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Non-Thermal Plasma Catalysis for Flexible Ammonia Production: Linking Reaction Chemistry, Materials, and Reactor Performance

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Abstract. Non-thermal plasma offers a route to synthesize ammonia from nitrogen and hydrogen at a low bulk-gas temperature and near-atmospheric pressure, with the operational flexibility needed for intermittent renewable electricity. Its principal challenge is not ammonia formation alone but the inefficient conversion of electrical energy into recoverable product. This review presents a reactor-centred interpretation of plasma catalysis. It first examines how electron-impact excitation, vibrationally excited nitrogen and radical chemistry establish a non-equilibrium reaction environment. It then considers how packed materials alter electric-field distribution, discharge mode, heat transfer and residence time while also supplying chemical sites for nitrogen adsorption and stepwise hydrogenation. Recent metal, support, defect and porous-material strategies are assessed in terms of these coupled physical and chemical roles. Particular attention is given to ammonia loss by plasma-induced decomposition and to reversible adsorption as a means of separating product formation from product recovery. The review argues that progress requires matched-power controls, complete nitrogen balances, energy yield reporting, transient measurements and long-duration testing. Rather than seeking a universally superior catalyst, future work should co-design the power supply, discharge geometry, accessible surface chemistry and product-removal step for a defined operating window. This paper provides discriminating criteria for delineating the boundary between "physical modification" and "chemical catalysis" in plasma catalysis, while offering a systematic attribution of the origins of energy yield discrepancies reported in the current literature.

Keywords: Non-Thermal Plasma, Ammonia Synthesis, Plasma Catalysis, Energy Efficiency, Process Intensification

1. Introduction

Ammonia is indispensable to modern agriculture and is also being considered as a carbon-free energy carrier. At the same time, rapid growth of renewable generation is increasing the value of chemical processes that can absorb fluctuating electricity. Global renewable power capacity and annual additions reached new records in 2025 [1]. The conventional Haber-Bosch process is

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effective at large, continuously operated sites, yet it depends on high pressure, elevated temperature and an upstream hydrogen supply that is commonly fossil-based [2]. The resulting energy use and carbon emissions have motivated research on lower-carbon feedstocks and more flexible synthesis routes. These routes should be viewed as alternatives that address the emerging demands of distributed production and trade, rather than replacements for the conventional process.

Non-thermal plasma (NTP) is attractive because electrical energy is delivered preferentially to electrons rather than to the entire gas inventory. Electron-impact collisions can excite or dissociate N_2 and H_2 while the average gas remains far cooler than a thermal-equilibrium reactor. Atmospheric-pressure dielectric barrier discharges can start rapidly, tolerate intermittent power and be built in modular units. These characteristics align with distributed power-to-X concepts [3]. They do not, however, remove the thermodynamic and kinetic difficulties of activating nitrogen, nor do they guarantee a competitive energy cost. A flexible reactor is useful only if a meaningful fraction of the deposited electrical energy reaches reaction pathways that end in collected NH_3.

The literature has expanded from proof-of-concept plasma reactors to studies of metals, oxides, ferroelectrics, porous supports and dynamically operated sorbents. Broad reviews have documented this development and the continuing gap between laboratory energy yield and industrial ammonia synthesis [4-7]. A recurring difficulty is that the word catalyst is used for materials that perform several jobs simultaneously. A pellet placed in a discharge changes capacitance, local field enhancement, microdischarge propagation, pressure drop, temperature and residence-time distribution. The same pellet may adsorb radicals, stabilise surface intermediates or retain ammonia. An increase in outlet concentration therefore cannot be assigned to surface catalysis without separating these effects.

This review uses the reactor as the unit of analysis. Section 2 describes the reaction environment created by plasma and packing. Section 3 evaluates how catalytic materials influence nitrogen and hydrogen chemistry and how porous or adsorbing phases affect product recovery. Section 4 discusses performance metrics, stability and scale-up. Within this framework, the review draws a clear boundary between the physical modification of the discharge by packed materials and their genuine surface-chemical functions, and traces the main sources of scatter in reported energy yield data to three experimental factors, namely power metering, nitrogen-balance closure, and product-recovery accounting, thereby offering a valuable reference for moving the field from concentration-enhancement demonstrations toward assessments grounded in energy efficiency and system integration.

