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# Recent advances in metal-mediated electrochemical ammonia synthesis towards commercialization

Craig Burdis[1,2,a], Suzanne Zamany Andersen[3,a], Jesús Barrio[2], Magda Titirici[2], Ifan E. L. Stephens[1] and Mattia Saccoccio[3]

Current commercial ammonia production occurs in large, centralized facilities *via* the Haber–Bosch process, emitting a significant portion of our global $CO_2$ emissions. In a world of increasing decentralized renewable energy production, there is therefore an urgent need to develop sustainable and decentralized technologies for ammonia synthesis, which can abate these emissions and take advantage of the decentralized nature of renewables: the electrochemical $N_2$ reduction field offers a promising alternative. Herein, only electrochemical metal-mediated ammonia synthesis processes have been proven across several laboratories and research groups to work at near-ambient pressure and temperature. This review will provide an overview of recent advances in the last two years laying a foundation for future industrial applications of the metal-mediated ammonia synthesis process.

**Addresses**
[1] Department of Materials, Royal School of Mines, Imperial College London, SW7 2AZ, UK
[2] Department of Chemical Engineering, Imperial College London, London, SW7 2AZ, UK
[3] Department of Physics, Technical University of Denmark, Kongens Lyngby, Denmark

Corresponding authors: Saccoccio, Mattia (ms@nitrovolt.com); Stephens, Ifan E. L. (i.stephens@imperial.ac.uk)
[a] These authors contributed equally to this work.

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## Background

Ammonia ($NH_3$) is a crucial compound for agriculture as it is used as a nitrogen-based fertilizer [1]. In 2023, the global production of ammonia was estimated to be 182 million metric tons, with over 80% being used for fertilizer production [2]. As the global population increases, so does the demand for ammonia. However, ammonia production consumes about 2% of worldwide fossil fuel due to the energy-intensive nature of the process [3]. This generates 0.5 Gt of carbon dioxide ($CO_2$) annually, which accounts for 1% of global greenhouse gas emissions [4]. The primary method of ammonia production is the Haber–Bosch process, which is carried out at large centralized industrial plants [3]. Coupling the centralized production with often very rural points of use of nitrogen-based fertilizers, there can also be significant amounts of $CO_2$ emissions associated with transport and distribution. The Haber–Bosch process converts hydrogen gas ($H_2$) and nitrogen gas ($N_2$) into ammonia [3]. This process requires extreme reaction conditions involving high pressure (150–350 atm) and temperature (400–550 °C), along with pure hydrogen, usually obtained from the steam reforming process of natural gas [3].

