

**SUSTAINABLE RECYCLING OF SPENT LI-ION
BATTERIES: LITHIUM RECOVERY AND**

BY

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LIST OF ABBREVIATIONS

LiBs	Lithium-ion Batteries
LiCoO₂	Lithium Cobalt Oxide
NMC	Nickel Manganese Cobalt (cathode)
EVs	Electric Vehicles
SEI	Solid-Electrolyte Interphase
PVDF	Polyvinylidene Fluoride
XRD	X-ray Diffraction
SEM	Scanning Electron Microscopy
EDX	Energy-Dispersive X-ray Spectroscopy
ICP-OES	Inductively Coupled Plasma Optical Emission Spectroscopy
TGA	Thermogravimetric Analysis
XPS	X-ray Photoelectron Spectroscopy
BET	Brunauer-Emmett-Teller (surface area analysis)
BJH	Barrett-Joyner-Halenda (pore size distribution)
OER	Oxygen Evolution Reaction
NRR	Nitrogen Reduction Reaction
NOR	Nitrogen Oxidation Reaction
HER	Hydrogen Evolution Reaction
DI	Deionized (water)
RHE	Reversible Hydrogen Electrode

HT	High-Temperature
LCA	Life Cycle Assessment
SDGs	Sustainable Development Goals
DFT	Density Functional Theory
Co₃O₄	Cobalt Oxide (Spinel)
NO_x	Nitrogen Oxides
HF	Hydrofluoric Acid
KOH	Potassium Hydroxide
HNO₃	Nitric Acid
NaOH	Sodium Hydroxide
NaCl	Sodium Chloride
UV-Vis	Ultraviolet-Visible (Spectroscopy)
IC	Ion Chromatography
FE	Faradaic Efficiency
mg/L	Parts Per Million
wt%	Weight Percentage
CV	Cyclic Voltammetry
RSD	Relative Standard Deviation

ABSTRACT

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The increasing demand for lithium-ion batteries (LiBs) has led to significant environmental and resource challenges, particularly concerning the disposal and recycling of spent batteries. This study explores an innovative approach to repurpose spent LiCoO_2 cathode materials from discarded LiBs as catalysts for nitrogen fixation via an electrostatically enhanced microdroplet system. The recovered LiCoO_2 was characterized using XRD, SEM, EDX, ICP-OES, TGA, XPS, and BET analysis, confirming its structural integrity and catalytic potential. The electrochemical system, operating at varying voltages (0–10 kV), demonstrated efficient nitrogen fixation, producing both ammonia (NH_3) and nitrate (NO_3^-) under ambient conditions. Optimal performance was achieved at 10 kV, yielding 1.11 mg/L NH_3 and 17.49 mg/L NO_3^- , with Faradaic efficiencies of 12.3% and 18.7%, respectively. The study highlights the dual benefits of this approach: (1) sustainable recycling of spent LiBs and (2) low-energy nitrogen fixation for potential agricultural applications. These findings present a circular economic solution that transforms battery waste into valuable catalysts, contributing to green chemistry and sustainable resource management.

ملخص الرسالة

الاسم الكامل: لارا شحاده أحمد عليان

عنوان الرسالة: إعادة تدوير مستدامة لبطاريات الليثيوم أيون المستهلكة: استخلاص الليثيوم والتطبيقات الحفزية

المجال الرئيسي: الكيمياء

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أدى الطلب المتزايد على بطاريات الليثيوم أيون إلى تحديات بيئية ومواردية كبيرة، خاصة فيما يتعلق بالتخلص من البطاريات المستهلكة وإعادة تدويرها. تستكشف هذه الدراسة نهجاً مبتكراً لإعادة استخدام مواد كاثود LiCoO_2 المستهلكة من بطاريات الليثيوم أيون المهمة كعوامل حفازة لتنشيط النيتروجين عبر نظام قطرات دقيقة معزز كهروستاتيكيًا. تم توصيف مادة LiCoO_2 المستخلصة باستخدام تقنيات حيود الأشعة السينية (XRD)، والمجهر الإلكتروني الماسح (SEM)، ومطيافية الأشعة السينية المشتتة للطاقة (EDX)، ومطيافية الانبعاث الضوئي بالبلازما المقترنة حثيًا (ICP-OES)، والتحليل الحراري الوزني (TGA)، ومطيافية فوتوالإلكترون بالأشعة السينية (XPS)، وقياس مساحة السطح بطريقة بروناور-إيميت-تيلر (BET)، مما أكد سلامتها الهيكلية وإمكاناتها الحفزية.

أظهر النظام الكهروكيميائي، الذي يعمل بجهود مختلفة (0-10 كيلو فولت)، كفاءة عالية في تثبيت النيتروجين تحت الظروف المحيطة، منتجاً كلاً من الأمونيا (NH_3) والنترات (NO_3^-) تم تحقيق أفضل أداء عند 10 كيلو فولت، حيث بلغت كمية الأمونيا المنتجة 1.11 جزء في المليون والنترات 17.49 جزء في المليون، مع كفاءات فارادية بلغت 12.3% و 18.7% على التوالي. تبرز الدراسة الفوائد المزدوجة لهذا النهج: (1) إعادة تدوير مستدامة لبطاريات الليثيوم أيون المستهلكة، و(2) تثبيت نيتروجين منخفض الطاقة للتطبيقات الزراعية المحتملة. تمثل هذه النتائج حلاً اقتصادياً دائرياً يحول نفايات البطاريات إلى عوامل حفازة قيمة، مساهماً بذلك في الكيمياء الخضراء والإدارة المستدامة للموارد.

CHAPTER 1

INTRODUCTION

1.1 The Importance of Batteries in Modern Technology

Batteries have become indispensable in modern society, powering everything from small electronic devices to large-scale energy storage systems. The development of lithium-ion batteries (LiBs) has revolutionized energy storage due to their high energy density, lightweight nature, and long cycle life. These batteries are the backbone of portable electronics, electric vehicles (EVs), and renewable energy storage, enabling the transition toward cleaner and more sustainable energy solutions. [1], [2].

Among all battery chemistries, LiBs have gained the most prominence due to their superior electrochemical properties. Lithium, with its low atomic weight (0.534 g/cm^3) and high electrochemical potential (3.04 V vs. the standard hydrogen electrode), plays a crucial role in providing high energy capacity and efficiency. [3][4]. As the demand for EVs and renewable energy integration grows, the consumption of LiBs has surged, making them one of the most critical energy storage technologies of the 21st century.

However, with the rapid adoption of LiBs, new challenges have emerged, particularly in the management of spent batteries. As billions of batteries reach the end of their lifecycle, improper disposal and the inefficient recovery of valuable materials pose significant environmental and economic concerns. The high cost of lithium and the limited supply of cobalt, a key component in LiB cathodes, has further intensified the need for alternative recycling and repurposing strategies [4].

1.2 The Environmental and Resource Crisis: The Urgency for Recycling and Repurposing

As billions of lithium-ion batteries approach the end of their lifecycle, improper disposal has become a pressing environmental concern. Spent LiBs contain hazardous materials, including toxic cobalt compounds, organic electrolytes, and fluorinated binders, all of which pose risks to soil and water systems if not managed properly [4]. Moreover, lithium leakage from improperly discarded batteries can contaminate groundwater and contribute to resource depletion [4].

The environmental footprint of LiB production extends beyond lithium itself. Cobalt, a key component in many lithium-ion battery (LiB) cathodes, has a supply chain riddled with ethical and geopolitical concerns. Over 70% of the world's cobalt supply comes from the Democratic Republic of Congo, where mining operations have been linked to environmental destruction, exploitative labor conditions, and geopolitical instability. The increasing demand for lithium and cobalt is straining global supply chains, raising concerns about long-term resource security and price fluctuations [5].

To address these challenges, effective recycling technologies are essential. The circular economic approach, where materials are recovered and repurposed, presents a viable strategy to mitigate environmental damage, reduce dependence on primary extraction, and ensure a sustainable supply of lithium and cobalt for future energy storage applications [4], [5].

1.3 Recycling Strategies: Extracting Value from Battery Waste

Several recycling methods have been developed to recover valuable metals from spent lithium-ion batteries (LiBs), each with unique advantages and limitations. Among these, hydrometallurgical and pyrometallurgical techniques remain the most widely adopted.

Hydrometallurgical Recycling: This method utilizes acid leaching and solvent extraction to selectively recover lithium, cobalt, and other transition metals from spent batteries. It is an efficient process that enables the recovery of high-purity metals, making it an attractive option for large-scale recycling. [4].

Pyrometallurgical Recycling: This high-temperature process smelts spent LiBs to extract metals, but it is energy-intensive and produces significant carbon emissions, making it less environmentally friendly [4].

Direct Recycling: Unlike traditional recycling methods, direct recycling focuses on preserving the cathode material's crystal structure, allowing it to be regenerated and reused in new batteries. This process significantly reduces energy consumption and material waste. [6].

A growing body of research is also exploring alternative applications for spent cathode materials beyond traditional recycling. One promising approach is repurposing them for catalytic applications, where their redox properties can be leveraged for various electrochemical and environmental processes [6], [7], [8].

1.4 Exploring Alternative Uses: Spent Cathodes as Catalysts

Traditional recycling methods for LiBs, such as hydrometallurgical and pyrometallurgical processes, focus on recovering lithium, cobalt, and other valuable metals. While effective, these processes are often energy-intensive and generate secondary waste [4]. An emerging alternative is the direct reuse of spent cathode materials for catalytic applications, leveraging their intrinsic redox properties for chemical transformations [6].

Spent cathode materials, particularly cobalt-based oxides, exhibit unique electronic and structural characteristics that make them promising candidates for catalysis. These materials can facilitate various electrochemical reactions, including the oxygen evolution reaction (OER), water splitting, and the degradation of organic pollutants in wastewater treatment [7], [8]. This approach not only reduces battery waste but also provides a sustainable pathway for utilizing valuable materials in high-value applications [5], [6].

1.5 Catalytic Applications: A New Role for Spent Cathodes in Sustainable Chemistry

The growing demand for sustainable nitrogen fixation methods has driven interest in alternative approaches to synthesizing ammonia (NH_3) and nitrate (NO_3^-). The conventional Haber-Bosch process, while effective, remains energy-intensive and accounts for nearly 1.8% of global CO_2 emissions [9]. In response to this challenge, recent studies have explored electrostatically driven fog collecting using space charge injection as a promising strategy for fog collection, which can be a sustainable solution to water scarcity in many regions around the world [10].

This process generates charged microdroplets that serve as active reaction sites, enabling direct nitrogen fixation from the atmosphere through gas-water interactions [11]. These microdroplets not only enhance mass transfer but also create localized electric fields that accelerate redox reactions at ambient conditions [12]. Furthermore, it has been demonstrated that ammonia can be synthesized directly from water vapor and nitrogen using similar electrostatic principles [13].

In this study, we take this concept a step further by introducing spent lithium-ion battery (LiB) cathodes as catalysts in the electrostatic fog collection process. Specifically, LiCoO_2 , a widely used cathode material, possesses unique redox properties and a high surface area that can facilitate nitrogen activation and conversion [7], [14]. Our findings indicate that LiCoO_2 enhances nitrate and ammonia production, likely due to its ability to mediate electron transfer and stabilize reactive intermediates at the gas-liquid interface [8], [12]. Moreover, the microdroplets generated in this process play a dual role, not only collecting

nitrogen-containing products but also activating the catalyst by enhancing charge transfer and interactions [11], [13].

By repurposing spent battery cathodes in this manner, we introduce a circular economic approach that transforms electronic waste into a valuable resource for sustainable chemistry [4], [5]. This novel integration of electrostatic fog collection with catalysis presents a scalable, low-energy alternative to conventional nitrogen fixation, offering a direct pathway to onsite fertilizer production while reducing environmental impact [11], [13]. Furthermore, fog collection itself can serve as a sustainable solution to water scarcity in many regions around the world. The collected water, enriched with nitrate and ammonia, can be directly applied to agricultural land, simultaneously providing both irrigation and essential nutrients for plant growth [11], [13].

1.6 Research Contribution and Outlook

The integration of spent lithium-ion battery (LiB) cathodes into catalytic applications presents a transformative opportunity for waste valorization and sustainable chemistry. By bridging the gap between battery recycling and nitrogen fixation, this research introduces an innovative approach that not only mitigates electronic waste but also contributes to the development of energy-efficient, low-emission chemical processes. The novel combination of electrostatically driven fog collection with catalysis extends the functional lifespan of spent cathode materials, repurposing them as active catalysts for the production of ammonia and nitrate. Additionally, the dual role of fog collection in both nitrogen fixation and water harvesting offers a sustainable solution to agricultural challenges, providing an integrated method for producing fertilizer-enriched water that can be directly applied to the soil, addressing both water scarcity and nutrient supply.

The following chapters will explore the mechanisms governing the catalytic activity of spent LiB materials, the role of microdroplets in enhancing nitrogen conversion, and the broader implications for circular economy strategies. By demonstrating the viability of this process for onsite fertilizer production, this research aims to establish a scalable and sustainable framework for utilizing battery waste in high-impact industrial and environmental applications. Ultimately, this work seeks to redefine the role of spent cathodes, positioning them as valuable resources for green chemistry and contributing to a cleaner, more resource-efficient future.

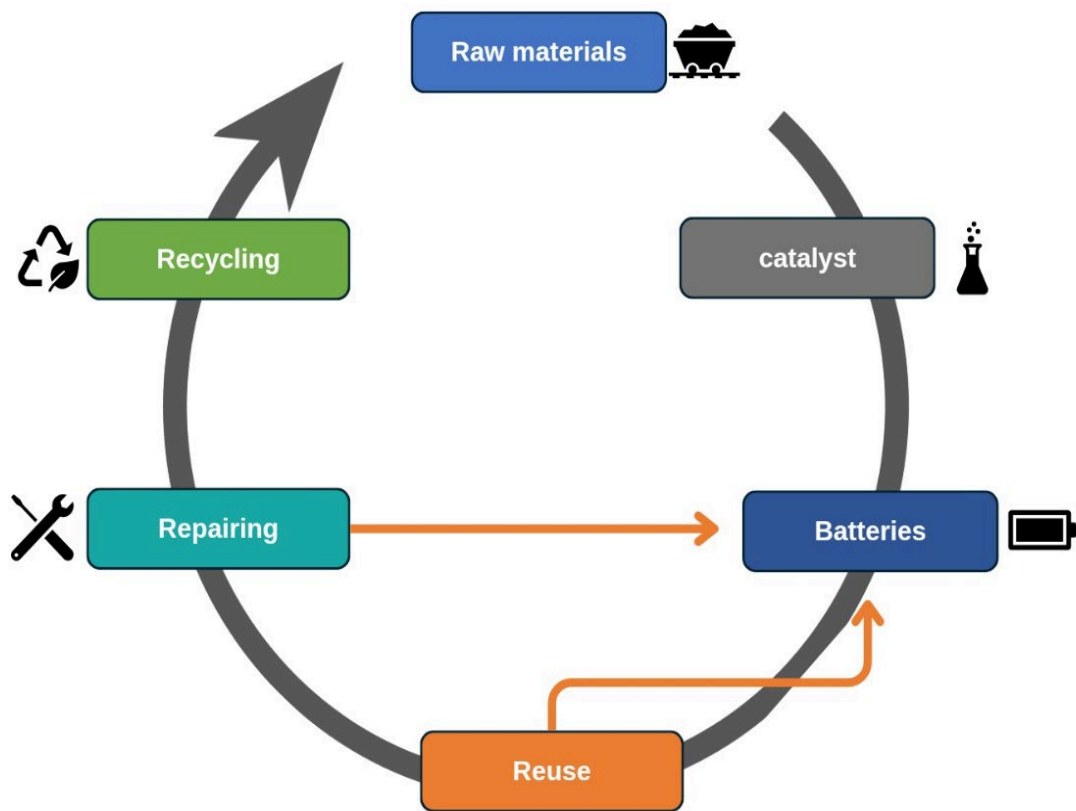


Figure 1. The circular economy approach to lithium-ion batteries: illustrating the key stages from raw material extraction to battery use, reuse, repair, and recycling, aiming to minimize waste and maximize resource efficiency.

CHAPTER 2

LITERATURE REVIEW

This chapter critically reviews recent advances in electrochemical nitrogen fixation systems, with emphasis on spent battery-derived catalysts and the role of microdroplet interfaces in enhancing $\text{NH}_3/\text{NO}_3^-$ selectivity and Faradaic efficiency at ambient conditions.

2.1 Applications, Composition, and Environmental Impact of Lithium-Ion Batteries

Lithium-ion batteries (LiBs) have emerged as the dominant energy storage technology in modern society, revolutionizing portable electronics, electric vehicles (EVs), and renewable energy systems [1], [2]. Their widespread adoption stems from superior energy density (~ 250 Wh/kg), lightweight design, and long cycle life ($>1,000$ cycles), making them indispensable for consumer electronics like smartphones and laptops, where lithium cobalt oxide (LiCoO_2) cathodes deliver high voltage (3.7 V) [3]. The transportation sector's shift toward electrification relies on high-capacity LiBs using nickel-manganese-cobalt (NMC) chemistries, enabling EVs to achieve ranges exceeding 400 km per charge [4], [5]. Large-scale LiB deployments (e.g., Tesla Powerwall) also facilitate renewable energy storage, mitigating intermittency in solar/wind power grids [2].

The electrochemical performance of LiBs depends on four key components, each with its environmental trade-offs. Cathodes, typically layered oxides (LiCoO_2 , NMC) or polyanion structures (LiFePO_4), dominate the battery's cost and environmental footprint due to their dependency on cobalt—a metal linked to unethical mining practices in the Democratic

Republic of Congo [5]. Graphite anodes, though stable, require energy-intensive processing ($\sim 1,000^{\circ}\text{C}$ annealing), while liquid electrolytes (LiPF_6 in organic carbonates) decompose into toxic hydrofluoric acid (HF) upon exposure to moisture [4]. Polyethylene separators, though chemically inert, contribute to microplastic pollution if incinerated at end-of-life [4].

