

Electronic Supplementary Information

A Zn-nitrite battery as an energy-output electrocatalytic system for high-efficiency ammonia synthesis by carbon-doped cobalt oxide nanotubes

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S1. Experiment section

Chemicals

Potassium sulfate (K_2SO_4 , 99%), potassium nitrite (KNO_2 , 99.99%), hydrazine monohydrate ($\text{N}_2\text{H}_4\cdot\text{H}_2\text{O}$, > 98%), Deuterium oxide (D_2O , 99.9 atom % D), hydrochloric acid (HCl , 37%), sodium hydroxide (NaOH , > 98%), cobalt chloride hexahydrate ($\text{CoCl}_2\cdot 6\text{H}_2\text{O}$, > 99%), sodium oxalate ($\text{Na}_2\text{C}_2\text{O}_4$, 99%), hydrogen peroxide (H_2O_2 , 30%), sodium hypochlorite solution (NaClO , available chlorine 4.0 %), p-Dimethylaminobenzaldehyde ($\text{C}_9\text{H}_{11}\text{NO}$), salicylic acid ($\text{C}_7\text{H}_6\text{O}_3$, 99%), ethanol ($\text{C}_2\text{H}_5\text{OH}$, 99.7%), polyvinyl pyrrolidone (PVP), trisodium citrate ($\text{C}_6\text{H}_5\text{Na}_3\text{O}_7$, 98%), sodium nitroferricyanide dehydrate ($\text{C}_5\text{FeN}_6\text{Na}_2\text{O}\cdot 2\text{H}_2\text{O}$, 99%), ethylene glycol ($\text{C}_2\text{H}_8\text{O}_2$, 99%) were purchased from Shanghai Macklin Biochemical Co., Ltd. W1S1005 carbon cloth (CC) was purchased from Fuel Cell Store Ltd. Bipolar membrane (Fumasep® FBM) was purchased from Fumatech GmbH. A commercial Nafion 117 membrane and typical Nafion solution (5 wt.%) was purchased from DuPont™ (E. I. du Pont de Nemours and Company).

Synthesis of the $\text{C}_2\text{O}_4^{2-}$ doped CoC_2O_4 precursor

Typically, 2 mmol of $\text{CoCl}_2\cdot 6\text{H}_2\text{O}$ and 8 mmol of $\text{Na}_2\text{C}_2\text{O}_4$ were dissolved in 40 mL of ethylene glycol to form a homogeneous solution under continuous magnetic stirring. The solution was then transferred to a 50 mL Teflon-lined autoclave. The reactor was sealed and kept at 200 °C for 16 h in the oven. After the reaction was completed, the

pink-colored precipitate was collected by centrifugation, washed with deionized water and ethanol for several times, and dried in a vacuum oven at 60 °C for 12 h.

Synthesis of the Co₃O₄ and C/Co₃O₄

Briefly, 2 g of PVP ($M_w = 1\,300\,000$) was added into 30 mL of ethanol. After 1 h stirring, 0.4 g of the obtained C₂O₄²⁻ doped CoC₂O₄ was dispersed into the above solution. After 12 h stirring, the C₂O₄²⁻ doped CoC₂O₄/PVP complex was harvested by centrifugation and dried at 60 °C for 12 h. To obtain C/Co₃O₄, the C₂O₄²⁻ doped CoC₂O₄/PVP complex was transferred into a box furnace and annealed in air at 400 °C for 2 h with a heating rate of 2 °C min⁻¹. Undoped Co₃O₄ was prepared by calcinating C₂O₄²⁻ doped CoC₂O₄ nanorods directly in air at 400 °C for 2 h.

Characterization

The crystalline, morphologies and microstructures of samples were investigated by XRD using a Bruker D2 Phaser diffractometer with Cu K α irradiation ($\lambda = 1.54\text{ \AA}$), field-emission scanning electron microscopy (FEI Quanta 450 FEG) and JEOL-2001F field-emission TEM (JEOL-2001F). The surficial chemical states and compositions of the as-obtained products were investigated by XPS (ESCALB 250) with an Al K α X-ray beam ($E = 1486.6\text{ eV}$). ¹H-NMR measurements were performed on Bruker 400MHz ASCEND AVANCE III HD Nuclear Magnetic Resonance System (NMR-400). UV-Vis spectroscopy measurements were carried out using a UV/Vis Spectrometer DB-20. FTIR measurements were carried out using Spectrum Two FTIR Spectrometers by PerkinElmer. KPFM experimental results were obtained by Bruker Dimension Icon.

Computational Details

All the computations were conducted based on the density functional theory (DFT) using the Cambridge Sequential Total Energy Package (CASTEP) code of the Materials Studio 2018 software. The generalized gradient approximation (GGA) with the Perdew-Burke-Ernzerhof (PBE) functional were used to describe the electronic exchange and correlation effects. The kinetic-energy cutoff was set as 500 eV. The geometry optimization within the conjugate gradient method was performed with forces on each atom less than 0.05 eV/Å. An energy tolerance is 5.0×10^{-6} eV per atom, and a maximum displacement of 0.001 Å was considered. A Co_3O_4 (311) surface with a three-layer model of the crystal plane composed of 54 atoms was used for calculation with a sufficient vacuum gap of 15 Å. Bottom atomic Co-O layer was fixed while other layers and the adsorbates were fully relaxed during structural optimizations. The free energies for each reaction were given after correction: $\Delta G = \Delta E - T\Delta S$. Where ΔE is the reaction energy obtained by the difference between reactants and products; ΔS is the change in entropy for each reaction. Entropy values are taken from the standard database in the NIST webbook (J. Phys. Chem. Ref. Data 1996, 25, 551- 603.). The entropies of adsorbate and adsorption site are negligible.

Electrochemical measurements

Before the NO_2 -RR tests, the Nafion 117 membrane was pretreated in 5 wt% H_2O_2 aqueous solution at 80 °C for 1 h and then in ultrapure water at 80 °C for another 1 h. All electrochemical measurements were carried out in the argon atmosphere. The CC-supported catalysts ($1 \times 1 \text{ cm}^2$), Ag/AgCl (3 M KCl), and graphite rod were used as the

work electrode, the reference electrode and the counter electrode, respectively. For the preparation of the working electrode, 10 mg of samples was dispersed in 0.95 mL of isopropyl alcohol and 50 μL of 0.25% Nafion solution. The mixed solution was sonicated for 30 min to form a homogeneous ink. 50 μL of the catalyst ink was drop cast onto CC ($1 \times 1 \text{ cm}^2$) and dried at room temperature with a loading mass of 0.5 mg/cm^2 . All electrochemical tests were carried out at ambient conditions. LSV tests were conducted with the scan rate of 5 mV/s in the three-electrode system. The chronoamperometry tests were conducted at a series of applied potentials in a typical H-type cell that contains 50-mL electrolyte separated by a membrane (Nafion 117) on the electrochemical workstation (CHI 760E, Chenhua, China). All potentials in this work refer to RHE, $E(\text{RHE}) = E(\text{Ag}/\text{AgCl}) + 0.222 + 0.059\text{pH}$. Different sweep rates in cyclic voltammetry measurements were performed and a linear relation was demonstrated on the basis of the current densities ($J_{\text{anodic}} - J_{\text{cathodic}}$) at the midpoint of the sweep voltage interval against scan rate. The C_{dl} was then obtained by halving the slope and the ECSA could be calculated by the following formula: $\text{ECSA} = (C_{\text{dl}}/C_s) \times S$ in which C_s and S respectively represent the specific capacitance (0.04 mF cm^{-2}) and the area of electrode (1 cm^2).