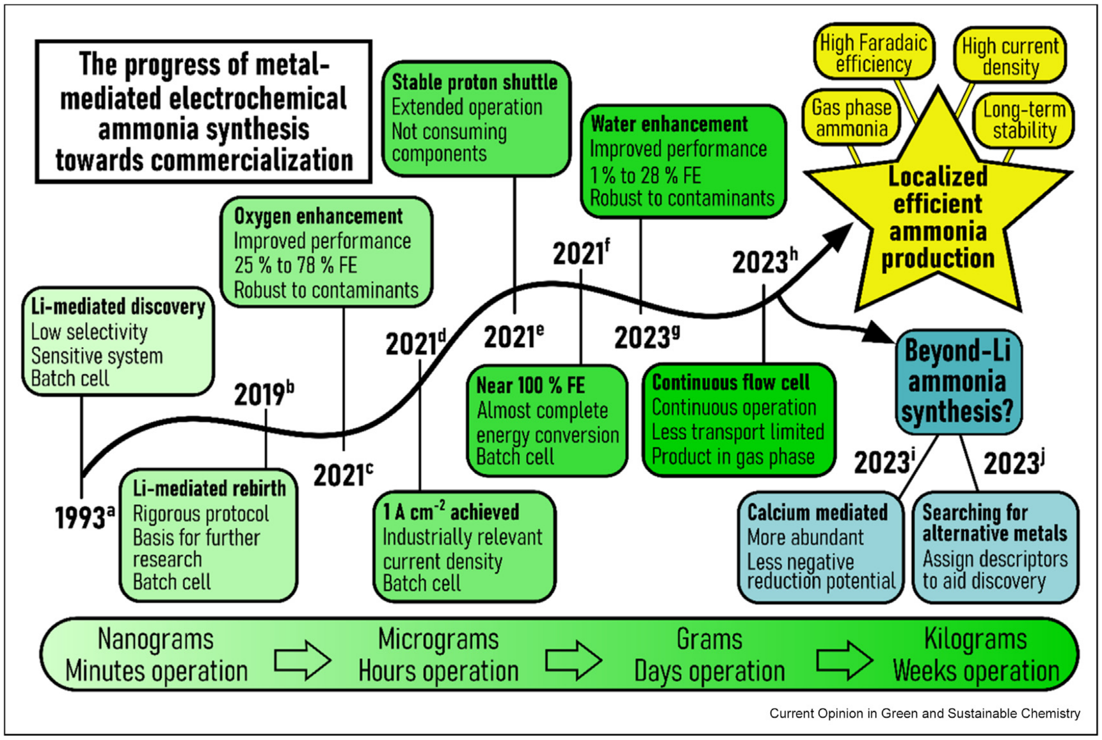
An alternative, novel approach is electrochemical $N_2$ reduction, where electricity is used as the driving force for the catalytic reaction: here $N_2$ reduction occurs at the cathode, coupled with proton generation at the anode. The electrochemical process is attractive because it can proceed at ambient or near-ambient pressure and room temperature. In theory, this process could be completely decentralized in a container-sized system placed on a farm, requiring only air, water, and electricity as the input. This circumvents not only the $CO_2$ emissions from the production and use of fossil fuels *via* the Haber–Bosch process but also the emissions tied to the distribution and transportation. For electrochemical processes, the lithium-mediated $N_2$ reduction reaction is one of the most-studied systems and multiple laboratories have rigorously verified the process. This method, which uses a nonaqueous electrolyte and relies on *in-situ* electrodeposition of lithium metal, was discovered in 1930 by Fichter et al. [5] and later brought to light by Tsuneto et al. in the 1990s [6,7]. Fichter et al. operated at high pressure and employed an electrolyte in which the solvent was an alcohol [5], whereas Tsuneto et al. used tetrahydrofuran (THF), an ether, as the solvent with a small amount of alcohol acting as a proton source [6,7] which is more similar to the systems studied at present. More recently, hydrogen oxidation has been employed to provide protons for the reaction which will be discussed in further detail in a later section. After being buried in a sea of false positives [8], the lithium-mediated process was revisited by Andersen et al., who also suggested the protocol for rigorous experimentation within the field to identify working processes [8]. Lazouski et al. then expanded upon the method by investigating the kinetics of the reaction using a batch system [9]. This review will cover published works on this method from the last 2 years, with the aim of elucidating future commercial applications; the key publications have been visualized on a timeline (Figure 1).

## Electrolyte, additives and contaminants

The basis of the electrolyte in the electrocatalytic reaction is a nonaqueous solvent, a Li-based salt and a proton carrier. The ideal reaction is proposed by Schwalbe et al. [18] and Lazouski et al. [19] to occur *via* the following mechanism: 1) Li-ions are reduced to metallic lithium on the cathode (which will in turn react with the organic electrolyte, forming a solid electrolyte interphase) [20], 2) the metallic lithium reacts with $N_2$ to form a lithium nitride, 3) protons in solution, which can be generated at the anode *via* the hydrogen oxidation reaction (HOR), are supplied *via* the proton carrier and 4) protonation of the lithium nitride on the cathode leads to the formation of ammonia [18,19]. The solid electrolyte interphase mentioned in step 1 is a complex yet vital aspect of the lithium-mediated system, and this will not be the focus of this review; for an extensive review on the role of the solid electrolyte interphase, we recommend the article by Chang et al. in 2024 [21]. There are numerous combinations of solvents, additives, salts, and proton carriers which can impact the system in different ways. Generally, it is seen that peak Faradaic efficiency can be found by varying the concentration of additive, salt, or proton carrier. Until recently, most studies have employed a batch reactor [10, 13, 14, 17, 22, 23] as opposed to the more industrially relevant flow reactor. This will have marked effects on the system, particularly in relation to step 3. Batch reactors experience solvent oxidation and decomposition as the counter reaction to $N_2$ reduction [24], whereas in a flow reactor, the counter reaction is hydrogen oxidation. Certain trends seen in a batch system will therefore not necessarily translate into the more commercially relevant flow-cell system. This was exemplified by Li et al. with respect to the solvent, whereby diglyme was more suitable than THF for use in the flow cell [25]. This is contrary to the findings in the batch cell by Sažinas et al. who concluded diglyme to be less stable than THF [24].

