

Supporting Information

Electrochemical Ammonia synthesis from dilute gaseous Nitric Oxide Reduction at Ambient Conditions

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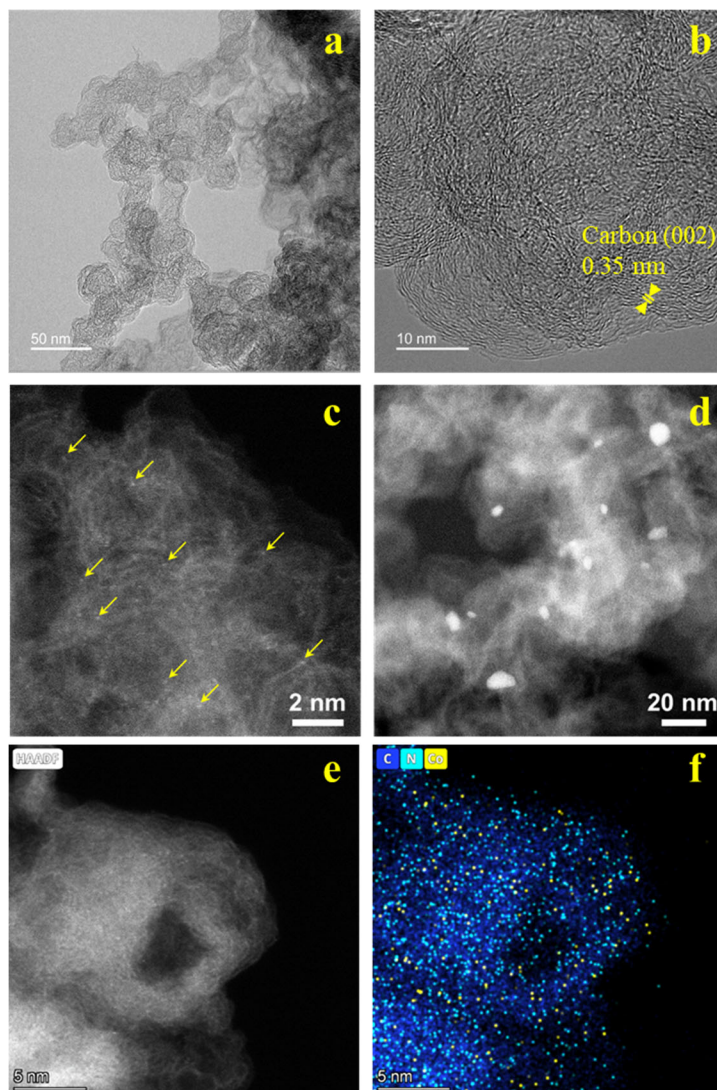


Figure S1. Structural characterization of Co-NC: (a) Low magnification FE-TEM image, (b) HR-TEM image, (c-e) STEM images, and (f) EDS elemental mapping, respectively.

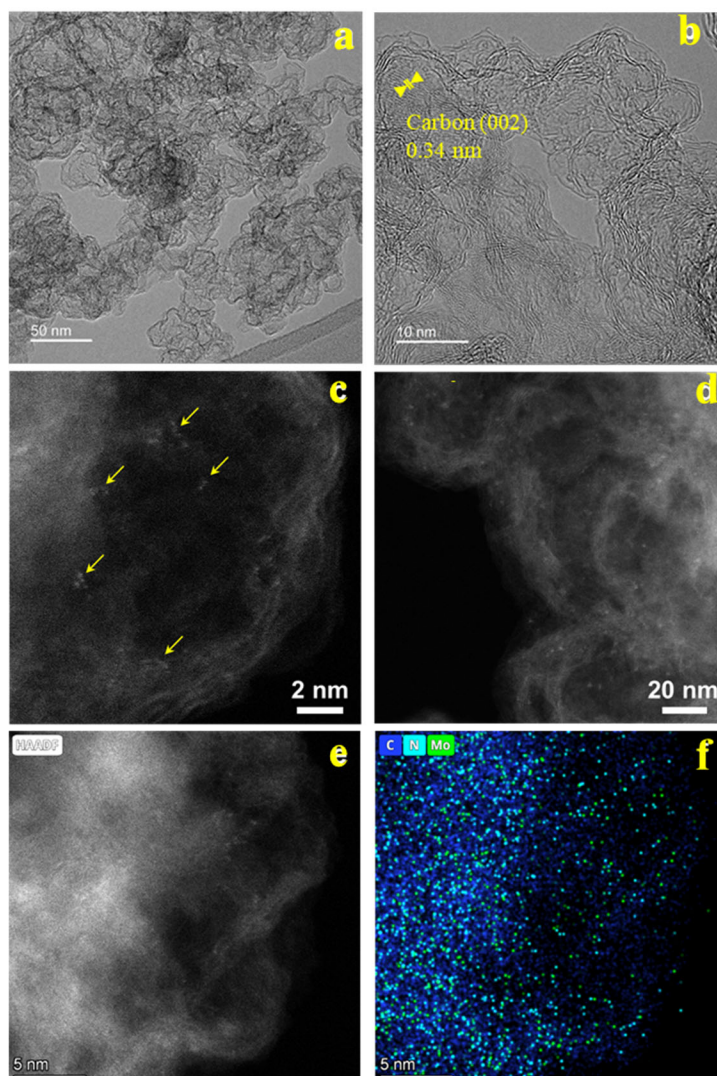


Figure S2. Structural characterization of Mo-NC: (a) Low magnification FE-TEM image, (b) HR-TEM image, (c-e) STEM images, and (f) EDS elemental mapping, respectively.

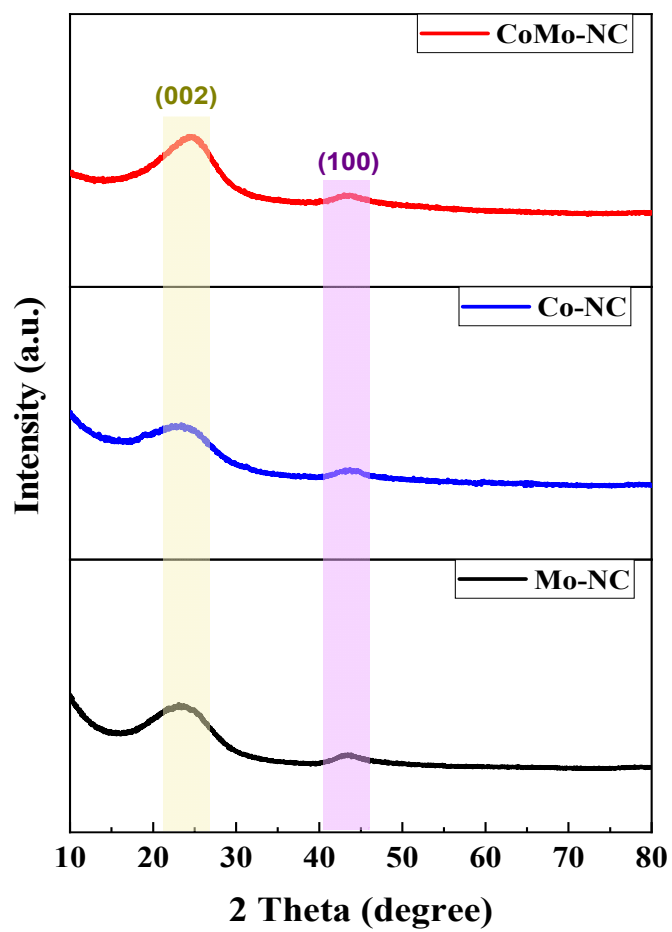


Figure S3. XRD patterns of Mo-NC, Co-NC, and CoMo-NC (Bottom to top).