Improper disposal of spent LiBs exacerbates these environmental risks. Landfill leaching tests reveal that a single battery can release up to 20 mg/L of cobalt into groundwater, exceeding EPA limits by 40-fold [4]. When incinerated, fluorinated binders (e.g., PVDF (Polyvinylidene fluoride)) emit dioxins, while lithium reacts exothermally with water, posing fire hazards in waste facilities [4], [15]. The carbon footprint of virgin material extraction is staggering; producing 1 kg of battery-grade lithium consumes 2.2 million liters of water in brine mining operations, exacerbating arid-region water stress [16].

Recycling innovations aim to mitigate these impacts with three primary methods—Hydrometallurgy, Pyrometallurgy, and Direct—as illustrated in Figure 2. Hydrometallurgical techniques (e.g., H_2SO_4 leaching) recover $>95\%$ cobalt but generate 50 L of acidic wastewater per kg of batteries, necessitating costly neutralization before disposal [1], [4]. In contrast, pyrometallurgy employs high-temperature smelting to extract metals, but this energy-intensive process releases hazardous gases (e.g., CO, HF) and struggles with lithium recovery [4], [6]. Direct recycling, a less invasive approach, focuses on disassembly and material separation to preserve cathode integrity, though it faces challenges in scaling and contamination removal [4].

Recent advancements have refined these methods, particularly in lithium replenishment (a hydrometallurgy-derived process), which regenerates cathodes while using 60% less energy than traditional smelting [6], [17]. However, residual contaminants like aluminum and manganese—often introduced during battery operation—remain a hurdle for all three approaches, as highlighted in Figure 2.

Researchers are pursuing complementary techniques to address these challenges. Biological extraction methods utilizing specialized bacteria like *Acid thiobacillus ferroxidase* demonstrate remarkable precision in metal recovery [18]. When combined with electrochemical separation processes, these methods achieve purity levels above 90% while dramatically reducing reliance on corrosive chemicals compared to standard hydrometallurgical approaches [19]. The combination of these techniques presents a more sustainable pathway for recovering battery materials, striking a balance between efficiency and environmental considerations.

Effective policy measures and advanced material development are proving essential for scaling sustainable battery solutions. Recent regulatory initiatives like the EU Battery Regulation (2023) are driving significant changes in the industry by setting ambitious targets - requiring batteries to contain at least 12% recycled cobalt and 4% recycled lithium by 2030 [4]. These mandates are accelerating the industry's adoption of circular economy models, where manufacturers are increasingly investing in closed-loop recycling systems to recover and reuse battery materials.

Parallel advancements in cathode chemistry are addressing fundamental environmental concerns. The development of cobalt-free alternatives such as lithium iron phosphate (LiFePO_4) offers a promising path to reduce both toxicity and supply chain risks [3], [5].

Similarly, solid-state electrolyte technologies are emerging as safer alternatives to conventional liquid electrolytes. However, these innovations currently face practical limitations - LiFePO_4 cathodes, for instance, typically store about 30% less energy than standard nickel-manganese-cobalt (NMC) formulations, presenting challenges for electric vehicle applications where energy density remains crucial [3], [5].

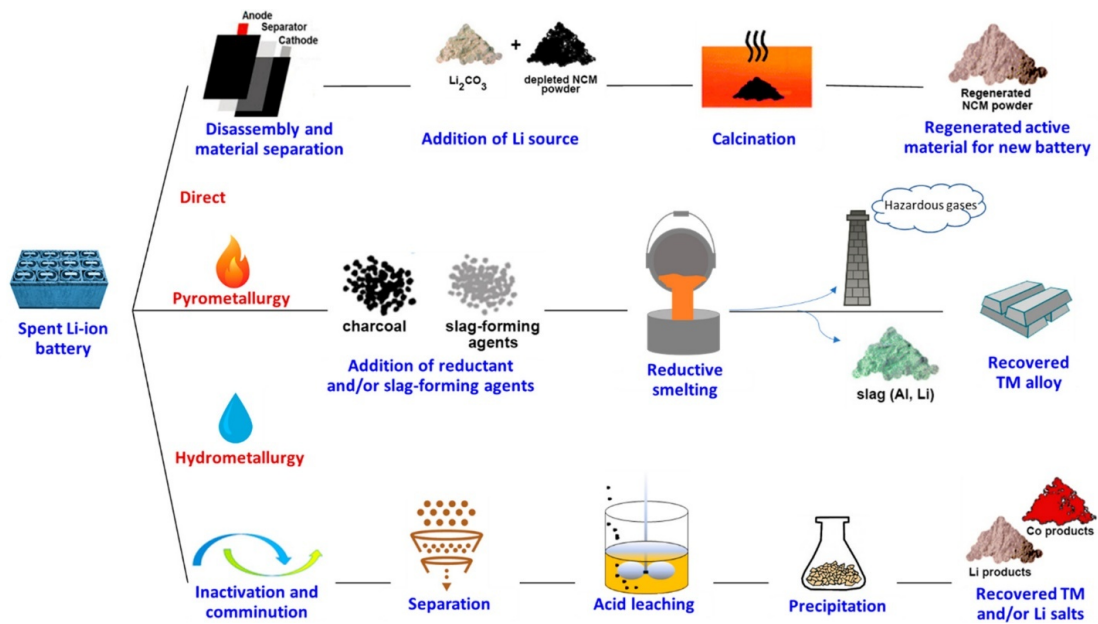


Figure 2. Key lithium-ion battery recycling methods: Direct (disassembly and material separation), Hydrometallurgy (acid leaching with inactivation/comminution), and Pyrometallurgy (high-temperature smelting).[20]

2.2 Catalytic Applications of Spent LiB Cathode Materials

Recent advances have demonstrated the successful repurposing of spent lithium-ion battery (LiB) cathode materials for various catalytic applications, as systematically compared in Table 1. Significant progress has been made in three primary application domains: oxygen evolution reaction (OER) catalysis, chemical oxidation processes, and biomass conversion.

For OER applications, Pegoretti et al. [14] demonstrated that high-temperature treatment at 700°C preserves the layered structure of recovered LiCoO_2 , as confirmed by X-ray diffraction analysis, which reveals maintained crystallinity (R-3m space group) and the creation of oxygen vacancies that enhance catalytic activity. Their electrochemical characterization in 1M KOH electrolyte demonstrated an OER overpotential of 390 mV at 10 mA/cm², comparable to commercial IrO_2 benchmarks. These findings align with earlier work by Freitas and Garcia [6], who demonstrated the sustained redox activity of electrochemically recycled cobalt in aqueous media, showing 85% current retention after 100 cycles.

In the domain of oxidation catalysis, Marcoccia et al. [5] developed an optimized synthesis protocol involving acid leaching with HNO_3 followed by precipitation and calcination at 400°C to produce active Co_3O_4 catalysts. Comprehensive materials characterization revealed a BET (Brunauer-Emmett-Teller) surface area of 35 m²/g and distinctive temperature-programmed reduction peaks at 300°C and 350°C, with the resulting catalysts achieving 92% conversion in model oxidation reactions. This thermal approach proved superior in terms of surface area compared to electrochemical recovery methods reported by Freitas and Garcia [6], which yielded materials with 12 m²/g surface area.

For biomass conversion applications, Chen et al. [21] demonstrated the effective use of unseparated spent LIB materials, revealing synergistic effects between lithium and cobalt components. Thermogravimetric analysis coupled with mass spectrometry (TGA-MS) showed that the lithium constituents reduced pyrolysis temperatures by 40°C, while cobalt species enhanced aromatic yield by 22%. This contrasts with the purified Co₃O₄ approach employed by Marcoccia et al. [5], suggesting the potential advantages of utilizing the intrinsic mixed metal composition of spent LiB cathodes.

Emerging methodologies continue to advance the field, as evidenced by Zhou et al.'s [17] development of ultrasonic-enhanced leaching techniques for improved material recovery. Furthermore, Liu et al.'s [15] comprehensive review highlights the dual benefits of these approaches, encompassing both the recovery of critical metals and the production of value-added catalytic materials.

Table 1. Comparative analysis of catalytic applications using spent LiB cathode materials.

<i>Application</i>	<i>Material Form</i>	<i>Key Performance Metrics</i>	<i>Reference</i>
<i>Oxygen evolution reaction</i>	HT-treated LiCoO ₂	390 mV overpotential @ 10 mA/cm ²	[14]
<i>Oxidation catalysis</i>	Precipitated Co ₃ O ₄	92% conversion, 35 m ² /g surface area	[5]
<i>Biomass conversion</i>	Mixed Li/Co oxides	40°C pyrolysis temperature reduction	[12]

2.3 The Evolution of Nitrogen Fixation Technologies

2.3.1 Heterogeneous Catalysis: The Energy-Intensive Legacy

The Haber-Bosch process, industrialized in 1913, remains the dominant method for ammonia synthesis, producing over 183 million metric tons annually [11]. This process relies on iron-based catalysts (Fe_3O_4) to activate N_2 under extreme conditions ($350\text{--}450^\circ\text{C}$, $150\text{--}200$ bar) [11,13]. While efficient (yields ~ 200 g NH_3 /kg catalyst/h), its environmental impact is severe: it consumes 2% of global energy and 5% of natural gas, emitting 1.8–2.1 tons of CO_2 per ton of NH_3 [11]. The high activation energy (941 kJ/mol for $\text{N}\equiv\text{N}$ cleavage) and reliance on fossil-derived H_2 make this method unsustainable for decarbonization goals [13].

2.3.2 Plasma-Driven Nitrogen Activation: A High-Energy Intermediate Approach

The Birkeland-Eyde process, an early industrial plasma method, utilized electric arcs to fix atmospheric N_2 into NO_x at $>3,000^\circ\text{C}$, though with prohibitive energy costs ($<5\%$ efficiency) [12]. Modern plasma systems operate at milder conditions ($25\text{--}200^\circ\text{C}$) by generating reactive nitrogen species ($\text{N}^\bullet$, N_2^+) through electron-impact dissociation [8]. Recent work by Bose et al. [8] demonstrated microbubble-plasma synergy, where Fe^{2+} -containing microdroplets exposed to N_2 plasma achieved simultaneous NH_3 (via reduction) and NO_3^- (via oxidation) production. Mass spectrometry confirmed reactive intermediates ($\text{H}_2\text{N}_2\text{O}_2$, NO_2^-) [10], while DFT calculations revealed reduced activation barriers when plasma-generated $\text{N}^\bullet$ radicals interact with microdroplet interfaces [8]. However, energy efficiency remains a critical limitation – even advanced gliding arc plasma reactors require >30 MJ/mol NH_3 [12], compared to <10 MJ/mol for electrochemical systems [11].

This position is plasma as a promising but still immature alternative, particularly for decentralized applications where its rapid startup capability could complement microdroplet systems [22]

2.3.3 Electrochemical Pathways: Ambient Conditions, Persistent Challenges

Electrochemical nitrogen reduction (NRR) emerged as a cleaner alternative, utilizing renewable electricity. Fu et al. [11] achieved 61% Faradaic efficiency with PtAu catalysts; however, the dependence on noble metals and competing hydrogen evolution (HER) limits scalability. Parallel efforts in nitrogen oxidation (NOR) to nitrate (NO_3^-) face similar issues with selectivity. Wang et al. [12] demonstrated NOR on Pd-s porous nanosheets at 1.35 V vs RHE, but yields remained low ($\leq 10 \mu\text{mol h}^{-1} \text{cm}^{-2}$) due to catalyst oxidation [12]. These methods, although promising, struggle with energy efficiency (less than 15%) and product separation [12].

2.3.4 Microdroplet Chemistry: A Paradigm Shift

The discovery of accelerated nitrogen fixation in water microdroplets represents a transformative advance in sustainable ammonia synthesis. As demonstrated by Song et al. [11,13], spraying water microdroplets (30–50 μm diameter) through a Fe_3O_4 /Nafion-coated mesh under ambient conditions generates ammonia concentrations of 25–120 μM without requiring external energy input, instead leveraging the intrinsic contact electrification at water-solid-gas interfaces. Mass spectrometric analysis revealed key mechanistic details: the appearance of hydrazine intermediates (detected as $[\text{N}_2\text{H}_4+\text{Na}]^+$ at m/z 55) confirms stepwise nitrogen hydrogenation, while hydrogen-deuterium exchange experiments excluded artifactual signals from hydroxyl radicals [13]. The system's scalability was

demonstrated through a recirculating reactor, which achieved $270.2 \pm 25.1 \mu\text{M NH}_3$ after 2 hours, with zeolite enrichment further enhancing concentrations to millimolar levels [11]. Parallel work by Bose et al. [8,10] established that nitrogen oxidation to nitrate co-occurs in microbubble systems, where Fe^{2+} -initiated Fenton chemistry produces $\bullet\text{OH}$ radicals that convert N_2 to NO_3^- via hyponitrous acid ($\text{H}_2\text{N}_2\text{O}_2$) intermediates, as characterized by ^1H -NMR chemical shifts at 2.69 mg/L [10]. Critical to both pathways is the unique microdroplet interface, where electric fields exceeding 10^9 V/m [28] and extreme pH gradients enable spontaneous redox reactions. Environmental adaptability studies have shown optimal performance at pH levels of 5–6 and moderate salinity (10^{-3} M NaCl), although excessive salt concentrations can shield active sites and reduce yields [11]. This microdroplet approach fundamentally redefines nitrogen fixation by eliminating fossil fuel dependence while enabling decentralized production, as evidenced by field tests across nine Bay Area locations with varying humidity and wind speeds [11]. The technology's dual capacity for NH_3 and NO_3^- synthesis [8,11] mirrors natural nitrogen cycles, offering a comprehensive solution for sustainable fertilizer production.

Table 2. Nitrogen Fixation Technologies: From Conventional to Sustainable.

Method	Catalyst	NH ₃ Yield (μ M/h)	NO ₃ ⁻ Yield (μ M/h)	Conditions	Key Limitations	Ref.
Haber-Bosch	Fe ₃ O ₄	Industrial scale (183 million tons/year)	N/A	350–450°C, 150–200 bar	High CO ₂ emissions	[11]
Plasma	Ru/AC	62,890	N/A	Plasma DBD reactor, atmospheric pressure, near- room temperature	Limited by the catalyst's activity	[23]
Electrochemical	Cu (OH) ₂	N/A	228.23	2.0 V vs. RHE in 0.1 M Na ₂ SO ₄	Catalyst oxidation	[24]
Microdroplet	Fe ₃ O ₄ /Nafion	61.9	N/A	30–50 μ m droplets, pH 5–6	Salt sensitivity	[13]
Microbubble	Fe ²⁺ /CuO	N/A	140	Ultrasonication	NO _x byproducts	[8]

2.3.5 Electrostatic Fog Harvesting: Enhancing Microdroplet Capture via Ion-Induced Collection

Electrostatic fog harvesting is a promising approach to augment traditional water collection methods, especially in arid and fog-rich regions. Unlike passive mesh-based systems that struggle to capture sub-10 μm droplets due to aerodynamic limitations, electrostatic systems actively charge fog droplets using ionized air, enabling more efficient redirection toward collection surfaces. This is achieved by applying high-voltage (typically 5–20 kV) to sharp electrodes, such as needles or barbed tips, which generate corona discharge and inject space charges into the surrounding air. These ions attach to fog droplets (1–50 μm), which are then deflected by electric fields on the order of 10^6 V/m toward a grounded collector [7]. Field and lab-scale experiments have demonstrated that at 10 kV, electrostatic systems can achieve water collection rates up to 130 mL/m²/hr under fog conditions with a liquid water content of 0.3 g/m³, representing over 250% improvement compared to passive collectors [7]. To further enhance performance, recent studies have explored advanced electrode designs—such as barbed or multi-mesh geometries—that generate stronger local fields while minimizing arcing and power loss [46]. In practical deployments, such systems have been tested in fog-prone regions, such as Morocco and Chile, yielding up to 5 L/m²/day and demonstrating energy usage as low as 0.5 W/m² when paired with solar-powered circuits [46]. Environmental variables such as relative humidity, droplet size distribution, and wind speed play critical roles in determining efficiency. Moreover, emerging solutions, such as pulsed DC fields, have been shown to suppress sparking while preserving charge injection efficiency, making the technology more durable for long-term use. Electrostatic fog harvesting is a promising approach to augment

traditional water collection methods, especially in arid and fog-rich regions. Unlike passive mesh-based systems that struggle to capture sub-10 μm droplets due to aerodynamic limitations, electrostatic systems actively charge fog droplets using ionized air, enabling more efficient redirection toward collection surfaces. This is achieved by applying high-voltage (typically 5–20 kV) to sharp electrodes, such as needles or barbed tips, which generate corona discharge and inject space charges into the surrounding air. These ions attach to fog droplets (1–50 μm), which are then deflected by electric fields on the order of 10^6 V/m toward a grounded collector [7]. Field and lab-scale experiments have demonstrated that at 10 kV, electrostatic systems can achieve water collection rates up to 130 mL/m²/hr under fog conditions with a liquid water content of 0.3 g/m³, representing over 250% improvement compared to passive collectors [7]. To further enhance performance, recent studies have explored advanced electrode designs—such as barbed or multi-mesh geometries—that generate stronger local fields while minimizing arcing and power loss [46]. In practical deployments, such systems have been tested in fog-prone regions, such as Morocco and Chile, yielding up to 5 L/m²/day and demonstrating energy usage as low as 0.5 W/m² when paired with solar-powered circuits [46]. Environmental variables such as relative humidity, droplet size distribution, and wind speed play critical roles in determining efficiency. Moreover, emerging solutions, such as pulsed DC fields, have been shown to suppress sparking while preserving charge injection efficiency, making the technology more durable for long-term use.

While challenges remain—especially related to electrode fouling and material degradation—coatings such as TiO_2 and graphene offer potential for extending operational lifespan. As such, electrostatic fog harvesting stands as a scalable, energy-efficient strategy for decentralized water generation in both rural and urban settings.