If the inputs for a decentralized lithium-mediated electrochemical ammonia synthesis system are water

**Figure 1**



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Timeline of advances in metal-mediated electrochemical ammonia synthesis that is relevant towards commercialization of the process in which key metrics have improved over time. Such key metrics for good performance are Faradaic efficiency (FE), which is the percentage of the input electricity that goes towards the product, current density which corresponds to conversion rates per unit area, producing ammonia in the gas phase to remove product separation costs, and long-term device stability to enable continued production without intervention. References: a [6], b [8], c [10], d [11], e [12], f [13], g [14], h [15], i [16], j [17].

and air, an electrolyzer would be required to split the water to produce hydrogen and an air separation unit (ASU) to purify the $N_2$. These systems can add significant cost to the overall product, depending on how pure the inputs need to be. To mitigate these costs, an important investigation would be looking towards reducing the input purity requirement for this system. Typically, the gases supplied to the system in a research setting are ultra-pure (99.9999% and higher) to avoid contamination [10, 13–15, 17, 22, 23], but in 2021, Li et al. saw an interesting and counter-intuitive increase in Faradaic efficiency by adding small amounts of oxygen to a batch system under 20 bar of pressure [10]. Li et al. found that by increasing the oxygen content from 0.0 mol% to 0.8 mol%, the Faradaic efficiency would increase from 25% to 78% [10]. These experiments were conducted in a batch cell. However, the role of gas purity in a flow cell system must also be understood, in view of the commercialization of this process, due to the energy and capital cost of an electrolyzer and ASU.

Another inevitable contaminant is water, which is omnipresent, in small amounts even in high-purity commercial chemicals. Experiments are often conducted in an argon-filled glove box, partly to prevent false positives but also to ensure the electrolyte is not contaminated with deleterious species [8]. Water was believed to be one of the species that must be excluded until Spry et al. discovered that increasing water concentration to 33.3 mM in fact improved Faradaic efficiency towards ammonia from 1.4% (no water added to the electrolyte) to 27.9% under ideal conditions [14]. Although still sensitive, this suggests that the lithium-mediated electrochemical ammonia synthesis may be more robust to unavoidable contaminants, thus improving its scalability.

Aside from contaminants, the choice of essential electrolyte components (lithium salt, solvent, and proton donor) inevitably has a pronounced impact on scalability of lithium-mediated electrochemical ammonia synthesis. One key distinction, in relation to step 4 of the proposed mechanism, is the difference between a proton donor and a proton shuttle. Ideally, a proton shuttle would transport protons produced at the anode to the cathode and return to the anode for re-protonation without being consumed; often the proton donor is partially consumed over the course of a reaction. A phosphonium salt introduced by Suryanto et al. in 2021 addressed this issue, which was discovered to be stable under operating conditions for extended periods of time [12]. The ability of the phosphonium salt to operate reversibility, i.e. without significant consumption, was studied *via* $^{31}P$ NMR analysis in a modelling chemical reaction whereby the phosphonium salt was protonated and deprotonated by adding acid and base, respectively [12]. The reversibility of the salt's protonation was not monitored in the lithium-mediated system, but multinuclear NMR was used to highlight the stability of the salt after 20 h of operation under $N_2$ reduction conditions [12]. A study with a similar goal by Fu et al. identified phenol to be a suitable proton shuttle in a flow cell as it can cycle between phenol and phenoxide, identified by $^{1}H$ NMR [26]. Phenol also exhibited an improved Faradaic efficiency compared to the literature standard proton donor ethanol, 72 ± 3% vs. 67%, respectively [26].

The lithium salt used impacts the system because the salt anion affects both the deposited solid electrolyte interphase layer at the cathode and the HOR at the anode and thereby the overall performance of the system [13, 27]. Du et al. showed that by switching from $LiClO_4$ to $LiNTf_2$, they could achieve nearly 100% Faradaic efficiency with a yield rate of 150 nmol $s^{-1}$ $cm^{-2}$ [13]. Li et al. furthermore showed that by increasing the electrochemical surface area and using $LiBF_4$, they could achieve 71% Faradaic efficiency at 1000 mA $cm^{-2}$ with a yield rate of 2500 nmol $s^{-1}$ $cm^{-2}$ [11]. While both these results were reported in batch systems without HOR on the anode, a recent work by Fu et al. [27] in a flow cell system demonstrated how the salt choice also affects the anode reaction. This indicates that further investigation into the impact of salt is required for the continuous, commercially relevant system.

## Beyond lithium: metal-mediated ammonia synthesis

Lithium-mediated electrochemical ammonia synthesis has seen huge improvements in many aspects of the system. However, the system is intrinsically limited by the extremely negative standard reduction potential (−3.04 V vs. the standard hydrogen electrode [SHE]) which must be achieved to reduce lithium [16]. Therefore, researchers are taking inspiration from battery technologies towards searching for a different reducing metal [16, 17]. A focal point of battery research is the quest for more abundant alternatives to lithium [16, 17]. Metal batteries have been developed based on materials such as sodium, magnesium, and calcium [17]. These technologies provide inspiration for electrochemical ammonia synthesis as there are key commonalties, namely plating the metal onto an electrode and the formation of a solid electrolyte interphase, which are vital for both applications [17].

