

## Supplementary Information

### Experimental section

**Materials:** Sodium Tungstate Dihydrate ( $\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$ ), Copper chloride hexahydrate ( $\text{CuCl}_2 \cdot 6\text{H}_2\text{O}$ ), Anhydrous sodium sulfate ( $\text{Na}_2\text{SO}_4$ ), Sodium nitrite ( $\text{NaNO}_2$ ), acetone ( $\text{C}_3\text{H}_6\text{O}$ ), anhydrous ethanol ( $\text{C}_2\text{H}_6\text{O}$ ), ammonium chloride ( $\text{NH}_4\text{Cl}$ ), sodium hydroxide ( $\text{NaOH}$ ), salicylic acid ( $\text{C}_7\text{H}_6\text{O}_3$ ), sodium citrate dihydrate ( $\text{C}_6\text{H}_5\text{Na}_3\text{O}_7 \cdot 2\text{H}_2\text{O}$ ), p-dimethylamino benzaldehyde ( $\text{C}_9\text{H}_{11}\text{NO}$ ), and sodium nitroferricyanide dihydrate ( $\text{C}_5\text{FeN}_6\text{Na}_2\text{O} \cdot 2\text{H}_2\text{O}$ ) were purchased from Chengdu Kelong Ltd.

**Preparation of  $\text{CuWO}_4$ :** Typically, 1mmol of  $\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$  and 1mmol of  $\text{CuCl}_2 \cdot 6\text{H}_2\text{O}$  were dissolved in 30 mL of deionized water, and stirred for one hour. Finally, the solution was transferred to a 50 ml sealed Teflon-lined stainless steel autoclave. Then the autoclave was kept at 180 °C for 16 h. After the autoclave cooled down to room temperature, the catalysts were taken out and washed with water and ethanol several times, and subsequently dried at 60 °C overnight. The catalyst was then calcined in air at 500 °C for 2 h. Finally, the catalyst  $\text{CuWO}_4$  was obtained.

**Preparation of  $\text{CuO}$ :** Typically, dissolve 5 mmol of copper nitrate and 30 mmol of urea in 40 mL of deionized water, stirring for one hour. The solution is then transferred into a 100 mL sealed stainless steel autoclave lined with polytetrafluoroethylene. The autoclave is subsequently maintained at 90°C for 14 h. After cooling to room temperature, the catalyst is removed, washed multiple times with water and ethanol, and dried overnight at 60°C. It is then calcined at 300°C for 2 h in air, yielding the final

CuO catalyst.

**Preparation of WO<sub>3</sub>:** A solution of Na<sub>2</sub>WO<sub>4</sub> (0.33 g dissolved in 20 mL of water) was placed under stirring. Then, 20 mL of a 0.1 M HCl solution was slowly added dropwise under vigorous stirring. The solution is then transferred into a 100 mL sealed stainless steel autoclave lined with polytetrafluoroethylene. and maintained at 180 °C for 16 h. After the reaction, the autoclave was allowed to cool down to room temperature naturally. The product was collected by centrifugation, washed several times with water and ethanol, and dried overnight at 60 °C. Finally, the obtained sample was calcined in air at 500 °C for 2 h to remove organic residues and enhance crystallinity, yielding WO<sub>3</sub> nanoparticles.

**Characterizations:** XRD data were acquired by an X-ray diffractometer with Cu K $\alpha$  radiation (DX-2700B). SEM measurements were carried out on an X-ray diffractometer with Cu K $\alpha$  radiation (DX-2700B). The absorbance data were measured on UV–vis spectrophotometer of SHIMADZU UV-2600. TEM image was obtained from an atomic-resolution scanning transmission electron microscopy (FEI Talos F200S Super) operated at 200 kV. XPS measurements were performed with Thermo Fischer ESCALAB Xi<sup>+</sup>.

**Electrochemical measurements:** All electrochemical measurements were carried out in an H-shaped electrochemical cell separated by Nafion 117 membrane using a CHI 760E electrochemical workstation (Chenhua, Shanghai). The CuWO<sub>4</sub> nanoparticles loaded on carbon cloth serve as the working electrode. The area of the working electrode immersed in the electrolyte is 0.25 cm<sup>2</sup>. LSV was performed in Ar-saturated

0.5 M Na<sub>2</sub>SO<sub>4</sub> with 0.1 M NaNO<sub>2</sub> at a scan rate of 5 mV s<sup>-1</sup>. All potentials reported in this work were converted to a reversible hydrogen electrode (RHE) scale, and current densities were normalized to the geometric surface area. All experiments were carried out at room temperature (25 °C).

DEMS was conducted using an in-situ mass spectrometer (Linglu QAS100) combined with an electrochemical workstation. A small electrolytic cell with a volume of 2 mL was used, and the electrolyte was bubbled with high-purity Ar gas for 30 min to remove O<sub>2</sub> and N<sub>2</sub>. A Pt wire and an Ag/AgCl electrode were used as the counter and reference electrodes, respectively. In situ mass spectrometry was performed simultaneously with constant potential electrolysis tests to detect products and intermediates.

H<sub>2</sub> determination was performed using a gas chromatograph (Shimadzu GC-2014C) and an electrochemical workstation. An H-type cell was employed, with a platinum sheet and Ag/AgCl electrode serving as the counter electrode and reference electrode, respectively. The electrolyte was purged with high-purity Ar gas to remove O<sub>2</sub> and N<sub>2</sub>. GC testing was conducted concurrently with the constant-potential electrolysis experiments. The generated H<sub>2</sub> was quantified using a gas chromatograph equipped with a thermal conductivity detector (TCD). Ar was used as the carrier gas. During the experiment, a flow meter is used to monitor and control the carrier gas flow rate. The system is equipped with an automatic sampler to ensure stable and repeatable injection volumes. H<sub>2</sub> quantification is accomplished using a calibration curve established with certified standard mixed gases.

**Determination of NH<sub>3</sub>:** The NH<sub>3</sub> concentration in the electrolyte was determined (the obtained electrolyte was diluted 20 times) by the indophenol blue method. Specifically, 2 mL of electrolyte collected after electrolysis was mixed with 2 mL of coloring solution (1 M NaOH containing 5% salicylic acid and 5% sodium citrate) and 1 mL of oxidizing solution (0.05 M NaClO). Then, 0.2 mL oxidation solution (0.05 M NaClO) mL catalyst solution (1 wt% C<sub>5</sub>FeN<sub>6</sub>Na<sub>2</sub>O 2H<sub>2</sub>O) were dropped into the collected solution. After standing in the dark for 2 h, the concentration of NH<sub>3</sub> was determined by UV–Vis at a specific wavelength of 655 nm. The concentration–absorbance curve was calibrated using the standard NH<sub>4</sub>Cl solution with known concentrations of 0.0, 0.25, 0.5, 1.0, 1.5, 2.0, and 2.5 µg mL<sup>-1</sup> in 0.5 M Na<sub>2</sub>SO<sub>4</sub>. The fitting curve ( $y = 0.41418x + 0.02591$ ,  $R^2 = 0.9998$ ) shows a good linear relation of absorbance value with NH<sub>3</sub> concentration.

