Enhanced NH$_3$ Synthesis from Air in a Plasma Tandem-Electrocatalysis System Using Plasma-Engraved N-Doped Defective MoS$_2$

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**ABSTRACT:** We have developed a sustainable method to produce NH$_3$ directly from air using a plasma tandem-electrocatalysis system that operates via the N$_2$−NO$_x$−NH$_3$ pathway. To efficiently reduce NO$_2^−$ to NH$_3$, we propose a novel electrocatalyst consisting of defective N-doped molybdenum sulfide nanosheets on vertical graphene arrays (N-MoS$_2$/VGs). We used a plasma engraving process to form the metallic 1T phase, N doping, and S vacancies in the electrocatalyst simultaneously. Our system exhibited a remarkable NH$_3$ production rate of 7.3 mg h$^{-1}$ cm$^{-2}$ at −0.53 V vs RHE, which is almost 100 times higher than the state-of-the-art electrochemical nitrogen reduction reaction and more than double that of other hybrid systems. Moreover, a low energy consumption of 2.4 MJ mol$^{-1}$ of NH$_3$ was achieved in this study. Density functional theory calculations revealed that S vacancies and doped N atoms play a dominant role in the selective reduction of NO$_2^−$ to NH$_3$. This study opens up new avenues for efficient NH$_3$ production using cascade systems.

**KEYWORDS:** sustainable NH$_3$ production, plasma electrocatalysis, defective N-MoS$_2$, plasma engraving, density functional theory

**INTRODUCTION**

Ammonia (NH$_3$) is essential for modern agriculture and is being explored as a promising carbon-free hydrogen carrier to tackle climate change and energy challenges. However, the conventional centralized Haber−Bosch process for industrial NH$_3$ production requires extremely harsh conditions, resulting in high energy demands and significant CO$_2$ emissions. Therefore, developing sustainable, environmentally friendly, and distributable routes for NH$_3$ production is critical.

One promising approach is electrochemical nitrogen reduction reaction (eNRR) to NH$_3$, which can be powered by renewable electricity using abundant N$_2$/air and H$_2$O under ambient conditions. The performance of electrocatalysts is crucial for eNRR kinetics. Among the noble-metal-based transition-metal-based metal-free electrocatalysts explored, Mo, especially MoS$_2$, is of particular interest due to its natural occurrence in biological nitrogen fixation and structural similarity to nitrogenase.

Recently, there has been a surge in efforts to enhance catalyst reactivity through phase transformation, heteroatom doping, and defect engineering. Nevertheless, the ultrastrong N≡N bond and poor solubility of N$_2$ in the electrolyte have limited the NH$_3$ production rate to <0.1 mg h$^{-1}$ cm$^{-2}$.

To address these challenges, a promising two-step strategy has been proposed that uses plasma to activate inert N$_2$ into more accessible and clean nitrogen oxides (NO$_x$), followed by enhanced electroreduction of NO$_2^−$ into NH$_3$. Although NO$_2^−$ may be abundant in wastewater, it would be unsuitable for this application due to the presence of impurities. The plasma electrocatalysis approach uses air instead of pure nitrogen, eliminating the need for energy-intensive air separation and purification processes, and is suitable for intermittent and decentralized operation using renewable energy sources as a turnkey process. While an NH$_3$ production rate of up to 3.0 mg h$^{-1}$ cm$^{-2}$ has been achieved, investigations into this strategy remain extremely limited.

Further development of new electrochemical NO$_x$ reduction reaction (eNO$_x$−RR) processes, including system design and high-performance catalysts such as MoS$_2$, is critical to improve the slow conversion of NO$_x^−$ to NH$_3$ in the second
step. Moreover, existing catalyst engineering strategies typically require high temperature (>300 °C) and long-duration treatment (>2 h), calling for a more facile and rational catalyst fabrication approach. Furthermore, a molecular-level understanding of the complex eNO \(^{-}\)RR mechanism remains limited, requiring in-depth experimental and theoretical studies.

Herein we propose a plasma tandem-electrocatalysis system to generate NH\(_3\) from air (see Plasma Tandem-Electrocatalysis Setup and Figure S1). We engineered a novel defective N-MoS\(_2\) electrocatalyst supported by vertical graphene arrays on carbon cloth (VGs/CC) with exceptional eNO \(^{-}\)RR reactivity. Advanced physical characterizations and density functional theory (DFT) calculations were conducted to elucidate the eNO \(^{-}\)RR reaction pathways with N-MoS\(_2\)/VGs.

