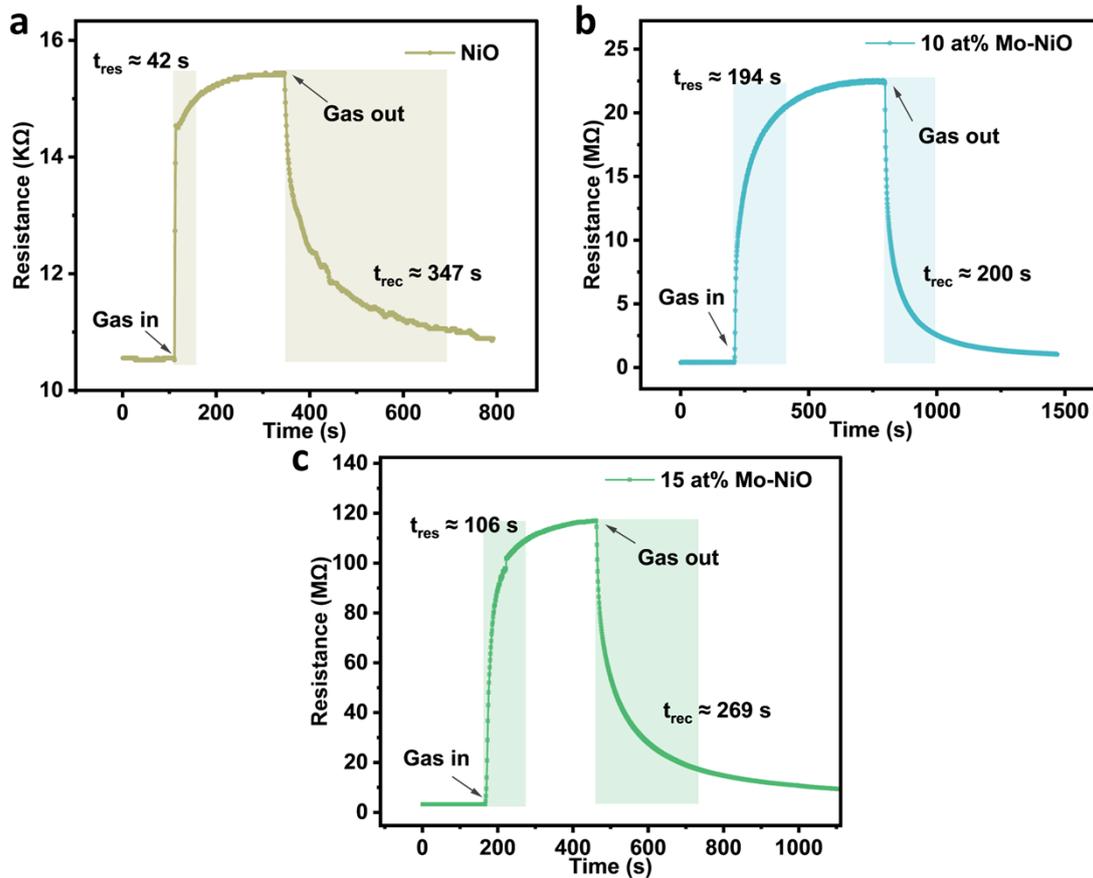**Fig. S3.** SEM images of (a) NiO and (b) 12.5 at% Mo-NiO.

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Fig. S4. Particle size distributions of (a) NiO and (b) 12.5 at% Mo-NiO.
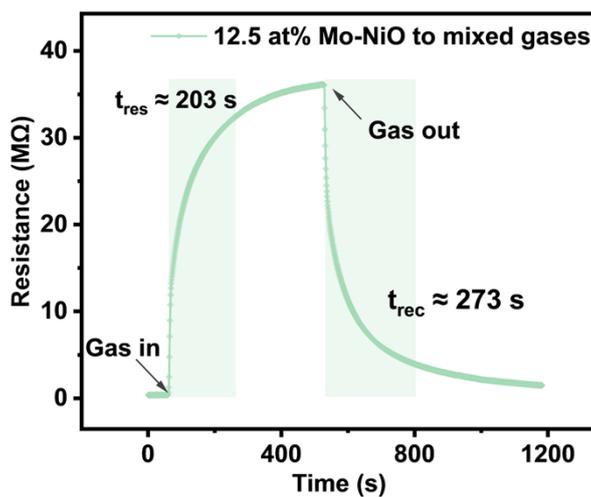
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39 **Fig. S5.** Response-recovery curves to 20 ppm xylene of the (a) NiO, (b) 10 at% Mo-

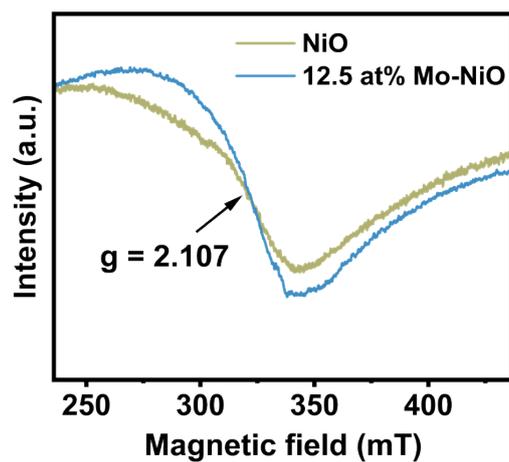40 NiO, and (c) 15 at% Mo-NiO sensors at 265 °C.

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43 **Fig. S6.** Response-recovery curve of 12.5 at% Mo-NiO sensor to 20 ppm mixed gases

44 containing benzene, toluene, ethylbenzene, and xylene at 265 °C.

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Fig. S7. EPR spectra of NiO and 12.5 at% Mo-NiO.

Table S1. Fitting results of Ni 2p XPS spectra of the NiO and 12.5 at% Mo-NiO.

Sample	Ni^{2+}	Ni^{3+}	Ni^{3+}/Ni^{2+}
NiO	25.9 %	74.1 %	2.86
10 at% Mo-NiO	25.2 %	74.8 %	2.97
12.5 at% Mo-NiO	24.1 %	75.9 %	3.14
15 at% Mo-NiO	25.3 %	74.7 %	2.94

53 **Table S2.** Fitting results of O 1s XPS spectra of the NiO and 12.5 at% Mo-NiO.

Sample	Oxygen species	Relative percentage (%)
NiO	Lattice oxygen	55.2
	Oxygen vacancy	22.7
	Chemisorbed oxygen	22.1
10 at% Mo-NiO	Lattice oxygen	50.3
	Oxygen vacancy	23.1
	Chemisorbed oxygen	26.6
12.5 at% Mo-NiO	Lattice oxygen	42.6
	Oxygen vacancy	28.0
	Chemisorbed oxygen	29.4
15 at% Mo-NiO	Lattice oxygen	48.1
	Oxygen vacancy	26.4
	Chemisorbed oxygen	25.5

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56 **Table S3.** The results of N_2 adsorption-desorption analysis.

Sample	Specific surface area (m²/g)	Total pore volume (ml/g)	Average pore size (nm)
NiO	25.1	0.2	25.2
10 at% Mo-NiO	157.7	0.5	11.6
12.5 at% Mo-NiO	150.8	0.3	7.2
15 at% Mo-NiO	172.2	0.4	8.3

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