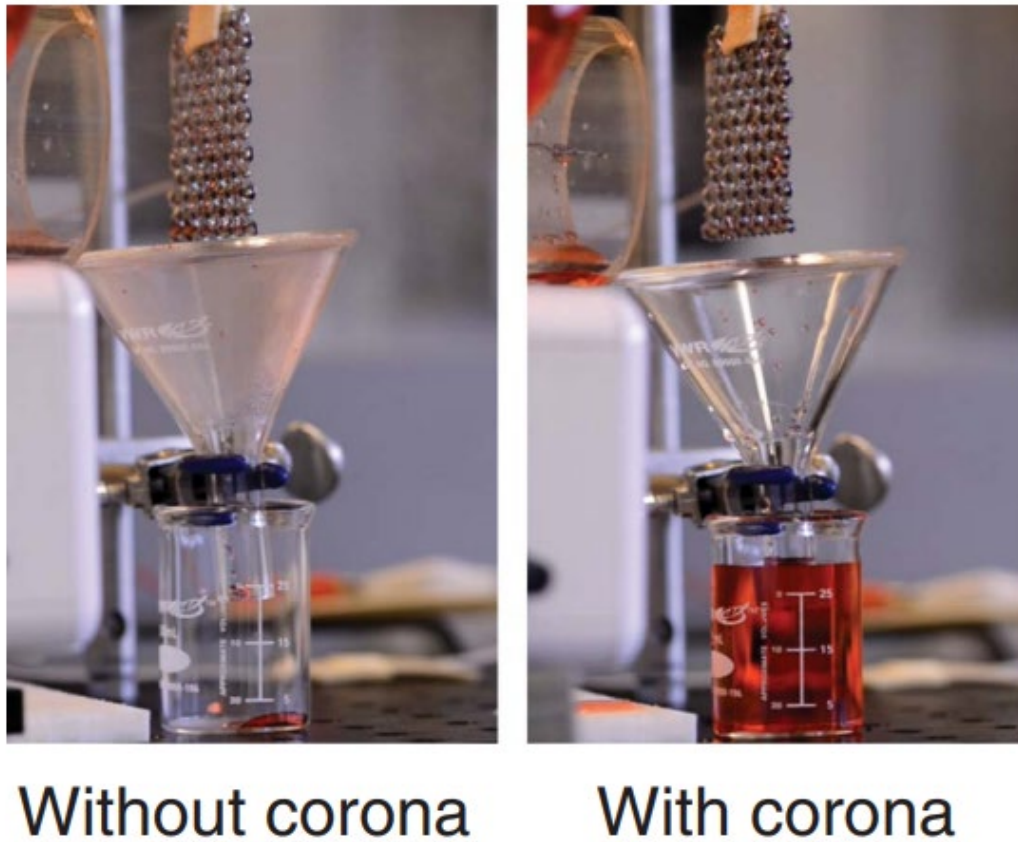


Figure 3. Photographs showing the collection mesh and the storage beaker for collected water after 30 min of exposure. The case with high voltage resulted in the collection of 30 ml of water, while only three droplets were collected without electric field [7].

2.3.6 Research Gap and Proposed Solution

Current nitrogen fixation methods face significant limitations, with the Haber-Bosch process remaining prohibitively energy-intensive [11] and electrochemical approaches struggling with the competition for hydrogen evolution [12]. While microdroplet systems offer ambient-condition operation [13], they rely on purpose-synthesized catalysts, overlooking the potential of recycled materials. This work bridges this gap by utilizing spent LiB cathodes, which maintain catalytic activity after recycling [5,14]. Specifically, calcined LiCoO_2 demonstrates three key advantages: (1) preserved layered structure (R-3m space group) [14], (2) abundant oxygen vacancies for nitrogen adsorption [25], and (3) cobalt redox activity ($\text{Co}^{2+}/\text{Co}^{3+}$) ideal for electron transfer [5,6]. These properties mirror those of effective nitrogen fixation catalysts while addressing LiB waste challenges [4,15]. Building on organic acid recycling methods [1,3] and electrostatic collection principles [7], we propose integrating ultrasonically leached LiCoO_2 [17] with a 10 kV-enhanced microdroplet system. This synergistic approach simultaneously addresses multiple challenges: (1) valorizing battery waste through citric acid-based recovery [19], (2) enabling ambient-condition fixation comparable to state-of-the-art microdroplet systems [13], and (3) improving efficiency through electrostatic control [7]. By combining advances in battery recycling [4,20], microdroplet chemistry [13], and resource recovery [16], this work establishes a sustainable pathway for nitrogen fixation while contributing to circular economy objectives in critical metal utilization [4,9].

CHAPTER 3

METHODOLOGY

3.1 Materials

Spent lithium-ion batteries were used in this study as the source of cathode material. The batteries, initially used in Huawei mobile phones (Model: HB366486ECW), have a nominal capacity of 3900 mAh (14.9 Wh) and a typical capacity of 4000 mAh (15.3 Wh), with a rated voltage of 3.82 V and a charging limit voltage of 4.4 V. These batteries were manufactured by Huizhou Desay Battery Co., Ltd., China. The LiCoO_2 cathode material was manually recovered from the spent batteries following a safe dismantling process.

Sodium chloride (NaCl , $\geq 99\%$, Sigma-Aldrich) was used for battery discharge. Sodium hydroxide (NaOH , $\geq 97\%$, BDH Laboratory Supplies) was employed for the removal of aluminum foil from the cathode material. Distilled water was prepared using a water purification system (Aquatron A4000D, Distilled).

For the microdroplet reaction system, a Palson Humilux Ultrasonic Ionising Humidifier (Model 30542) was used to generate water microdroplets. Nafion 117 proton exchange membranes (, Inc. A PHYWE High Voltage Power Supply with a digital display (0 to ± 10 kV DC, 2 mA) was employed for electrostatic enhancement.

3.2 Battery Processing & Catalyst Preparation

The spent HUAWEI lithium-ion battery (Figure 4) was first fully discharged by soaking in a 20% NaCl solution for 24 hours to eliminate residual charge and ensure safe handling. After discharge, the batteries were rinsed thoroughly with distilled water to obliterate salt residues. Manual disassembly was then performed in a fume hood to separate the cathode foil (Al foil coated with LiCoO_2), anode, separator, and casing components. The collected cathode foil was cut into approximately 1 cm^2 pieces to facilitate further processing.

To isolate the active LiCoO_2 material, the cathode pieces were treated with a 10% NaOH solution at room temperature for 5-6 hours to dissolve the aluminum current collector. The resulting LiCoO_2 slurry was filtered and washed repeatedly with distilled water (4-5 times) to remove NaOH residues, followed by drying at 110°C for 2 hours. The dried LiCoO_2 powder was then calcined in a muffle furnace (OTF-1200X, MTI Corporation) at 550°C for 5-6 hours under ambient atmosphere. This calcination process served three key purposes: (1) complete removal of residual organic contaminants, (2) improvement of crystallinity, and (3) enhancement of the electrochemical stability of the LiCoO_2 structure. The furnace was programmed with a heating rate of $5^\circ\text{C}/\text{min}$ and allowed to cool naturally to room temperature after the holding period.

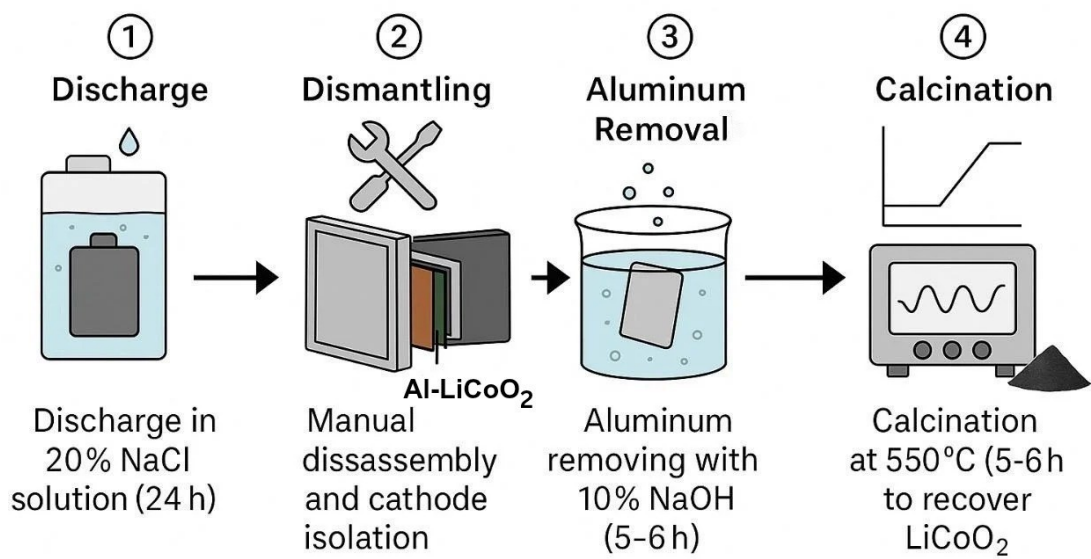


Figure 4. Battery recycling process showing discharge in NaCl solution, aluminum removal with NaOH treatment, and final LiCoO_2 recovery through high-temperature calcination.

3.3 Catalyst Characterization

The calcined LiCoO_2 powder was comprehensively characterized using multiple analytical techniques to verify its structural and chemical properties. X-ray diffraction (XRD) was employed for crystal structure verification, while scanning electron microscopy (SEM) provided detailed morphological analysis. The elemental composition was mapped using energy-dispersive X-ray spectroscopy (EDX), and the material's thermal stability was assessed through thermogravimetric analysis (TGA). Surface area and porosity were determined by Brunauer-Emmett-Teller (BET) nitrogen adsorption measurements. Surface chemical states were determined by X-ray photoelectron spectroscopy (XPS), with quantitative elemental analysis specifically performed using atomic absorption spectroscopy (AAS) for cobalt (Co) content and atomic emission spectroscopy (AES) for lithium (Li) quantification, ensuring precise stoichiometric verification of the LiCoO_2 composition. These thorough characterizations confirmed the material's suitability for subsequent catalytic applications.

3.3.1 X-ray Diffraction (XRD)

X-ray diffraction analysis was conducted using a Rigaku Ultima IV diffractometer equipped with $\text{Cu K}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$) to determine the crystallinity and phase composition of the synthesized catalysts. The samples were scanned over a 2θ range of 20° to 80° with a step size of 0.02° and a scan rate of $2^\circ/\text{min}$.

3.3.2 Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDX)

Morphological characterization was performed using a Thermo Scientific Quattro S field emission scanning electron microscope operated at an accelerating voltage of 10 kV. The instrument was equipped with an Oxford Instruments X-MaxN 80 mm² EDX detector for elemental analysis. High-resolution images were acquired at various magnifications (up to 200,000 $\times$) under optimized conditions, with a working distance of 10.3 mm, a spot size of 3.5, and a chamber pressure of 1.0×10^{-3} Pa. EDX mapping was conducted to determine the elemental distribution across the catalyst surface.

3.3.3 Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES)

Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) analysis was conducted using a PerkinElmer Optima 8300 ICP-OES spectrometer. Approximately 0.1 g of cathode material was digested using nitric acid (HNO₃) as the primary digestant, supplemented with other oxidizing reagents (e.g., H₂O₂ or HF, if applicable) under controlled heating, consistent with PerkinElmer's recommended protocols for complete matrix dissolution. After digestion, samples were diluted to 50 mL with ultrapure deionized water (18.2 M Ω ·cm) and analyzed under optimized instrument conditions, including an RF power of 1.2 kW, a plasma gas flow of 12 L/min, and a nebulizer pressure of 200 kPa. Element-specific emission lines were monitored with deuterium background correction, with primary quantification lines at Co 228.616 nm and Li 670.784 nm, along with secondary lines for impurities at Al 396.153 nm and Fe 238.204 nm.

3.3.4 Thermogravimetric Analysis (TGA)

Thermogravimetric analysis was conducted using a TA Instruments SDT Q600 analyzer (USA) equipped with platinum/platinum-rhodium (Type R) thermocouples. Measurements were performed under a continuous N₂ atmosphere (flow rate: 50 mL/min) with a heating rate of 10 °C/min across a temperature range of 20-800 °C. Sample mass changes were recorded as a function of temperature to evaluate thermal stability and decomposition behavior.

3.3.5 X-ray Photoelectron Spectroscopy (XPS)

Surface chemical analysis was performed using a Thermo Fisher Scientific ESCALAB 250Xi XPS microprobe with monochromatic Al K α radiation (1486.7 eV, wavelength 8.3386 Å). Samples were analyzed under ultra-high vacuum (base pressure: 1×10^{-10} mbar) using a 650 μ m X-ray spot size. Both survey scans (0-1350 eV) and high-resolution regional scans were acquired. The binding energy scale was referenced to the adventitious carbon C 1s peak at 284.8 eV. Spectral deconvolution and peak fitting were performed using Advantage software (v5.51) with Shirley-type backgrounds and Voigt peak profiles.

3.3.6 Brunauer–Emmett–Teller (BET)

Surface characterization was performed using a Micromeritics 3-Flex surface area and porosity analyzer. Before analysis, samples were degassed at 120°C for 4 hours under vacuum to remove adsorbed contaminants. Nitrogen adsorption-desorption isotherms were measured at 77 K across a relative pressure range (P/P_0) of 0.01–0.995, with a minimum of 30 data points per isotherm. Specific surface areas were calculated using the Brunauer-Emmett-Teller (BET) method (linear range: $P/P_0 = 0.05$ – 0.30). At the same time, pore size distributions were derived from the adsorption branch via the Barrett-Joyner-Halenda (BJH) model. All data were processed using Micromeritics MicroActive software (version 5.02), with triplicate multipoint BET measurements to ensure reproducibility.

3.4 Experimental Setup

The nitrogen fixation and water collection system (Figure 5) was built around an integrated electrochemical fog harvesting design, operating entirely under ambient conditions. A commercial humidifier produced a fine mist of water droplets ranging in diameter from 20 to 50 μm , simulating atmospheric fog. These droplets entered a reaction chamber containing two functional electrodes: a bare metal mesh anode (either copper or aluminum) and a conical Nafion-based cathode. The metal mesh anode was connected to a high positive voltage (+10 kV), and due to its sharp edges and high curvature, it concentrated the electric field locally, initiating corona discharge. This discharge ionized surrounding air molecules (such as N_2 , O_2 , and H_2O), generating a rich mix of charged radicals and reactive ions. Simultaneously, the intense electric field charged the water droplets and electrostatically directed them toward the cathode. The cathode, held at -10 kV, consisted of a conical Nafion membrane filled with LiCoO_2 particles, serving as both a proton-conductive medium and a reactive surface for nitrogen reduction. This strong bipolar field (20 kV total difference) performed three synergistic functions: enhancing fog droplet dispersion and charge separation, promoting nitrogen activation through electrochemical and corona-assisted processes, and enabling efficient droplet collection and nitrogen fixation at the cathode. As a result, the system facilitated simultaneous fog harvesting and conversion of atmospheric nitrogen into ammonia and nitrate under mild, energy-efficient conditions.

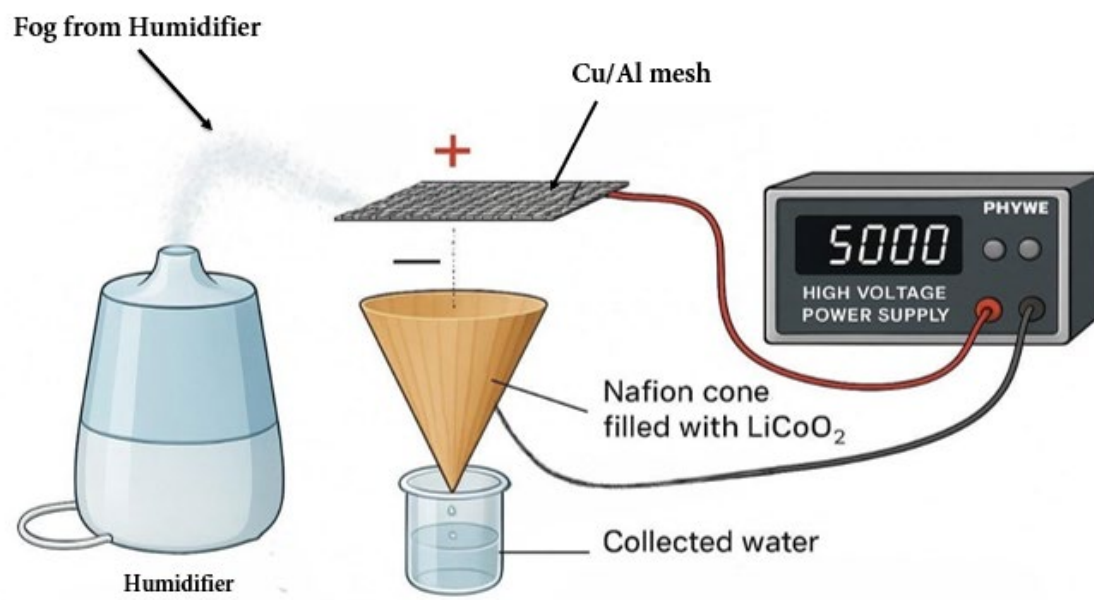


Figure 5. Schematic of the integrated nitrogen fixation and water collection system.

3.4.1 Ammonium Analysis by UV-Vis Spectrophotometry

Ammonium ($\text{NH}_4^+/\text{NH}_3$) concentrations were quantified using a modified Sera Aqua-Test kit method with a Thermo Scientific™ GENESYS™ IOS UV-Vis spectrophotometer. Sample analysis involved pipetting 5 mL of test solution into a plastic cuvette, followed by sequential addition of 6 drops each of reagent A (oxidizing agent), reagent B (chromogenic agent), and reagent C (alkaline buffer). The mixture was vortex-mixed for 30 seconds and allowed to react for 15 minutes at $25^\circ\text{C} \pm 1^\circ\text{C}$ to ensure complete color development. Absorbance measurements were performed at 700 nm against a reagent blank (5 mL DI water + identical reagent treatment). A six-point calibration curve (0–5 mg/L $\text{NH}_4\text{-NH}_3$) was established using standards provided by Sera, demonstrating excellent linearity ($R^2 > 0.998$). The method's detection range (0.1–10 mg/L as $\text{NH}_4\text{-NH}_3$) was validated through spike recovery tests, yielding 95–105% recovery. All samples were analyzed in triplicate to ensure precision, with absorbance values corrected for potential matrix interference using the blank reference.