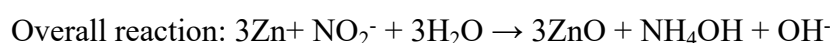
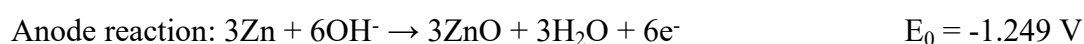
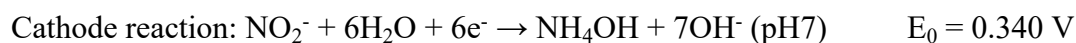
Assembly of the Zn-NO_2^- battery and electrochemical test.

The CC-supported catalysts (1 cm^2) and Zn plate (1 cm^2) were employed as the cathode for zinc-nitrate battery. A typical H-type cell that contains 20-mL cathode electrolyte ($0.5 \text{ M K}_2\text{SO}_4 + 50 \text{ mM KNO}_2$) and 20-mL anode electrolyte (1 M KOH). Considering our Zn-NO_2^- battery has an alkali anodic electrolyte and a neutral cathodic electrolyte,

a bipolar membrane consisting of an anion exchange layer and a cation exchange layer is used in the Zn-nitrite battery, where H^+ and OH^- are carriers at the cathode and anode, respectively. H^+ and OH^- ions migrate along the electric field to the corresponding electrodes and are consumed. The discharging polarization curves with a scan rate of 5 mV/s and galvanostatic tests were conducted using a CHI 760E workstation and Land 2001A battery test system at room temperature, respectively. After electrochemical test, the electrolyte was diluted for the next detection.

The power density (P) of zinc-nitrate battery was determined by $P = I \times V$, where I and V are the discharge current density and voltage, respectively.

The electrochemical reactions in Zn-nitrite battery are presented as following:



$$E_{\text{Zn-nitrite battery}} = 1.089 \text{ V} \quad \text{pH14}$$

$$E_{\text{Zn-nitrite battery}} = 1.589 \text{ V} \quad \text{pH7}$$

$$E_{\text{Zn-nitrite battery}} = 2.146 \text{ V} \quad \text{pH0}$$

Though the Zn- NO_2^- battery system can simultaneously realize the NH_3 synthesis and electricity supply, the Zn dissolution and depletion of NO_2^- in the electrolyte will make the battery system less robust. To overcome the first problem, we can design a rechargeable Zn- NO_2^- battery to recycle Zn in the anodic chamber. However, the

oxygen evolution reaction ($4\text{OH}^- \rightarrow \text{O}_2 + 2\text{H}_2\text{O} + 4\text{e}^-$) and nitrite oxidation reaction ($\text{NO}_2^- + 2\text{OH}^- \rightarrow \text{NO}_3^- + \text{H}_2\text{O} + \text{e}^-$) may simultaneously occur in the cathode. The generated NO_3^- can then be reduced to NH_3 over the $\text{C}/\text{Co}_3\text{O}_4$ when discharging. Therefore, a selective electrocatalyst should be rationally designed. For the second problem, the flow system with continuously stirring can be introduced a steady NO_2^- concentration and a steady current density. Future research should be focused on designing and preparing efficient and selective electrocatalysts applied in a flow Zn- NO_2^- battery system.

Determination of ammonia

The amount of the produced NH_3 was detected with the indophenol blue method. To minimize the experimental error in quantification of ammonia, all data was recorded three times to obtain the error bar. In detail, a certain amount of electrolyte was taken out after NO_2^- -RR test and diluted with electrolyte, and 1.25 mL of solution containing 0.625 M NaOH, 0.36 M salicylic acid and 0.17 M sodium citrate was added. Then 150 μL sodium nitroferricyanide solution (10 mg/mL) and 75 μL NaClO (available chlorine 4.0 wt%) solution were added. After 2 h under ambient conduction, UV-Vis absorption spectrum was recorded, and the absorbance value was obtained at the wavelength of 658 nm. The standard NH_4^+ solutions with the given concentrations of $(\text{NH}_4)_2\text{SO}_4$ in 0.5 M K_2SO_4 were prepared for building the calibration curve. The amount of the produced NH_3 was also determined by ^1H NMR. The pH value of the electrolyte after NO_2^- -RR test was adjusted by 2 mol/L H_2SO_4 to achieve a weak acid. Then, 400 ppm of $\text{C}_4\text{H}_4\text{O}_4$ was added into the solution for a quantitative determination. Subsequently,

adding 50 μL D_2O into 0.5 mL of above mixture for NMR detection. Finally, the peak area ratio of NH_4^+ and $\text{C}_4\text{H}_4\text{O}_4$ was recorded and the concentration could be determined according to the standard calibration curves. The calibration curve was obtained by collecting the peak area ratios of NH_4^+ with given various concentrations and $\text{C}_4\text{H}_4\text{O}_4$ in 0.5 M K_2SO_4

Determination of hydrazine

Hydrazine in the electrolytes was also detected by the Watt-Chrisp method. A mixture of ethanol (100 mL), para(dimethylamino) benzaldehyde (2.0 g) and HCl (concentrated, 12 mL) were used as a color reagent. 2-mL color reagent was added into 2-mL of diluted electrolyte. After 30 min, the absorbance was measured at a wavelength of 458 nm. The standard hydrazine monohydrate solutions with the given concentrations of hydrazine in 0.5 M K_2SO_4 were also prepared for building the calibration curves.

Faradaic efficiency and ammonia yield rate

FEs and area-normalized yield rates of NH_3 , NO_2^- , and N_2H_4 were calculated:

$$\text{FE}(\text{NH}_3) = (6F \times C \times V \times n) / Q$$

$$\text{Yield rate}(\text{NH}_3) = (C \times V \times n) / (t \times A)$$

$$\text{FE}(\text{N}_2\text{H}_4) = (12F \times C \times V \times n) / Q$$

$$\text{Yield rate}(\text{N}_2\text{H}_4) = (C \times V \times n) / (t \times A)$$

Where F is the Faraday constant (96485 C/mol), C is the measured NH_3 concentration, V is the volume of electrolyte, Q is the total charge passed through the electrode, n is the dilution factor and A is the geometric area of the working electrode ($1 \times 1 \text{ cm}^2$).

S2. Fig.s and tables

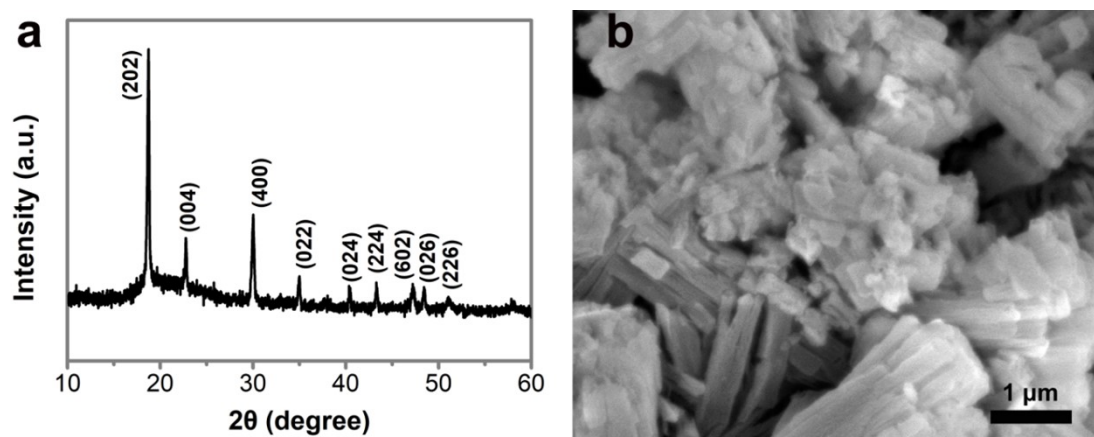


Fig. S1. XRD pattern and SEM image of $\text{C}_2\text{O}_4^{2-}$ -doped CoC_2O_4 .

