Supporting information

Oxygen Vacancy-Enriched NiO Nanozymes Achieved by Facile Annealing in Argon for Detection of L-Cys

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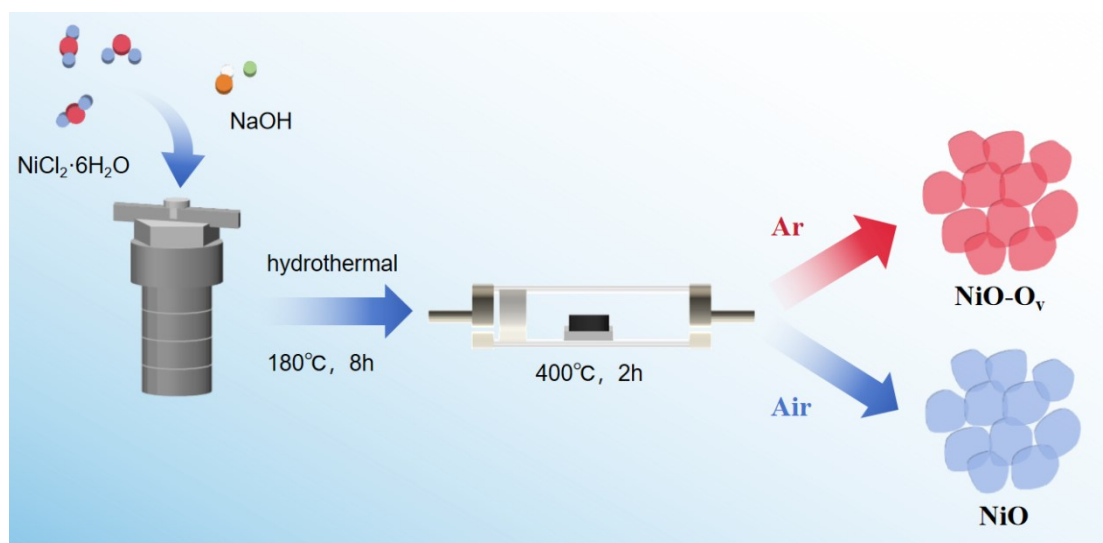
[d] Sun Yat Sen University, School of