Supporting Information

Sustainable Two-Step Electrochemical Conversion of N_2 to Ammonia Using

Rhodium Nanoparticles on Carbon Nanosheets

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Table S7. eNOR production rate and Faradaic efficiency of commercial 5 wt.% Rh/C under N_2 and air atmosphere.



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Calibration Data

Result: PASS

Equation: y = -40.7605x + 76.0037

	Result	Minimum	Maximum		Calibration	Recorded
Slope:	-40,7605	-62.0000	-53,0000		Standard	Standard
Intercent	76 0037	-100 0000	100 0000		0.1000	115.8730
Correlation:	0.9986	0.9900	1 0000		1.0000	77.7860
Correlation:	0.5500	0.5500	1.0000	10.0000	34.3520	

Figure S2. UV-Visible spectrophotometric data of standard ammonia samples measured by Indophenol blue method (a & c) in 0.1 M KOH solution and (b & d) 1 mM H_2SO_4 solution, and (e) calibration plot of ammonia ion selective electrode using 0.1, 1.0 and 10.0 ppm standard ammonia solutions.



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 Table S1. Summary of XRD, Raman and ICP-OES results of as synthesized catalysts

 compared with commercial 5 wt.% Rh/C.

	Crystallite size	I _D /I _G ratio	Rh wt.%
Sample name	(from XRD – (111)	(from	(from ICP-

	plane)	Raman)	OES)
Commercial 5 wt.% Rh/C	127.3 nm	1.14	5.2 ± 0.2%
2 wt.% Rh/C	12.8 nm	0.69	$2.2 \pm 0.2\%$
5 wt.% Rh/C	14.5 nm	0.75	$5.6\pm0.4\%$
20 wt.% Rh/C	34.2 nm	0.85	$20.7 \pm 1.1\%$
40 wt.% Rh/C	63.5 nm	0.89	$40.5 \pm 1.8\%$



Figure S4. (a) SEM image, (b) EDX layered mapping, (c) Rh, (d) C mapping, and (e) corresponding EDX spectra of as synthesized 5 wt.% Rh/C catalyst.



Figure S5. (a and b) STEM images of 5 wt.% Rh/C synthesized in different batches, and (c and d) corresponding Rh particle size distribution; (e) TGA data of "as synthesized" 20 wt.% Rh/C catalyst.



Figure S6. (a & b) Chronoamperometric curve of 5 wt.% Rh/C in N₂ and air saturated 0.1 M KOH at different applied potential; (c & d) UV-Vis spectrometric data of the electrolyte sample after eNOR electrolysis at different potential under N₂ and air atmosphere; (e) linear sweep voltammogram of carbon support without Rh loading at a scan rate of 5 mV s⁻¹ in Ar and N₂-saturated 0.1 M KOH.



Figure S7. (a) Linear sweep voltammogram at a scan rate of 5 mV s⁻¹ in N₂-saturated 0.1 M KOH for different Rh/C; (b), (c) and (d) NO_3^- formation rate and Faradaic efficiency at different selected applied potentials for 2 wt.% Rh/C, 20 wt.% Rh/C and 40 wt.% Rh/C catalysts respectively in N₂-saturated 0.1 M KOH.



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Table S2. Resistance (obtained from EIS at 1.7 V vs. RHE) and ECSA values of Rh/C catalyst
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Electrode	$R_{S} (\Omega \text{ cm}^{2})$	R_{CT} (Ω cm ²)	ECSA (in cm²/g _{Rh})
2 wt.% Rh/C	7.53	111.52	63.95
5 wt.% Rh/C	7.38	84.77	300.01
20 wt.% Rh/C	7.48	61.45	176.20
40 wt.% Rh/C	7.25	30.82	154.53



Figure S9. UV-Vis spectrometric data of the electrolyte sample after eNOR electrolysis stability test of 5 wt.% Rh/C at 1.7 V vs. RHE in (a) N_2 and (b) air saturated 0.1 M KOH.



Figure S10. (a) XRD pattern, and (b) Raman spectra of 5 wt.% Rh/C coated Toray carbon electrode before and after 24 hours eNOR at 1.7 V vs. RHE in N₂ and air saturated 0.1 M KOH.



