

## Supporting Information

# Constructing Fe<sub>2</sub>O<sub>3</sub> Nanoparticles in Nitrogen-Doped Carbon Materials to Enhance the Electrochemical Sensing Performance of Pb<sup>2+</sup> and Cd<sup>2+</sup>

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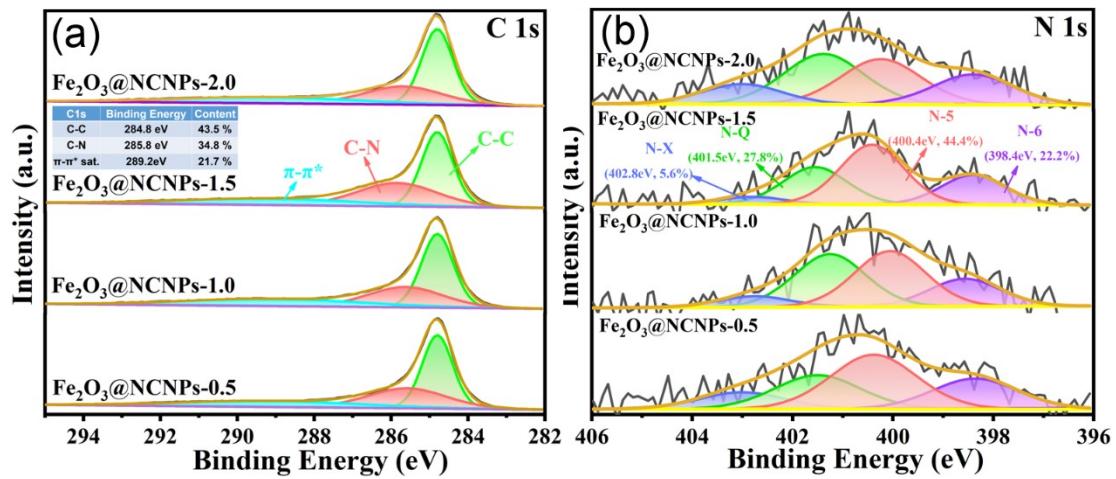
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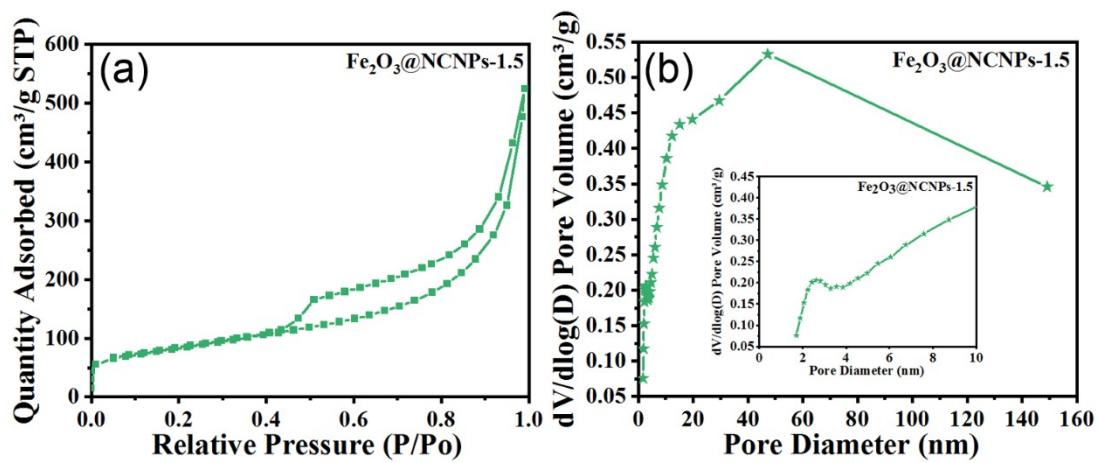
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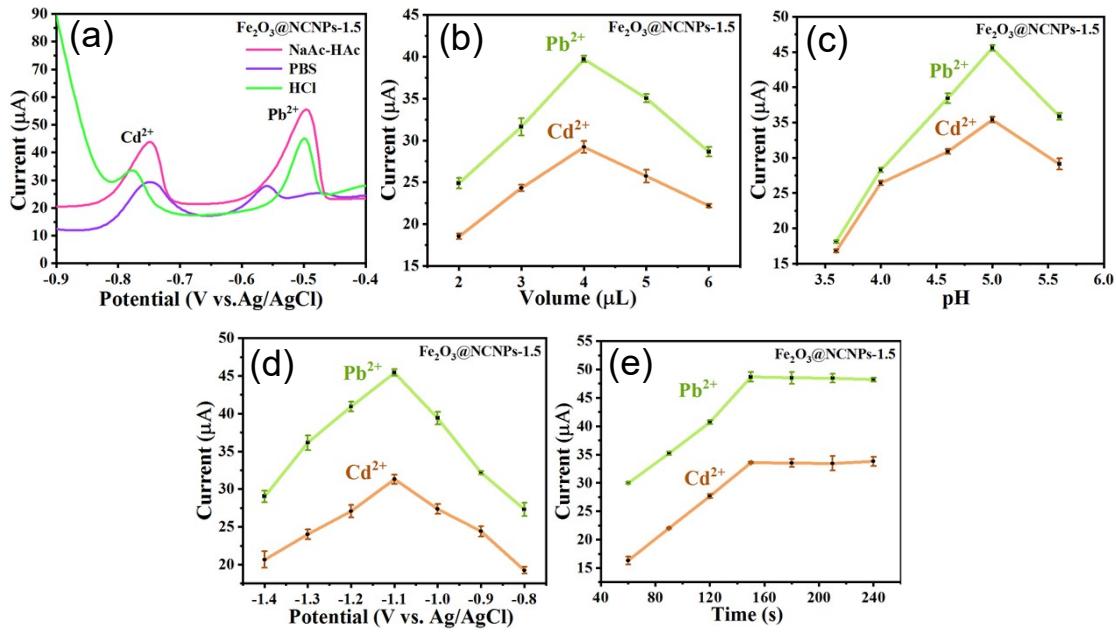
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**Fig. S1** High resolution (a) C 1s and (b) N 1s XPS spectrum of  $\text{Fe}_2\text{O}_3@\text{NCNPs-}x$ .