Chemical Engineering and Technology, Zhuhai 519000, China.
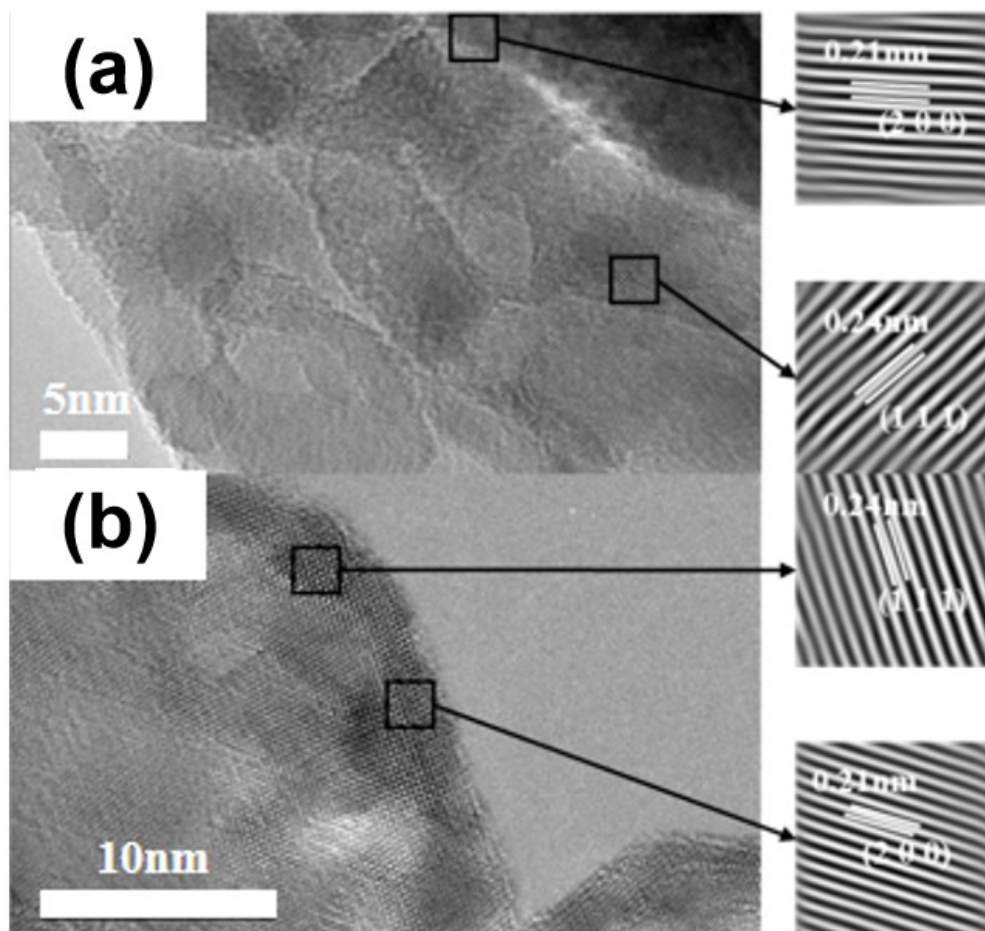
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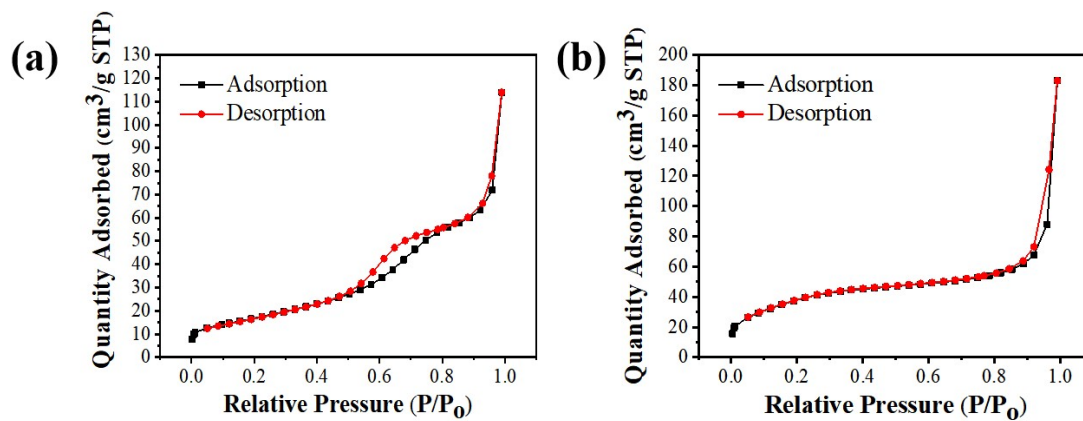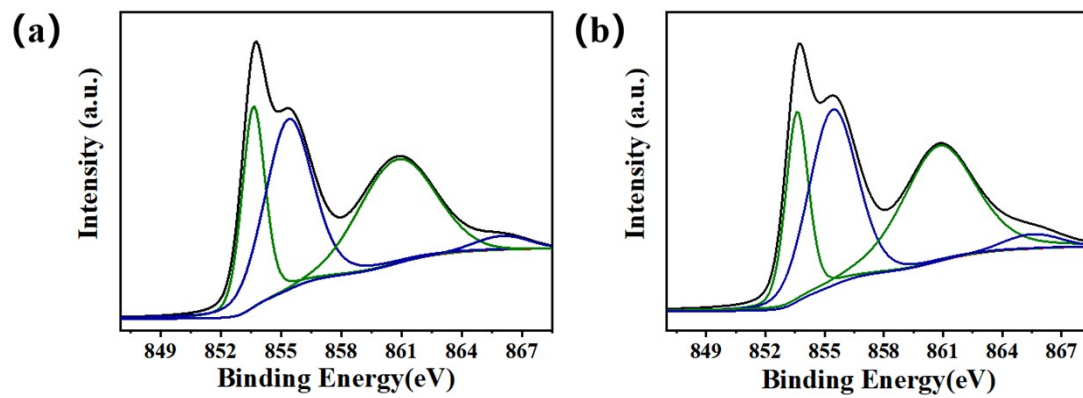
1. Supplementary Figures