Figure S11. (a) XPS survey spectra, and (b) deconvoluted O 1s spectra of 5 wt.% Rh/C before and after eNOR stability studies at 1.7 V vs. RHE.

Table S3. Summary of XPS results of 5 wt.% Rh/C before and after eNOR stability studies at1.7 V vs. RHE under air.

Sample	Rh 3d (peak area %)		C 1s (peak area %)			O 1s (peak area %)		
Sample	Rh ⁰	Rh ¹⁺	C-C /C=C	C-0	С=О	Rh-O	C-0	Rh-O-H

Pristine	58.9	41.1	46.7	35.7	17.6	1.0	50.7	48.3
After Stability	46.8	53.2	48.7	13.2	38.1	-	79.5	20.5



Figure S12. The comparison of commercial and synthesized 5 wt.% Rh/C catalysts in (a) nitrate formation rate, and (b) Faradaic efficiency at 1.7 V vs. RHE in N₂ and air saturated 0.1 M KOH solution.



Figure S13: (a-c) STEM images of commercial 5 wt.% Rh/C material at different spots.



Figure S14. UV-Vis spectra of elctrolyte after eNOR at different atmosphere using 5 wt.% Rh/C catalyst in 0.1 M KOH at 1.7 V vs. RHE.



Figure S15. UV-vis spectra of Indophenol ammonia quantification in (a) electrolyte, and (b) trap after eNO₃RR on RuO₂; (c) electrolyte, and (d) trap after eNO₃RR on 5 wt.% Rh/C

electrode at selected applied potentials in Ar saturated 0.1 M KOH containing 1.0 mM KNO₃ electrolyte.



Figure S16. (a) eNOR chronoamperometric plot of 5 wt.% Rh/C electrode under air atmosphere at 1.7 V vs. RHE, (b) ion chromatography data of electrolytes after eNOR at 1.7 V vs. RHE at N_2 and air; (c) UV-Vis spectra of Indophenol ammonia quantification of electrolyte after eNO₃RR on RuO₂ catalyst at -0.0 V vs. RHE, and (d) their corresponding ammonia formation rate with Faradaic efficiency at the same potential.

 Table S4. Comparison of nitrate and ammonia concentration analysed by different quantification techniques.

Atmospher	[NO ₃ -] (μM)			[NH ₃] (µM)			
e	UV	IC	Deviation	Indophenol	ISE	Deviation	

N ₂	243.5	261.3	7.3%	83.2	77.8	6.5%
Air	496.0	510.2	2.9%	283.6	260.2	8.2%



Figure S17. (a) N₂-TPD results of 5 and 20 wt.% Rh/C samples; (b) O₂, (c) N₂, and (d) He mass spectra of N₂ and air (79% N₂ + 21% O₂)-TPD results of 5 wt.% Rh/C sample.



Figure S18. (a) XRD pattern, and (b) Raman spectra of 5 wt.% Rh/C catalyst before and after N_2 and air TPD (25-500 °C).



Figure S19. Mass spectra recorded during the eNOR using 5 wt.% Rh/C catalyst in (a) air, and (b) N₂ saturated 0.1 M KOH.

 Table S5. eNOR production rate and Faradaic efficiency comparison of various reported catalysts.

S. No	Catalyst	Conditions	Potential (V vs. RHE)	Nitrate formation rate (µg h ⁻¹ mg _{cat} ⁻¹)	Faradaic efficiency %	Referenc e
1	Ru doped TiO ₂ /RuO ₂	0.1 M Na ₂ SO ₄ , N ₂ , 10 h	2.20	0.1	2.50	1
2	Co ₃ O ₄	0.1 M Na ₂ SO ₄ , N ₂ , 10 h	1.70	0.09	20.40	2
3	Fe-SnO ₂	0.05 M H ₂ SO ₄ , N ₂ , 2 h	1.96	0.04	0.84	3
4	Ru-Mn ₃ O ₄	0.1 M Na ₂ SO ₄ , N ₂ , 2 h	2.00	35.3	6.33	4
5	np-B ₁₃ C ₂	0.1 M Na ₂ SO ₄ , N ₂ , 2 h	2.40	165.8	4.90	5