**Determination of FE and NH<sub>3</sub> yield:**

The NH<sub>3</sub> FE is estimated from the charge consumed for NO<sub>2</sub><sup>-</sup> reduction and the total charge passed through the electrode:

$$FE = 6 \times F \times V \times [NH_3] / (Q \times 17) \times 100\%$$

The yield rate of NH<sub>3</sub> (aq) is calculated:

$$NH_3 \text{ yield} = V \times [NH_3] / (A \times t \times 17)$$

Where [NH<sub>3</sub>] is the concentration of NH<sub>3</sub>, F is the Faradaic constant (96485 C mol<sup>-1</sup>), V is the volume of electrolyte in the anode compartment (45 mL), Q is the total charge passing the electrode, t is the electrolysis time, and A is the geometric surface area.

**DFT calculation details:** First-principles calculations with spin-polarized were performed based on density functional theory (DFT) implemented in the VASP package.<sup>1</sup> The interaction between valence electrons and ionic core was expanded using the projector augmented wave (PAW)<sup>2</sup> approach with cutoff of 500 eV and 450 eV for the bulk and slab models, respectively. Perdew-Burke-Ernzerhof functional (PBE) with semi-empirical corrections of DFT-D3 was adopted to describe the exchange-correlation functional effect<sup>3</sup> based on general gradient approximation (GGA). CuWO<sub>4</sub> (111) slab with the thickness of the vacuum region is >15 Å was built. The Hubbard U model was implemented with an effective U = 7.5 eV for Cu, while the U correction is unnecessary for the W atoms.<sup>4</sup> The Brillouin zone was sampled by 4 × 4 × 1 special k-points using the Monkhorst Pack scheme for structural configuration optimizations.<sup>5</sup> The force convergence thresholds are 0.02 eV/Å and the total energy is less than 1E-5 eV, respectively.

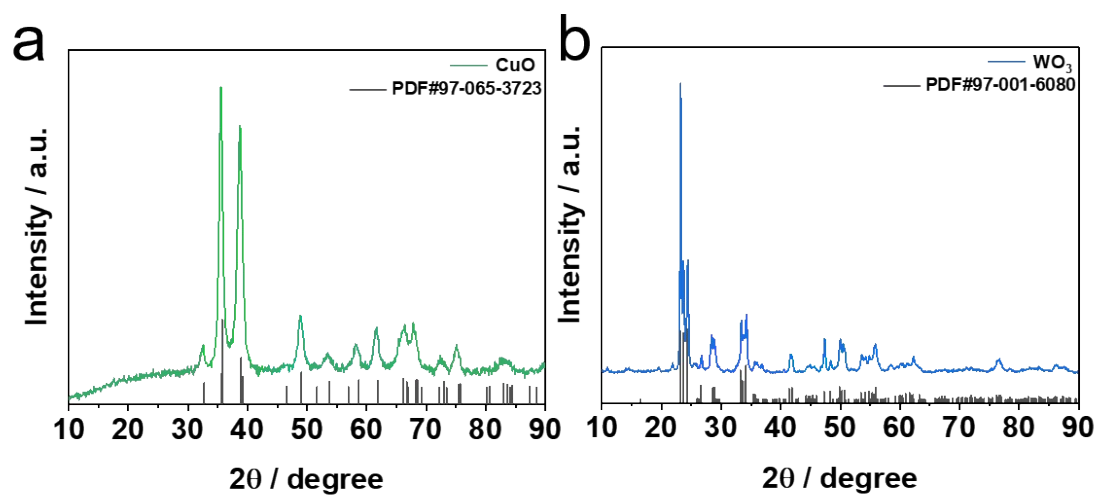


Fig. S1 XRD spectra of (a)  $\text{WO}_3$  and (b) CuO.

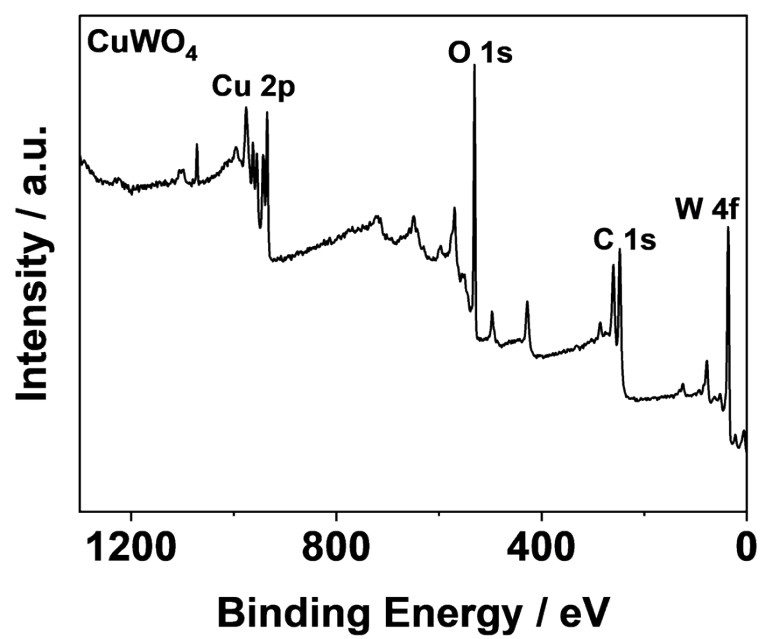


Fig. S2 XPS survey spectrum of  $\text{CuWO}_4$ .

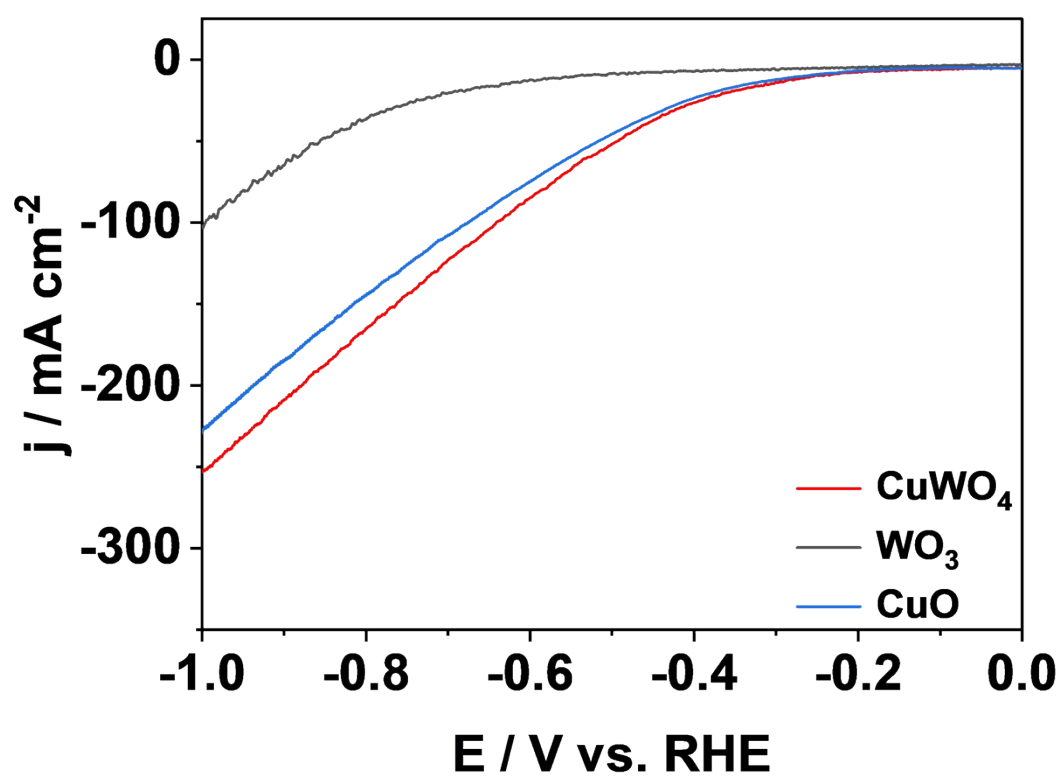


Fig. S3 LSV curves of  $\text{CuWO}_4$ ,  $\text{WO}_3$ , and  $\text{CuO}$  tested in 0.5 M  $\text{Na}_2\text{SO}_4$  solution with 0.1 M  $\text{NaNO}_2$ .