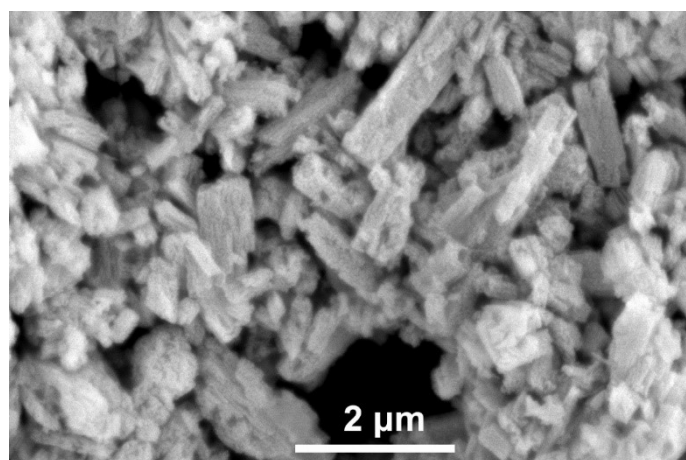


Fig. S2. SEM image of the $\text{C}/\text{Co}_3\text{O}_4$.

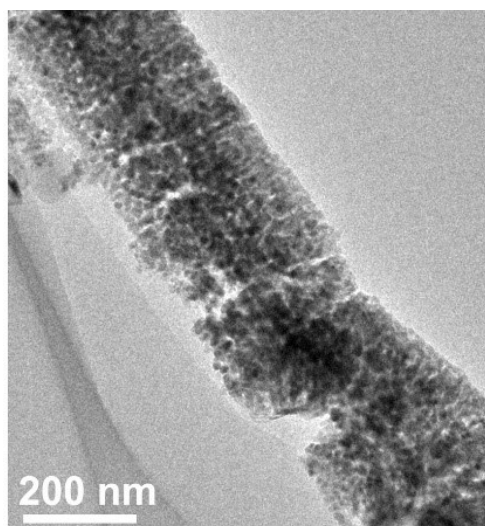


Fig. S3. TEM images of the Co_3O_4 .

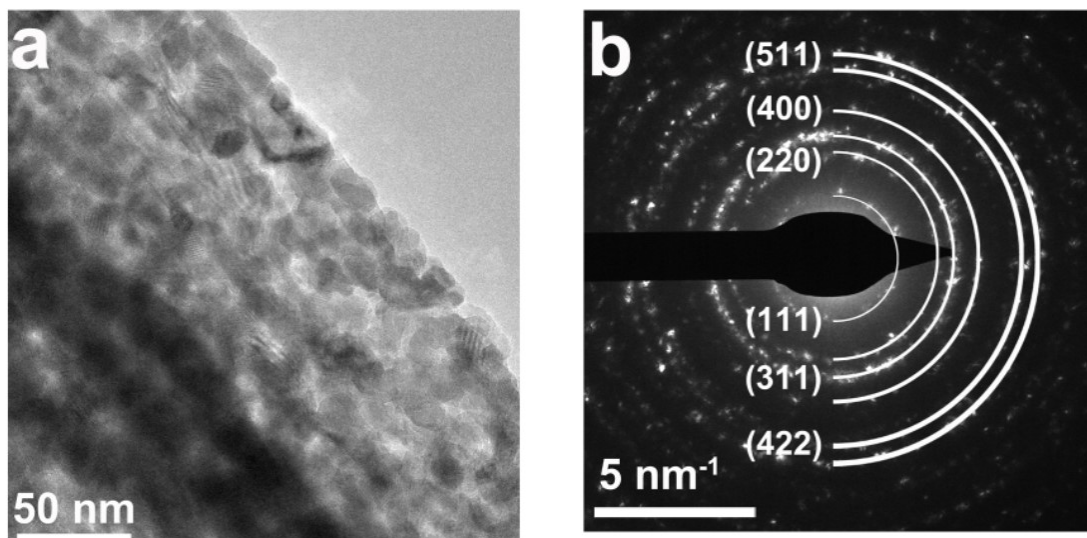


Fig. S4. (a) TEM images of the $\text{C}/\text{Co}_3\text{O}_4$ shell. (b) SEAD pattern of the $\text{C}/\text{Co}_3\text{O}_4$ nanocrystal.

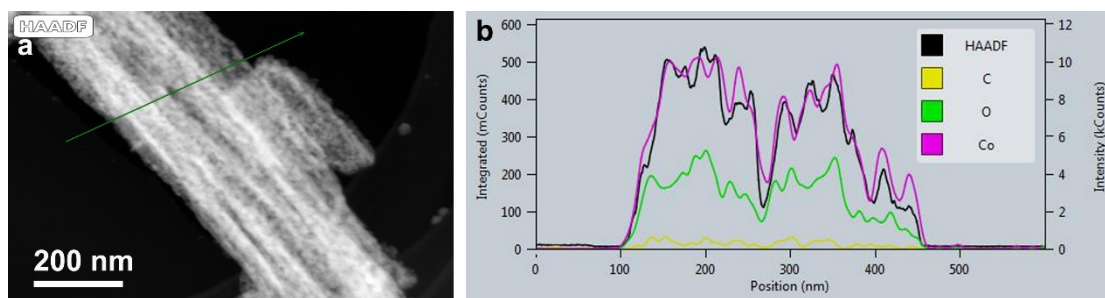


Fig. S5. EDS compositional line profile of C/Co₃O₄.

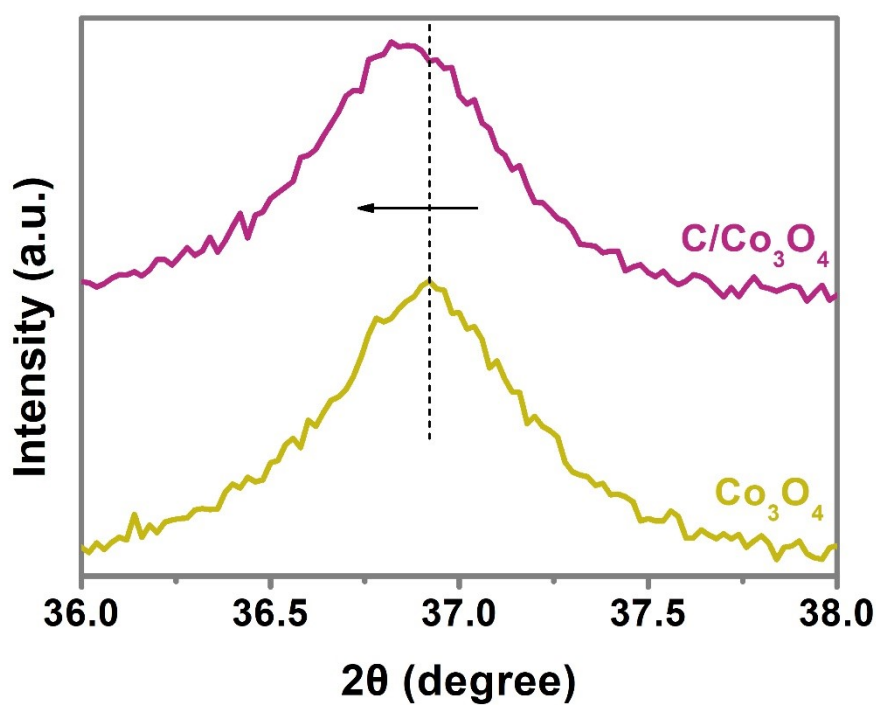


Fig. S6. Magnified view of the XRD patterns for doped and undoped Co₃O₄.