Figure S1. The synthetic route of NiO an NiO-Ov samples.

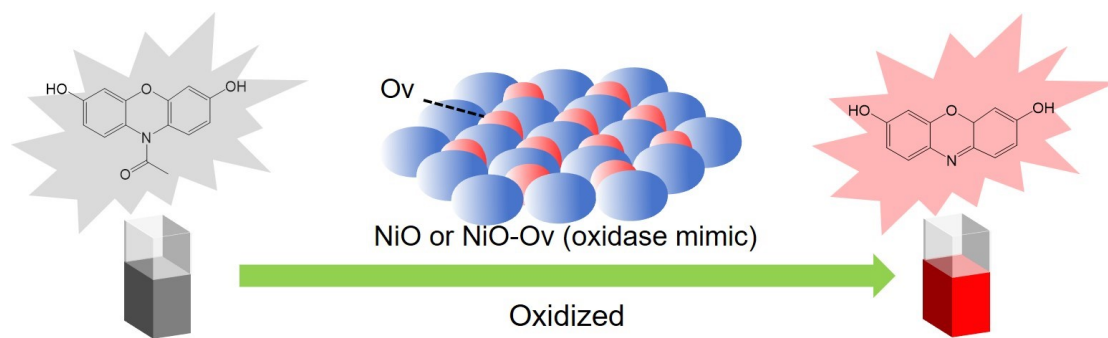**Figure S2.** High-resolution TEM of (a) NiO; (b) NiO-Ov (The black boxes correspond to the Fourier-transformed lattice stripes)

Figure S3. BET test curve comparison： (a) NiO; (b) NiO-O$_V$.

Figure S4. XPS spectra of different samples of Ni2p: (a) NiO; (b) NiO-O$_V$.

Figure S5. Schematic diagram of Fluorescent turn-on process for catalysis of NiO or NiO-Ov nanozyme on AR to resorufin.

2. Supplementary Tables

Table S1. Structural parameters of NiO samples calcined in different atmospheres

Sample	2θ (°)	FWHM	Grain size[a] (nm)	BET[b] (m²/g)	Pore size (nm)
NiO	43.240	0.641	2.97	62.01	9.39
NiO-O$_V$	43.220	0.820	2.32	136.55	12.37

[a] calculated by the method of Scherrer's equation based on the diffraction of the (200) peak of NiO at 2θ = 43.1°.

[b] calculated by the method of specific surface area.

Table S2. The summary of XPS results of different NiO samples.

Sample	Binding energy (eV)					Atomic ratio(%)		EPR area
	Ni 2p$_{3/2}$		O 1s			Ni^{3+}/Ni^{2+}	$O_\beta/(O_\alpha+O_\beta+O_\gamma)$	
	Ni^{2+}	Ni^{3+}	O_α	O_β	O_γ			
NiO	853.6	855.43	529.15	531.18	532.14	1.72	21.4	8.1×10^4
NiO-O$_V$	853.63	855.45	529.18	531.08	532.91	1.85	32.8	1.1×10^5

Table S3. Comparison of kinetic parameters (K_m and V_{max}) corresponding to different nanozymes

Substrate	Nanozymes	K_m (μM)	V_{max} (nM s^{-1})	Ref.
	Bi–Au NPs	89.3	15.0	1
	ZiF-67	5.28	28.2	2
	FeP@C	2.30	—	3
Amplex Red	Au/AgCl	17	4.6	4
	MFNP1:1	34.2	244	5
	CoOxH-GO	4.87	0.839	6
	NiO-O_V	2.83	26.7	this work

Table S4. Detection range and detection limit of L-Cys by different systems

System	Method	Linear range (μM)	Detection limit (nM)	Ref.
Co_4S_3	fluorescence	0.25-2.5	75	7
AuNRs/Au-Ag NCs	fluorescence	5-100	1.73×10^3	8
AuNCs-AuNPs	fluorescence	1.5-35	1.4×10^3	9
Si-CDs	fluorescence	20-100	410	10
CDs	chemiluminescence	10-100	8.8×10^3	11
$OV-Mn_3O_4$	colorimetric	5-800	1.31×10^3	12
$Gd(OH)_3$	colorimetric	0.2−75	2.6×10^3	13
2D Co_3S_4	colorimetric	0.2−100	2.7×10^3	14
VS_4	colorimetric	5-100	2.5×10^3	15
$[Ag_2(bit)_2]_2[Mo_8O_{26}]$	colorimetric	1-100	220	16
rGO-GP	colorimetric	2-30	100	17
MnO_2 nanobelts	colorimetric	0-35	100	18
$MnO_2@Co_3O_4$	colorimetric	1.25-25	1.1×10^3	19
$NiO-O_V$	fluorescence	0.05-2	37.8	this work

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