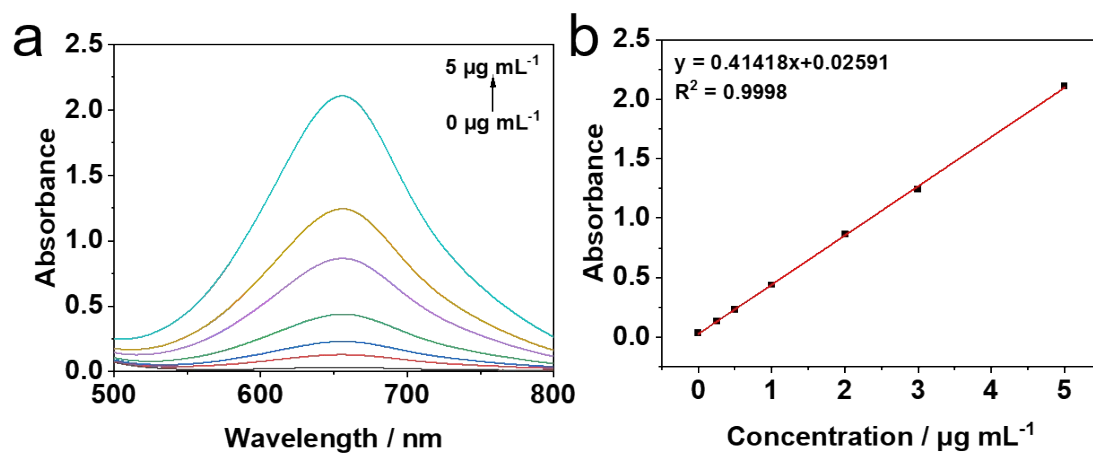


Fig. S4 (a) UV–Vis spectra and (b) corresponding calibration curves were used to calculate  $\text{NH}_4^+$ .

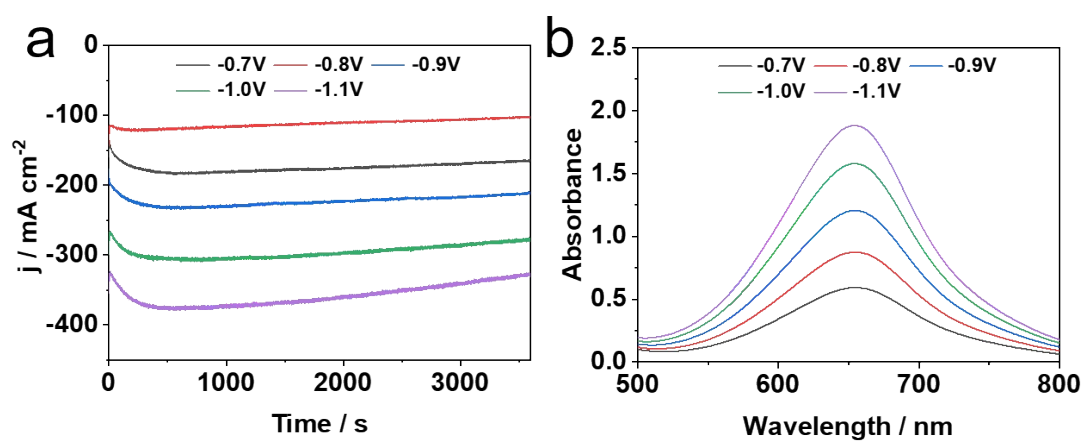


Fig. S5 (a) Chronoamperometry curves and (b) corresponding UV-Vis spectra of  $\text{CuWO}_4$  from -0.7 V to -1.1 V.

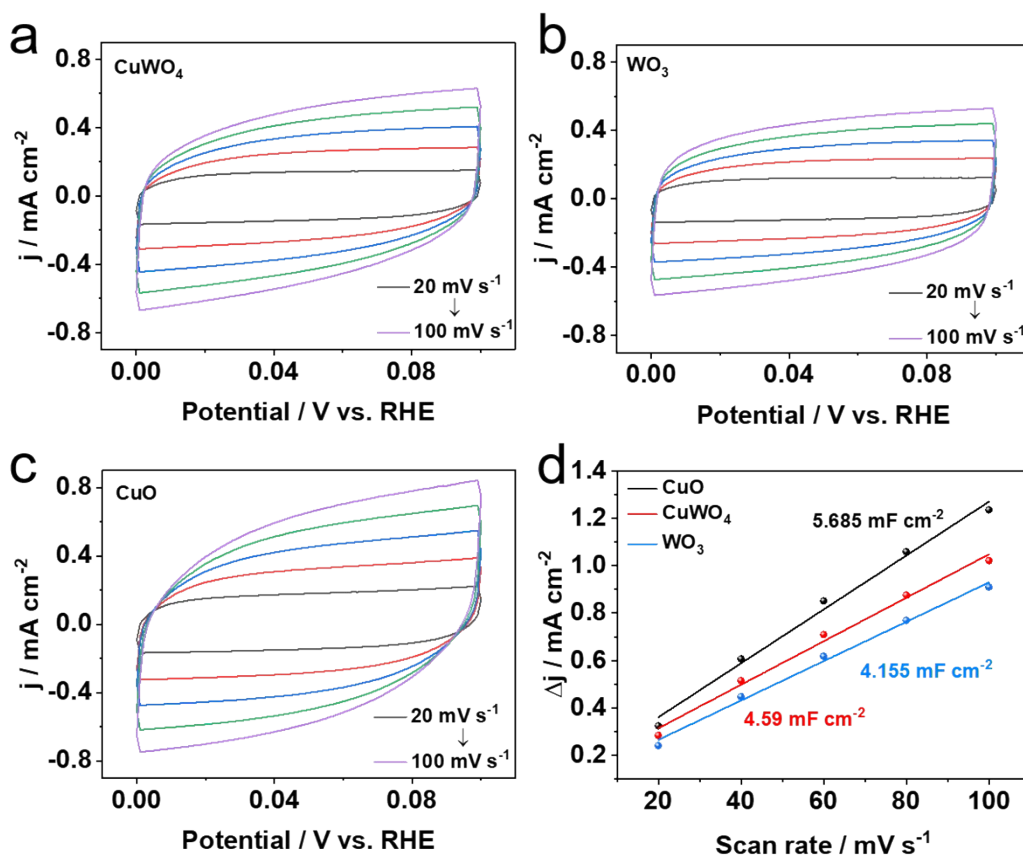


Fig. S6 CV curves of (a) CuWO<sub>4</sub>, (b) WO<sub>3</sub>, and (c) CuO at different scan rates (20–100 mV s<sup>-1</sup>). (d) C<sub>dl</sub> of CuWO<sub>4</sub>, WO<sub>3</sub>, and CuO.