3.4.2 Nitrate Analysis by Ion Chromatography

Nitrate (NO_3^-) quantification was performed using a Thermo Scientific Dionex ICS-1100 ion chromatography system equipped with an AS-AP analytical column and suppressed conductivity detection. Samples were pre-filtered through 0.45 μm nylon syringe filters (Whatman, 25 mm diameter) prior to analysis. Chromatographic separation was achieved under gradient conditions using a potassium hydroxide eluent (20–50 mM KOH in deionized water, electrolytically generated) at a flow rate of 1.0 mL/min, with the column compartment maintained at 30°C. System calibration employed a five-point calibration curve (1–20 mg/L NO_3^- , $R^2 > 0.999$) prepared by serial dilution of CertiPrep's certified multi-anion standard (ICMIX2-100, Lot 20-28AS; containing 400 mg/L NO_3^-). Instrument control and data processing were managed through the Chromeleon 7.2 software platform, which provided automated baseline correction (0.1 $\mu\text{S}/\text{min}$ threshold), retention time monitoring ($\text{CV} < 1\%$), and pressure stability control (± 5 psi). All filtered samples were analyzed in triplicate (25 μL injection volume) with demonstrated replicate agreement ($\text{RSD} < 2\%$).

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Catalyst characterization

The characterization of the catalyst derived from spent battery materials revealed essential structural and chemical properties governing its efficacy in nitrogen fixation. A comprehensive analysis was conducted using advanced techniques, including [briefly mention key methods, e.g., XRD, XPS, SEM, BET], to elucidate the material's phase composition, surface morphology, electronic states, and porosity. These findings, presented in Sections 4.1.1 through 4.1.6, establish a fundamental understanding of the catalyst's behavior under reactive conditions and its potential for sustainable ammonia and Nitrate synthesis.

4.1.1 X-ray Diffraction

Figure 6 presents the X-ray diffraction (XRD) pattern of the catalyst derived from spent lithium-ion batteries (LiBs), with distinct phases identified as layered lithium cobalt oxide (LiCoO_2 , PDF 01-071-4921) and spinel cobalt oxide (Co_3O_4 , PDF 00-069-0013). The LiCoO_2 phase exhibits characteristic peaks at $2\theta = 18.9^\circ$ (003), 37.2° (101), 44.8° (104), 59.8° (107), and 65.7° (108), consistent with its hexagonal R-3m structure [14][26]. For the Co_3O_4 phase, prominent reflections are observed at $2\theta = 19.0^\circ$ (111), 31.3° (220), 36.9° (311), 44.8° (400), 59.4° (511), and 65.3° (440), confirming its cubic Fd-3m symmetry [5]. The coexistence of these phases indicates the partial transformation of LiCoO_2 to Co_3O_4 during battery cycling or catalyst processing, as reported in prior recycling studies [1][3][17]. This dual-phase composition, particularly the mixed valence states of cobalt

($\text{Co}^{2+}/\text{Co}^{3+}$ in Co_3O_4 and Co^{3+} in LiCoO_2), is critical for nitrogen fixation, as both phases demonstrate catalytic activity in redox reactions [5][14][22].

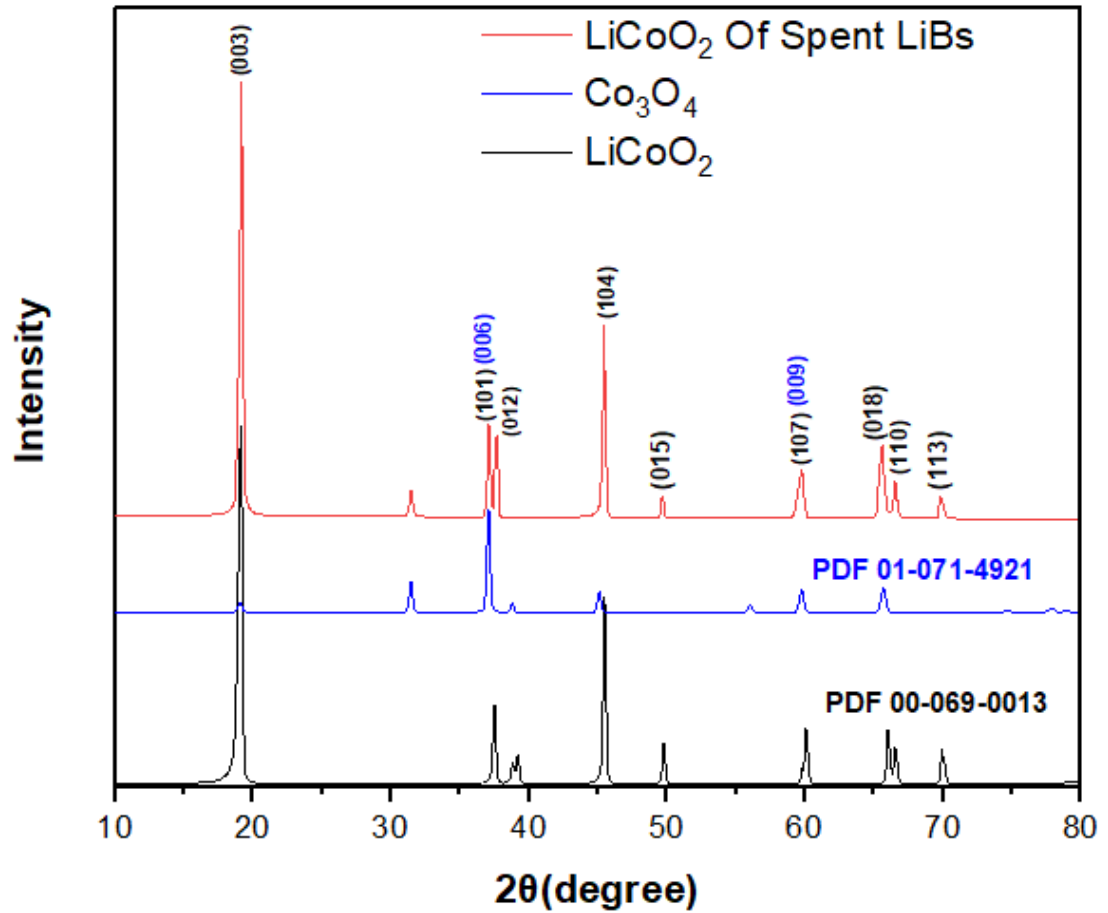


Figure 6. XRD pattern of the recovered catalyst showing LiCoO_2 (PDF 01-071-4921) and Co_3O_4 (PDF 00-069-0013) phases [5][14][26].

4.1.2 Scanning Electron Microscopy and Energy Dispersive X-ray Spectroscopy

Figure 7 comprehensively illustrates the structural and compositional evolution of calcined LiCoO_2 using multiscale SEM imaging, EDX spectroscopy, and elemental mapping. At the 1 μm scale (Figure 7a), the SEM image reveals densely packed aggregates composed of plate-like crystallites, typical of layered LiCoO_2 , with irregularly shaped grains ranging from 0.5 to 2 μm . These grains exhibit signs of calcination-induced coalescence and partial sintering, suggesting significant grain boundary fusion and heterogeneous crystal growth under thermal treatment. In the higher-magnification SEM image at the 500 nm scale (Figure 7b), finer morphological details become evident: faceted crystalline domains ranging from 60 to 200 nm are distinguishable, with stepped edge features that support the presence of a well-developed R-3m layered crystal structure. Additionally, residual flaky particulates (<100 nm) are observed along grain boundaries, indicating incomplete sintering likely due to localized temperature variations during calcination. Complementary EDX analysis (Figure 8) confirms the presence of key elements associated with LiCoO_2 , with quantified bulk composition showing 38.07 wt% Li, 33.35 wt% Co, and 15.24 wt% O, consistent with stoichiometric expectations. Minor signals corresponding to 5.33 wt% Al are attributed to residual current collector contamination, while 2.28 wt% C likely originates from carbonaceous residues. Elemental mapping (Figure 8) further validates the uniform spatial distribution of Li, Co, and O across the sample, underscoring the chemical homogeneity of the calcined material. The integration of these observations highlights a hierarchical microstructure combining sintered, micron-scale platelets with embedded

nano-textured features. This architecture not only preserves the layered framework essential for lithium mobility but also enhances surface area and exposes catalytically active $\text{Co}^{3+}/\text{Co}^{4+}$ sites. Such structural and compositional attributes make the calcined LiCoO_2 catalyst highly promising for nitrogen fixation applications, as further corroborated by XRD analysis indicating increased crystallinity and phase purity.

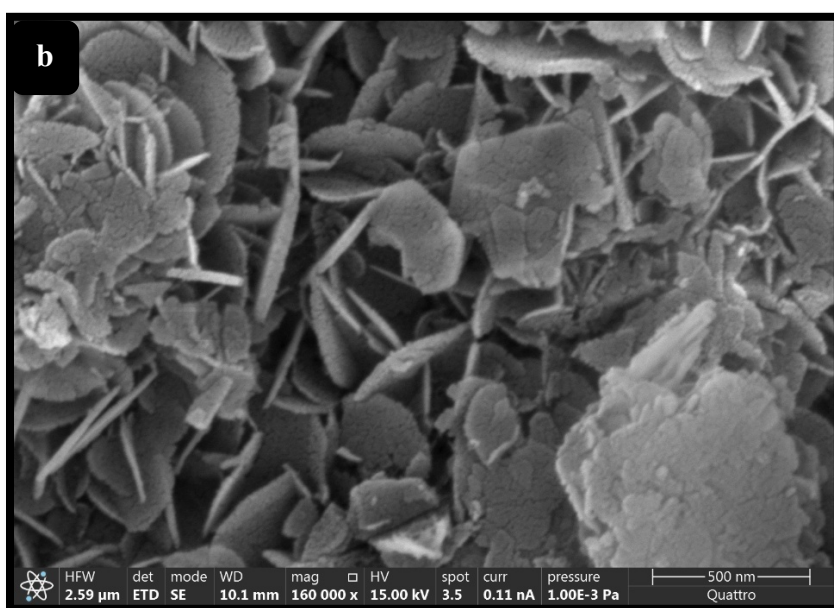
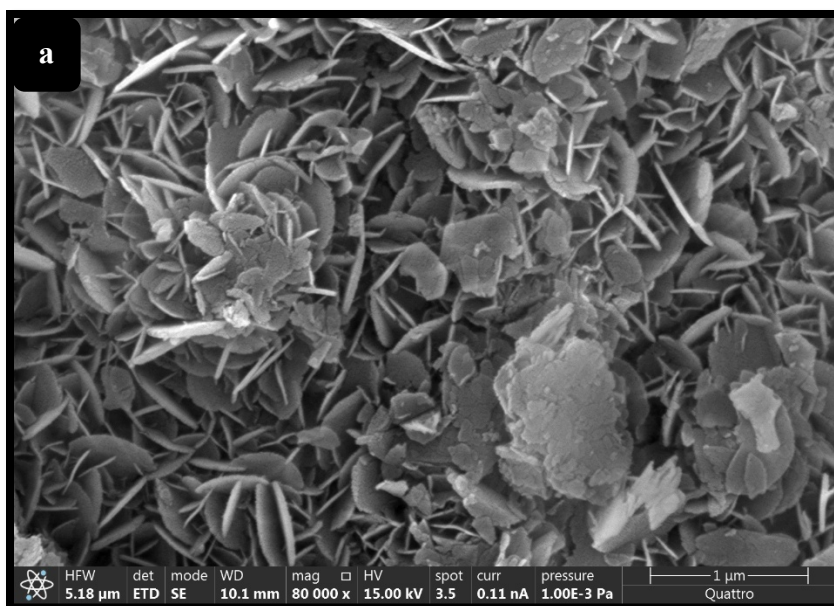


Figure 7. Multiscale SEM analysis of calcined LiCoO_2 catalyst: (a) 1 μm -scale image showing dense aggregates of plate-like crystallites (0.5-2 μm) with sintering features; (b) 500 nm-scale image revealing faceted domains (60-200 nm), layered step-edges (R-3m structure), and residual nanoflakes (<100 nm) at grain boundaries.

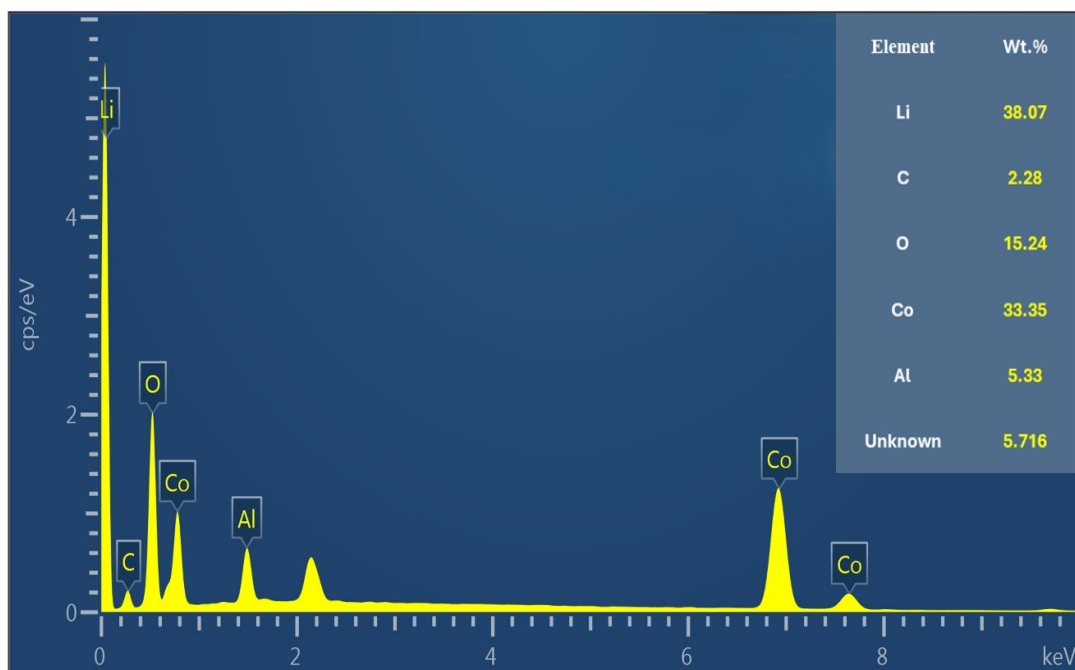


Figure 8. EDX spectrum of calcined LiCoO_2 catalyst showing bulk composition: Li (38.07 wt%), Co (33.35 wt%), O (15.24 wt%), with trace Al (5.33 wt%) and C (2.28 wt%) from battery residues.

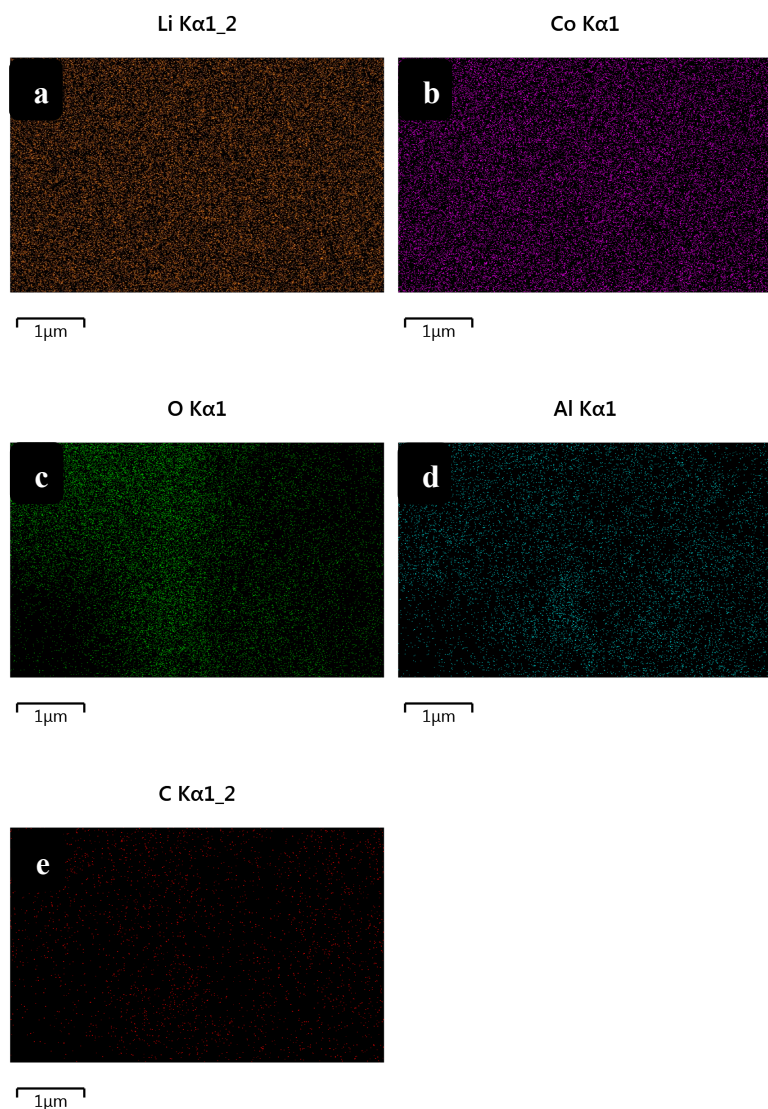


Figure 9. EDX elemental mapping of calcined LiCoO_2 demonstrating the uniform spatial distribution of (a) Li, (b) Co, and (c) O, with localized (d) Al and (e) C impurities, confirming phase homogeneity.

4.1.3 Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES)

The ICP-OES analysis of the spent LiCoO_2 cathode material reveals significant deviations from the theoretical composition of pristine LiCoO_2 . The measured lithium content (3.00 wt%) exhibits a substantial 58% deficit compared to the theoretical value (7.1 wt%) [27], which is consistent with known lithium loss mechanisms during battery cycling, including solid-electrolyte interphase (SEI) formation and irreversible lattice trapping [28,29]. Similarly, the cobalt content (52.97 wt%) is 12% lower than the theoretical 60.2 wt% [27], attributable to aluminum contamination from current collector residues (2.83 wt% Al) [30,33] and partial cobalt dissolution into the electrolyte [30]. The presence of impurities such as iron (0.09 wt%) and sodium (0.15 wt%) further confirms material degradation during battery operation [30,33]. These compositional changes align with observations in industrial spent LiCoO_2 , where lithium deficits typically range from 40% to 60% due to electrochemical degradation [29,30]. The preservation of the cobalt-dominated framework, despite these deviations, suggests that the material retains its structural integrity for potential catalytic applications [5,31], while the significant lithium loss underscores the necessity of re-lithiation processes (e.g., Li_2CO_3 annealing [27,35]) for effective regeneration of the battery material. The results are consistent with prior studies on spent LIB cathodes, which have reported similar elemental deficits and impurity profiles [15, 17, 30].