6	Tensile- Strained Palladium Porous Nanosheets	1.0 М КОН, N ₂	1.75	18.6	1.55	6
7	Rh nanoparticles	0.5 M SO ₄ ²⁻ in 0.1 M KOH, N ₂ ,	1.90	10.4	2.30	7
8	Fluidized V ₂ O ₅ nanorods	0.1 M Na ₂ SO ₄ , N ₂ , 1 h	2.40	1388.0	7.80	8
9	Mo doped TiO ₂	0.1 M K ₂ SO ₄ , N ₂ , 12 h	2.10	5.4±0.24	2.88±0.13	9
10	Ferriporphyrin- based MOF	0.1 M HCl, N ₂ , 2 h	1.60	110.9	70.70	10
11	D-RuO ₂	0.05 M H ₂ SO ₄ , N ₂ , 2 h	1.44	767.9	0.94	11
12	Mo-(O-C ₂) ₄	0.1 M Na ₂ SO ₄ , N ₂ , 2 h	2.35	217.1±13.5	7.8±0.5	12
13	5 wt.% Rh/C	0.1 M KOH, N ₂ , 1 h	1.70	106.5	28.20	Present work

		NH ₃ formation	Faradaic	
Route	Conditions	rate (µg h ⁻¹	efficiency	
		mg_{Ru}^{-1})	(%)	
Direct	RuO_2 catalyst, 0.1 M KOH, N ₂ , -0.15 V	0.2	0.2	
eNRR ¹³	vs. RHE	8.2	0.3	
Direct	RuO ₂ catalyst, 0.1 M KOH + 1000 μ M			
eNO ₃ RR	NO_3^- , -0.0 V vs. RHE in Ar	25.7	11.9	
D 1	Step1 : 5 wt.% Rh/C catalyst, 0.1 M			
Dual step	KOH, Air, 1.7 V vs. RHE (eNOR); Step			
eNOR +		45.1	5.8	
NO PP	2 : RuO_2 catalyst, -0.0 V vs. RHE			
en03KK	(eNO ₃ RR) in Ar			

 Table S6. Comparison of ammonia synthesis efficiencies using different electrochemical routes.

Table S7. eNOR production rate and Faradaic efficiency of commercial 5 wt.% Rh/C under N_2 and air atmosphere.

	Rate unde	Rate under N ₂		Rate under Air					
Potential					FE % (N ₂)		FE % (Air)		
	$(\mu g/h/cm^2)$		$(\mu g/h/cm^2)$						
(V vs.									
	Commercial	In-	Commercial	In-	Commercial	In-	Commercial	In-	
RHE)									
		house		house		house		house	
1.6	14.43	16.46	29.46	41.47	22.55	37.22	35.2	99.58	
1.7	12.22	21.28	21.81	46.93	17.31	21.01	26.79	30.91	
1.8	10.22	18.86	18.15	48.64	7.21	8.49	10.28	12.03	
1.9	6.30	16.42	10.66	39.35	2.37	3.24	5.55	4.17	

2.0	5.97	13.75	8.23	30.52	1.02	2.16	2.34	2.26

Proposed Working Model-Continuous NH₃ Synthesis by Coupling of eNOR and eNO₃RR

In the present study, the electrochemical conversion of N_2 to NH_3 via the formation of NO_3^- was demonstrated in two steps, i.e., oxidation of N_2 oxidation to NO_3^- in the first step via eNOR followed by the reduction of the formed NO_3^- to NH_3 via eNO_3RR . To address the inefficiencies associated with the hydrogen evolution reaction (HER) and oxygen evolution reaction (OER) during eNOR and eNO_3RR , a continuous flow cell setup (Figure S20) is designed to operate in a cascade configuration. In this system, nitrate generated at the anode is directly utilized at the cathode for ammonia production.