Electrochemically active surface area (ECSA) can be estimated from double-layer capacitance (C<sub>dl</sub>) using the following formula:  $ECSA = C_{dl} / C_s$ , where C<sub>dl</sub> is the experimentally measured double-layer capacitance and C<sub>s</sub> is the specific capacitance per unit true surface area, whose value depends on material properties and electrolyte type. For copper-based materials, the literature commonly employs the accepted value of C<sub>s</sub> = 40 μF cm<sup>-2</sup> (see Power Sources, Appl. Surf. Sci., 2022, 585, 152757). Thus, the electrochemical surface area calculations are as follows:  $ECSA (CuWO_4) = 4.59 / 40 \times 10^{-3} \text{ cm}^2 = 114.75 \text{ cm}^2$   $ECSA (CuO) = 5.685 / 40 \times 10^{-3} \text{ cm}^2 = 142.125 \text{ cm}^2$ ,  $ECSA (WO_3) = 4.155 / 40 \times 10^{-3} \text{ cm}^2 = 103.875 \text{ cm}^2$ .

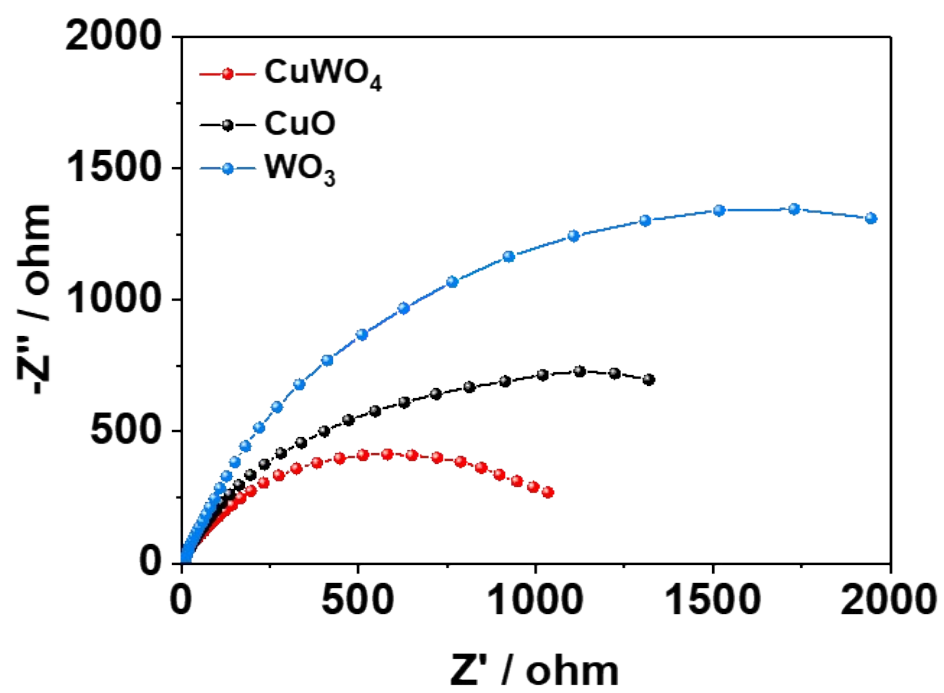


Fig. S7 Nyquist plots for  $\text{CuWO}_4$ ,  $\text{WO}_3$ , and  $\text{CuO}$ .

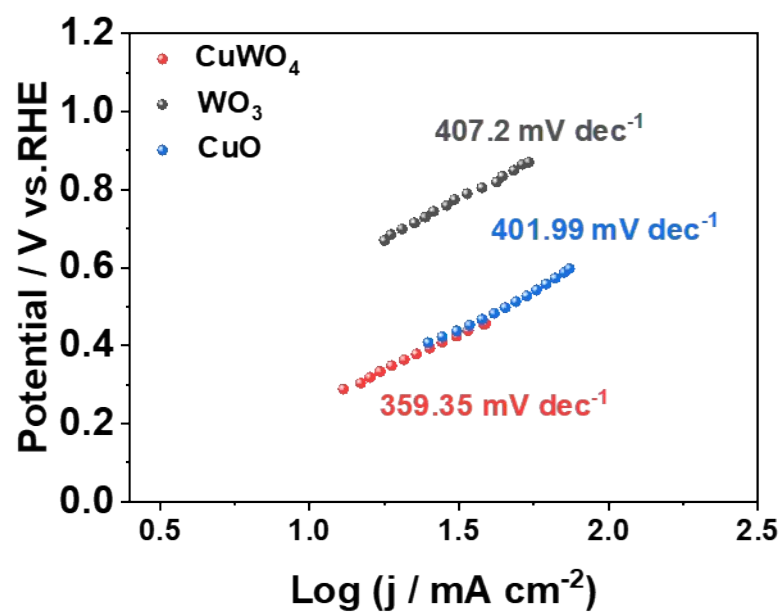


Fig. S8 Tafel slopes of CuWO<sub>4</sub>, WO<sub>3</sub>, and CuO.

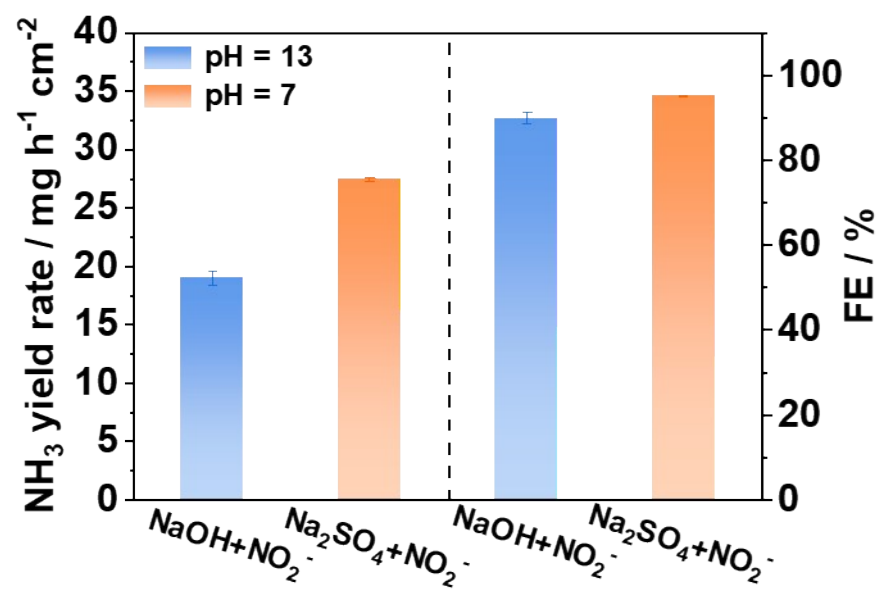


Fig. S9  $\text{NH}_3$  yield and FE at different pH at -1.0V.