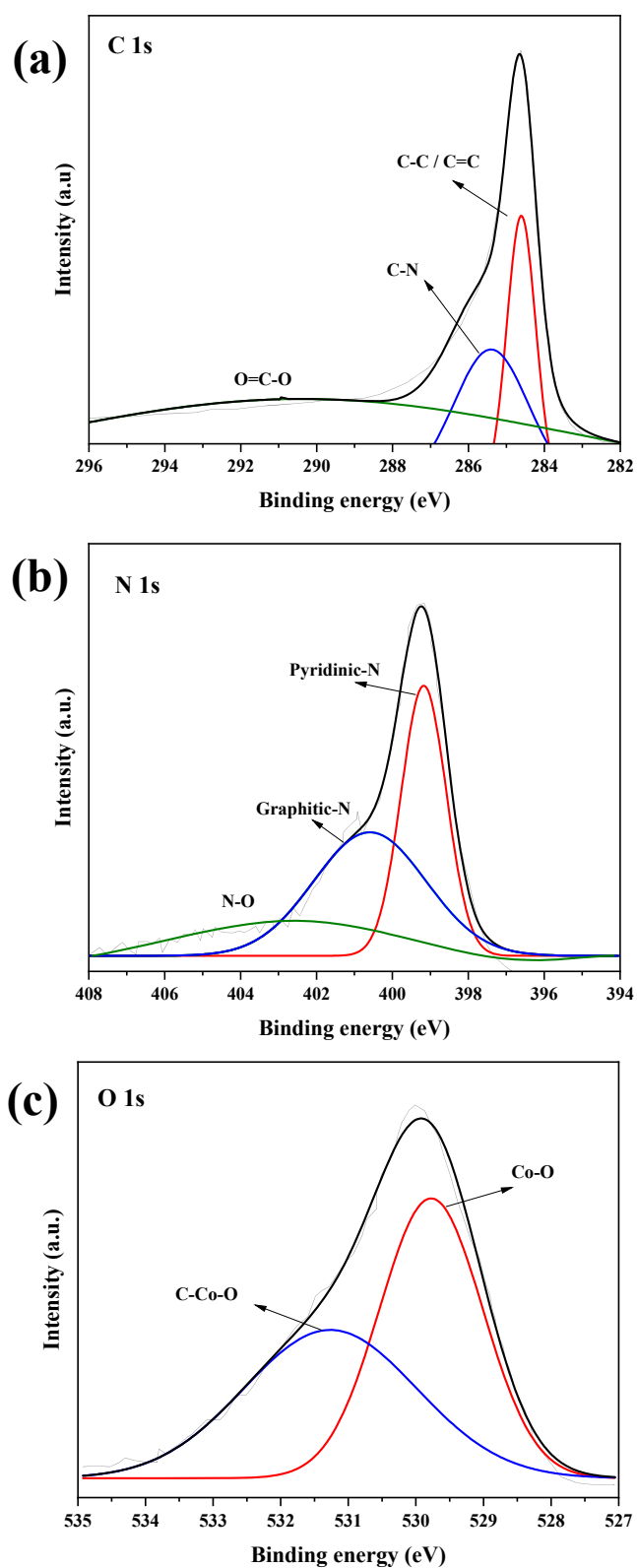


Figure S4. High-resolution XPS spectra of Co-NC: (a) Carbon (C 1s), (b) Nitrogen (N 1s), and (c) Oxygen (O 1s).

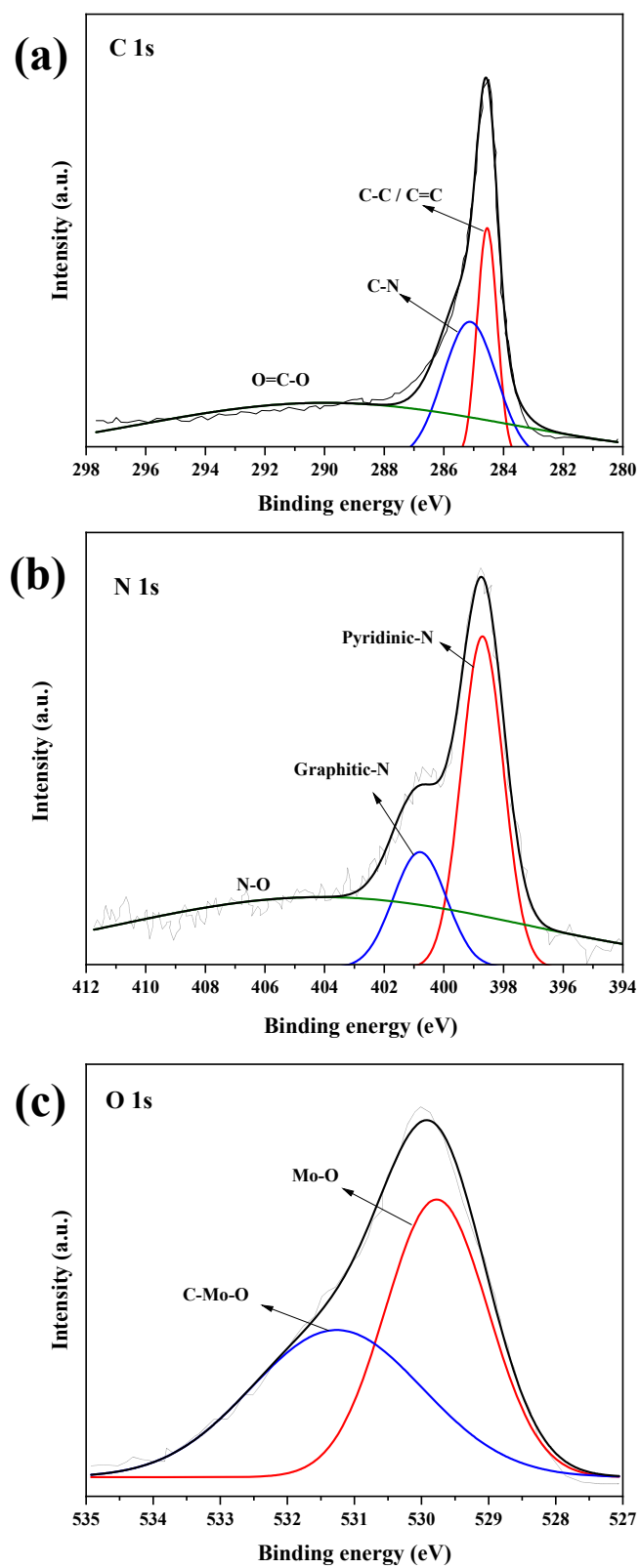


Figure S5. High-resolution XPS spectra of Mo-NC: (a) Carbon (C 1s), (b) Nitrogen (N 1s), and (c) Oxygen (O 1s).

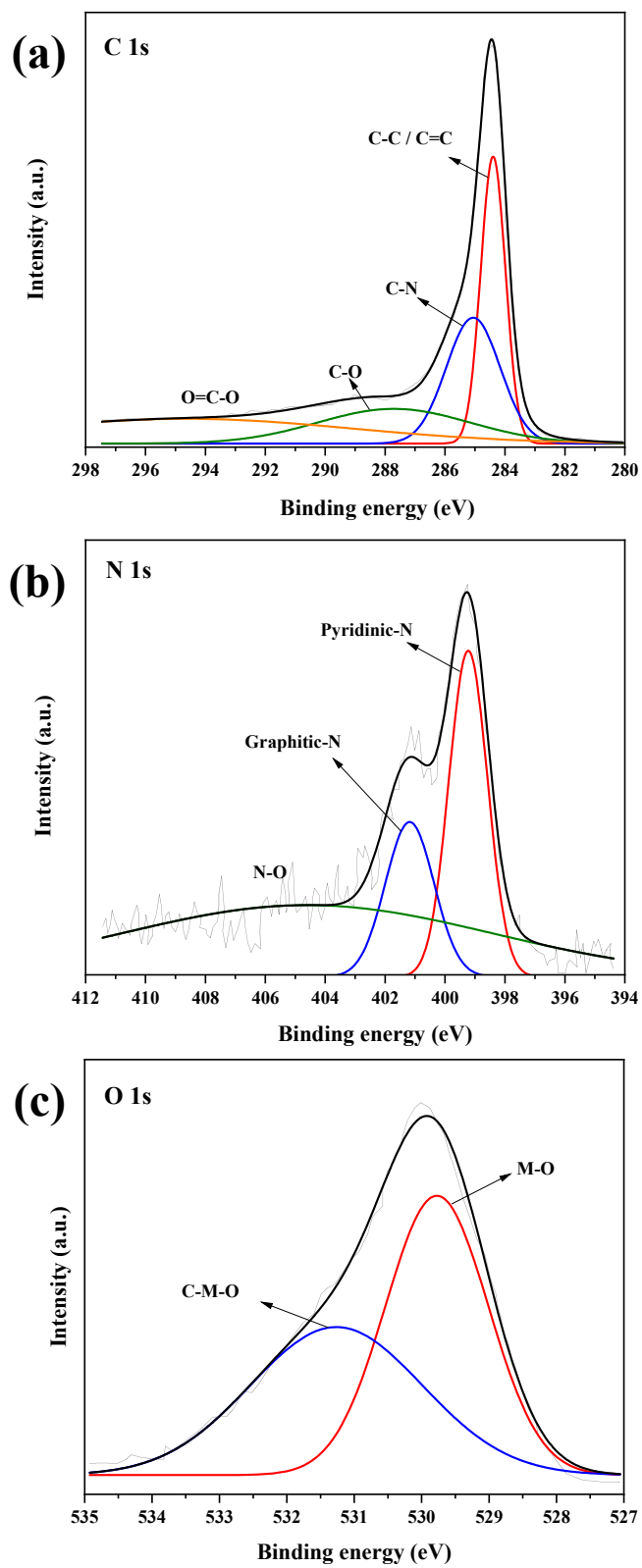


Figure S6. High-resolution XPS spectra of CoMo-NC: (a) Carbon (C 1s), (b) Nitrogen (N 1s), and (c) Oxygen (O 1s).