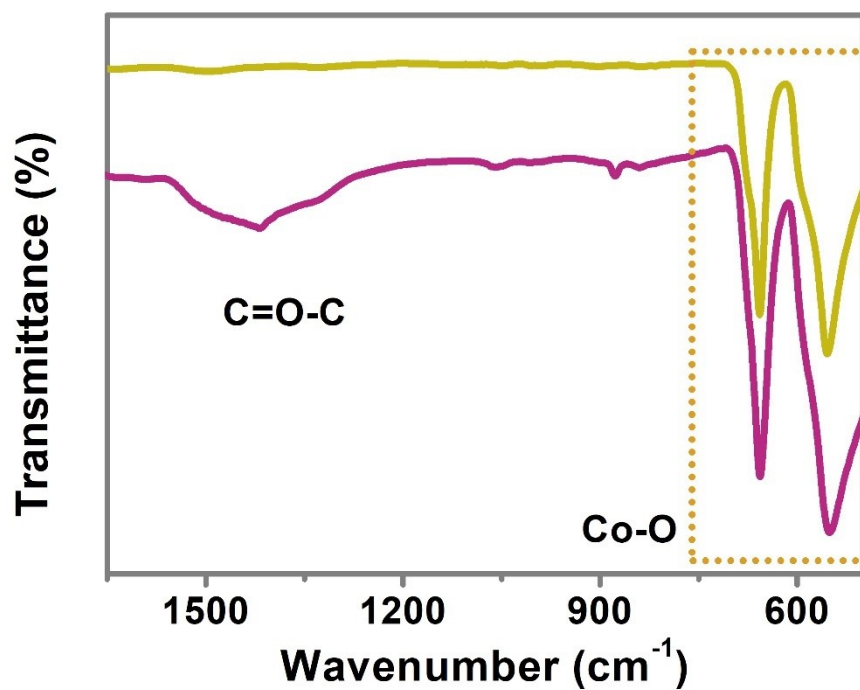


Fig. S7. FTIR spectra of the undoped Co₃O₄ and C-doped Co₃O₄.

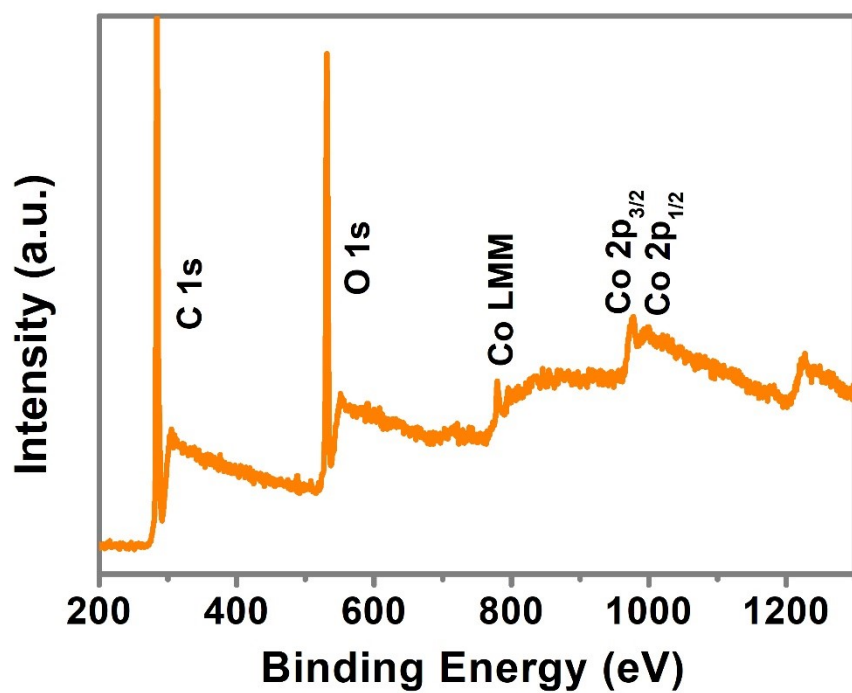


Fig. S8. XPS survey spectrum of the C/Co₃O₄.

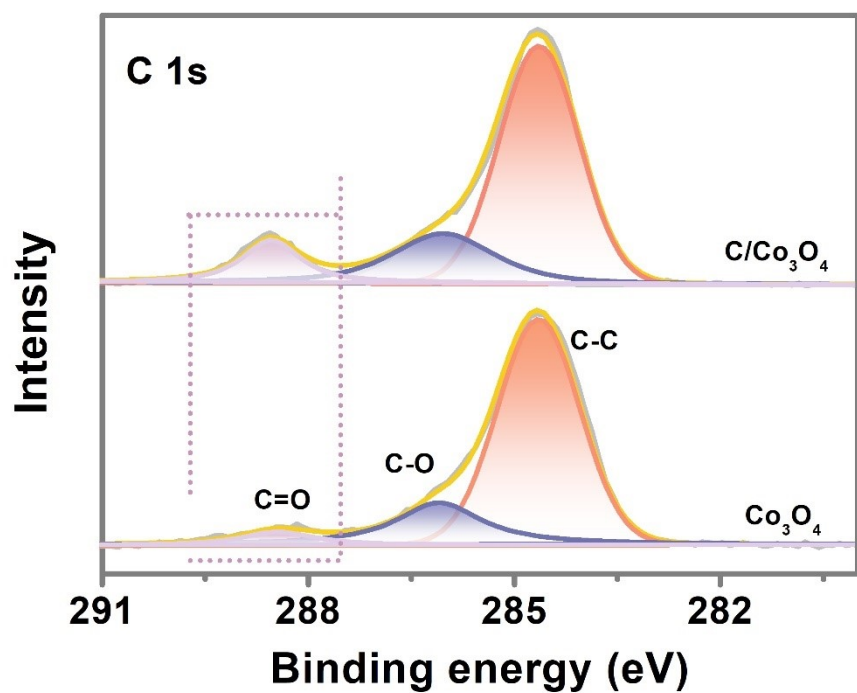


Fig. S9. XPS spectrum of the C/Co₃O₄ and Co₃O₄ in C 1s region.

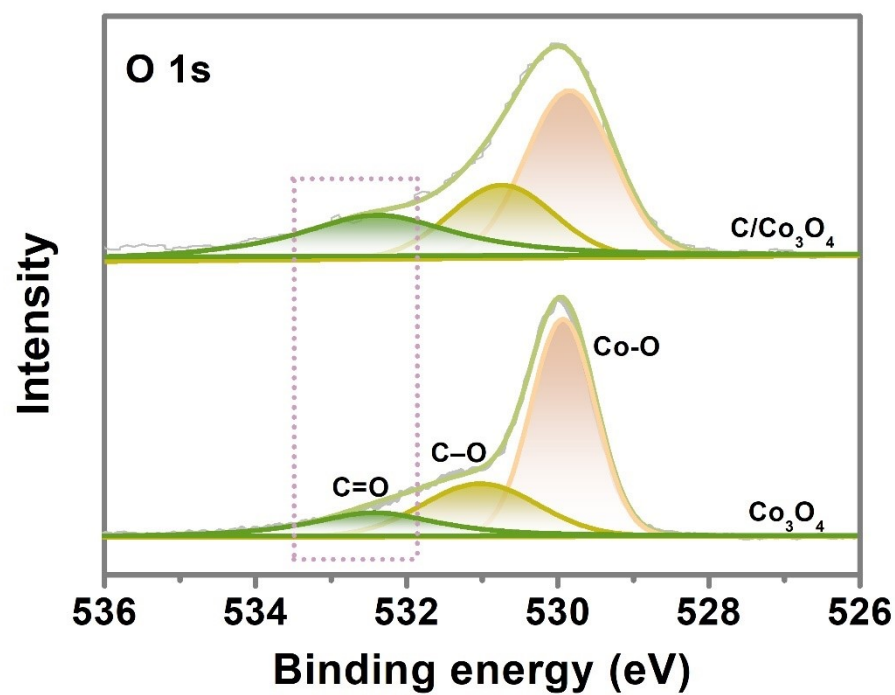


Fig. S10. XPS spectrum of the C/Co₃O₄ and Co₃O₄ in O 1s region.