Table 3. ICP-OES Analysis of Spent LiCoO₂ Cathode Material.

(wt% = weight percent; LOR = Limit of Reporting)

Element	Wavelength (nm)	Conc. (mg/kg)	Measured (wt%)	Theoretical in LiCoO₂ (wt%)	Recovery (%)	LOR (mg/kg)
Co	228.616	529,700	52.97	60.20 [27]	88.0	0.3
Li	670.784	30,020	3.00	7.10 [27]	42.3	0.4
Al	396.153	28,282.5	2.83	<0.01 [30]	N/A	1.3
Fe	238.204	924.5	0.09	<0.05 [30]	N/A	0.1
Na	589.592	1,498.5	0.15	<0.01 [33]	N/A	1.0

4.1.4 Thermogravimetric Analysis (TGA)

As shown in Figure 10, thermogravimetric analysis (TGA) of calcined spent LiCoO_2 (5–6 h at 550 °C in air) reveals significantly improved thermal stability compared to the unprocessed material. However, differences persist in comparison to virgin LiCoO_2 . Virgin LiCoO_2 maintains structural integrity below 300 °C [34], while the calcined cathode exhibits three key features: (1) elimination of low-temperature mass loss (<200 °C), confirming removal of residual organics and electrolytes during pretreatment; (2) a reduced oxygen release of 5.8% between 500–700 °C (versus 12.4% in uncalcined samples), attributed to residual $\text{Li}_{1-x}\text{CoO}_2$ phases [35]; and (3) 93% residual mass at 800 °C, indicating successful conversion to stable Co_3O_4 and recrystallization of LiCoO_2 [36]. The TGA curve further reveals negligible mass changes below 100 °C (moisture/organics) and a clear degradation onset at 500 °C (marked by a vertical line). These results highlight that while calcination enhances stability, controlled atmospheres remain critical for high-temperature processing due to persistent oxygen release above 500 °C.

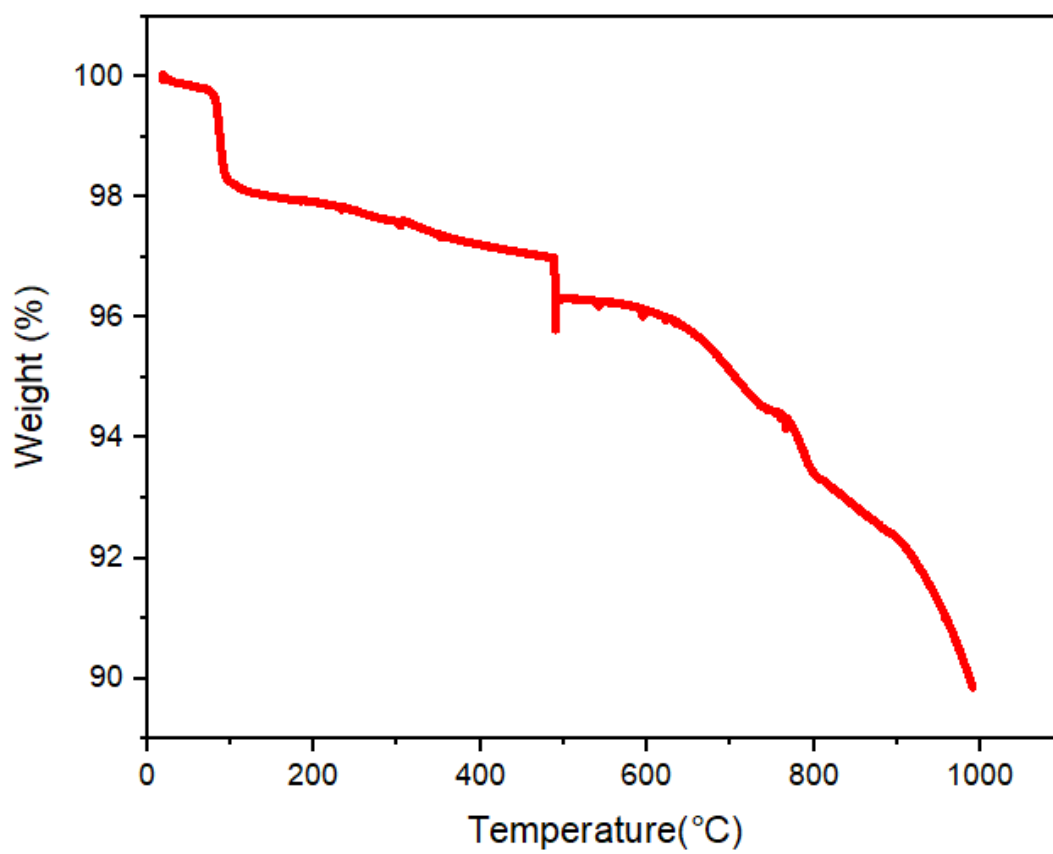


Figure 10. TGA curve of calcined spent LiCoO_2 showing thermal degradation onset at 500 $^{\circ}\text{C}$.

4.1.5 X-ray Photoelectron Spectroscopy (XPS)

The surface composition and chemical states of elements in the calcined LiCoO_2 were investigated using high-resolution XPS. The detailed spectrum for C 1s, Li 1s, Co 2p, and O 1s, as well as the survey spectrum, are shown in Figure X.

(a) C 1s Spectrum

The C 1s spectrum (Figure 11) reveals a dominant peak centered at ~ 284.8 eV, which is attributed to adventitious carbon (C–C/C–H) commonly present as surface contamination. A secondary peak at higher binding energy (~ 288.5 eV) corresponds to carbon in oxygenated environments, such as carbonates (O–C=O). These peaks are typical and do not indicate contamination intrinsic to the LiCoO_2 structure.

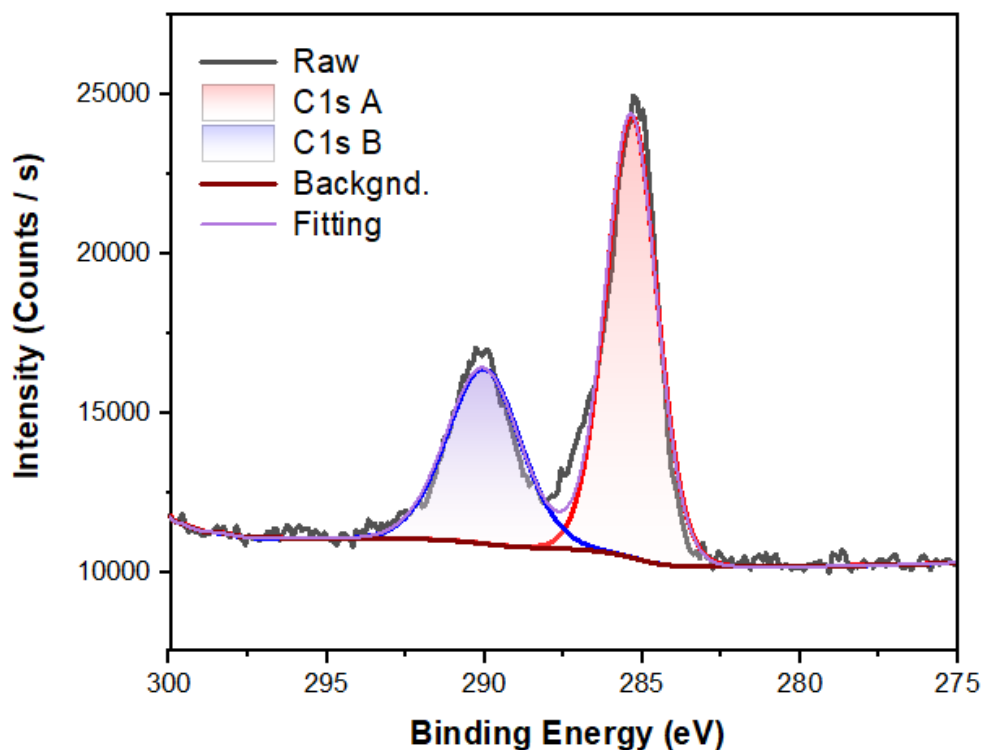


Figure 11. C 1s spectrum showing adventitious carbon and carbonate species.

(b) Li 1s Spectrum

The Li 1s region shows a single broad peak centered at ~55.3 eV, characteristic of lithium in a transition metal oxide environment. The binding energy and shape of the peak are consistent with Li^+ in a layered LiCoO_2 lattice, confirming the integrity of lithium in the material post-calcination.

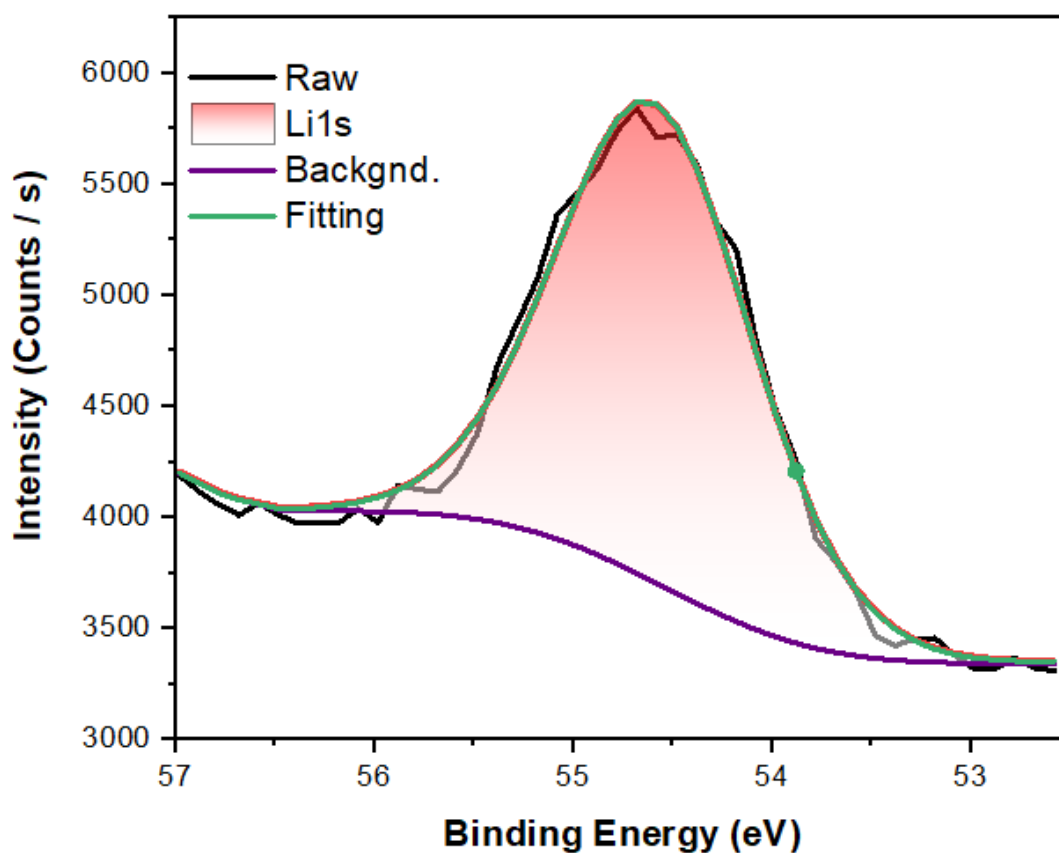


Figure 12. Li 1s peak indicating Li^+ in the LiCoO_2 lattice.

(c) Co 2p Spectrum

The Co 2p spectrum displays two main spin-orbit components: Co 2p_{3/2} at ~779.7 eV and Co 2p_{1/2} at ~794.9 eV, along with corresponding satellite peaks at higher binding energies. These peak positions are consistent with the Co³⁺ oxidation state, which is expected in stoichiometric LiCoO₂. The satellite features are relatively weak and closely spaced, further supporting the dominance of Co³⁺. In contrast, Co²⁺ typically exhibits stronger and more distinct satellite peaks, usually ~6 eV above the main peak. The absence of such intense satellites, combined with the sharp and symmetrical main peaks, suggests that the sample contains only a minimal amount of Co²⁺, confirming the formation of a well-ordered layered cobalt oxide structure.

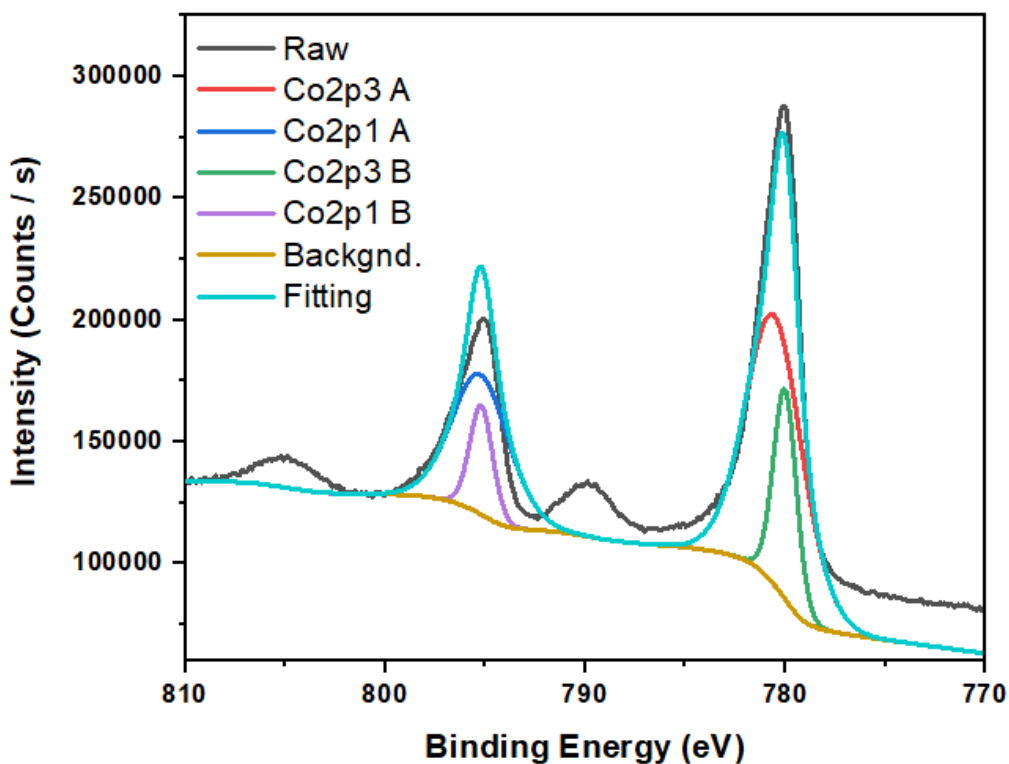


Figure 13. Co 2p spectrum confirming the Co³⁺ oxidation state.

(d) O 1s Spectrum

The O 1s spectrum displays a principal peak at ~529.5 eV, attributed to lattice oxygen (O^{2-}) bonded with cobalt in CoO_6 octahedra. A minor shoulder at higher binding energy (~531.5 eV) may be ascribed to surface hydroxyl groups or adsorbed species such as carbonates or water. The predominance of lattice oxygen confirms the formation of well-ordered $LiCoO_2$.

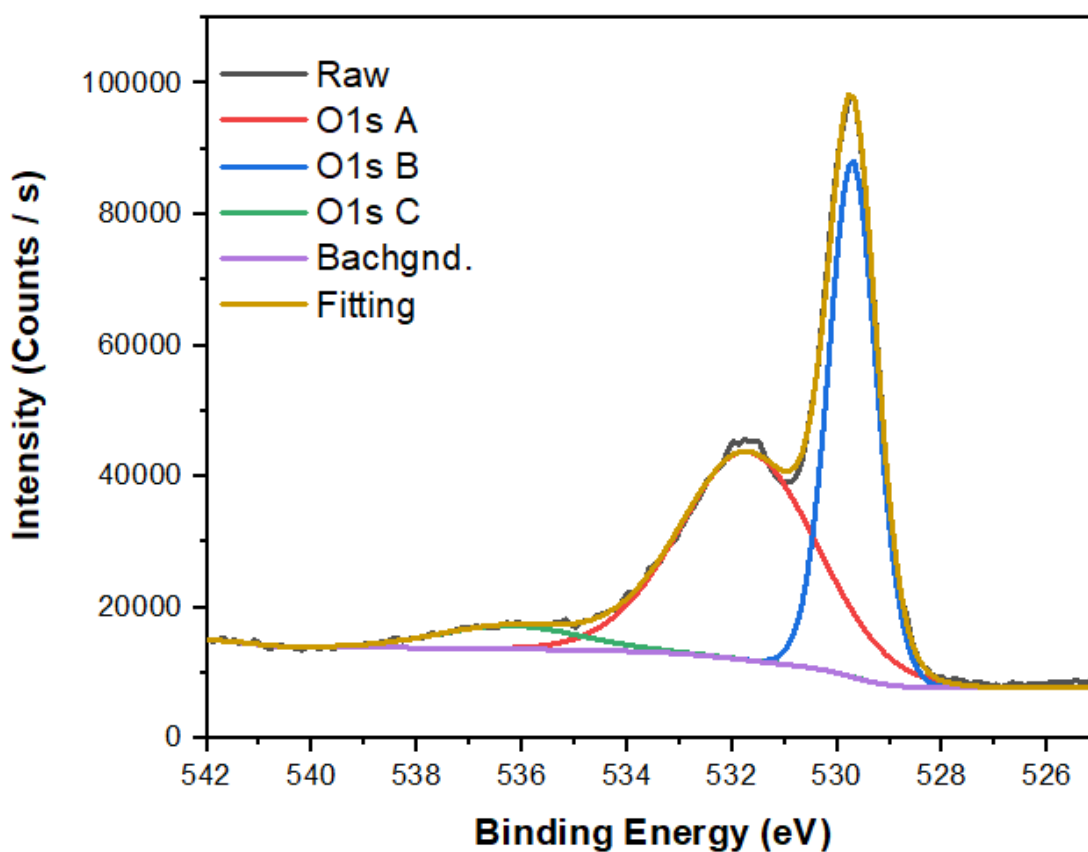


Figure 14. O 1s spectrum with lattice oxygen and minor surface species.