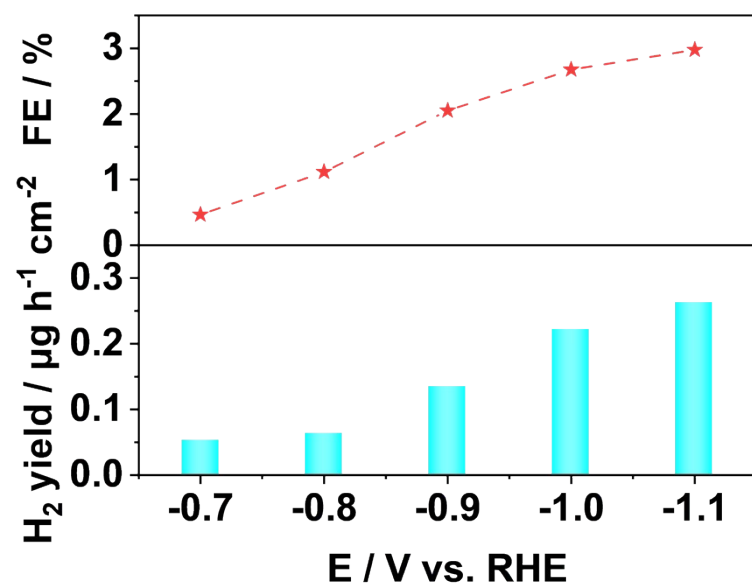


Fig. S10 H<sub>2</sub> yield and FE during NO<sub>2</sub><sup>-</sup>RR electrolysis at various potentials.

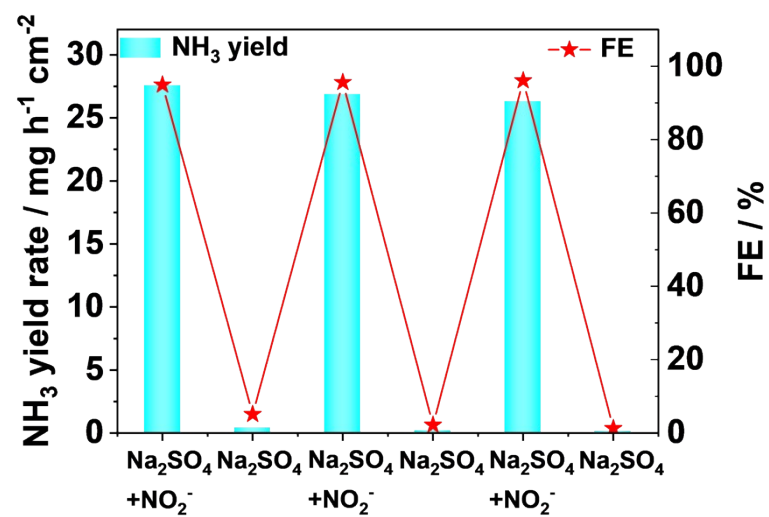


Fig. S11 NH<sub>3</sub> yield and FE of CuWO<sub>4</sub> were evaluated during alternating cycling tests.



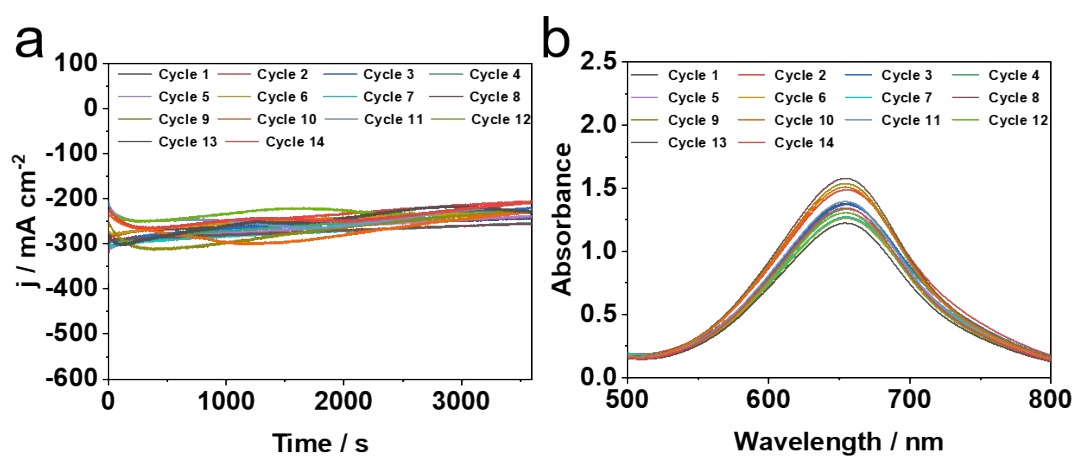


Fig. S12 (a) Chronoamperometry curves and (b) corresponding UV-Vis absorption spectra of  $\text{CuWO}_4$  during cycling tests in 0.5 M  $\text{Na}_2\text{SO}_4$  with 0.1 M  $\text{NO}_2^-$  at  $-1.0$  V.

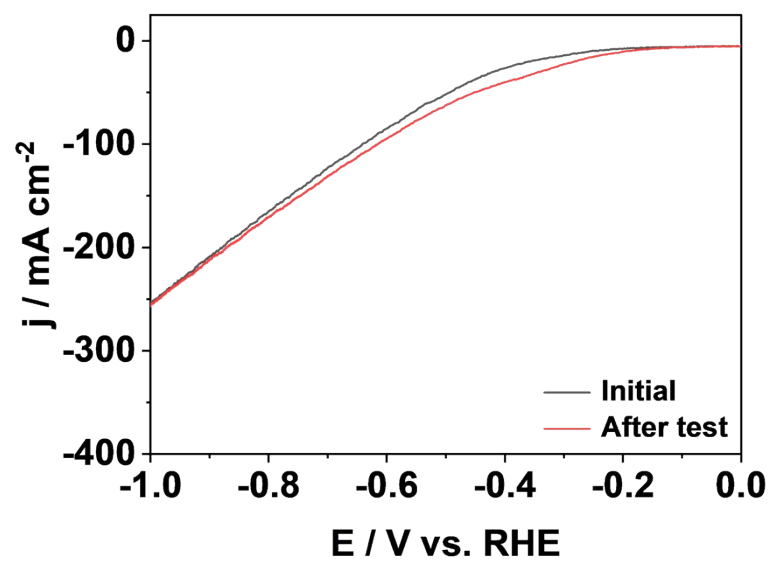


Fig. S13 LSV curves before and after the stability test.

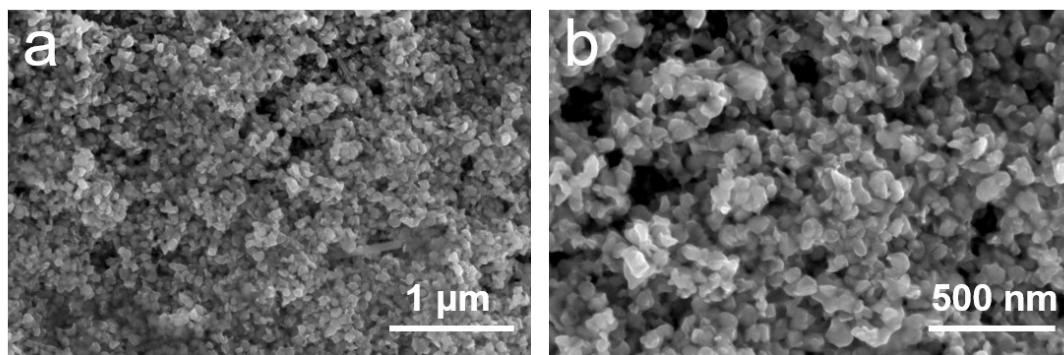


Fig. S14 SEM images of CuWO<sub>4</sub> nanoparticle after long-term electrolysis.