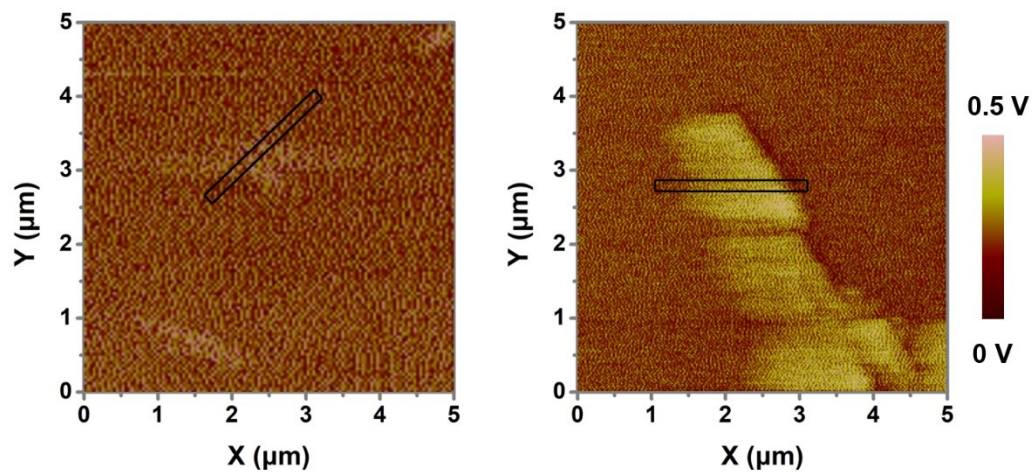


Fig.

S11. KPFM images of the Co_3O_4 (left) and the $\text{C}/\text{Co}_3\text{O}_4$ (right).

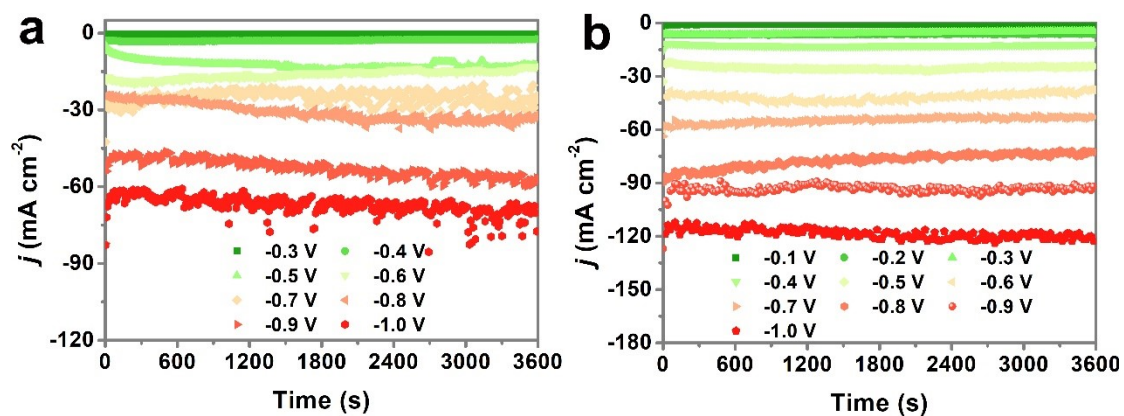


Fig. S12. Chronoamperometry curves of (a) Co_3O_4 and (b) $\text{C}/\text{Co}_3\text{O}_4$ in 0.5 M K_2SO_4 electrolyte with 50 mM KNO_2 at different potentials.

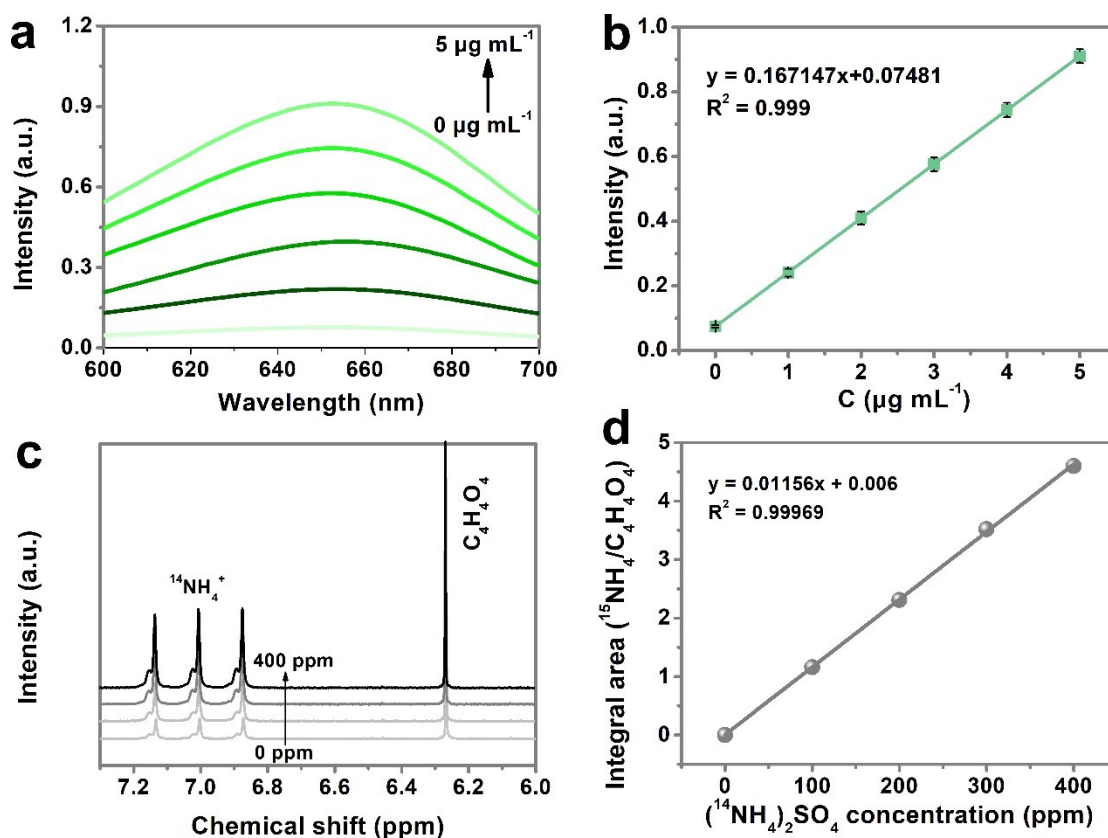


Fig. S13. (a) The UV-Vis adsorption curves and (b) calibration curve of the electrolyte with the given concentrations of $(\text{NH}_4)_2\text{SO}_4$. (c) ^1H NMR spectra of standard sample $(\text{NH}_4)_2\text{SO}_4$ solution with different concentrations. (d) Calibration curve of the electrolyte with the given concentrations of $(\text{NH}_4)_2\text{SO}_4$.