2. The coupled plasma-reactor environment

A low-temperature discharge contains electrons, ions, radicals, vibrationally and electronically excited molecules, photons and neutral gas [8]. Their populations depend on reduced electric field, pulse width, frequency, gas composition and collisional history. Nitrogen activation can proceed through direct electron-impact dissociation, stepwise vibrational excitation or reactions involving metastable species. Hydrogen is more readily dissociated, but a large hydrogen-radical population is not necessarily beneficial if it consumes energy without encountering nitrogen-derived intermediates. Distinct operating regimes have been observed as specific energy input and gas residence time vary [9]. This explains why data collected at one voltage or flow rate cannot be extrapolated to another condition based on catalyst identity alone.

Packing a dielectric barrier discharge introduces a second level of complexity. Contacts between particles concentrate the electric field, while polarization changes the charge accumulated during each half cycle. Particle diameter, shape, relative permittivity and bed voidage can shift a diffuse

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discharge toward filamentary microdischarges or redistribute the locations where streamers terminate. Experimental studies consequently describe packed catalysts as plasma modifiers as well as chemical materials [10]. Kinetic modelling and time-resolved electrical measurements further show that apparent improvements may originate from changes in deposited power and gas-phase reaction history [11]. A credible comparison should therefore report absorbed power, current-voltage waveforms and bed temperature in addition to applied voltage.

Surface chemistry becomes important when plasma-produced species reach the solid surface before recombining. On nickel catalysts, evidence supports a pathway in which nitrogen-containing species formed in the plasma interact with adsorbed hydrogen and surface intermediates at relatively low temperature [12]. The surface may lower barriers for sequential hydrogenation even when it does not perform the initial N_2 dissociation. This division of labour differs from classical thermal catalysis, where dissociative nitrogen adsorption is often the defining elementary step. It also means that an active metal hidden deep within a pore can be less useful than a modestly active site positioned near the plasma-solid interface. The relevant descriptor is accessible reactive area during discharge; not total surface area measured after synthesis.

Pulsed excitation can change the balance between species generation and surface utilisation. A nanosecond-pulsed discharge combined with Ni-MOF-74 produced a different electrical response and a higher ammonia output than the corresponding plasma-only condition [13]. Such results illustrate the promise of coordinating pulse duration with adsorption and reaction timescales. They also show why power matching is essential: peak voltage, average power and energy per pulse describe different aspects of the excitation. Without a common energy basis, a change attributed to catalyst chemistry may instead reflect a different electron-energy distribution or a different number of discharge events per unit gas volume.

Mechanistic analysis must finally account for competing reactions. NH_3 can form through gas-phase radical sequences, Eley-Rideal reactions between plasma species and adsorbates, or Langmuir-Hinshelwood steps involving two surface species. The relative importance of these channels changes with pressure, surface coverage and the distance between the active plasma region and the catalyst. Isotopic and species-resolved studies demonstrate that formation pathways operate on different timescales [14]. A single steady concentration is unable to identify the dominant route. Transient switching, isotope labelling and operando spectroscopy are therefore more informative than post-reaction surface characterisation alone.

Feed purity and wall chemistry add further uncertainty. Small quantities of water or oxygen may change electron attachment, introduce OH-containing radicals and oxidise catalyst surfaces. Nitrogen obtained from air separation and hydrogen produced by electrolysis will not necessarily have the same impurity profile as research-grade cylinders. Reactor walls and dielectric surfaces can also store adsorbates between runs, producing history-dependent behaviour that resembles catalyst activation or deactivation. Blank conditioning protocols, controlled impurity addition and repeated experiments after cleaning are therefore necessary. A material that is highly active only under exceptionally dry laboratory gas may be unsuitable for a distributed system unless purification energy and maintenance are included in the process boundary.

No single diagnostic can resolve this environment. Electrical waveforms quantify charge transfer and energy deposition, optical emission spectroscopy tracks selected excited species, and laser-based methods can provide spatially resolved radical or temperature information. Surface-sensitive infrared and X-ray techniques probe adsorbates and oxidation states, while mass spectrometry records transient products. The strongest mechanistic study combines measurements that share the same operating point and time base. Diagnostics performed in a separate reactor or after the

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discharge may miss the active state. Future platforms should synchronise electrical, gas-phase and surface signals so that a change in NH_3 formation can be connected to a measured change in energy delivery and surface coverage.

3. Catalytic materials and product recovery

Material selection should begin with a defined plasma environment and a specified chemical task. For nitrogen-rich surfaces, the limiting step may be hydrogen supply or removal of strongly bound NH_x species. For hydrogen-rich surfaces, additional N_2 activation or nitrogen capture may be required. Nickel remains attractive because of cost and because its surface chemistry can complement plasma-generated nitrogen. Spatial placement is crucial. External nickel sites on mesoporous silica were combined with internal pore volume that reduced ammonia decomposition and retained a recoverable inventory [15]. This architecture suggests that formation and protection can be assigned to different regions of one particle rather than forced onto the same site.