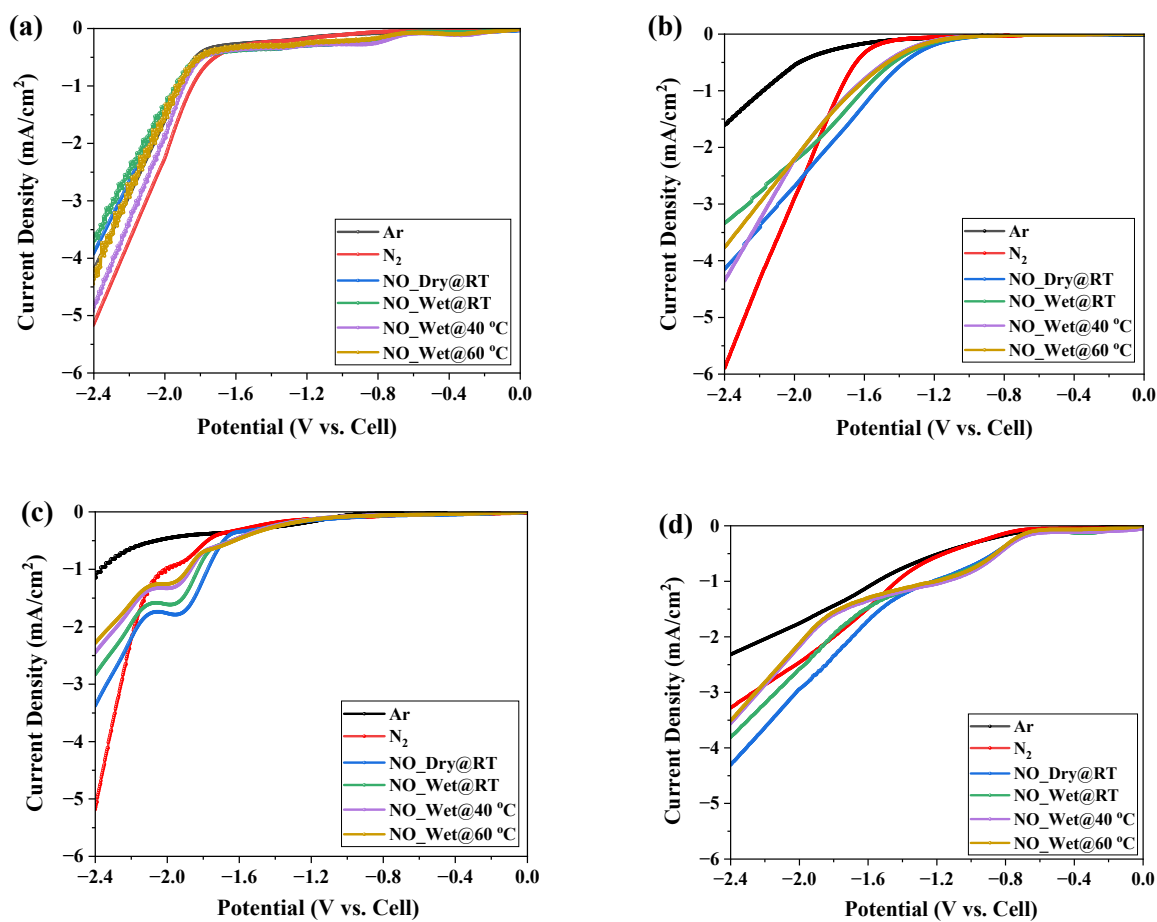


Figure S7. LSV tests of electrocatalysts: (a) Co-VC, (b) Co-NC, (c) Mo-NC, and (d) CoMo-NC at a scan rate of 10 mV s⁻¹.

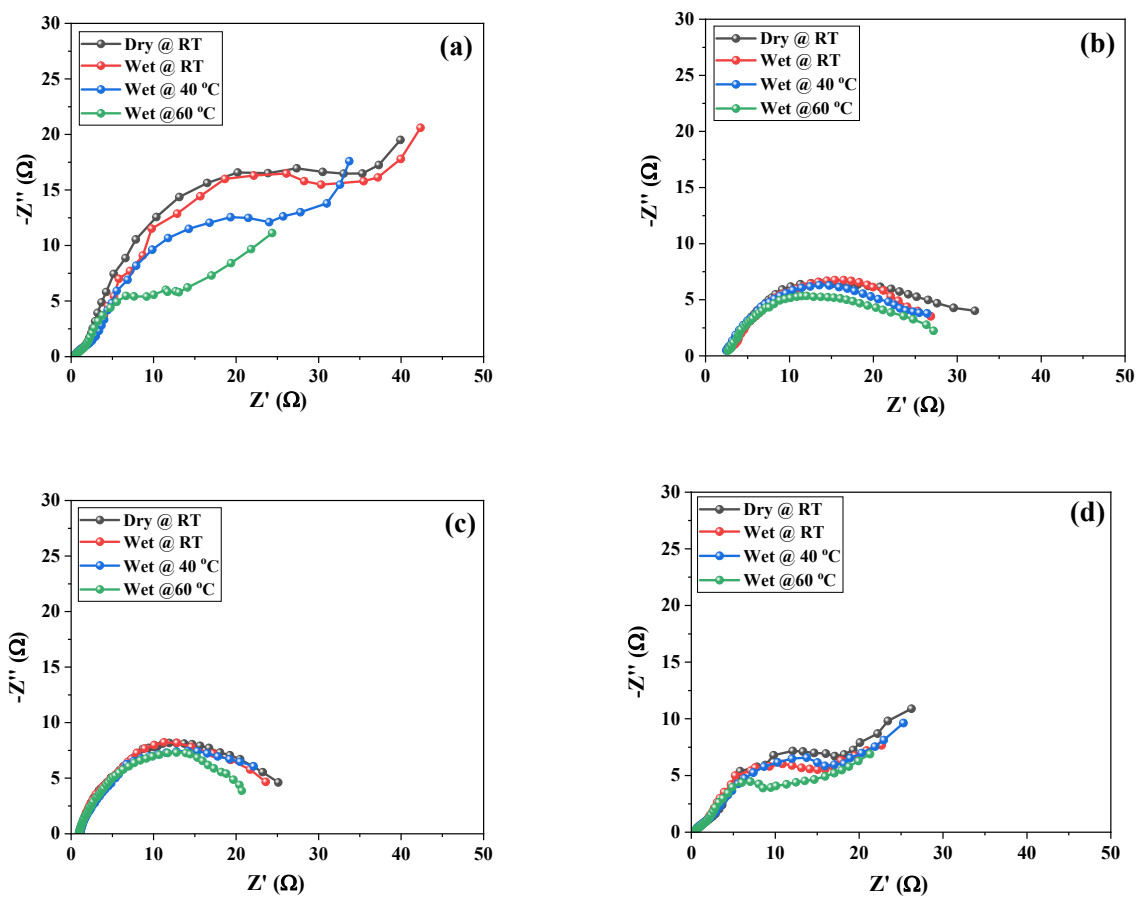


Figure S8. Electrochemical impedance spectroscopy (EIS) measurement in the range of 100 kHz to 100 mHz under potentiostatic mode at (a) -1.6, (b) -1.8, (c) -2.0, and (d) -2.2 V_{cell} at excitation amplitude of 20 mV for Co-VC.