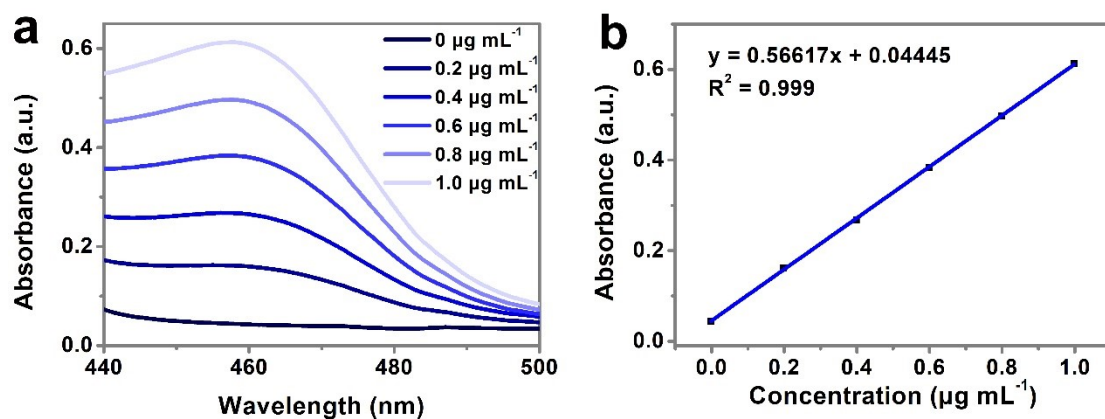


Fig. S14. (a) The UV-Vis adsorption curves and (b) Calibration curve of the electrolyte with the given concentrations of N_2H_4 .

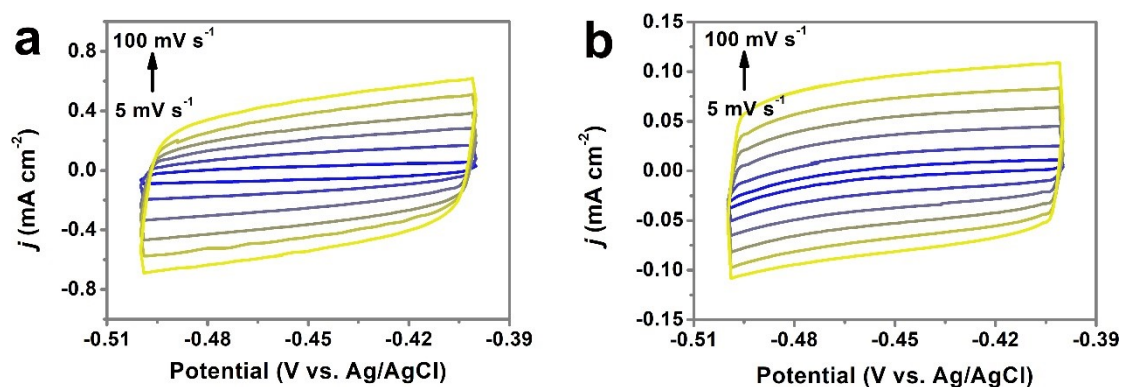


Fig. S15. The cyclic voltammetry profiles obtained on the (a) Co_3O_4 and (b) C/Co_3O_4 at the sweep rates of 5, 20, 40, 60, 80 and 100 $mV s^{-1}$.

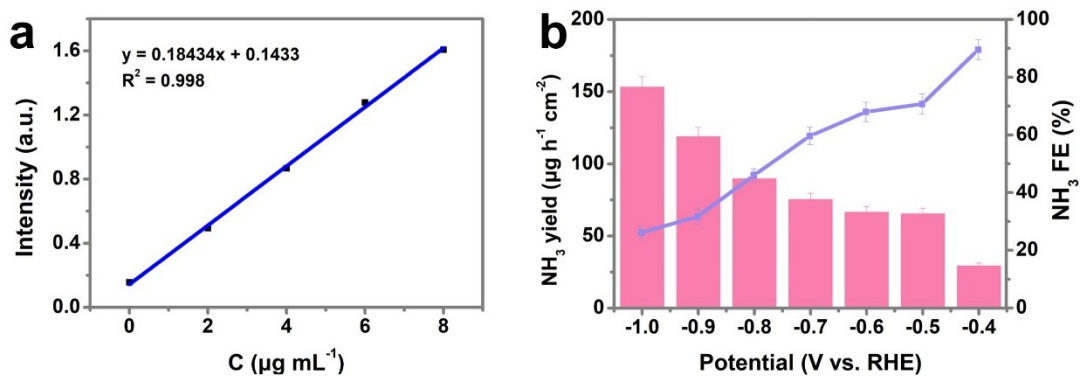


Fig. S16. Calibration curve in NH₃ measurement with different (NH₄)₂SO₄ concentration. (b) NH₃ yield and the corresponding FE of the C/Co₃O₄ catalyst for the NO₂⁻RR in 0.5 M K₂SO₄ + 200 ppm NO₂⁻.

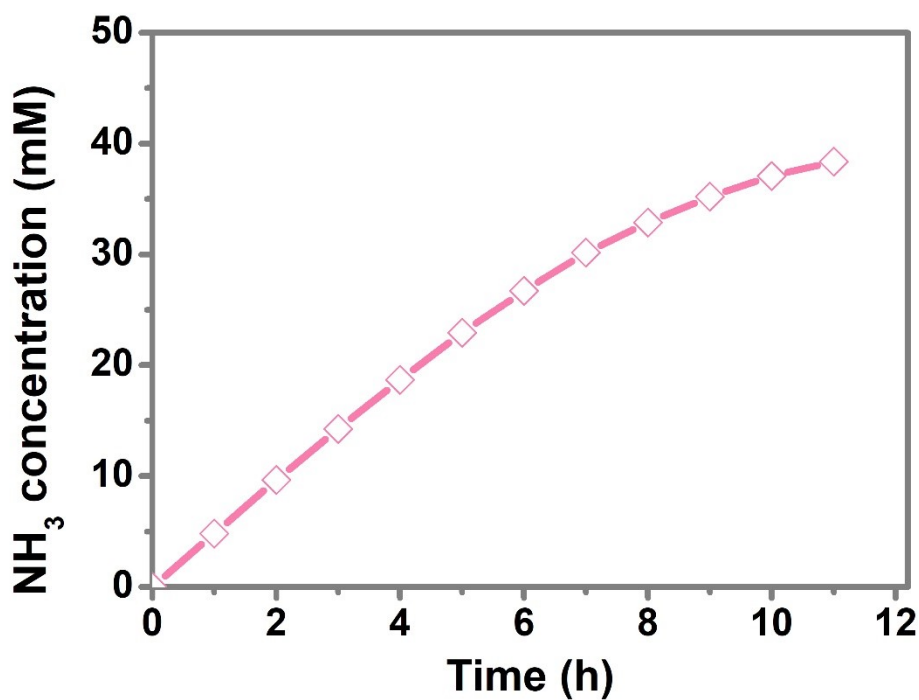


Fig. S17. The NH₃ concentration of the electrolyte after consecutive 12-h electrolysis.