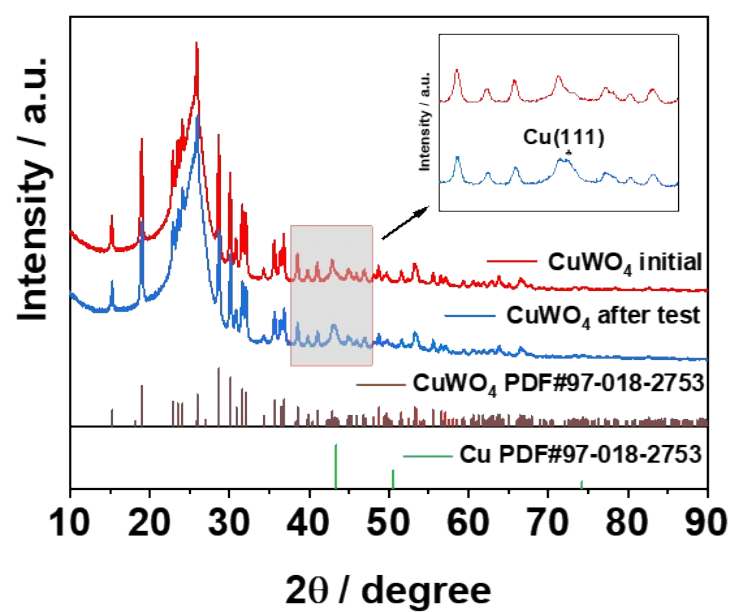


Fig. S15 XRD spectra of CuWO<sub>4</sub> nanoparticles loaded on carbon cloth before and after long-term electrolysis.

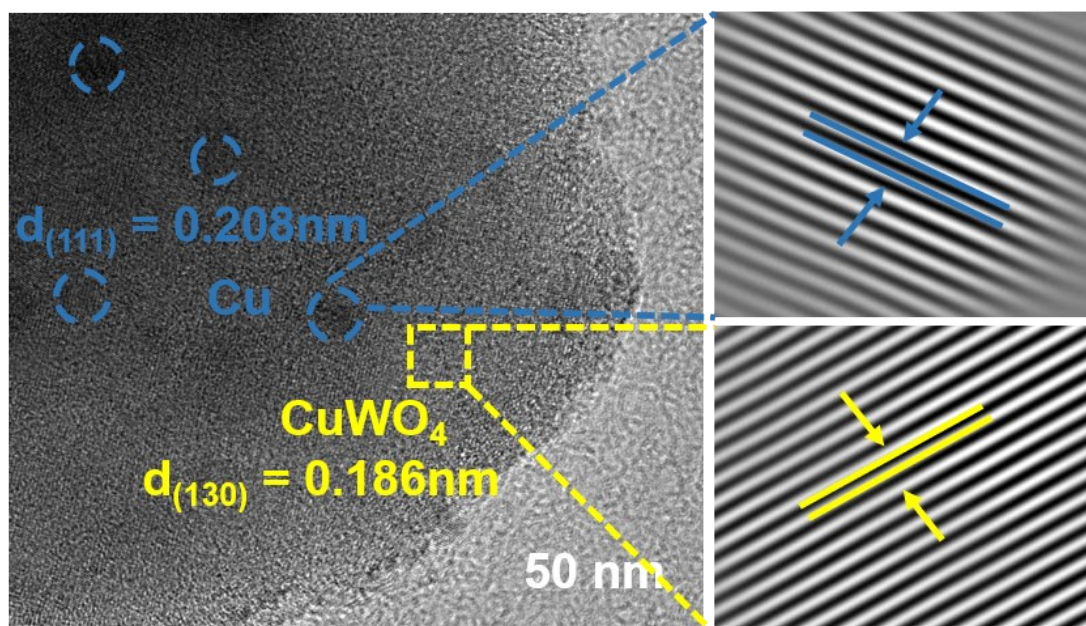


Fig. S16 HRTEM images of  $\text{CuWO}_4$  nanoparticle after long-term electrolysis.

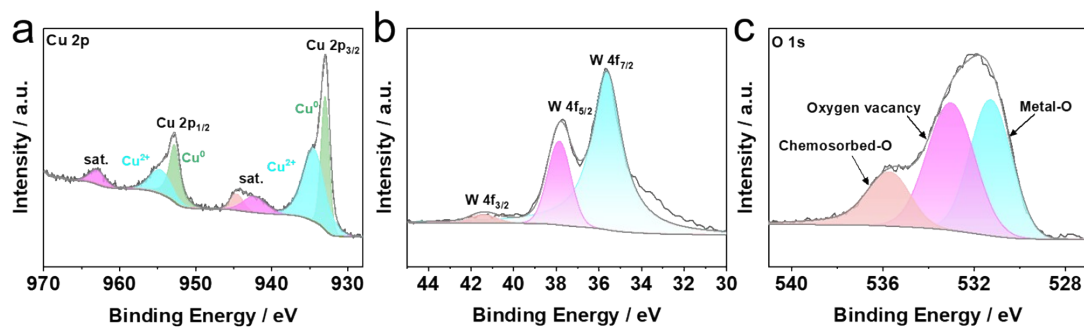


Fig. S17 High-resolution XPS spectra of (a) Cu 2p, (b) W 4f, and (c) O 1s of CuWO<sub>4</sub> nanoparticles after long-term electrolysis.

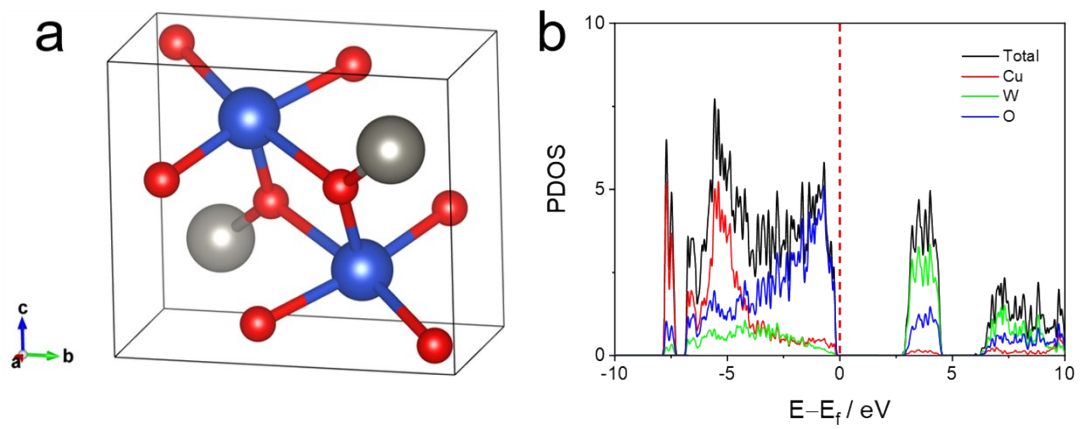


Fig. S18 (a) Atomic structure of  $\text{CuWO}_4$  after lattice optimization and (b) corresponding PDOS.