### RESULTS AND DISCUSSION

The MoS\(_2\) nanosheets were assembled by a hydrothermal (HT) method and supported by vertical graphene arrays on carbon cloth (VGs/CC) with exceptional eNO \(^{-}\)RR reactivity. Advanced physical characterizations and density functional theory (DFT) calculations were conducted to elucidate the eNO \(^{-}\)RR reaction pathways with N-MoS\(_2\)/VGs.

Notably, we pioneered a radiofrequency (RF) NH\(_3\) plasma strategy for atomic-level surface engineering of MoS\(_2\) (Figure 1a). By creation of a plasma sheath on the sample surface, ablated Mo atoms reacted with N atoms in the plasma to form N-MoS\(_2\). Furthermore, the plasma treatment also generated crystal vacancies via ion sputtering and etching effects of NH\(_3\). This method required only a short duration (20 min) and low temperature (25 °C). The formation of octahedrally coordinated 1T-MoS\(_2\) after the plasma engraving was confirmed by high-angle annular dark-field (HAADF) (Figure 1e) and spherical aberration corrected scanning transmission electron microscopy (AC-STEM) along the (100) zone axis (Figure 1f). The HRTEM images of MoS\(_2\)/VGs and N-MoS\(_2\)/VGs further evidenced the distinct phase transformation from 2H to 1T (Figures S2 and S3).

Additionally, energy-dispersive X-ray (EDX) elemental mapping images demonstrated the uniform distribution of Mo, S, and N in the N-MoS\(_2\)/VGs (Figure 1g). X-ray diffraction (XRD) patterns (Figure 2a) reveal characteristic peaks at 14.37°, 32.68°, and 39.54° that...
correspond to the (002), (100), and (103) planes of MoS$_2$ (PDF no. 37-1492) for N-MoS$_2$/VGs, MoS$_2$/VGs, and VGs. The 1T phase of N-MoS$_2$/VGs is further confirmed by Raman spectroscopy (Figure 2b). The E$_{2g}$ and A$_{1g}$ peaks at 380 and 405 cm$^{-1}$, respectively, are observed in both MoS$_2$/VGs and N-MoS$_2$/VGs, with N-MoS$_2$/VGs also exhibiting three additional peaks (148, 207, and 335 cm$^{-1}$) associated with vibrational modes of the 1T phase. The E$_{2g}$ peak (380 cm$^{-1}$) is also found in N-MoS$_2$/VGs due to the well-organized tetragonal symmetry.

X-ray photoelectron spectroscopy (XPS) (Figure 2c–e) revealed the presence of Mo, S, and N in N-MoS$_2$/VGs and the absence of N in MoS$_2$/VGs. The Mo 3d spectrum of MoS$_2$/VGs can be deconvoluted into four peaks, with two dominant peaks at 232.0 eV (Mo 3d$_{3/2}$) and 229.1 eV (Mo 3d$_{5/2}$) assigned to the 1T phase and two other peaks at 233.5 eV (Mo 3d$_{3/2}$) and 229.6 eV (Mo 3d$_{5/2}$) allocated to the 2H phase. Conversely, only two typical 1T phase peaks at 231.2 eV (Mo 3d$_{3/2}$) and 229.1 eV (Mo 3d$_{5/2}$) are detected in N-MoS$_2$/VGs. Furthermore, the positive shift of N-MoS$_2$/VGs to MoS$_2$/VGs also confirms the phase transformation from 2H to 1T. Notably, the S:Mo ratio decreased from 1.74 to 1.37, according to quantized EDX data (Table S1), likely due to the substitution of N and possible S vacancies induced by plasma engraving. This hypothesis is explicitly proven by the electron paramagnetic resonance (EPR) spectroscopy results (Figure 2f), which display a prominent signal at $g = 2.004$ assigned to S vacancies in N-MoS$_2$/VGs and no signal in MoS$_2$/VGs, suggesting the partial deletion of thiol–Mo bonds in N-MoS$_2$/VGs.