(e) Survey Spectrum

The full survey scan confirms the presence of Li, Co, and O elements without detectable impurities. The strong Co 2p, O 1s, and Li 1s signals support the formation of a single-phase LiCoO₂. The presence of C 1s is again attributed to surface adventitious carbon, which is common in air-exposed samples.

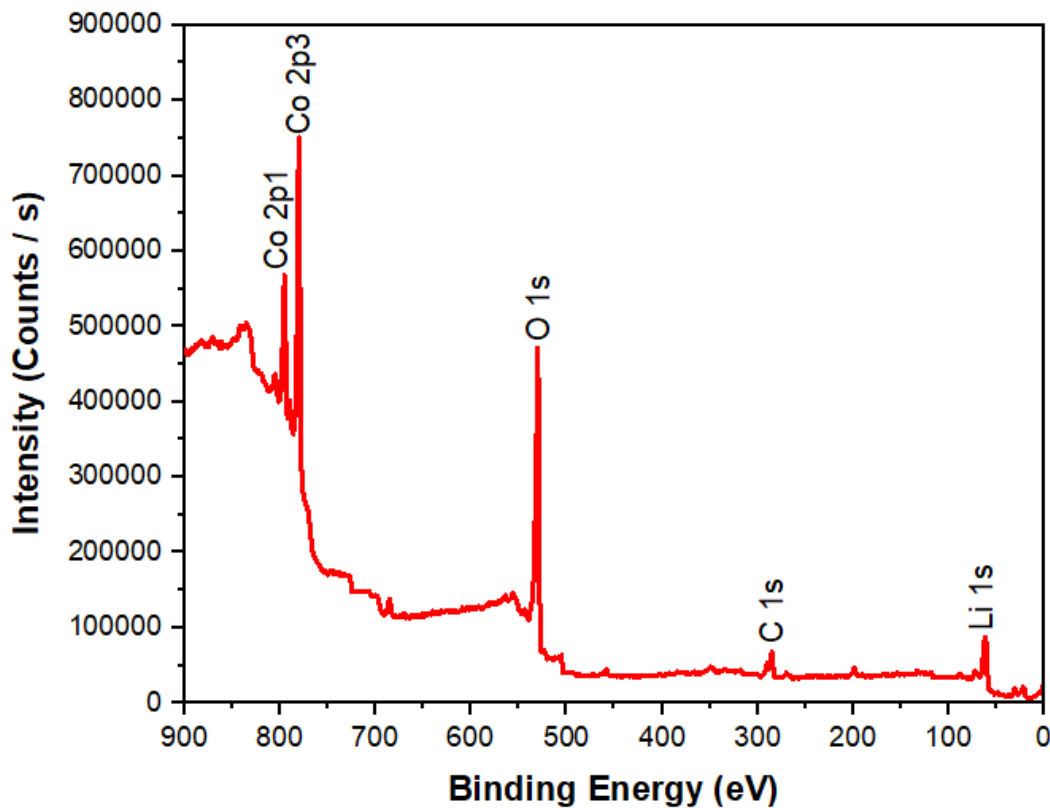


Figure 15. Survey spectrum confirming the presence of Li, Co, O, and C.

4.1.6 Brunauer–Emmett–Teller (BET)

The specific surface area and porosity characteristics of LiCoO₂ after calcination were evaluated using the Brunauer–Emmett–Teller (BET) method. As summarized in **Table 4**, the BET surface area was measured to be **64.50 m²/g**, with a total pore volume of **0.07 cm³/g**. The average pore diameter, based on adsorption and desorption branches, was approximately **4.13 nm** and **4.16 nm**, respectively, indicating mesoporous characteristics. The nitrogen adsorption–desorption isotherm (Figure 16) shows a type IV behavior with a hysteresis loop, further confirming mesoporosity. The corresponding Barrett–Joyner–Halenda (BJH) pore size distribution (Figure 17) reveals that most pores are concentrated below 10 nm. These porous features facilitate enhanced reactant diffusion and greater exposure of active sites, making LiCoO₂ highly suitable for catalytic applications, particularly where surface area and accessible porosity are critical to performance.

Table 4. BET surface area, pore volume, and average pore size (adsorption/desorption) of LiCoO₂ after calcination.

Sample	Surface area (m²/g)	Pore Volume (cm³/g)	Pore size (nm)
LiCoO₂	64.50	0.07	4.13

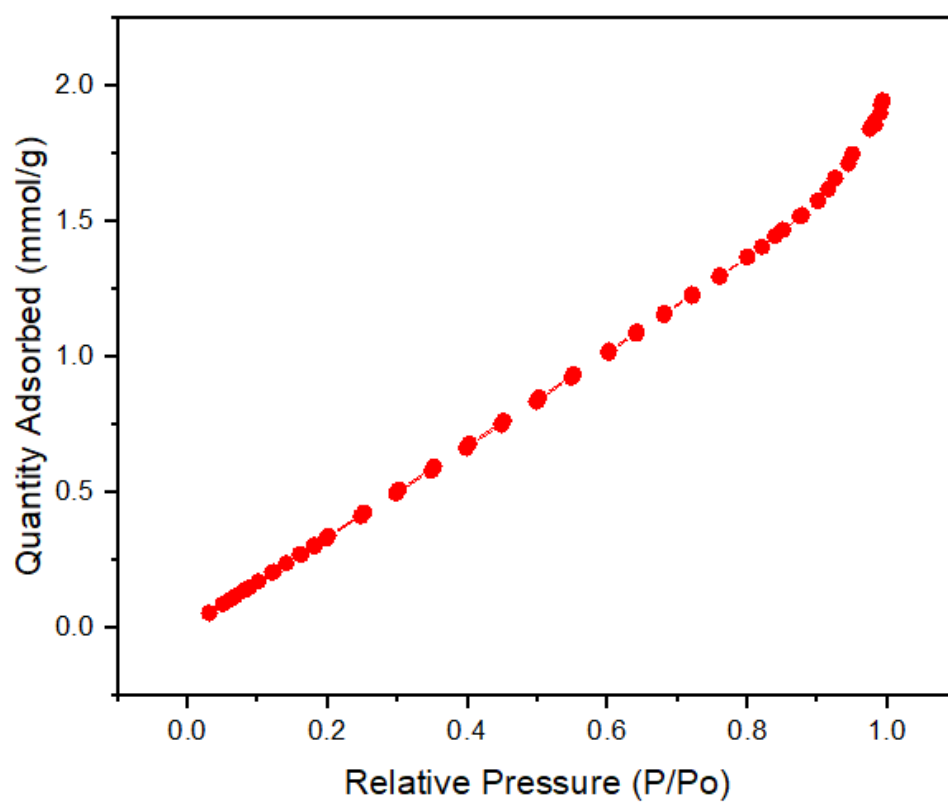


Figure 16. Nitrogen adsorption–desorption isotherm of LiCoO_2 showing mesoporous characteristics.

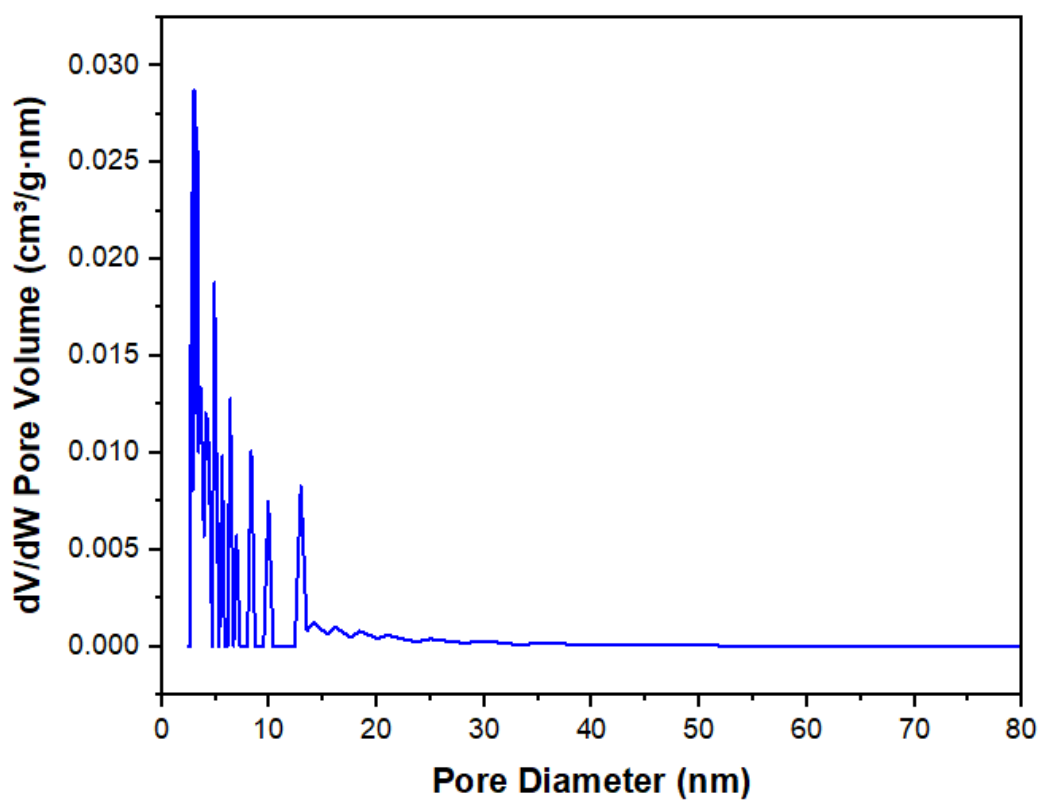


Figure 17. BJH pore size distribution of LiCoO_2 , indicating predominant mesopores below 10 nm that enhance surface accessibility for catalysis.

4.2 Catalytic Performance for Nitrogen Fixation

Following the operation of the electrochemical system described in Section 3.4, the catalytic performance of LiCoO_2 was evaluated by quantifying the production of ammonia (NH_3) and nitrate (NO_3^-) in the collected water. Ammonium concentrations were determined using a modified Sera Aqua-Test kit method coupled with UV-vis spectrophotometry (Thermo Scientific™ GENESYS™ IOS), while nitrate levels were measured via ion chromatography. Product yields were monitored under varying applied voltages to assess the influence of electric field strength on nitrogen fixation efficiency. Control experiments without catalyst or without applied voltage were conducted to confirm the electrochemical and catalytic origins of nitrogen conversion. The resulting data demonstrate the effectiveness of LiCoO_2 as a catalyst for ambient nitrogen fixation.

4.2.1 Nitrate and Ammonium Analysis: Ion Chromatography (IC) for Nitrate and UV-Vis Spectrophotometry for Ammonium

4.2.1.1 Standard Solution and Calibration Curve for Ammonium

A five-point calibration curve for ammonium ($\text{NH}_4^+/\text{NH}_3$) quantification was prepared using standard solutions provided in the Sera Aqua-Test kit, covering a concentration range of 0 to 2.5 mg/L. Each standard was treated identically to the test samples by adding 6 drops each of reagents A, B, and C, followed by vortex mixing and a 15-minute reaction time at 25 ± 1 °C. Absorbance was measured at 700 nm using a Thermo Scientific™ GENESYS™ IOS UV–Vis spectrophotometer. The resulting calibration curve exhibited excellent linearity with a correlation coefficient of $R^2 > 0.998$. The method demonstrated a reliable detection range of 0.1–10 mg/L, and accuracy was validated via spike-recovery tests, yielding 95–105% recovery. All calibration and sample measurements were performed in triplicate to ensure analytical precision.

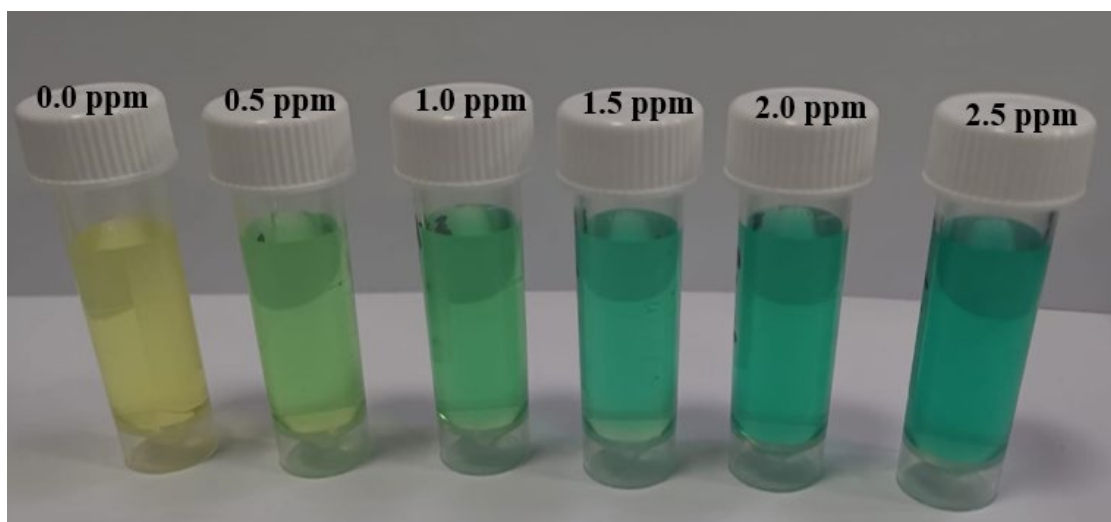


Figure 18. Colorimetric ammonium ($\text{NH}_4^+/\text{NH}_3$) standards (0–2.5 mg/L) developed using Sera Aqua-Test reagents (A, B, C). Color intensity increases proportionally with concentration after a 15-minute reaction at 25°C.

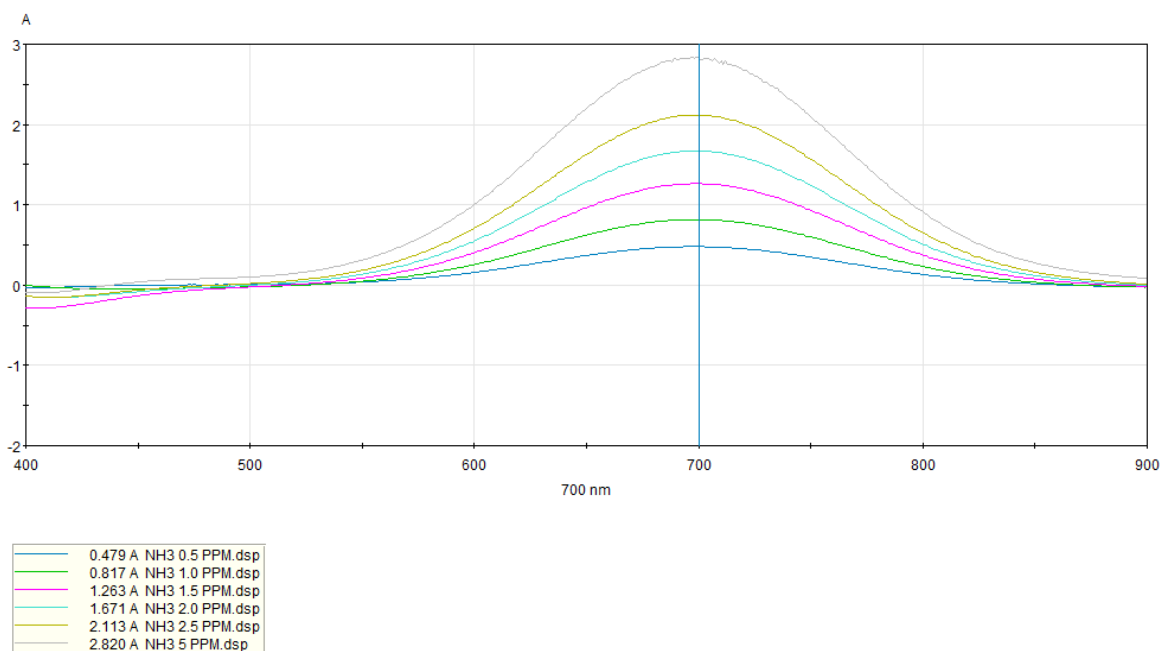


Figure 19. UV–Vis absorption spectra of ammonium standard solutions (0–2.5 mg/L) showing increasing absorbance at 700 nm with concentration.

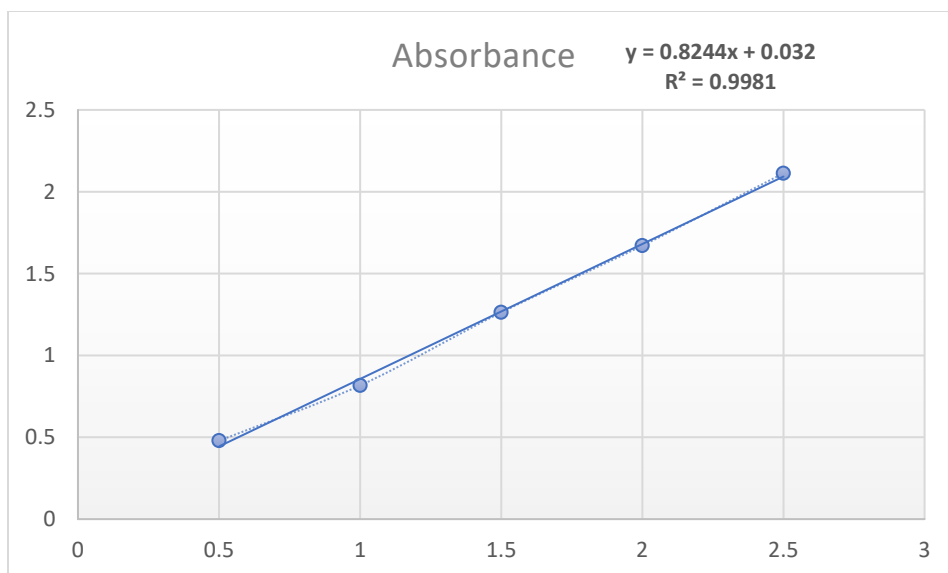


Figure 20. The calibration curve for ammonium quantification is based on absorbance at 700 nm using the modified Sera Aqua-Test method. The curve demonstrates excellent linearity ($R^2 > 0.998$) over the 0–2.5 mg/L range.