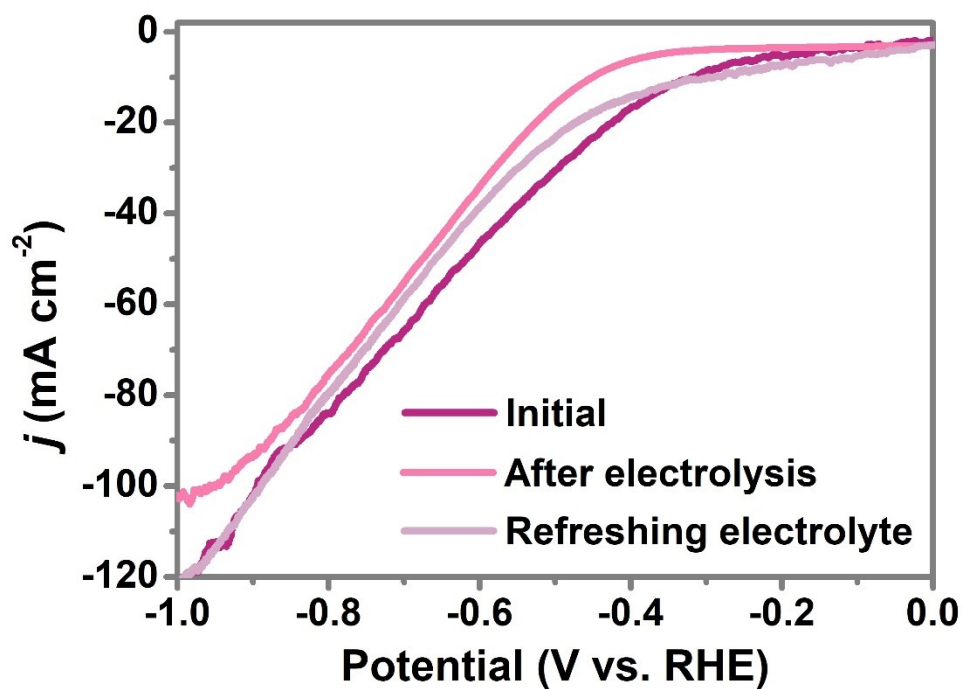


Fig. S18. LSV curves of the C/Co₃O₄ under different test conditions.

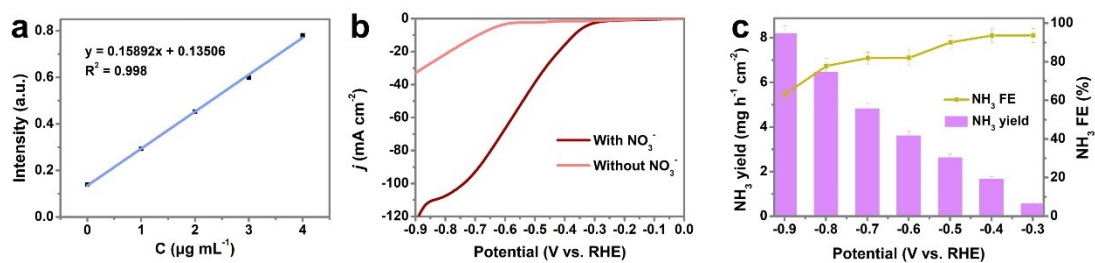


Fig. S19. (a) Calibration curve in NH₃ measurement with different (NH₄)₂SO₄. (b) Polarization curves of the C/Co₃O₄ in 0.5 M K₂SO₄ electrolyte with or without 50 mM NO₃⁻. (c) NH₃ yield and NH₃ FE of the C/Co₃O₄ in 0.5 M K₂SO₄ with 50 mM NO₃⁻ electrolyte.

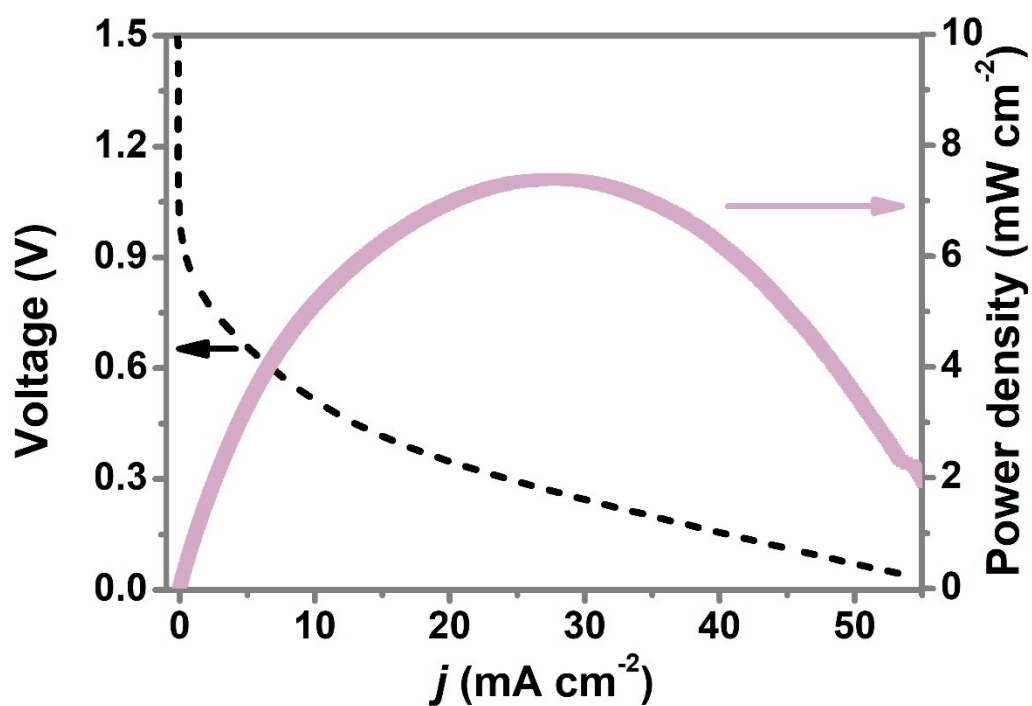


Fig. S20. Discharge polarization curves and the corresponding power density of the C/Co₃O₄-based Zn-NO₂⁻ battery with a Nafion 117 membrane.

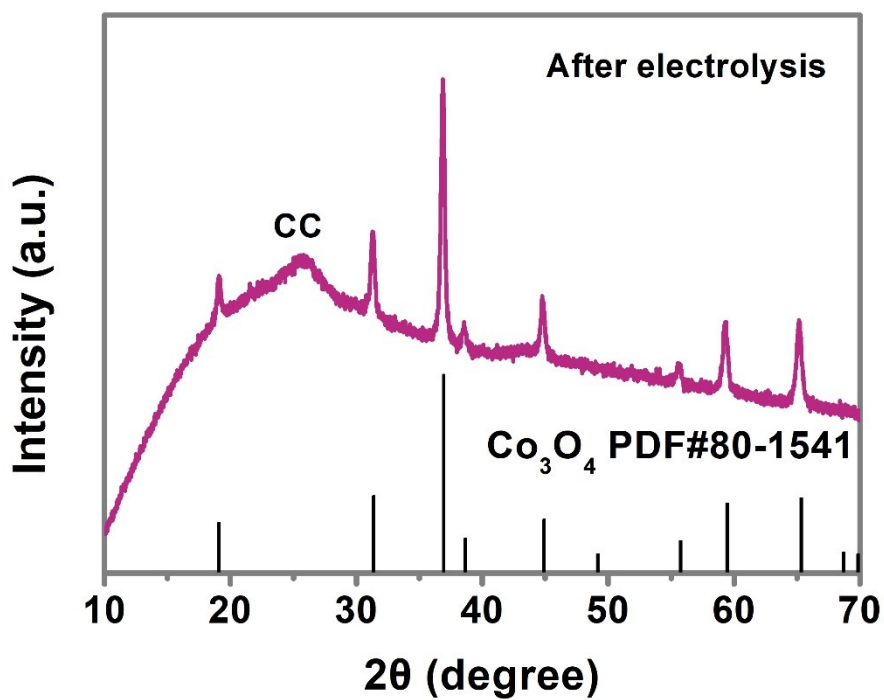


Fig. S21. XRD pattern of the C/Co₃O₄ after stability operation.