Comparative experiments in the plasma or electrocatalysis alone system and the plasma electrocatalysis system with different catalysts were performed at $-0.33$ V versus reversible hydrogen electrode (V vs RHE). As shown in Figure 3a, neither the plasma- or electrocatalysis-alone system nor the eNO$_x$RR over the substrates (CC and VGs/CC) yielded significant NH$_3$. However, the MoS$_2$ catalyst dramatically
enhanced the NH₃ production rate (5.2 mg h⁻¹ cm⁻²) by a factor of approximately 38 over the substrates. Notably, the defective N-MoS₂/VGs catalyst further improved the catalytic reactivity by over 20%, reaching a production rate of up to 6.2 mg h⁻¹ cm⁻². This enhancement is attributed to the improved electron conductivity, higher charge polarization, and increased active sites of N-MoS₂/VGs, as discussed above.

In this study, plasma tandem-electrocatalysis was utilized by coupling double electrocatalyst cells, and for comparison, a plasma single-electrocatalysis system was also tested. The results, depicted in Figure 3b,c, demonstrated that increasing potentials had a positive effect on the average NH₃ production rate but had a negative impact on the Faradaic efficiency (FE) in both modes. A maximum production rate of 4.6 mg h⁻¹ cm⁻² (FE = 44%) and a highest FE of 97.6% (NH₃ production = 1.12 mg h⁻¹ cm⁻²) were achieved, respectively, at −0.53 and −0.13 V vs RHE in the single-electrocatalysis mode. The tandem-electrocatalysis system significantly increased the production rate to 7.3 mg h⁻¹ cm⁻² (FE = 38.1%) at −0.53 V vs RHE (Figure 3c) (see Figures S6–S10 for more comparative results).

The air plasma produced a substantial NOₓ concentration (up to 1.87% with NO: NO₂ ≈ 5:1) for subsequent eNOₓ−RR process. To elucidate the origin of NH₃ in eNOₓ−RR, we measured the concentrations of NO₂⁻, NO₃⁻, and NH₄⁺ in the electrolyte during plasma discharge with and without electrocatalysis. NO₂⁻ was found to be the dominant NOₓ− (Figure 3d) with a formation rate of 28 mM h⁻¹, and it was significantly consumed in the presence of electrocatalysis, strongly indicating that NH₃ is primarily produced from NO₂⁻ (referred to as eNO₂− RR hereafter).

The catalyst stability was validated by successive repetitive (24 times in 24 h) tests at −0.33 V vs RHE (Figure 3e). The scanning electron microscopy (SEM) images of the N-MoS₂/VGs, as discussed above.

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Figure 3. (a–c) NH₃ production rates and FEs for (a) plasma only, N-MoS₂/VGs electrocatalysis only, and plasma electrocatalysis with different catalysts, (b) plasma single-electrocatalysis, and (c) plasma tandem-electrocatalysis. (d) Time-dependent concentrations of nitrate, nitrite, and NH₄⁺ with/without electrocatalysis in the hybrid system. (e) Stability tests. (f) ¹H NMR (600 MHz) spectra in ¹⁵N₂ or ¹⁴N₂ atmosphere after N-MoS₂/VGs electrocatalysis. (g) Comparison of eNRR using different MoS₂ catalysts (green symbols), other plasma-electrocatalysis systems (indigo symbols), and this work (red star). See Tables S6 and S7 for more details.
Figure 4. (a) Differential charge densities of adsorbed NO\textsuperscript{2−} on 1T-MoS\textsubscript{2} and 2H-MoS\textsubscript{2}. (b) DOS plots of 1T-MoS\textsubscript{2} and 2H-MoS\textsubscript{2}. (c) HER and (d) NRR Gibbs free energy diagrams on various adsorption sites.

Moreover, we conducted DFT calculations to elucidate the enhanced eNO\textsuperscript{2−} RR kinetics of the N-MoS\textsubscript{2} system (see Section S-VII-1 in the Supporting Information (SI) for details). Our simulation model (Figure S15) featured N-MoS\textsubscript{2}(100) with S vacancies, which correlated with the catalyst characterization results. Figures 4a, S16, and S17 illustrate significant differences in the differential charge density of adsorbed NO\textsuperscript{2−} on 2H and 1T phases of MoS\textsubscript{2}. In the 1T phase, the electron accumulation (yellow areas) and electron loss (blue areas) are more extensive, reflecting greater electron delocalization. Bader charge calculations\textsuperscript{31} revealed that NO\textsuperscript{2−} chemisorption is more favorable on the metastable 1T-MoS\textsubscript{2} with an average charge transfer of ~0.86e\textsuperscript{−}, compared to ~0.79e\textsuperscript{−} on 2H-MoS\textsubscript{2}. Furthermore, Figure 4b compares the densities of states (DOS) of 1T-MoS\textsubscript{2} and 2H-MoS\textsubscript{2}, showing that the d-band center position of 1T-MoS\textsubscript{2} (ε\textsubscript{d}=−3.99 eV) is closer to the Fermi level (E\textsubscript{F}) than that of 2H-MoS\textsubscript{2} (ε\textsubscript{d}=−4.17 eV), which favors NO\textsuperscript{2−} adsorption. The partial density of states also indicates that 1T-MoS\textsubscript{2} has a larger overlap of the Mo d valence orbitals with the N p valence orbitals than 2H-MoS\textsubscript{2} (Figure S18), suggesting that 1T-MoS\textsubscript{2} has more occupied Mo orbitals and a stronger binding with N atoms from adsorbates such as NO\textsuperscript{2−}.