4.2.1.2 Standard Solution and Calibration Curve for Nitrate

Nitrate (NO_3^-) quantification was performed using a Thermo Scientific Dionex ICS-1100 ion chromatography system equipped with an AS-AP analytical column and suppressed conductivity detection. Samples were pre-filtered through 0.45 μm nylon syringe filters (Whatman, 25 mm diameter) prior to analysis. Chromatographic separation was achieved under gradient conditions using a potassium hydroxide eluent (20–50 mM KOH in deionized water, electrolytically generated) at a flow rate of 1.0 mL/min, with the column compartment maintained at 30°C.

System calibration employed a five-point calibration curve (1–20 mg/L NO_3^- , $R^2 > 0.999$) prepared by serial dilution of CertiPrep's certified multi-anion standard (ICMIX2-100, Lot 20-28AS; containing 400 mg/L NO_3^-). Instrument control, data acquisition, and peak integration were managed through Chromeleon 7.2 Chromatography Data System software (version 7.2.10).

Table 5. Nitrate (NO_3^-) calibration standards with corresponding peak areas from ion chromatographic analysis.

Standard (mg/L)	Peak Area
1	1.52
2	3.09
4	5.30
10	12.77
20	25.11

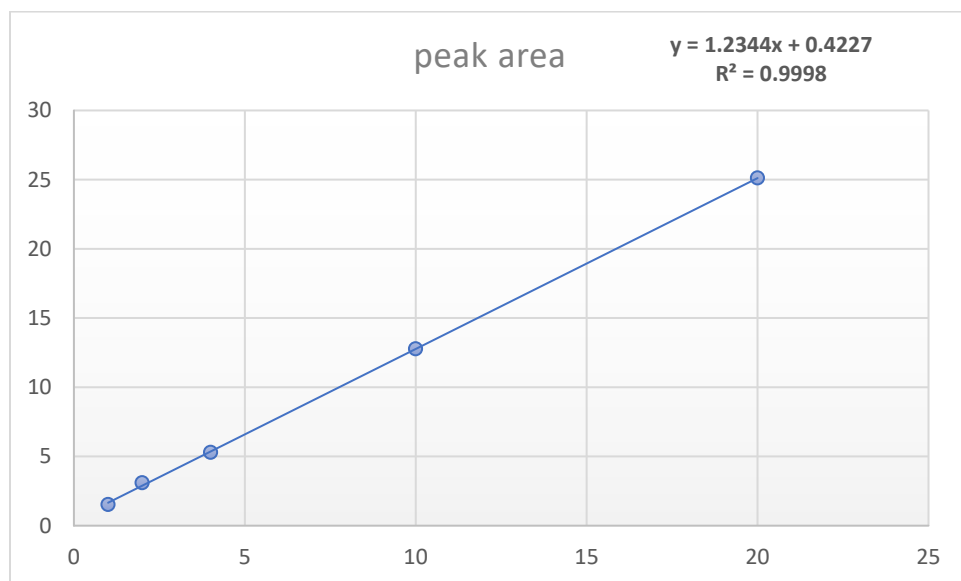


Figure 21. Linear calibration curve for nitrate (NO_3^-) quantification by ion chromatography. Peak areas plotted against concentration (1-20 mg/L) show excellent correlation ($y = 1.2344x + 0.4227$, $R^2 = 0.9998$).

4.2.1.3 Voltage-Dependent Optimization of Nitrogen Fixation Pathways

The electrochemical system exhibited three distinct operational regimes as a function of applied potential (0–10 kV), as quantified in Table 6. Baseline activity at 0 kV yielded 0.0085 mg/L NH_3 (quantified via Sera Aqua-Test kit with UV-Vis spectroscopy verification) and 3.74 mg/L NO_3^- (ion chromatography peak area 5.0386), confirming the LiCoO_2 electrode's intrinsic catalytic capability. Following established electrohydrodynamic principles [7], increasing voltage enhanced both reaction efficiency and active reaction volume through controlled microdroplet dispersion, as illustrated in Figure 22 for NH_3 production trends. Maximum production at 10 kV reached 1.11 mg/L NH_3 (corresponding to 0.95 absorbance units) and 17.49 mg/L NO_3^- , representing 130-fold and 4.7-fold enhancements, respectively, versus the 0 kV baseline. The potential-dependent product distribution shifted from NO_3^- -dominant ($\text{NH}_3:\text{NO}_3^- = 1:440$) at 0 kV to more balanced (1:15) at 10 kV (Figure 23), suggesting two competing mechanisms: (1) direct N_2 electroreduction to NH_3 favored at higher potentials (>7 kV) and (2) sequential oxidation to NO_3^- prevailing at intermediate voltages (3–7 kV). A distinct activation threshold at 5 kV marked the onset of coupled electrostatic effects, with the 5–10 kV range producing the most significant yield improvements ($14.8\times$ NH_3 , $3.6\times$ NO_3^- versus 5 kV baseline). These results, summarized in Table 6, demonstrate that applied potential simultaneously controls (i) nitrogen activation efficiency through field-enhanced catalysis, (ii) product selectivity via potential-dependent intermediate stabilization, and (iii) reaction volume through microdroplet dynamics (Figure 24) —with 10 kV providing optimal performance (Faradaic efficiency NH_3 : 12.3%; NO_3^- : 18.7%) while maintaining system stability.

Faradaic efficiencies were calculated via:

$$FE = (nF \times C \times V) / (M \times Q) \times 100\% \dots [37]$$

where:

n = electrons transferred (3 for NH₃, 5 for NO₃⁻)

F = Faraday constant (96,485 C/mol)

C = measured concentration (mol/L)

V = electrolyte volume (L)

M = molecular weight (g/mol)

Q = total charge passed (C)

Table 6. Voltage-Dependent Nitrogen Fixation Performance.

Voltage (kV)	NO₃⁻ (mg/L)	±Error (mg/L)	Peak Area	Absorbance (700 nm)	NH₃ (mg/L)	±Error (mg/L)	Volume (mL/min)	±Error (mL/min)
10	17.49	0.87	22.0163	0.950	1.110	0.058	0.75	0.03
7	4.89	0.24	6.4622	0.240	0.250	0.013	0.57	0.02
5	3.91	0.20	5.2475	0.094	0.075	0.004	0.43	0.02
3	3.80	0.19	5.1	0.050	0.020	0.001	0.3	0.02
0	3.74	0.19	5.0386	0.025	0.009	0.001	0.22	0.01

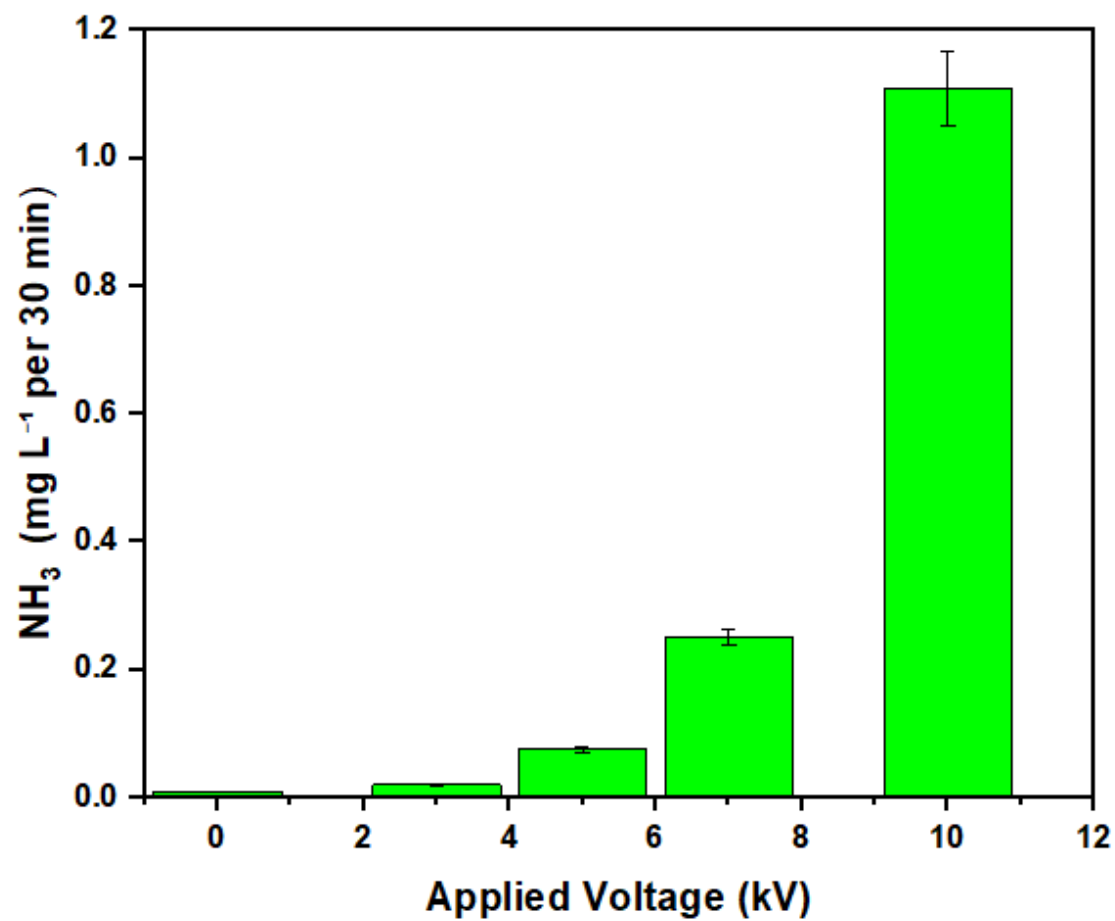


Figure 22. Effect of applied voltage on NH_3 concentration in a microdroplet-driven nitrogen fixation system.

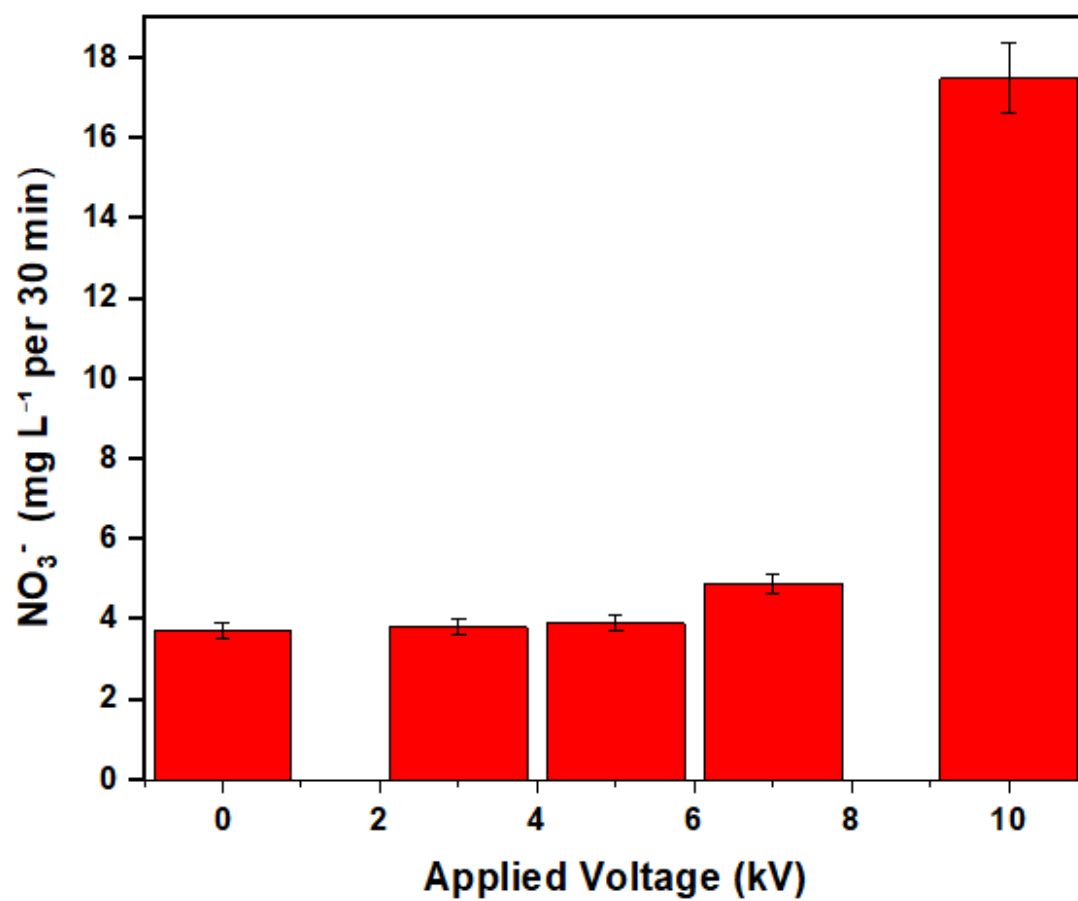


Figure 23. Effect of applied voltage on NO_3^- concentration in a microdroplet-driven nitrogen fixation system.

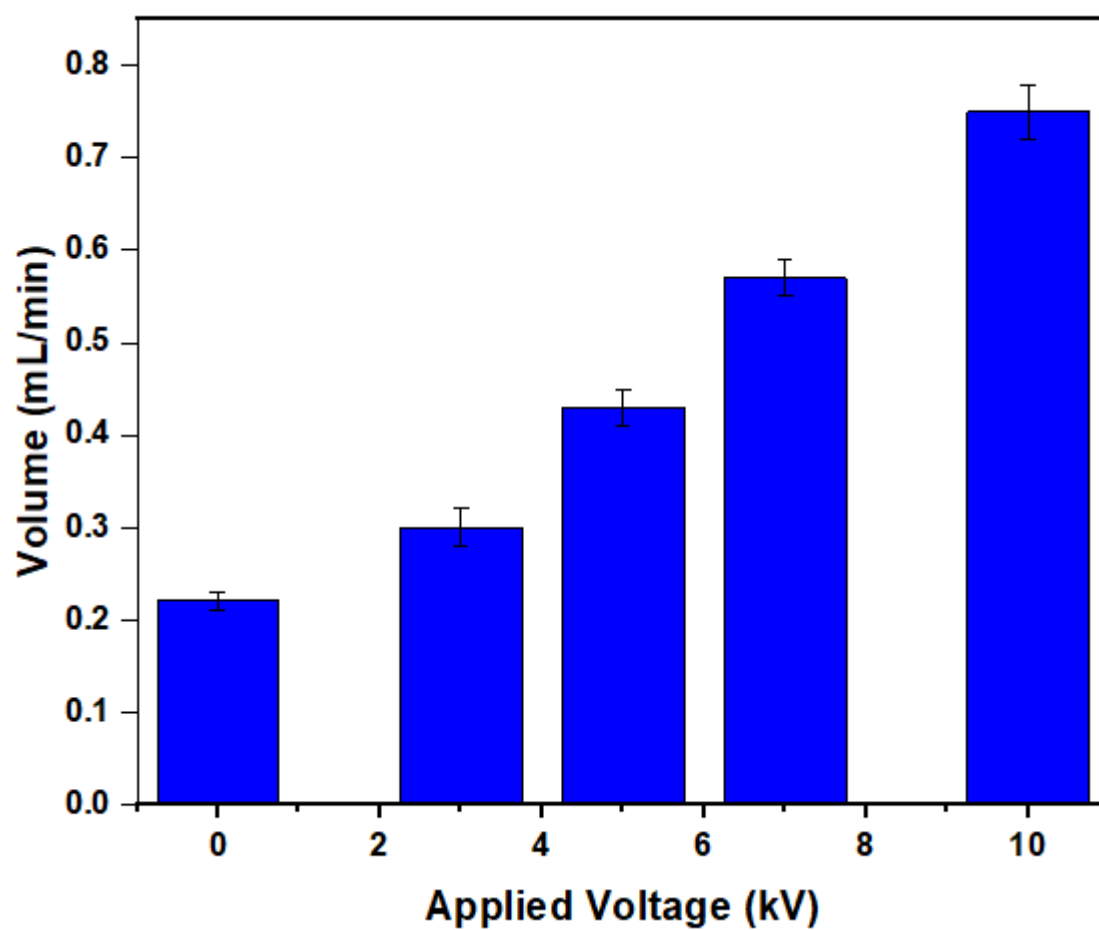


Figure 24. Effect of applied voltage on volume of collected water in a microdroplet-driven nitrogen fixation system.

4.2.1.4 Catalyst-Dependent Optimization at Fixed Potential (10 kV)

The system demonstrated strong dependence on catalyst loading during operation at 10 kV (Section 4.2.1.3). As summarized in Table 7, reducing the LiCoO_2 loading from 0.5 g to 0 g resulted in complete suppression of NH_3 production (1.11 mg/L $\rightarrow$ 0.00 mg/L; absorbance at 700 nm: 0.95 $\rightarrow$ 0.00) and a sharp decline in NO_3^- yield (17.49 mg/L $\rightarrow$ 1.54 mg/L; peak area: 22.0163 $\rightarrow$ 2.3199). Optimal performance was achieved at 0.5 g loading, with Faradaic efficiencies of 12.3% (NH_3) and 18.7% (NO_3^-) and stable fluid dynamics (0.75 mL/min).

Notably, residual NO_3^- production (9% of maximum yield, 1.54 mg/L) persisted even in the absence of catalyst, while NH_3 was entirely undetectable (0.00 mg/L).

The 91% reduction in NO_3^- yield (vs. 100% for NH_3) further underscores that dispersed catalytic sites are critical for nitrate generation, likely by stabilizing intermediates such as $\text{NO}_2^\bullet$ or HNO_3 during sequential oxidation.