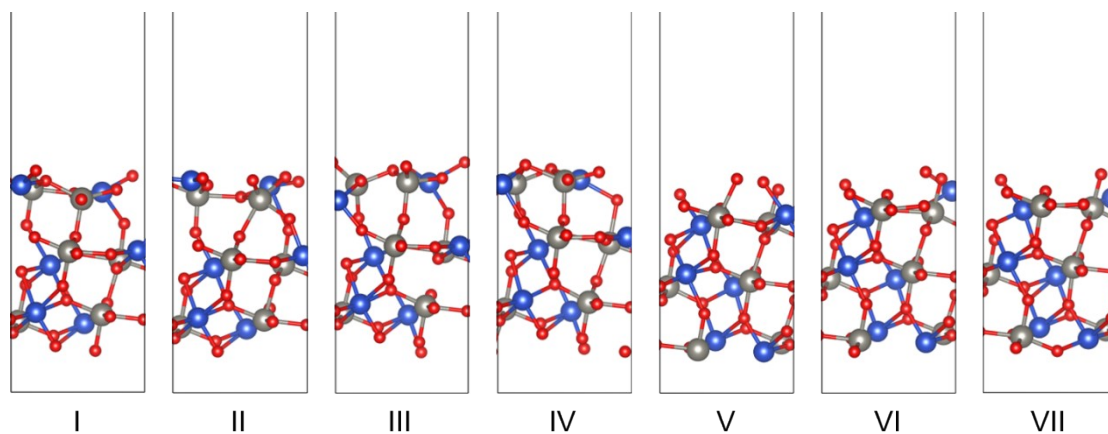


Fig. S19 Atomic structure of  $\text{CuWO}_4$  (111) slab model with various terminated surfaces. Blue, grey, and red represent Cu, W, and O, respectively.



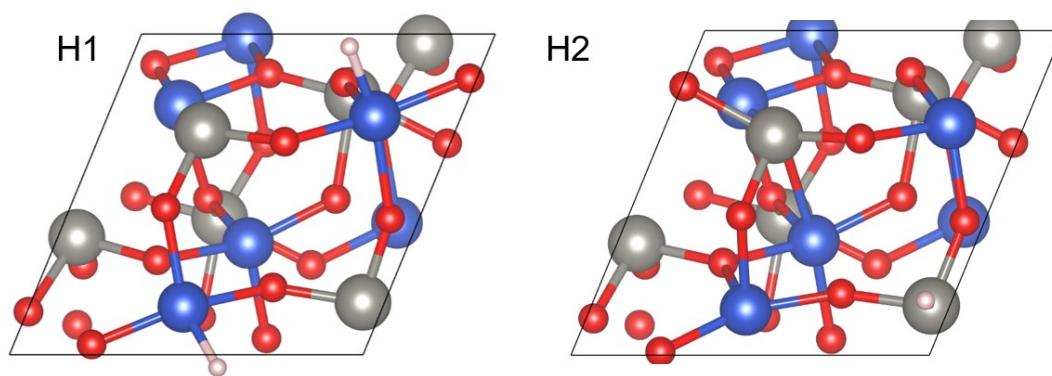


Fig. S20 Corresponding atomic structure of HER processing on different sites of CuWO<sub>4</sub> (111).

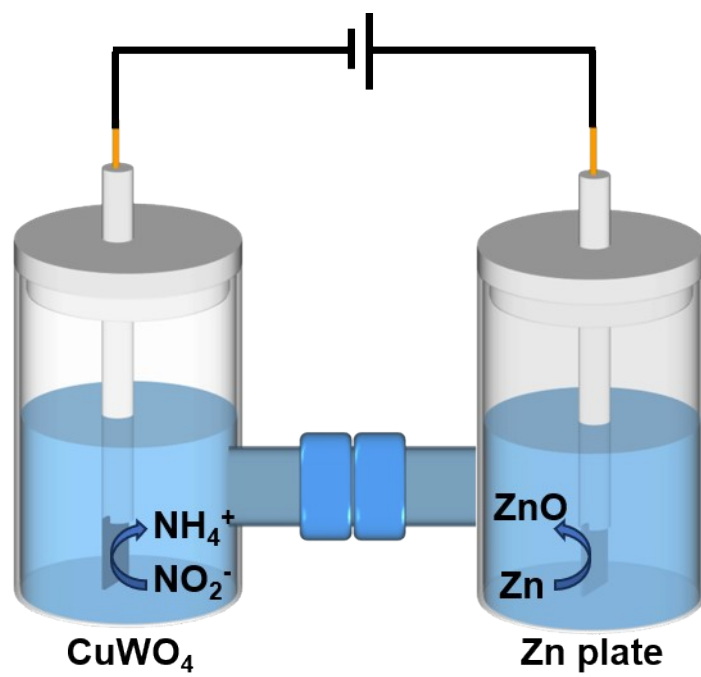


Fig. S21 Schematic diagram of a Zn-NO<sub>2</sub><sup>-</sup> battery.

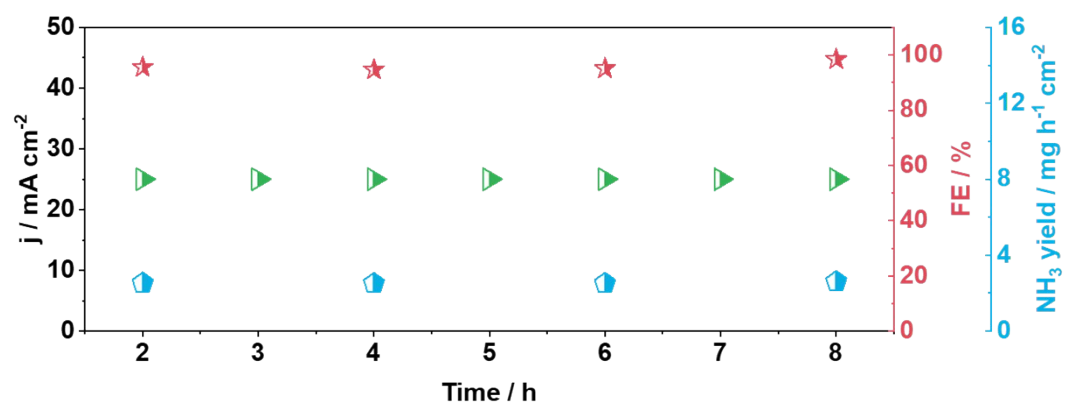


Fig. S22 Long-term  $\text{NO}_2^-$ RR experiment and the corresponding  $\text{NH}_3$  yields and FE with the  $\text{Zn-NO}_2^-$  battery.

**Table S1.** Comparison of the catalytic performance of CuWO<sub>4</sub> with other reported NO<sub>2</sub>-RR electrocatalysts.