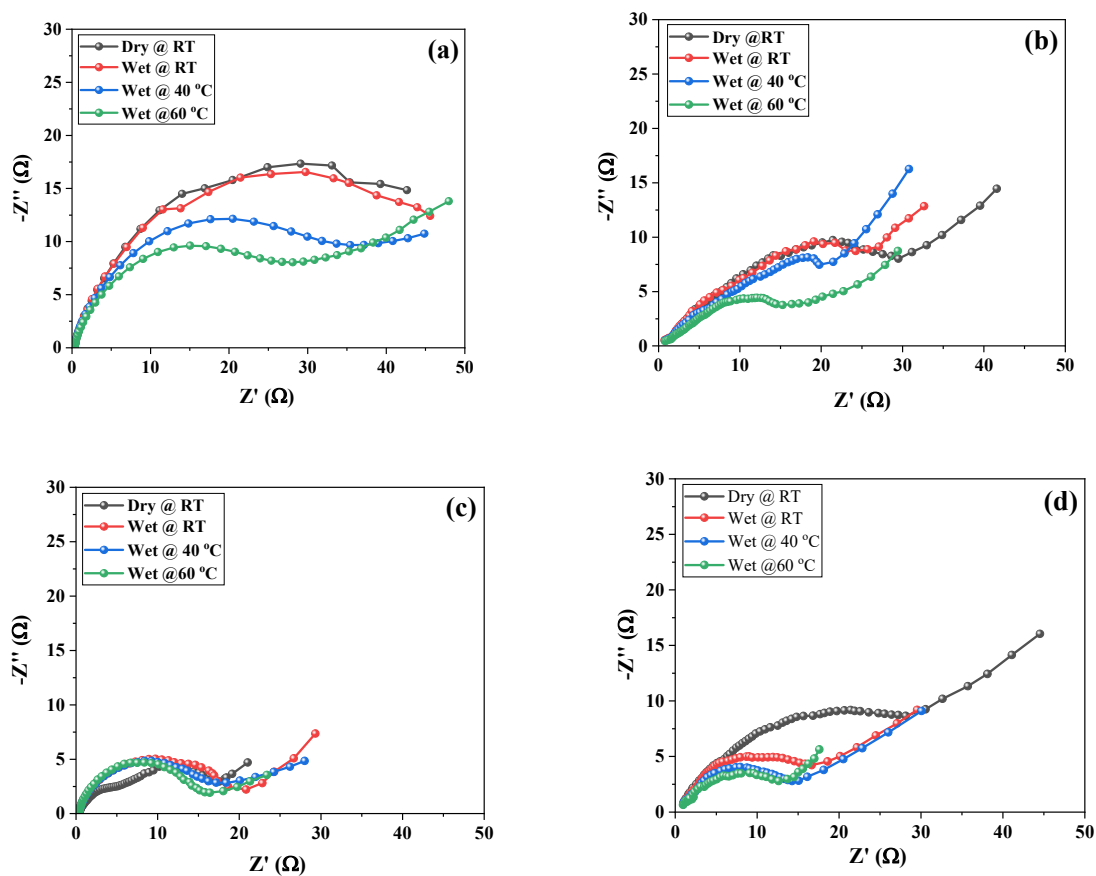


Figure S9. Electrochemical impedance spectroscopy (EIS) measurement in the range of 100 kHz to 100 mHz under potentiostatic mode at (a) -1.6, (b) -1.8, (c) -2.0, and (d) -2.2 V_{cell} at excitation amplitude of 20 mV for Co-NC.

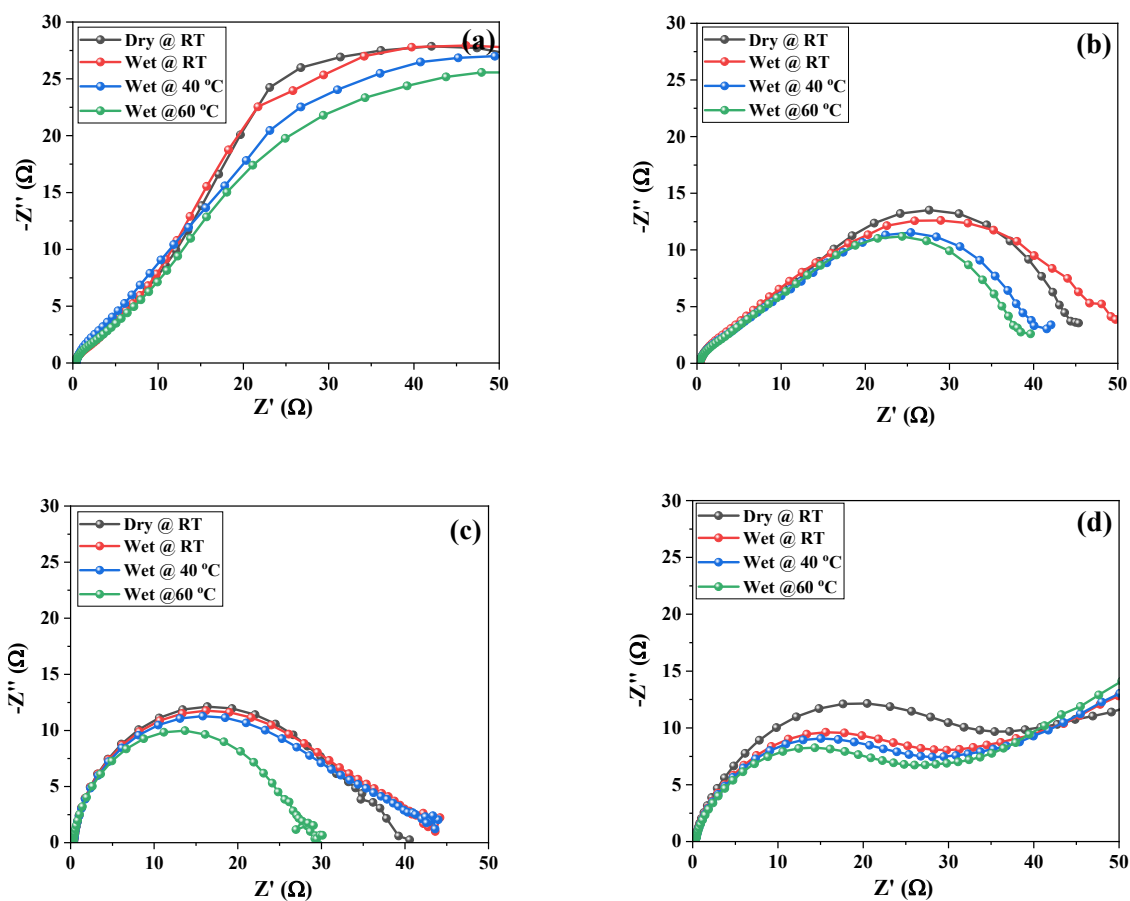


Figure S10. Electrochemical impedance spectroscopy (EIS) measurement in the range of 100 kHz to 100 mHz under potentiostatic mode at (a) -1.6, (b) -1.8, (c) -2.0, and (d) -2.2 V_{cell} at excitation amplitude of 20 mV for Mo-NC.

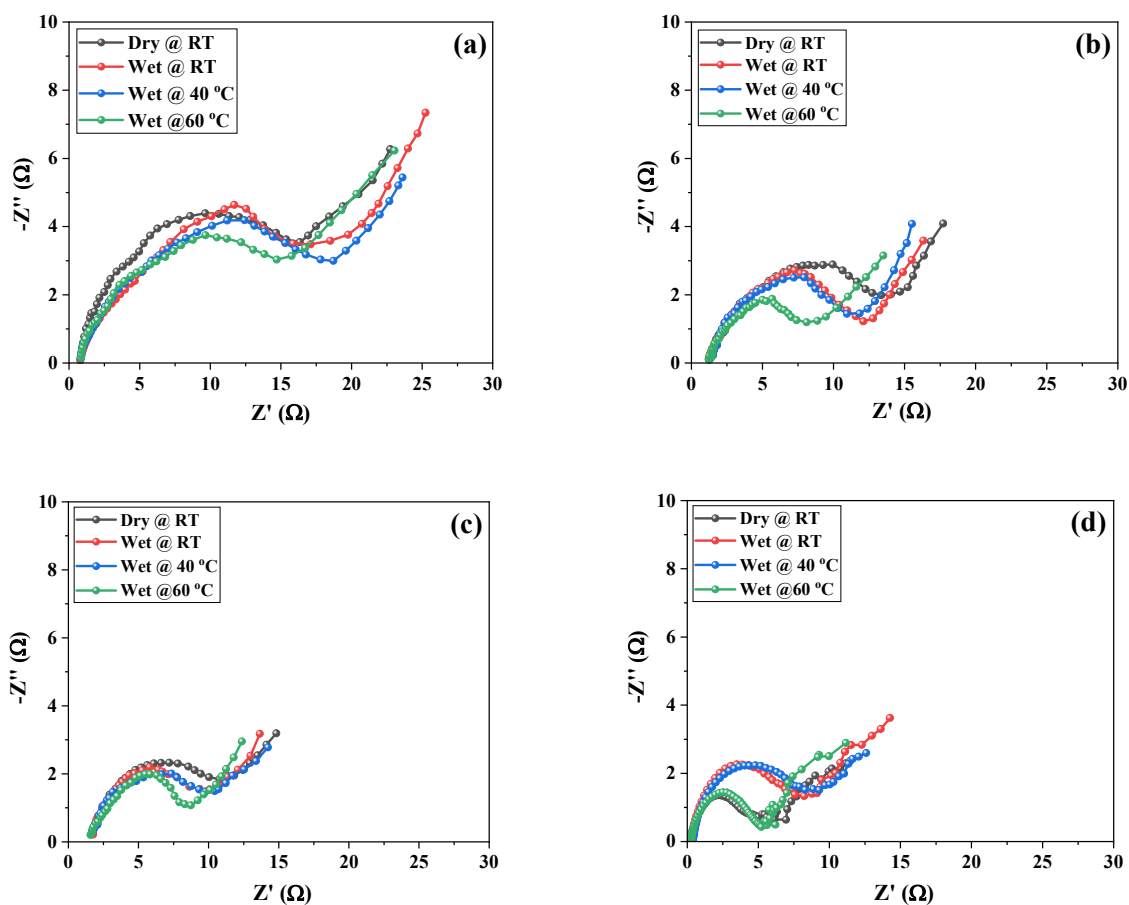


Figure S11. Electrochemical impedance spectroscopy (EIS) measurement in the range of 100 kHz to 100 mHz under potentiostatic mode at (a) -1.6, (b) -1.8, (c) -2.0, and (d) -2.2 V_{cell} at excitation amplitude of 20 mV for CoMo-NC.