The proposed continuous flow electrolyzer integrates anodic nitrogen oxidation (eNOR) and cathodic nitrate reduction (eNO₃RR) in a two separated compartments electrochemical cell that together can enable sustainable ammonia synthesis. In this design, the anode compartment is supplied with a N₂/air-saturated KOH electrolyte from Reservoir 1, where nitrogen is oxidized according to the reaction:

 $N_2 + 12OH^- \rightarrow 2NO_3^- + 6H_2O + 10e^- (E^o = 1.32 \text{ V vs. RHE})$

The electrochemically generated nitrate ions (NO_3) in the analyte are then transferred directly into the cathode compartment, where they undergo reduction to ammonia through the reaction:

$$NO_3^- + 6H_2O + 8e^- \rightarrow NH_3 + 9OH^-$$
 (E° = -0.12 V vs. RHE)

The catholyte, containing the generated ammonia and any unreacted nitrate, will collect in Reservoir 2. The electrolyte flow between compartments can be precisely regulated using a peristaltic pump, enabling continuous operation under an expected modest applied voltage of ~2.0 V. This is well-aligned with the thermodynamic requirement ($\Delta E^{\circ} \approx 1.44$ V), ensuring efficient energy utilization. An optimized anode and cathode size and other cell parameters including the pump-controlled flow rate between these compartments (shown in Figure S20) should provide a more efficient continuous flow process with higher yield. For example, higher ammonia reduction rate and efficiency at higher NO₃⁻ concentration provided from the larger anode size. The system can offer key advantages: high nitrogen utilization, minimized energy loss through operation near theoretical voltages, and a modular design suitable for commercial scale-up.

Therefore, matching catalyst activity and optimizing reactor design are essential future directions to ensure synchronized reaction rates. Overall, this cascade-flow electrolyzer represents a promising step forward in green ammonia synthesis, combining electrochemical innovation with process integration. With continued advancements in electrocatalyst design, reaction engineering, and system scalability, this approach has strong potential for industrial translation.



Figure S20. Schematic representation of proposed working model-Electrolysis cell with coupled eNOR and eNO₃RR.

Calculation of standard Gibbs energy

$$N_2 + 6H_2O + 6e^- \rightarrow 2NH_3 + 6OH^-$$
 (1)

$$N_2 + 10OH^- \rightarrow 2HNO_3 + 4H_2O + 10e^-$$
 (2)

$$NO_3^- + 6H_2O + 8e^- \rightarrow NH_3 + 9OH^-$$
(3)

The standard Gibbs free energy change of a reaction was calculated using the following equation:

$$\Delta^{G_{f(reaction)}} = \sum n \Delta G_{f}^{0} \text{ of products } - \sum n \Delta G_{f}^{0} \text{ of reactants}$$

Standard Gibbs Free Energy of Formation (ΔG^{o}_{f})

The standard Gibbs free energies of formation at 25°C (298 K)¹⁴ for the species involved:

- N_{2(g)}: 0 kJ/mol
- NO_{3⁻(aq)}: -110.6 kJ/mol
- NH_{3(aq)}: -26.5 kJ/mol
- OH⁻_(aq): -157.2 kJ/mol
- H₂O₍₁₎: -237.1 kJ/mol

Calculation of ΔG° for Each Reaction

Reaction (1): $N_2 + 6H_2O \rightarrow 2NH_3 + 6OH^2$

$$\Delta G_{1}^{0} = [2 \times (-26.5) + 6 \times (-157.2)] - [1 \times (0) + 6 \times (-237.1)] = 426.4 \text{ kJ/mol} \times 0.01036 = 4.42 \text{ eV}$$

Reaction (2): $N_2 + 10OH^- \rightarrow 2HNO_3 + 4H_2O$

$$\Delta G_2^{\circ} = [2 \times (-111.3) + 4 \times (-237.1)] - [1 \times (0) + 10 \times (-157.2)] = 401.4 \text{ kJ/mol} \times 0.01036 = 4.15 \text{ eV}$$

Reaction (3): $NO_3^- + 6H_2O \rightarrow NH_3 + 9OH^-$

$$\Delta G_3^{\circ} = [-26.5 + 9 \times (-157.2)] - [-111.3 + 6 \times (-237.1)] = 92.6 \text{ kJ/mol} \times 0.01036 = 0.96 \text{ eV}$$

Summary

- Electrochemical nitrogen reduction: $\Delta G^{\circ} = 4.42 \text{ eV}$
- Electrochemical nitrate reduction: $\Delta G^{\circ} = 0.96 \text{ eV}$
- Dual step eNOR+eNO₃RR: $\Delta G^{\circ} = 5.11 \text{ eV}$

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