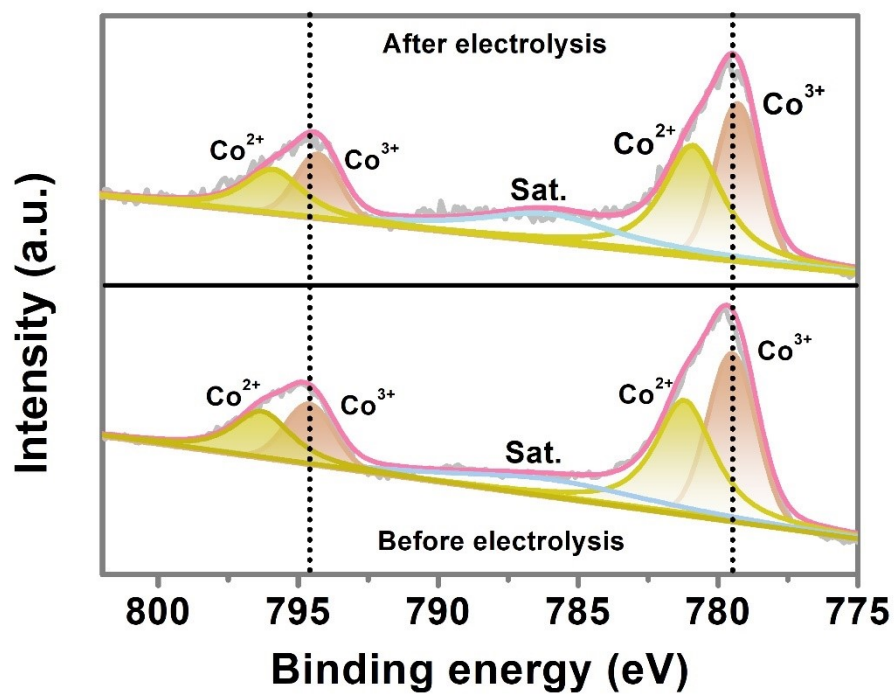


Fig. S22. XPS spectra of the C/Co₃O₄ in the Co 2p region before and after stability operation.

Supporting Information

Table S1. The comparison of NH₃ synthesis performance of NRR electrocatalysts and C/Co₃O₄.

Reactions	Catalysts	NH ₃ FE (%)	NH ₃ Yield (mg·cm ⁻² ·h ⁻¹)	Ref.
NRR	B ₄ C	15.95	25.67	[1]
	W ₁₈ O ₄₉ -16Fe	20	24.7	[2]
	Cu-TiO ₂	21.99	21.31	[3]
	SA Ru-Mo ₂ CT _x	25.77	40.57	[4]
	CoPc NTs	27.7	107.9	[5]
	Ru SAs/N-C	29.6	120.9	[6]
	Au nanocages	30.2	2.3	[7]
	CNT@C ₃ N ₄ -Fe&Cu	34	5.75	[8]
	Pd cubes	36.6	24.3	[9]
	RuFeCoNiCu/C	38.5	57.1	[10]
	PdH	43.6	0.0204	[11]
	Single Fe/N-C	56.55	7.48	[12]
	CN/C600	62.1	1.7	[13]
NO ₂ -RR	C/Co ₃ O ₄	100	4.175	This work

Supporting Information

Table S2. The comparison of NH_3 synthesis performance of NO_3^- -RR electrocatalysts and $\text{C}/\text{Co}_3\text{O}_4$.

Reaction	Catalysts	NH_3 FE (%)	NH_3 Yield ($\text{mg}\cdot\text{cm}^{-2}\cdot\text{h}^{-1}$)	Ref.
	CBTDA/Ag-Pd	90	0.006	[14]
NO_3^- -RR	Cu/Cu ₂ O NWAs	81.2	0.1633	[15]
	Co ₃ O ₄ /Ti	80	0.634	[16]
	TiO _{2-x}	85	0.765	[17]
	Cu@Cu ₂₊₁ O NWs	87.07	0.57653	[18]
	Pd/TiO ₂	92.1	1.12	[19]
	Fe SAC	75	~1.955	[20]
	Ti	82	1.58	[21]
	OD-Ag	89	~1.25	[22]
	CoP PANSs	94	2.2	[23]
	Fe-PPy SACs	100	2.75	[24]
	Co/CoO NSAs	91.2	3.298	[25]
	Cu/Cu-Mn ₃ O ₄	87.6	3.57	[26]
	Cu@Th-BPYDC	92.5	3.83	[27]
NO_2^- -RR	C/Co₃O₄	100	4.175	This work

Supporting Information

	Ni-NSA- V_{Ni}	89	0.236	[29]
	Cu_3P NA/CF	91.2	1.626	[30]
	CoP nanoarray	90	2.26	[31]
	Ni_2P/NF	90.2	2.6922	[32]
	Co@JDC	96.9	47.6	[33]
	FeOOH NTA/CC	94.7	11.937	[34]
NOR+NO _x R R	Co SAs/N-C	100	1.43	[35]
	$Ni_3B@NiB_{2.74}$	100	3.371	[36]
NORR	MoS ₂ nanosheet	76.6	1.69	[37]
	$Ru_{0.05}Cu_{0.95}$	64.9	0.3	[38]
	Single atom Nb	77.1	5.018	[39]
	FeNC	5.1	0.343	[40]
	Cu foil	61.9	1.69	[41]
NO₂-RR	C/Co₃O₄	99.5	4.175	This work

Table S3. The comparison of NH₃ synthesis performance of NO₂-RR, NOR+NO_xRR and NORR electrocatalysts with C/Co₃O₄.

Supporting Information

Table S4. The comparison of NH₃ synthesis performance of NORR electrocatalysts and C/Co₃O₄.

Batteries	Cathode	OCV (V)	FE(%)	NH ₃ Yield (mg·cm ⁻² ·h ⁻¹)	Power density (mW cm ⁻²)	Ref.
Na-N ₂	α-MnO ₂ nanowire	-	26	-	-	[42]
Al-N ₂	graphene/Pd	-	51.2	0.01084	-	[43]
Li-N ₂	carbon cloth	-	59	-	-	[44]
Li-N ₂	Mo ₂ C/NC	-	87	-	-	[45]
Zn-N ₂	metallic copper	~0.5	59	-	0.0101	[46]
Zn-N ₂	VN@NSC-900	~0.55	-	0.000172	0.01642	[47]
Zn-N ₂	NbS ₂	~1.3	-	0.0036	0.31	[48]
Zn-N ₂	CoPi/NPCS	~1.4	16.35	0.0147	0.49	[49]
Zn-N ₂	Fe _{1.0} HTNs	~0.6	-	0.000137	0.02765	[50]
Zn-N ₂	Vs-FePS3 NSs	~1.75	-	-	2.6	[51]
Zn-N ₂	CoPi/HSNPC	~1	24.42	-	0.31	[52]
Zn-NO ₃ ⁻	Pd/TiO ₂	0.81	81.3	0.54	0.87	[53]
Zn-NO	MoS ₂ nanosheets	2.03	85.0	0.4118	1.04	[54]
Zn-NO₂⁻	C/Co₃O₄	1.45	95.1	0.802	6.03	This work

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