We also investigated various adsorption sites of NO\textsuperscript{2−} and H\textsuperscript{+} on N-MoS\textsubscript{2}, including Mo top distal (TD), Mo−Mo bridge distal (BD), Mo with N-doped sites (ND), and Mo with S vacancy sites (SV) (Figure S19). We found that eNO\textsuperscript{2−} RR is inhibited on VGs due to the extremely high adsorption energy (1.54 eV; Table S3). As shown in the Gibbs free energy diagrams of HER in Figure 4c, compared to the adsorption sites for pristine MoS\textsubscript{2} (ΔG\textsubscript{H2}\textsubscript{H}=−0.06 eV and ΔG\textsubscript{H2}\textsubscript{H}=0.02 eV), N-doped sites and S vacancy sites in N-MoS\textsubscript{2} (ΔG\textsubscript{H2}\textsubscript{H}=1.17 eV and ΔG\textsubscript{H2}\textsubscript{H}=0.41 eV) yield higher energy barriers, confirming that the enhanced eNO\textsuperscript{2−} RR selectivity correlates with the suppression of HER. Importantly, the free energy changes of the eNO\textsuperscript{2−} RR pathways at different adsorption sites (Figure 4d; see Figures S21−S25 for more details) indicate that the energy barrier of the potential-determining step (PDS) for eNO\textsuperscript{2−} RR in N-doped MoS\textsubscript{2} (ΔG\textsubscript{PDS}\textsubscript{2}=0.43 eV and ΔG\textsubscript{PDS}\textsubscript{2}=0.68 eV) exhibits significantly better eNO\textsuperscript{2−} RR preference than MoS\textsubscript{2} (ΔG\textsubscript{PDS}\textsubscript{2}=1.19 eV and ΔG\textsubscript{PDS}\textsubscript{2}=1.67 eV). These findings lead us to conclude that 1T-MoS\textsubscript{2} facilitates stronger binding and faster electron transfer and that Mo with N doping and S vacancies is beneficial for NH\textsubscript{3} synthesis.

The optimal energy cost of the system is 1.41 MJ mol\textsubscript{NH\textsubscript{3}}\textsuperscript{−1} for plasma NO\textsubscript{x} generation and 0.99 MJ mol\textsubscript{NH\textsubscript{3}}\textsuperscript{−1} for eNO\textsuperscript{2−} RR, yielding a total energy cost of 2.4 MJ mol\textsubscript{NH\textsubscript{3}}\textsuperscript{−1}. This corresponds to a total of 39.2 kWh kg\textsubscript{NH\textsubscript{3}}\textsuperscript{−1}, which is at least 24.5% lower than that of the state-of-the-art plasma electrocatalysis systems (Table S7). Moreover, the proposed system has the potential to be extended to a cascade system by coupling plasma with multistage electrocatalyst cells, which can be powered flexibly by intermittent renewable electricity (Tables S8 and S9).

**CONCLUSIONS**

In summary, we have developed a plasma tandem-electrocatalysis system for NH\textsubscript{3} synthesis directly from air, achieving an impressive NH\textsubscript{3} production rate of 7.3 mg h\textsuperscript{−1} cm\textsuperscript{−2} at −0.53 V vs RHE. We engineered a novel defective N-MoS\textsubscript{2}/ VGs electrocatalyst, which incorporates metallic 1T-MoS\textsubscript{2} phase, N doping, and S vacancies through a one-step plasma engraving process. Experimental and DFT investigations confirmed the crucial role of plasma engraving in catalyst
fabrication. The adsorption sites of Mo with N doping and S vacancies possess a lower energy barrier for PDS for NH₃ formation than for H₂ generation, explaining the enhanced eNO₂⁻RR performance. This system provides a carbon-free and sustainable approach to NH₃ synthesis with an enhanced production rate. It also serves as a promising solution for chemical storage of carbon-neutral fuels with high location and scale flexibility, adapting readily to the irregular supply of renewable electricity.