Defect engineering offers another route to intercept activated species. Plasma-treated MgAl layered double hydroxides have been associated with changes in oxygen-vacancy concentration and ammonia production [16], while nitrogen vacancies in vanadium nitride have been proposed as adsorption and reaction centres [17]. Defects can influence local charge distribution, binding energy and metal dispersion, but their concentration often changes together with surface area, phase composition and dielectric behaviour. A correlation between a spectroscopic defect signal and NH_3 output is therefore a useful starting observation rather than proof of an elementary mechanism. Controlled defect series, site-blocking experiments and operando measurements are needed to establish causality. Bimetallic catalysts seek to divide nitrogen binding, hydrogen activation and intermediate transfer between neighbouring sites. $Ru/BaTiO_3$-based combinations have been interpreted through cooperative dual-metal centres [18]. More recent Ru-Co work shows that support acidity, surface area, metal dispersion and dielectric response can all influence performance [19]. These studies widen the available design space, but they also expose a general experimental problem: changing the support alters both the catalyst and the discharge. Strong evidence for a metal-support mechanism therefore requires a chemically meaningful material series, electrical diagnostics at matched deposited power and temperature measurements inside the packed zone.

Porous coordination materials and chemically unusual hydrogen carriers provide further options. Ni-modified ZIF-67 combines dispersed metal species with a porous framework and has shown improved plasma-catalytic ammonia formation [20]. Calcium imide introduces a lattice capable of participating in nitrogen-hydrogen transfer and represents a departure from conventional supported metals [21]. The practical value of such materials will depend on whether their active structure survives electrical discharge, water traces, repeated heating and long operating periods. Ex situ diffraction or spectroscopy after a short experiment cannot by itself establish structural stability under plasma. Time-resolved characterization and repeated start-stop testing are required.

Product management is as important as formation chemistry because ammonia remains vulnerable to electron-impact and radical-induced decomposition. Reversible adsorption can remove NH_3 from the most destructive region and release it during a separate step. In situ adsorption with zeolite 4A increased the cumulative energy yield relative to the corresponding steady outlet measurement [22]. Ordered mesoporous oxides with tunable acidity likewise show that adsorption strength and pore structure can influence the amount of recoverable ammonia [23]. The desirable sorbent is not the one with the strongest binding. It must capture product rapidly at reaction temperature, avoid blocking reactive transport and regenerate with a small energy penalty.

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Pore morphology controls both shielding and release. Comparisons of mesoporous gyroidal silica and macroporous particles reveal different uptake and plasma-assisted desorption behaviour [24]. Recent work on porous supports further indicates that moderate acid sites can store ammonia and create transient release peaks when power and temperature are changed [25]. Such peaks should not be reported as instantaneous synthesis enhancement unless fresh nitrogen is simultaneously converted. The correct quantity is the ammonia recovered over the complete capture-release cycle, divided by all electrical and thermal energy supplied. This cycle-level accounting prevents stored product from being counted twice and links material design to process performance.

Catalyst lifetime must be interpreted as a coupled electrical and chemical property. Filamentary discharges can create local hot spots, sputter electrodes and restructure nanoparticles even when the reported average temperature is low. Carbon-containing binders may degrade, porous frameworks may lose crystallinity, and repeated ammonia adsorption can alter acid sites. At the same time, gradual changes in bed impedance can shift deposited power and make a stable outlet concentration conceal a declining chemical activity. Long tests should periodically repeat plasma-only or inert reference measurements and should characterize the same material before and after operation. Separating catalyst decay from power-supply drift is essential for credible lifetime projections.

4. Performance assessment and scale-up priorities

Cross-study comparison is currently limited by inconsistent definitions. Ammonia concentration is easy to report but depends on dilution and flow. Production rate is more useful when normalised by reactor volume or catalyst mass, yet neither normalization indicates electrical efficiency. Energy yield, expressed as mass or moles of NH_3 per unit energy, should be calculated from deposited rather than nominal power whenever possible. Faradaic efficiency is not generally applicable to a gas-phase plasma reactor, and conversion alone may reward long residence time at negligible throughput. At a minimum, publications should provide feed composition, total flow, pressure, active volume, catalyst mass, residence-time definition, absorbed power, temperature, NH_3 rate and energy yield.

A minimum control set is equally important. The empty reactor identifies the contribution of plasma and walls. An inert packing with similar geometry and dielectric properties estimates the physical effect of introducing pellets. The candidate catalyst then adds chemical functionality. These three conditions should be compared at matched absorbed power, flow and temperature. If matching all variables is impossible, the remaining difference should be reported explicitly instead of being hidden inside a synergy factor. Analytical validation also matters: calibration must cover the full concentration range, and potential interference from water, hydrazine or nitrogen-containing by-products should be tested.