**Fig. S2** (a) the  $\text{N}_2$  adsorption/desorption isotherm and (b) the pore-size distribution of  $\text{Fe}_2\text{O}_3@\text{NCNPs-1.5}$  nanomaterial.



**Fig. S3** Investigation of optimal experimental conditions: the influence of (a) supporting electrolytes, (b) modified volume, (c) pH values, (d) deposition potential, and (e) deposition time on the current response of the  $\text{Fe}_2\text{O}_3@\text{NCNPs-1.5}/\text{GCE}$ . The error bars represented the standard deviations of triple measurements.

**Table S1** The curve fitting information of **C 1s** for Fe<sub>2</sub>O<sub>3</sub>@NCNPs-n.

C-C			
Sample	Binding Energy (eV)	FWHM	%Area
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-0.5	284.8	0.9	45.5
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.0	284.8	0.9	45.5
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.5	284.8	0.9	43.5
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-2.0	284.8	0.9	48.1

C-N			
Sample	Binding Energy (eV)	FWHM	%Area
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-0.5	285.6	2.4	31.8
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.0	285.6	2.3	29.5
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.5	285.8	2.4	34.8
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-2.0	285.7	2.4	28.8

$\pi-\pi^*$ satellite			
Sample	Binding Energy (eV)	FWHM	%Area
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-0.5	289.2	5.5	22.7
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.0	289.1	5.4	25.0
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.5	289.2	5.5	21.7
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-2.0	289.2	5.5	23.1

**Table S2** The curve fitting information of N 1s for Fe<sub>2</sub>O<sub>3</sub>@NCNPs-n.

N-6			
Sample	Binding Energy	FWHM	%Area
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-0.5	398.4	1.9	22.5
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.0	398.6	1.7	18.9
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.5	398.4	1.6	22.2
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-2.0	398.4	1.9	20.7

N-5			
Sample	Binding Energy	FWHM	%Area
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-0.5	400.4	1.9	40.0
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.0	400.1	1.7	37.7
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.5	400.4	1.6	44.4
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-2.0	400.2	1.9	31.0

N-Q			
Sample	Binding Energy	FWHM	%Area
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-0.5	401.5	1.9	25.0
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.0	401.3	1.7	35.9
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.5	401.5	1.6	27.8
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-2.0	401.4	1.9	34.5

N-X			
Sample	Binding Energy	FWHM	%Area
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-0.5	403.0	1.9	12.5
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.0	402.8	1.7	7.5
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.5	402.8	1.6	5.6
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-2.0	403.0	1.9	13.8

**Table S3** The curve fitting information of **O 1s** for Fe<sub>2</sub>O<sub>3</sub>@NCNPs-n.

Fe-O			
Sample	Binding Energy (eV)	FWHM	%Area
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-0.5	530.5	1.3	34.2
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.0	530.6	1.3	31.3
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.5	530.3	1.5	33.3
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-2.0	530.6	1.4	33.3