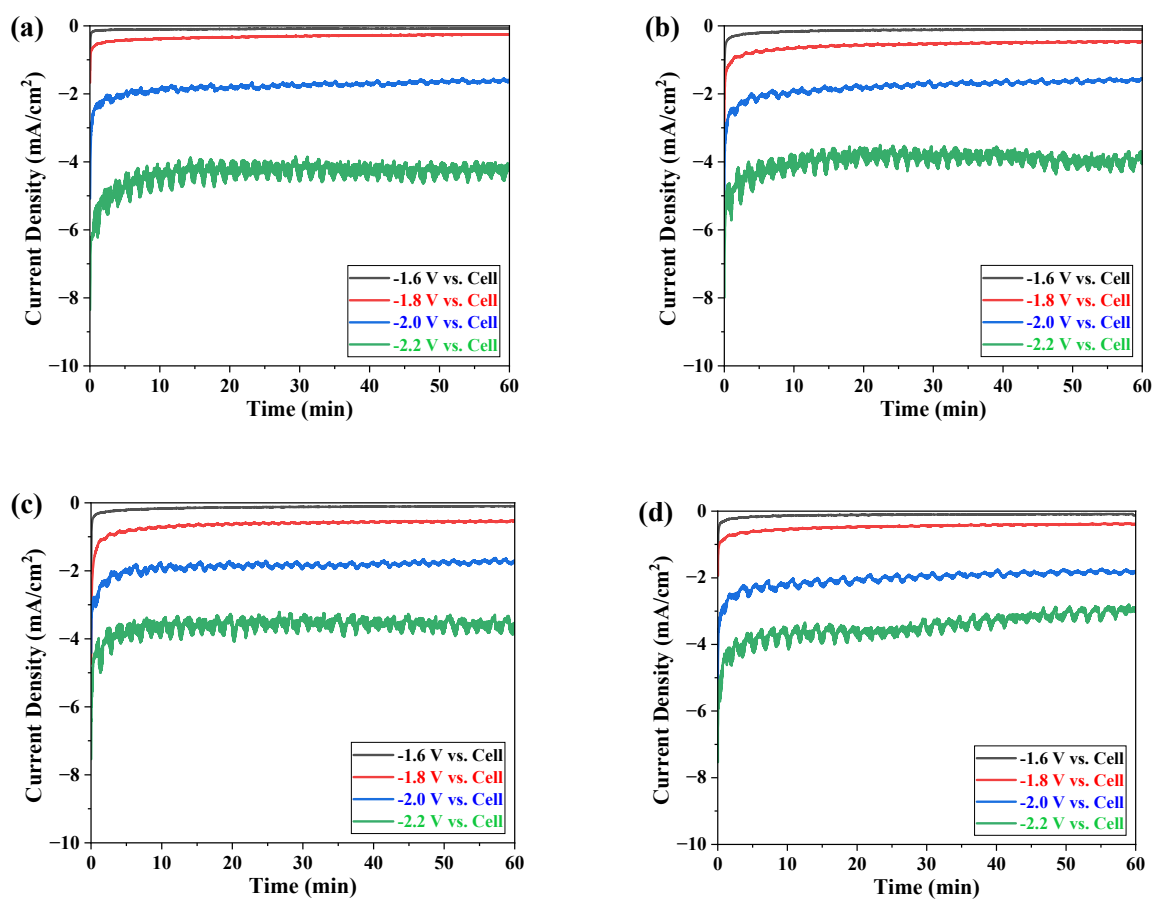


Figure S12. Chronoamperometry tests of Co-VC at a constant potential (vs. cell) for 1 h with (a) Dry@RT NO, (b) Wet@RT, (c) Wet@40 °C, and (d) Wet@60 °C condition.

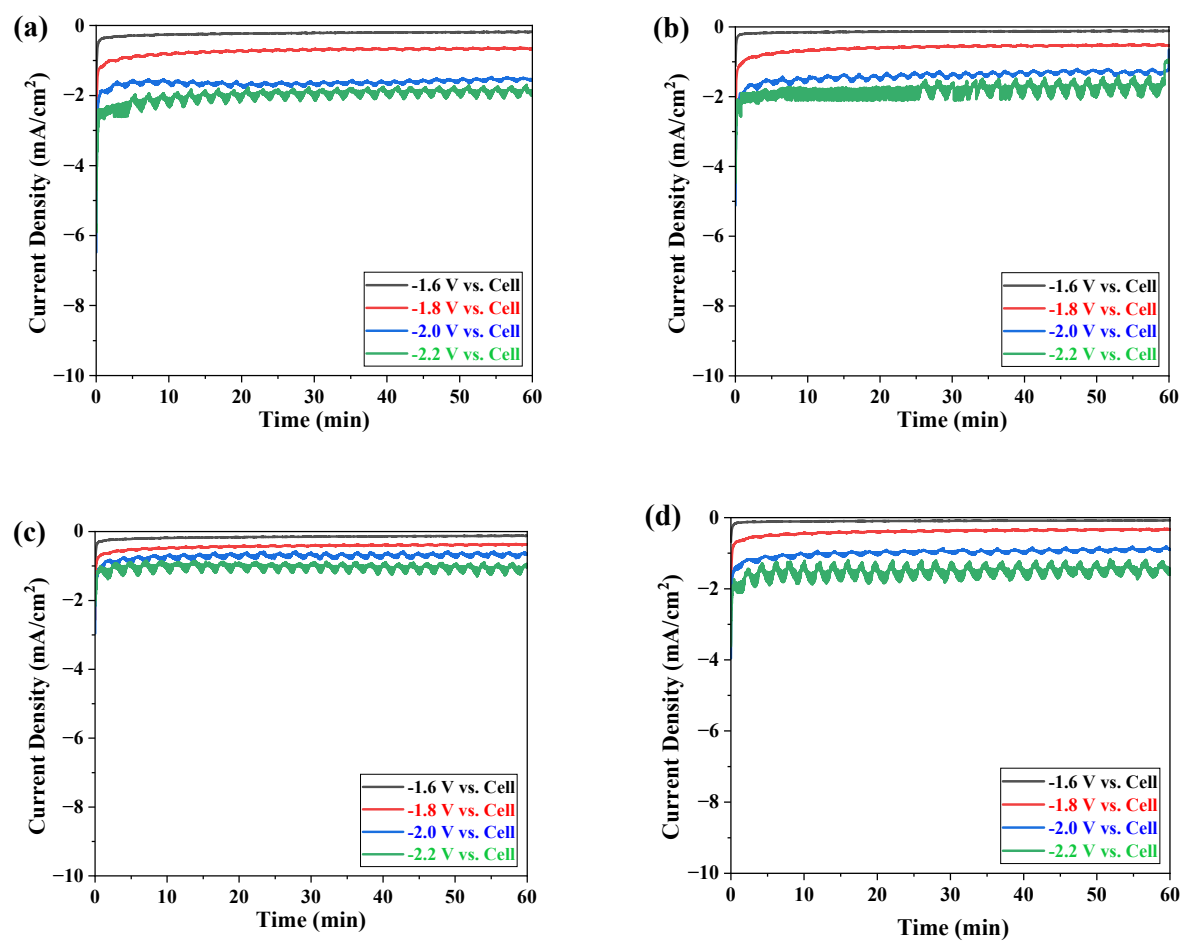


Figure S13. Chronoamperometry tests of Co-NC at a constant potential (vs. cell) for 1 h with (a) Dry@RT NO, (b) Wet@RT, (c) Wet@40 °C, and (d) Wet@60 °C condition.