Steady operation gives only part of the performance picture. Distributed units may experience rapid power changes, frequent shutdowns and seasonal capacity factors. A catalyst that performs well after thermal equilibration may respond poorly to repeated cold starts. Conversely, a material that stores ammonia may look inactive during the first part of a run and highly active during regeneration. Dynamic experiments should record both gas composition and stored inventory through complete cycles. The power supply should be characterised during transients because changes in bed temperature or adsorbate coverage may alter electrical impedance. Reporting only the final steady point discards the information most relevant to renewable operation.

Scale-up cannot be achieved by enlarging a laboratory dielectric barrier discharge while preserving only nominal voltage. Electrode area, gap distance, heat removal, pressure drop and the distribution of microdischarges all change with size. Numbering-up modular channels may preserve

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local discharge conditions more effectively, but it introduces manifold and power-electronics challenges. A useful scale-up framework should compare characteristic times for plasma-species generation, gas transport, surface reaction and product adsorption. Dimensionless groupings based on residence and reaction times could reduce the number of independent variables, provided that the underlying timescales are measured rather than fitted without physical interpretation. The economic target is also location dependent. Small plasma units may avoid part of the transport, storage and infrastructure burden in regions with remote renewable resources or limited fertilizer access. In regions already connected to efficient ammonia supply chains, the energy-efficiency gap will dominate. Consequently, future studies should couple reactor data with a defined deployment scenario: electricity price and carbon intensity, hydrogen source, annual operating hours, local ammonia demand, transport distance and product purity. This systems boundary prevents flexibility from being treated as a qualitative advantage and reveals the energy yield, lifetime and capital cost required for a specific market.

The highest research priority is coordinated optimisation rather than another isolated catalyst screen. Power waveform, reactor geometry, packing structure, active-site chemistry and product recovery should be varied within one experimental platform. Automated operation can map these interactions, but models must remain constrained by nitrogen balances and electrical measurements. Long-duration tests should quantify electrode erosion, catalyst restructuring, sorbent capacity loss and analytical drift. When these data are reported with uncertainty, plasma-catalytic ammonia synthesis can move from demonstrations of concentration enhancement toward engineering decisions about efficiency, lifetime and scale.

System analysis must include hydrogen and downstream separation. If H_2 is supplied by electrolysis, electrolyser efficiency, compression and buffering may dominate the energy balance even when the plasma reactor is improved. If ammonia leaves in a dilute gas stream, refrigeration, absorption or sorption is needed to reach a transportable product specification. Integrating reversible capture with the reactor may reduce decomposition and separation duty, but regeneration introduces heat and scheduling demands. A fair comparison with Haber-Bosch should therefore distinguish reactor energy yield from complete plant efficiency and should apply identical hydrogen and product-purity assumptions. Otherwise, an advantage assigned to plasma may actually come from an incomplete accounting boundary.

5. Conclusion and future perspectives

This review takes the reactor as the unit of analysis, examining how packed materials couple with discharge behaviour, surface reactions, and product recovery in NTP ammonia synthesis, and assessing the applicability of various catalysts and adsorbents as well as the comparability of reported performance data. The main conclusions are as follows. NTP creates chemical opportunities that are unavailable in a thermal-equilibrium gas, but it also couples electrical, transport and surface processes so tightly that catalyst rankings are easy to misinterpret. A packing may improve ammonia output by changing the discharge, by accelerating surface hydrogenation, by protecting product from decomposition or by temporarily storing NH_3. These functions should be identified separately before they are combined into a reactor design. Recent progress in nickel systems, vacancy-rich materials, bimetallic interfaces, porous frameworks and reversible sorbents demonstrates a broad materials toolbox, yet no composition removes the need for careful energy and mass balances.

The next stage of the field should be defined by comparable measurements and integrated operation. Matched-power plasma-only and inert-packing controls, transient nitrogen balances, cycle-integrated energy yield, spatial temperature measurements and durability testing are essential.

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Reactor and catalyst should then be co-designed for a specified power profile and product-recovery strategy. The main limitation of the present evidence is that many studies change electrical conditions, material chemistry and transport at the same time; future factorial experiments and operando diagnostics must separate these variables. If this methodological shift is achieved, NTP ammonia synthesis can be evaluated on its real strengths -- rapid response, modularity and compatibility with renewable electricity -- while its energy-efficiency and lifetime requirements remain quantitatively visible.

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