**EXPERIMENTAL SECTION**

**Plasma Tandem-Electrocatalysis Setup**

The plasma tandem-electrocatalysis system used in this study is schematically shown in Figure S1. A small-scale atmospheric gliding arc plasma (GAP) reactor was designed to generate NO₃ from air. The reactor consisted of a stainless steel inner anode rod with a diameter of 4 mm and a length of 58 mm and an outer cathode shell (grounded) with a convergent nozzle having a diameter of 21 mm and a thickness of 7.5 mm. The discharge was powered by an AC high voltage power supply (GK-10020L). Dry air was fed into the reactor at a flow rate of 9 L min⁻¹, which was controlled by a mass flow controller (MFC, TJ-700C). The arc was ignited at the narrowest gap (kept at 1 mm). The GAP reactor had three radial gas inlets to create more intense flow disturbances inside the reactor, allowing for the formation of an extended plasma area. The reactive plasma system effectively activated the nitrogen bond, leading to the formation of NO₂. To quantify the NO₂ produced with high concentration (NO and NO₂), the outlet gas was diluted with pure nitrogen and measured using a gas analyzer (MRU VARIO). The generated NO₂ was continuously fed into the electroreduction cells (single or tandem) for 1 h, absorbed by the alkaline electrolyte, and catalyzed into NH₃.

**Catalyst Synthesis**

VG arrays were first prepared using an RF PECVD method powered by a high-frequency generator (Kmate, HERO-500 W, 13.56 MHz). A VG arrays were first prepared using an RF PECVD method powered by a high-frequency generator (Kmate, HERO-500 W, 13.56 MHz). A high voltage power supply (GK-10020L). Dry air was fed into the reactor at a flow rate of 9 L min⁻¹, which was controlled by a mass flow controller (MFC, TJ-700C). The arc was ignited at the narrowest gap (kept at 1 mm). The GAP reactor had three radial gas inlets to create more intense flow disturbances inside the reactor, allowing for the formation of an extended plasma area. The reactive plasma system effectively activated the nitrogen bond, leading to the formation of NO₂. To quantify the NO₂ produced with high concentration (NO and NO₂), the outlet gas was diluted with pure nitrogen and measured using a gas analyzer (MRU VARIO). The generated NO₂ was continuously fed into the electroreduction cells (single or tandem) for 1 h, absorbed by the alkaline electrolyte, and catalyzed into NH₃.

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**Field-emission SEM images were recorded using a Hitachi SU8010. TEM images, EDS, elemental mappings, and a SAED pattern were collected using a JEOL 2100F microscope. HAADF images were collected using a FEM-ARM200F microscope. XRD patterns were obtained using Cu Kα radiation on a Rigaku D/Max-2550 diffractometer. Raman spectra were collected using a Renishaw-in Via Raman microscope with 514 nm laser excitation. XPS was obtained using an ESCALAB 250Xi X-ray photoelectron spectrometer. Specific surface area distributions were determined using a JW-BK112 analyzer. EPR spectra were taken using a JES FA-200 continuous-wave spectrometer with a sweeping magnetic field and an X band (9.2 GHz) at room temperature. Details of these methods are available in SI.

**ASSOCIATED CONTENT**

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/jacsau.3c00087.

Additional experimental details and methods; energy consumption calculations; phase transformation and kinetic enhancement enabled by plasma engraving; calibration curves of NH₃⁺ and N₂H₄ and ¹H NMR spectra, chronoenamperometry curves, UV–vis absorption spectra, and NRR performance for each reaction; morphology of the catalyst before and after stability tests; ammonia quantification by ¹⁵N and ¹⁴N isotope labeling; DFT calculation details; energy consumption comparison of plasma NO₂ generation and eNO₂⁻RR; summary of reported ammonia syntheses in conventional eNRR and plasma tandem-electrocatalysis systems; and summary of reported solar-powered plasma setups and solar-driven electrocatalysis and tables of performance comparison (PDF).
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Notes

The authors declare no competing financial interest.

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