OVs			
Sample	Binding Energy (eV)	FWHM	%Area
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-0.5	531.9	1.6	37.5
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.0	531.8	1.7	34.4
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.5	532.2	1.8	41.7
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-2.0	531.9	1.7	30.0

O <sub>2</sub>			
Sample	Binding Energy (eV)	FWHM	%Area
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-0.5	533.1	1.3	19.8
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.0	533.0	1.3	25.0
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.5	533.0	1.5	20.8
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-2.0	533.0	1.4	26.7

H-O-H			
Sample	Binding Energy (eV)	FWHM	%Area
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-0.5	534.4	1.4	8.5
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.0	534.4	1.4	9.3
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.5	534.8	1.5	4.2
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-2.0	534.3	1.4	10.0

**Table S4** The curve fitting information of **Fe 2p** for Fe<sub>2</sub>O<sub>3</sub>@NCNPs-n.

Fe 2p3/2-Fe <sup>2+</sup>			
Sample	Binding Energy	FWHM	%Area
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-0.5	710.6	1.5	22.1
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.0	710.5	1.5	19.2
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.5	710.4	1.5	21.7
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-2.0	710.5	1.6	14.3

Fe 2p3/2-Fe <sup>3+</sup>			
Sample	Binding Energy	FWHM	%Area
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-0.5	712.6	1.7	26.5
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.0	712.4	1.7	28.9
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.5	712.4	1.7	18.1
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-2.0	712.1	1.8	32.9

Fe 2p1/2-Fe <sup>2+</sup>			
Sample	Binding Energy	FWHM	%Area
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-0.5	724.1	1.7	16.2
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.0	723.9	1.7	11.5
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.5	724.2	1.7	13.3
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-2.0	723.9	1.8	9.6

Fe 2p1/2-Fe <sup>3+</sup>			
Sample	Binding Energy	FWHM	%Area
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-0.5	727.0	1.9	12.9
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.0	726.9	1.8	16.6
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.5	726.8	1.8	10.2
Fe <sub>2</sub> O <sub>3</sub> @NCNPs-2.0	726.9	1.8	18.3

**Table S5** The pore structure parameters of Fe<sub>2</sub>O<sub>3</sub>@NCNPs-1.5 nanomaterial.

Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.5	
S <sub>BET</sub> <sup>a</sup> (m <sup>2</sup> /g)	292
S <sub>micro</sub> <sup>b</sup> (m <sup>2</sup> /g)	30
V <sub>total</sub> <sup>c</sup> (cm <sup>3</sup> /g)	0.505
V <sub>micro</sub> <sup>d</sup> (cm <sup>3</sup> /g)	0.014
P <sup>e</sup> (nm)	6.92

- a.** The surface area (S<sub>BET</sub>) calculated by the Brunauer-Emmett-Teller (BET) method.
- b** and **d.** The surface area (S<sub>micro</sub>) and volume (V<sub>micro</sub>) of micropore calculated by the t-plot equation.
- c.** The total pore volume calculated by the N<sub>2</sub> adsorption/desorption isotherm at P/P<sub>0</sub>=0.95.
- e.** The main pore size.

**Table S6** The LOD and LOQ value of individual detection and simultaneous detection of  $\text{Pb}^{2+}$  and  $\text{Cd}^{2+}$  on  $\text{Fe}_2\text{O}_3@\text{NCNPs-1.5/GCE}$ .

Analyte		LOD (nM)	LOQ (nM)
Individual detection	$\text{Pb}^{2+}$	27.45	94.47
	$\text{Cd}^{2+}$	38.20	118.88
Simultaneous detection	$\text{Pb}^{2+}$	4.92	15.15
	$\text{Cd}^{2+}$	18.79	61.75

**Table S7** Comparison of the electrochemical performance of individual detection and simultaneous detection of Pb<sup>2+</sup> and Cd<sup>2+</sup> on Fe<sub>2</sub>O<sub>3</sub>@NCNPs-1.5/GCE and Fe<sub>2</sub>O<sub>3</sub> NPs/GCE at the same testing conditions.

	Analyte	Fe <sub>2</sub> O <sub>3</sub> NPs/GCE	Fe <sub>2</sub> O <sub>3</sub> @NCNPs-1.5/GCE
Simultaneous determination	LOD (nM)	Pb <sup>2+</sup> Cd <sup>2+</sup>	10.99 41.09
Individual determination	LOD (nM)	Pb <sup>2+</sup> Cd <sup>2+</sup>	9.48 38.31
			4.92 18.79 27.45 38.20