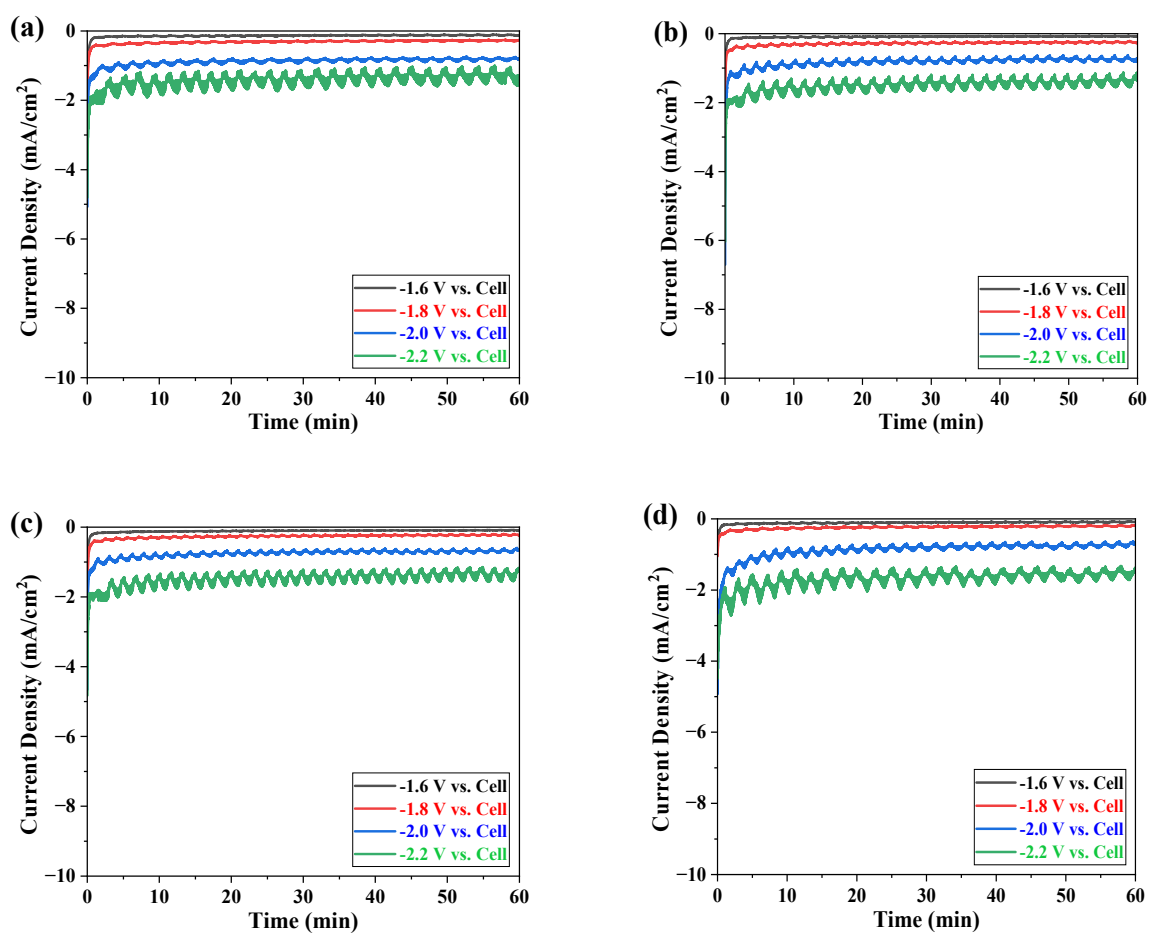


Figure S14. Chronoamperometry tests of Mo-NC at a constant potential (vs. cell) for 1 h with (a) Dry@RT NO, (b) Wet@RT, (c) Wet@40 °C, and (d) Wet@60 °C condition.

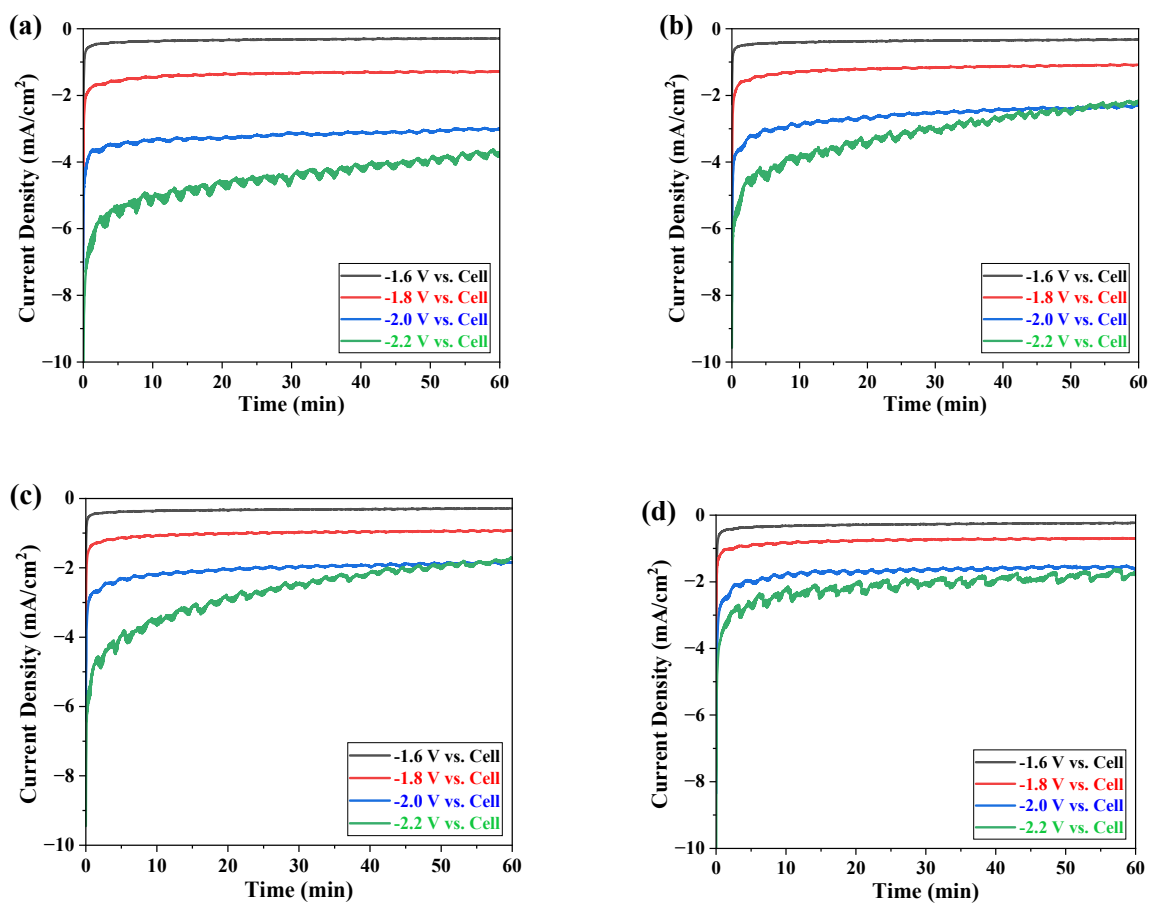


Figure S15. Chronoamperometry tests of CoMo-NC at a constant potential (vs. cell) for 1 h with (a) Dry@RT NO, (b) Wet@RT, (c) Wet@40 °C, and (d) Wet@60 °C condition.