Catalyst	Electrolyte	Potential (V vs RHE)	FE (%)	NH <sub>3</sub> yield rate	Refs.
CuWO <sub>4</sub>	0.5 M Na <sub>2</sub> SO <sub>4</sub> (0.1 M NaNO <sub>2</sub> )	−1.0 V	95.09	27.46 mg h <sup>−1</sup> cm <sup>−2</sup>	This work
NiWO <sub>4</sub> /NF	0.1 M NaOH (0.1 M NaNO <sub>2</sub> )	−0.4V	97.6	10.974 mg h <sup>−1</sup> cm <sup>−2</sup>	6
Cu <sub>3</sub> P NA/CF	0.1 M PBS (0.1 M NaNO <sub>2</sub> )	−0.5V	91.2±2 .5	1.626 ± 0.036 mg h <sup>−1</sup> cm <sup>−2</sup>	7
Mn <sub>1</sub> /MoO <sub>3-x</sub>	0.5 M Na <sub>2</sub> SO <sub>4</sub> (0.5 M NaNO <sub>2</sub> )	−0.6V	92.6	9.3 mg h <sup>−1</sup> cm <sup>−2</sup>	8
Ni-TiO <sub>2</sub> /TP	0.1 M NaOH (0.1 M NaNO <sub>2</sub> )	−0.5V	94.89	6.476 mg h <sup>−1</sup> cm <sup>−2</sup>	9
Ni@HPCF/CP	0.1 M NaOH (0.1 M NaNO <sub>2</sub> )	−0.8V	95.1	2.41 mg h <sup>−1</sup> cm <sup>−2</sup>	10
TiO <sub>2-x</sub> NBA/TP	0.1 M NaOH (0.1 M NaNO <sub>2</sub> )	−0.7V	92.7	7.898 mg h <sup>−1</sup> cm <sup>−2</sup>	11
WO <sub>2</sub> /W	0.1 M NaOH (0.1 M NaNO <sub>2</sub> )	−0.9V	94.32	14.964 mg h <sup>−1</sup> cm <sup>−2</sup>	12
Ru-Cu NW/CF	0.1 M PBS (500 ppm NO <sub>2</sub> −)	−0.6V	94.1	12.47 mg h <sup>−1</sup> cm <sup>−2</sup>	13
u-Cu/CF	0.5 M Na <sub>2</sub> SO <sub>4</sub> (0.1 M NaNO <sub>2</sub> )	−0.7V	94.7	8.4065 mg h <sup>−1</sup> cm <sup>−2</sup>	14
CF@Cu <sub>2</sub> O	0.1 M PBS (0.1 M NaNO <sub>2</sub> )	−0.6V	94.2	7.53 mg h <sup>−1</sup> cm <sup>−2</sup>	15
Co <sub>3</sub> S <sub>4</sub> /NF	0.1 M NaOH (0.1 M NaNO <sub>2</sub> )	−0.3V	96.9	19.2 mg h <sup>−1</sup> cm <sup>−2</sup>	16
Cu/N-SnS <sub>2-x</sub>	0.1 M NaOH (0.1 M NaNO <sub>2</sub> )	−0.835	95.7	3.775 mg h <sup>−1</sup> cm <sup>−2</sup>	17

FeCu DAC	0.1 M KOH (0.1 M NaNO <sub>2</sub> )	−0.6V	99.88	4.905 mg h <sup>−1</sup> cm <sup>−2</sup>	18
TiO <sub>2</sub> @CoFe-LDH	0.1 M NaOH (0.1 M NaNO <sub>2</sub> )	−0.6V	97.4	17.99 mg h <sup>−1</sup> cm <sup>−2</sup>	19
Sb <sub>1</sub> Cu	0.5 M Na <sub>2</sub> SO <sub>4</sub> (0.1 M NaNO <sub>2</sub> )	−0.6V	96.4	10.41 mg h <sup>−1</sup> cm <sup>−2</sup>	20
FeOOH NTA/CC	0.1 M PBS (0.1 M NaNO <sub>2</sub> )	−1.0V	94.7	11.96 mg h <sup>−1</sup> cm <sup>−2</sup>	21
CoWO <sub>4</sub> /NF	0.5 M Na <sub>2</sub> SO <sub>4</sub> (0.1 M NaNO <sub>2</sub> )	−0.7V	95.2	18.856 mg h <sup>−1</sup> cm <sup>−2</sup>	22
NiCo–TiO <sub>2</sub>	0.1 M NaOH (0.1 M NaNO <sub>2</sub> )	−0.4V	97.5	18.736 mg h <sup>−1</sup> cm <sup>−2</sup>	23

**Table S2.** Comparison of NH<sub>3</sub> yield and power density of our battery with recent Zn-N<sub>2</sub>, Zn-NO, Zn-NO<sub>2</sub><sup>-</sup>, or Zn-NO<sub>3</sub><sup>-</sup> battery systems.

Catalyst	Battery Type	Power density	Refs.
<b>CuWO<sub>4</sub></b>	<b>Zn-NO<sub>2</sub><sup>-</sup></b>	<b>7.63</b>	<b>This work</b>
Cu NDs	Zn-N <sub>2</sub>	0.01	24
Fe-2HCS-8	Zn-N <sub>2</sub>	0.15	25
NbS <sub>2</sub>	Zn-N <sub>2</sub>	0.31	26
CoPi/NPCS	Zn-N <sub>2</sub>	0.49	27
Ti <sub>2</sub> O <sub>3</sub>	Zn-N <sub>2</sub>	1.02	28
Nb <sub>2</sub> O <sub>5</sub> /Nb <sub>2</sub> CTX	Zn-N <sub>2</sub>	1.25	29
FePS <sub>3</sub>	Zn-N <sub>2</sub>	2.60	30
TiO <sub>2-x</sub> /TP	Zn-NO	0.84	31
MoS <sub>2</sub> /CP	Zn-NO	1.04	32
a-B <sub>2.6</sub> C@TiO <sub>2</sub> /Ti	Zn-NO	1.7	33
O-Fe-N <sub>6</sub> -Cu	Zn-NO	2.30	34
CoB/Co@C	Zn-NO	3.68	35
hcp-Co	Zn-NO	4.6	36
FeOCl-V <sub>Cl</sub>	Zn-NO	6.2	37
ITO@TiO <sub>2</sub> /TP	Zn-NO <sub>2</sub> <sup>-</sup>	1.22	38
MoO <sub>2</sub>	Zn-NO <sub>2</sub> <sup>-</sup>	2.94	39
NiMoO <sub>4</sub>	Zn-NO <sub>2</sub> <sup>-</sup>	3.6	40
WO <sub>2-x</sub>	Zn-NO <sub>2</sub> <sup>-</sup>	5.05	12
NiWO <sub>4</sub>	Zn-NO <sub>2</sub> <sup>-</sup>	5.55	6
Ni-Mo-P/TiO <sub>2</sub>	Zn-NO <sub>2</sub> <sup>-</sup>	6.0	41

Pd/TiO <sub>2</sub>	Zn-NO <sub>3</sub> <sup>-</sup>	0.87	42
RhCuM-tpp	Zn-NO <sub>3</sub> <sup>-</sup>	1.54	43
RuFe/NF	Zn-NO <sub>3</sub> <sup>-</sup>	1.9	10
Fe/Ni <sub>2</sub> P	Zn-NO <sub>3</sub> <sup>-</sup>	3.25	44
NiCo <sub>2</sub> O <sub>4</sub> /CC	Zn-NO <sub>3</sub> <sup>-</sup>	3.94	45
PdCuAg MTs	Zn-NO <sub>3</sub> <sup>-</sup>	4.8	46
IrSAC-Co <sub>3</sub> O <sub>4</sub>	Zn-NO <sub>3</sub> <sup>-</sup>	5.6	47

## References

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