In the search for a suitable candidate with a less-negative reduction potential, Tort et al. looked to define the criteria required for electrochemical $N_2$ reduction, assuming a dissociative mechanism [17]. Assisted by density functional theory calculations, Tort et al. attributed lithium's suitability for $N_2$ reduction to being able to dissociate $N_2$ and form a stable nitride, in addition to having a solid electrolyte interphase that can moderate access to lithium and protons while facilitating the passage of $N_2$ [17]. Analogous metals to lithium, like sodium and potassium, failed to fulfil the energetics criteria for metal-mediated ammonia synthesis [17]. Sodium was tested experimentally to act as a control experiment to evaluate the proposed energetics hypothesis, and ammonia was not made using sodium-based electrolytes which agreed with the hypothesis [17]. However, magnesium and calcium were identified as promising candidates but failed to produce ammonia experimentally, which was attributed to unoptimized experimental conditions [17]. In parallel to Tort et al., Fu et al. discovered that calcium could mediate electrochemical ammonia production by utilizing a calcium metal battery electrolyte found to facilitate calcium plating and stripping [16]. They achieved a Faradaic efficiency of 40% in a continuous electrochemical setup [16], lower than the current lithium-mediated electrochemical ammonia synthesis state of the art, but it is high considering it is the first instance of beyond-lithium electrochemical ammonia synthesis. The main benefit of calcium over lithium is that it is significantly more abundant (2075 times more abundant than lithium in the earth's crust, comparing $mg_{element}/kg_{crust}$) [28], which could lend itself to being more industrially relevant. However, with calcium there is not much of an improvement in terms of reduction potential (−2.87 V vs SHE) [16], and this will therefore still be restricted to low energy efficiency. Assuming no overpotential and 100% Faradaic efficiency, the energy efficiencies are 29.0% for calcium and 27.8% for lithium (see data repository for calculation details) [29].

A beyond-lithium catalyst or mediator which is more suited to commercialization should have a significantly less negative reduction potential whilst maintaining the reactivity and selectivity required for ammonia synthesis. Tort et al. highlighted that molybdenum and tungsten fit the energetics criteria and have significantly less negative standard reduction potentials. Therefore, providing that a suitable solid electrolyte interphase can be formed, we suggest that these metals should be researched for electrochemical ammonia synthesis [17].

## Flow cell and continuous measurements

In this field, most reported systems to date are batch processes, [12–14,17,22,23,30–34] where the electrodes are fully submerged in the electrolyte and are reliant on gas solubility in the electrolyte. This means that the nitride formation is expected to be $N_2$ mass transport limited, as in most reported electrolytes the gas solubility is expected to be quite low. This mass transport limitation is shown by the increase in Faradaic efficiency when the pressure of the batch system is increased, which increases the availability of $N_2$ at the cathode [7, 34]. Most reports employing a batch system also do not attempt hydrogen oxidation [12–14, 17, 22, 23, 30–34], leading to solvent oxidation on the anode as the counter reaction, which is not sustainable for commercial production. A flow-cell-based system allows for the availability of $N_2$ and $H_2$ gas at each electrode, thereby significantly reducing the mass transport limitation of these species and thus effectively increasing efficiency. Furthermore, a flow-cell-based system can also be operated continuously, which is better suited for commercial applications. However, it is nontrivial to design electrodes for nonaqueous flow-cell-based systems. Typically, the electrodes used in aqueous flow cells rely on a hydrophobic coating to keep the electrolyte on the one side and the gas on the other [35–37]. Hydrophobicity would not prevent a nonaqueous electrolyte from flooding the gas field; an omniphobic coating, which repels all substances including organic and aqueous liquids, would be required [38,39].