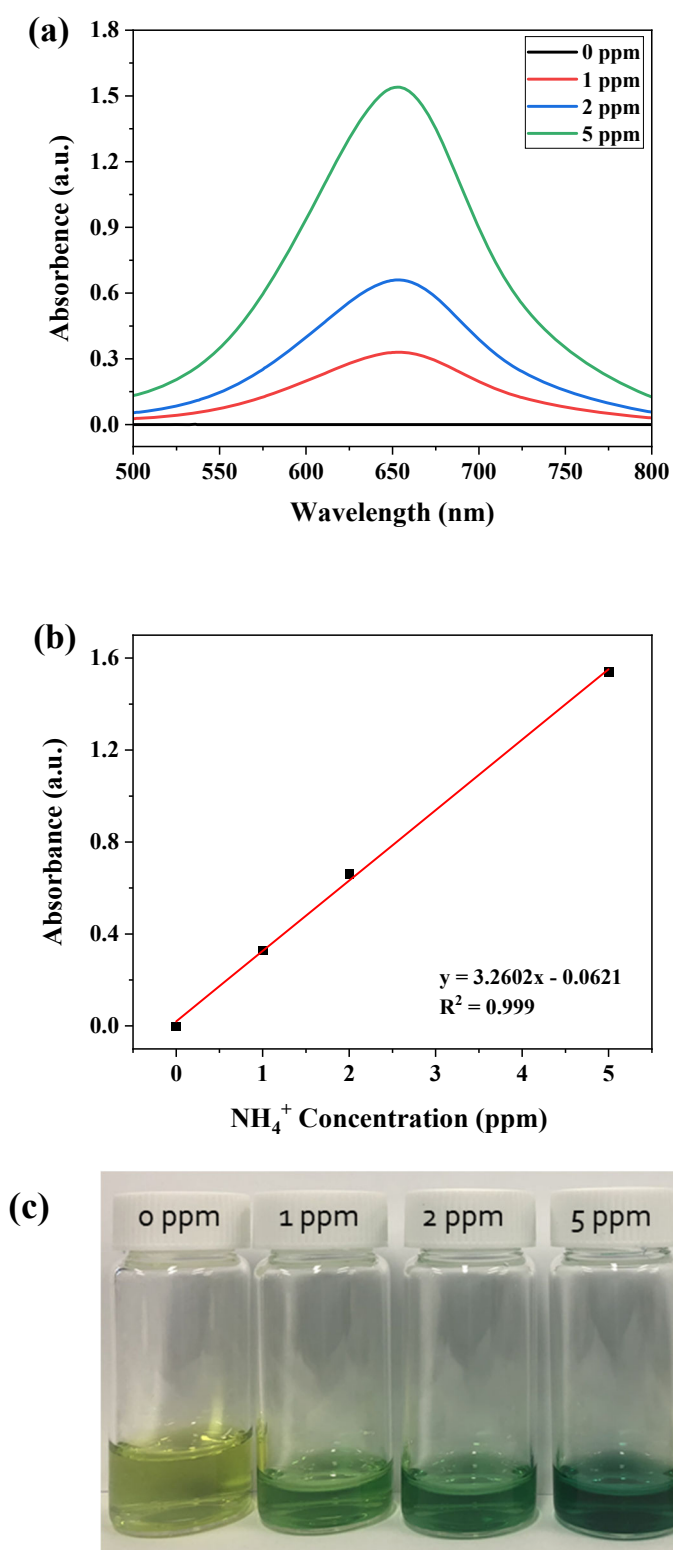


Figure S16. (a) UV-vis spectra (b) calibration curve and (c) photograph of standard NH_4^+ solutions.

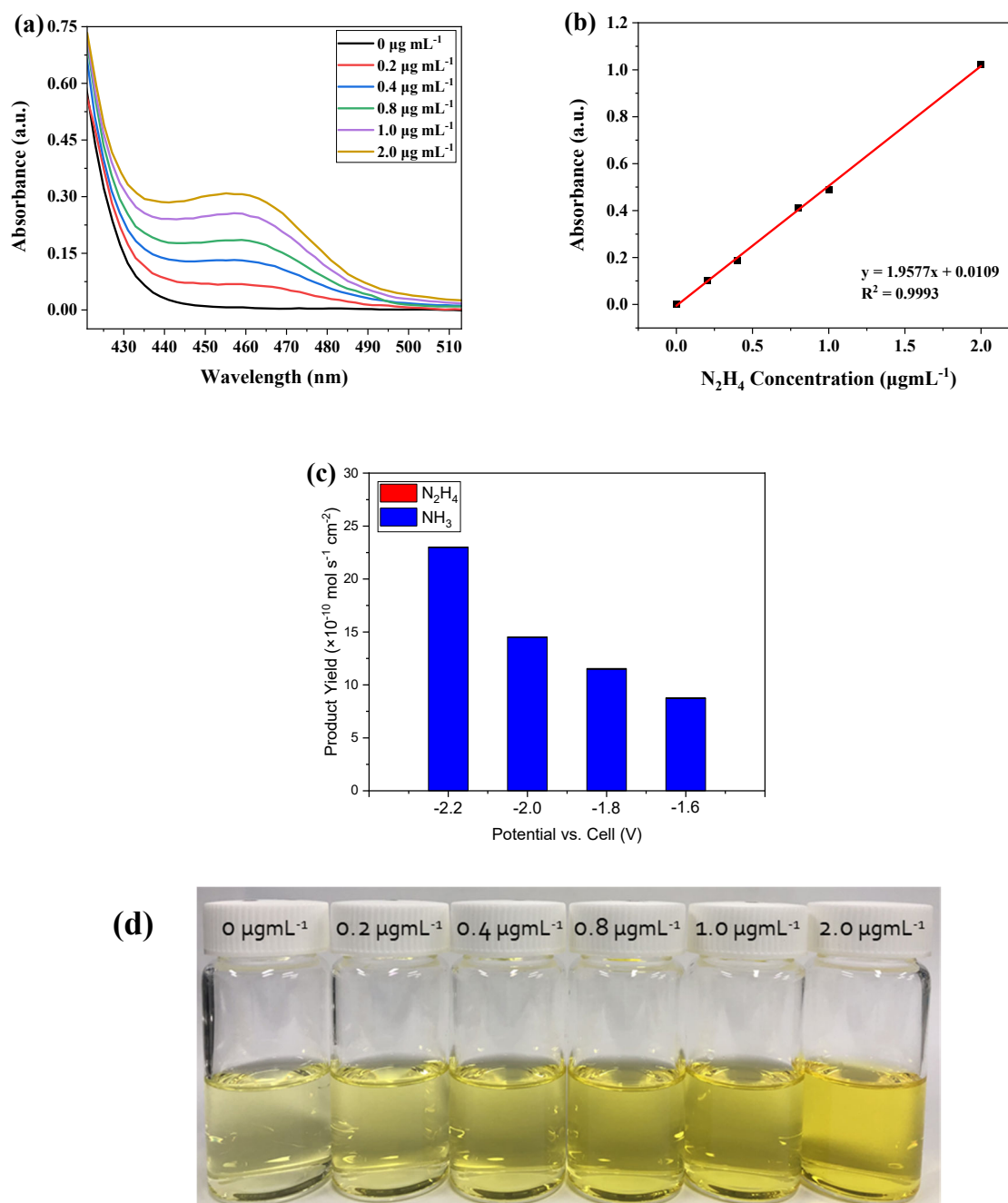


Figure S17. (a) UV-vis spectra (b) calibration curve, (c) NH_3 and N_2H_4 yield rates in NORR at different potentials, and (d) photograph of standard N_2H_4 solutions.

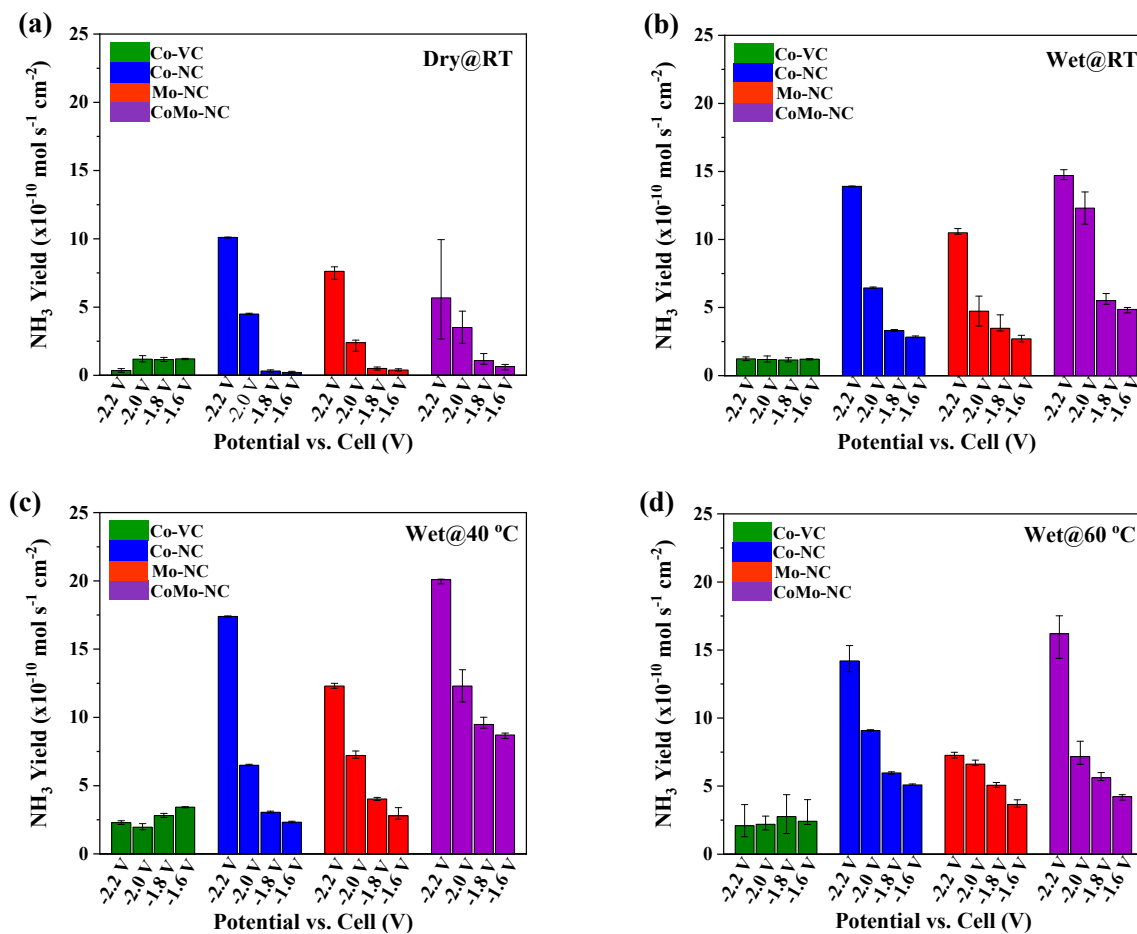


Figure S18. NH_3 yield rates at different potentials (vs. cell) and NO feed of (a) Dry@RT, (b) Wet@RT, (c) Wet@40 °C, and (d) Wet@60 °C of Co-VC, Co-NC, Mo-NC, and CoMo-NC.