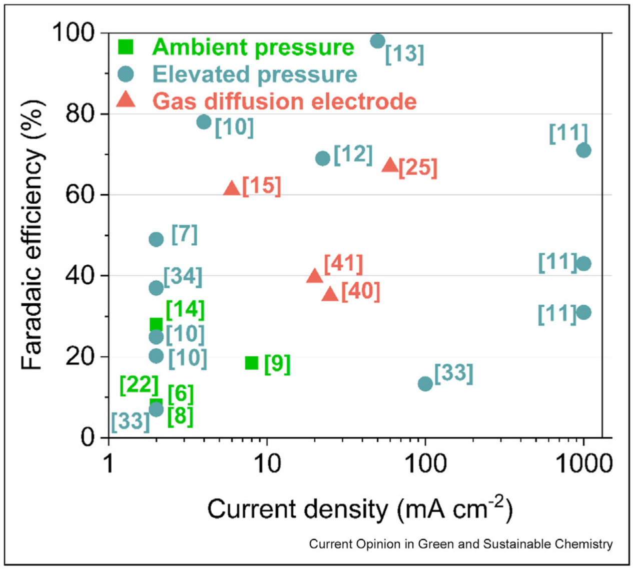
The first flow-cell-based system for the lithium-mediated ammonia synthesis process was published by Lazouski et al., using a stainless steel cloth and a small pressure difference between the gas and electrolyte to separate them based on the surface tension of the electrolyte [40]. This was the first reported instance of $H_2$ and $N_2$ flow at the anode and cathode, respectively, which was a major step forward in terms of commercialization for this process. The report achieved 35% Faradaic efficiency and at a total cell potential above 20 V [40]. The anolyte and catholyte were not circulated, and the experiments reported were only a few minutes long [40]. To overcome this high total cell potential, Fu et al. more recently designed a flow reactor that did not require a membrane, where both gases and the electrolyte are flowing continuously in separate circuits [15]. The separation of the gas and electrolyte phases was maintained as Lazouski et al. had previously reported. Fu et al. were the first to unambiguously prove hydrogen oxidation as the counter reaction by using deuterium rather than hydrogen gas and the subsequent measurement of deuterated ammonia species in the outlet of the $N_2$ gas [15]. They reported 61% Faradaic efficiency as well as a record high energy efficiency of 13% for the continuous lithium-mediated ammonia synthesis system [15].

More recently, Cai et al. showed a first-of-its-kind membrane electrode assembly system, completely circumventing the liquid electrolyte, using instead a lithium-doped polyethylene oxide as a polymer electrolyte [41]. The report achieved a 9% Faradaic efficiency towards ammonia with a total cell potential of 3.6 V [41], one of the lowest reported total cell potentials. Despite the system not being stable, the current density dropped to mA $cm^2$ over the course of an experiment, this work presented the first system without a liquid electrolyte. These results show a promising and much simpler approach for commercial application as maintaining a phase boundary between the gas and electrolyte will most likely be difficult at scale [41]. We have provided a summary of key advancements in cell design; for a more detailed examination, refer to the article by Fu et al. in 2023 [42].

## Outlook

Metal-mediated ammonia synthesis, particularly the Li-mediated system, has made significant progress in the past few years. The field has advanced in fundamental understanding, performance, and system engineering. The performance improvements have been remarkable, with Faradaic efficiencies exceeding 90% in a batch setup [13] and 81% in a flow cell [25]. Some of the recent advances in Faradaic efficiency have been plotted versus the current density (Figure 2), which highlights how the field has been progressing towards an ideal system which exhibits high selectivity at high current densities. While most of the research has focused on Faradaic efficiency as key metric, future efforts should explore operating industrially relevant cells (a flow cell) at high current densities for long periods of time. We expect that the electrode design for the cathode and anode must be optimized for these conditions, namely by increasing the surface area of electrodes. Initial steps towards this goal were taken by Fu et al. whereby 60 mA $cm^{-2}$ was achieved [25]. Alternative electrolyte systems with improved oxidative stability could also allow for extended operation at increased current density. The discovery of such systems would benefit from further understanding of electrolyte decomposition mechanisms. Development of beyond-lithium mediators would lead the field away from the energy requirement for lithium metal deposition, resulting in improved energy efficiencies and in turn commercial viability.

**Figure 2**



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Plot comparing the current density applied against the Faradaic efficiency for references in recent years. Data are available at data repository [29].

As the metal-mediated ammonia synthesis process has matured, it has also attracted commercial interest as a viable option to supply decarbonized and decentralized ammonia. This is evidenced by the emergence of at least two startup companies, Jupiter Ionics Pty Ltd in Australia and NitroVolt ApS in Denmark, both spun out from academic research groups who pioneered this nascent field. This, we are confident, will result in a positive feedback loop between academia and industry aimed at accelerating deployment and impact of this novel technology.

## Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Mattia Saccoccio reports a relationship with NitroVolt ApS that includes: equity or stocks. Suzanne Zamany Andersen reports a relationship with NitroVolt ApS that includes: equity or stocks. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

## Data availability

This work does not contain original data; all data presented are sourced from previously published studies, which are adequately referenced

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## Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.cogsc.2024.100964.

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