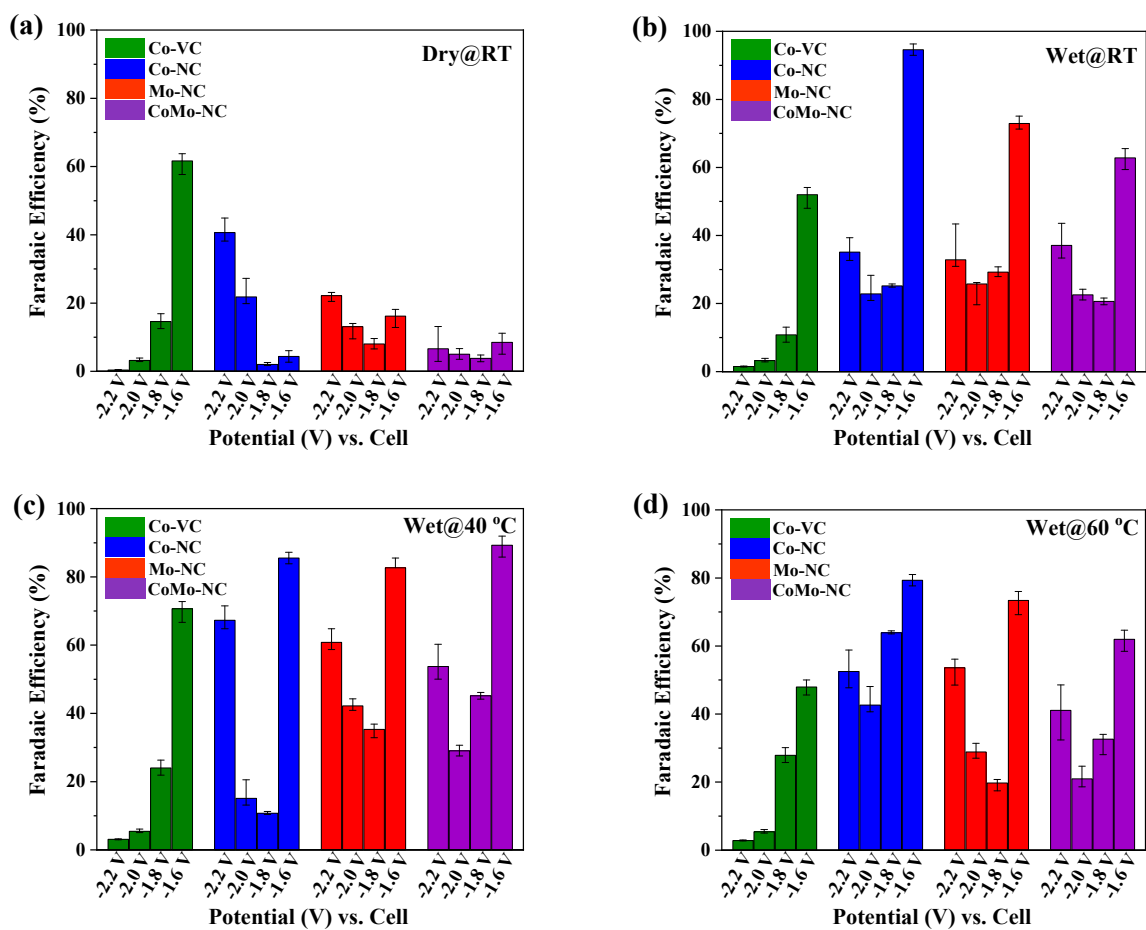


Figure S19. Faradaic efficiencies at different potentials (vs. cell) and NO feed of (a) Dry@RT, (b) Wet@RT, (c) Wet@40 °C, and (d) Wet@60 °C of Co-VC, Co-NC, Mo-NC, and CoMo-NC.

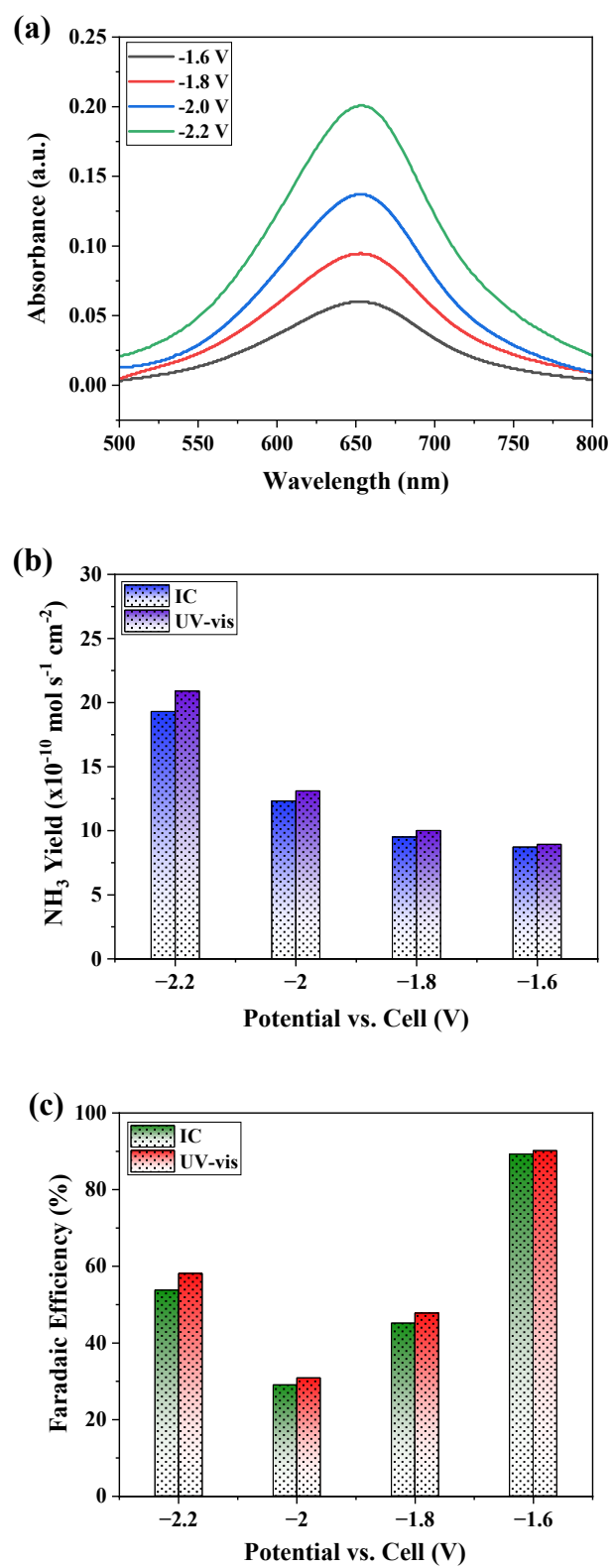


Figure S20. (a) NH_3 yield rates and (b) FE_{NH_3} quantified by IC and UV-vis (c) UV-vis absorption spectra at different potentials after 1 h electrolysis for NH_3 quantification.

Table S1. CoMo-C comparison with reported electrocatalysts for electrochemical ammonia synthesis

Reactant Gas	Electrocatalysts	Electrolyte	Cell Type	NH₃ Yield Rate ($\mu\text{mol h}^{-1} \text{cm}^{-2}$)	FE_{NH₃} (%)	Ref
Pure NO	Cu foam	0.25 M Li ₂ SO ₄	H-type	517.1	93.5	[4]
10% NO	K ₂ [Ni(CN) ₄]	Catholyte-free	Flow type	1128.9	89.5	[6]
1% NO	Ag Nanoparticles	FeEDTA+ 0.5 M PBS	Flow type	360	100	[13]
10% NO	Fe/C	0.5 M PBS	Flow type with GDE	908	77	[25]
10% NO	Fe/C	0.5 M H ₂ SO ₄	Flow type with GDE	1239	50.4	[25]
N ₂	Fe/CNT	Catholyte-free	H-type with GDE	0.013	0.05	[33]
N ₂	N-doped carbon nanospikes	0.25 M LiClO ₄	H-type	5.7	11.6	[34]
4.8% NO	Ru/C	Catholyte-free	Flow type	510	93	[35]
99.9% NO	Cu ₇₅ Ni ₂₅ @NC	1 M KOH	H-type	3.6	79	[36]
Pure NO	CuRh NSs	0.1 M Na ₂ SO ₄	H-type	436	93.1	[37]
1% NO	CoMo-NC	Catholyte-free	Flow type	8.34	94.6	This work