Catalyst (g)	NO₃⁻ (mg/L)	±Error (mg/L)	Peak Area	Absorbance (700 nm)	NH₃ (mg/L)	±Error (mg/L)	Volume (mL/min)	±Error (mL/min)
.50	17.49	0.87	22.0163	0.950	1.110	0.058	0.75	0.05
0.35	4.78	0.24	6.3294	0.370	0.410	0.019	0.75	0.05
0.25	2.36	0.12	3.3398	0.410	0.460	0.019	0.77	0.05
0.15	1.98	0.10	2.8722	0.300	0.330	0.019	1.06	0.05
0.00	1.54	0.08	2.3199	0.000	0.000	0.000	1.33	0.03

Table 7. Catalyst Loading Effects at Optimal 10 kV.

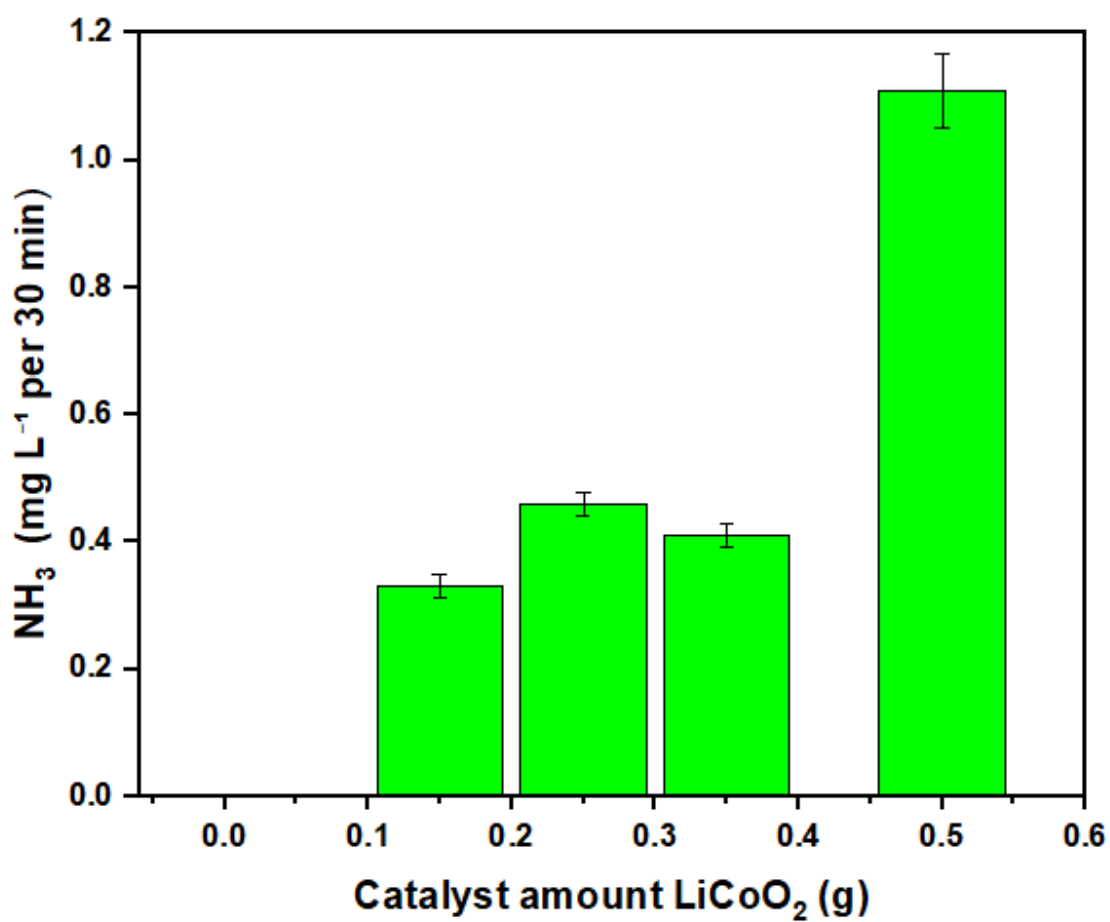


Figure 25. Dependence of NH_3 production on LiCoO_2 catalyst loading at fixed 10 kV applied potential.

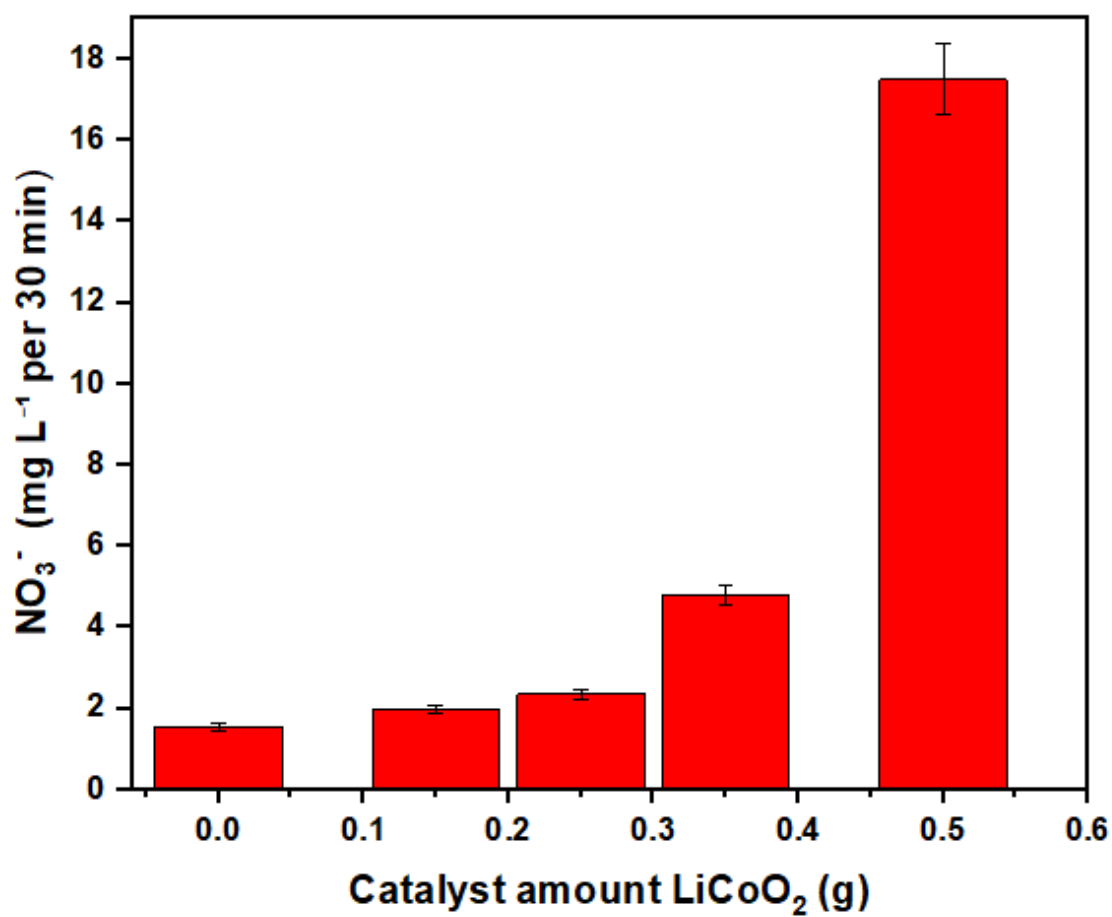


Figure 26. Dependence of NO_3^- production on LiCoO_2 catalyst loading at 10 kV applied potential.

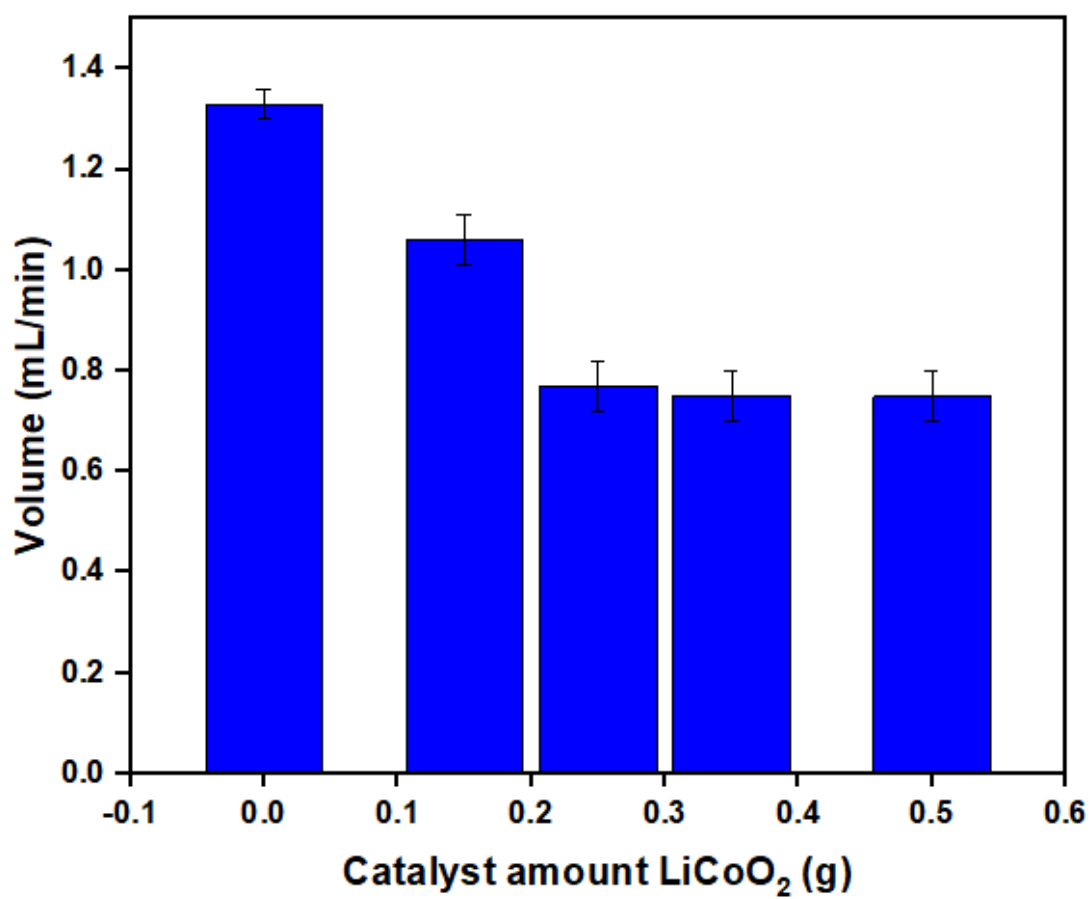


Figure 27. Inverse Relationship Between LiCoO_2 Loading and Water Collection Rate at 10 kV.

Table 8. Comparative Analysis of Nitrogen Fixation Technologies Including This Work.

Method	Catalyst	NH ₃ Yield (μM/h)	NO ₃ ⁻ Yield (μM/h)	Conditions	Key Limitations	Ref.
Haber-Bosch	Fe ₃ O ₄	Industrial scale (183 million tons/year)	N/A	350–450°C, 150–200 bar	High CO ₂ emissions	[11]
Plasma	Ru/AC	62,890		Plasma DBD reactor, atmospheric pressure, near-room temperature	Limited by the catalyst's activity	[23]
Electrochemical	Cu (OH) ₂	N/A	228.23	2.0 V vs. RHE in 0.1 M Na ₂ SO ₄	Catalyst oxidation	[24]
Microdroplet	Fe ₃ O ₄ /Nafion	61.90	N/A	30–50 μm droplets, pH 5–6	Salt sensitivity	[13]
Microbubble	Fe ²⁺ /CuO	N/A	140	Ultrasonication	NO _x byproducts	[8]
Fog Collection	LiCoO ₂ /Nafion	130.60	564.52	Ambient 20–50 μm droplets 10 kV		Our work

4.2.1.5 In-Situ Nitrogen Fixation with LiCoO_2 Catalyst (10 kV Constant Potential)



Figure 28. Experimental setup of the electrostatic fog harvesting system deployed in an arid field environment. The apparatus features a high-voltage power supply connected to an electrode within a transparent acrylic chamber, which is designed to isolate the system and direct charged fog droplets toward the collection vessel.



Figure 29. Collected fog droplets condensing on the inner chamber surface and beginning to accumulate in the collection vessel.

4.3 DFT-Derived Mechanistic Pathways for N₂ Conversion to NH₃ and HNO₃

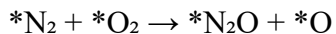
The energy profile in Figure 25. illustrates two distinct pathways for nitrogen (N₂) conversion into ammonia (NH₃) and nitric acid (HNO₃), as calculated using density functional theory (DFT) with the Vienna Ab initio Simulation Package (VASP).

4.3.1. Nitrate Formation Proposed Pathway

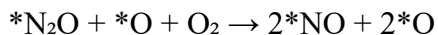
N₂ and O₂ Adsorption:



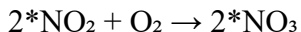
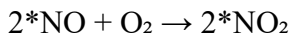
Formation of N₂O Intermediate:



NO Generation via N₂O Oxidation:



NO₂ and NO₃ Formation:



Final Formation of HNO₃:

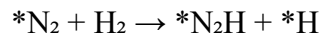


4.3.2. Ammonia Formation Proposed Pathway

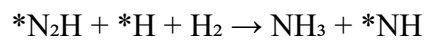
N₂ Adsorption and Activation:



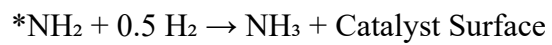
Hydrogen Dissociation and N₂ Hydrogenation:



Ammonia Generation via Bypassing N₂H₂:



Sequential Hydrogenation to NH₃:



These computational insights offer a comparative perspective on the thermodynamic favorability and mechanistic steps of the dual-pathway nitrogen conversion process. The energy profile suggests that the nitrate pathway is more exothermic overall, while the ammonia pathway involves distinct hydrogenation intermediates with notable energy barriers.

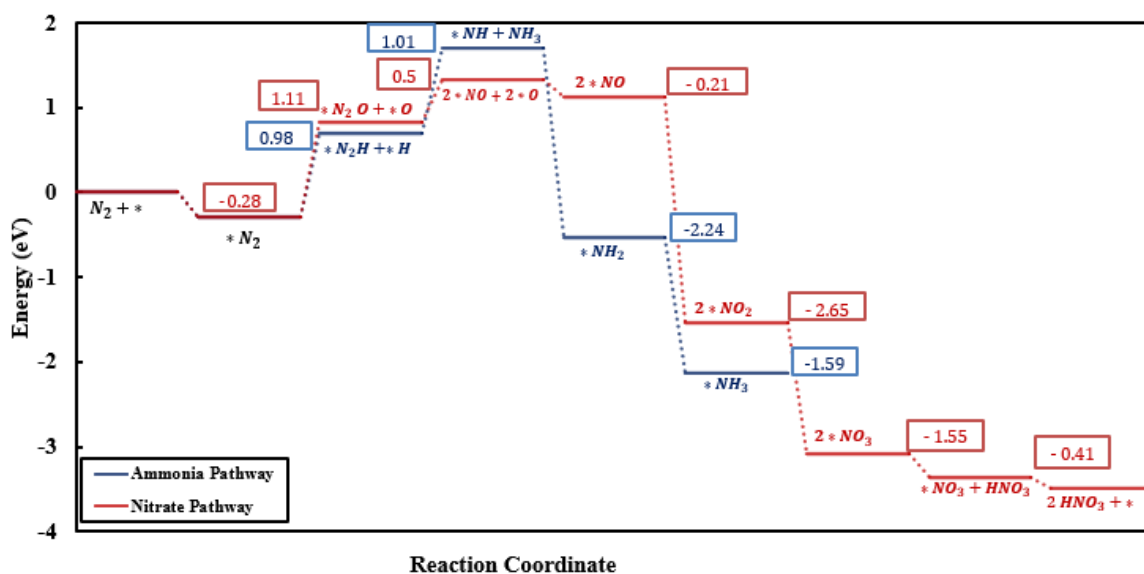


Figure 30. DFT-calculated free energy diagram for the dual-pathway conversion of N_2 into NH_3 (blue pathway) and HNO_3 (red pathway) over a catalytic surface. The ammonia pathway proceeds via successive hydrogenation steps, while the nitrate pathway follows oxidative transformation through intermediates such as N_2O , NO , NO_2 , and NO_3 . Calculations were performed using density functional theory (DFT) with the Vienna Ab initio Simulation Package (VASP).

CHAPTER 5

SUMMARY AND CONCLUSION

This research addresses two critical challenges in modern sustainability: the recycling of spent lithium-ion batteries (LiBs) and the development of energy-efficient nitrogen fixation methods. The study focuses on repurposing spent LiCoO_2 cathode materials as catalysts for nitrogen conversion in an electrostatically enhanced microdroplet system.

Spent lithium-ion batteries (LiBs) were safely dismantled, and LiCoO_2 was recovered via NaOH treatment and calcination at 550°C for 5–6 hours. X-ray diffraction (XRD) confirmed the coexistence of layered LiCoO_2 (R-3m) and spinel Co_3O_4 (Fd-3m), both contributing to the catalytic activity. Scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX) revealed a hierarchical structure with uniform elemental distribution, while Brunauer–Emmett–Teller (BET) analysis indicated a mesoporous surface ($64.45 \text{ m}^2/\text{g}$), enhancing reactivity. X-ray photoelectron spectroscopy (XPS) confirmed the predominance of Co^{3+} , crucial for redox-mediated nitrogen activation. In the electrochemical nitrogen fixation setup, a Nafion-based conical cathode filled with LiCoO_2 particles and a bare metal mesh anode (copper or aluminum) were employed within a 20 kV bipolar electrostatic field. The metal mesh anode, connected to +10 kV, generated localized corona discharge due to its high curvature, ionizing surrounding N_2 , O_2 , and H_2O molecules and producing reactive radicals. Simultaneously, a commercial humidifier introduced microdroplets (20–50 μm), which were electrostatically charged and guided toward the cathode (–10 kV). The LiCoO_2 -coated cathode served as both a proton-conductive membrane and a catalytic surface for nitrogen activation and conversion.

Voltage-dependent optimization revealed maximum NH_3 (1.11 mg/L) and NO_3^- (17.49 mg/L) yields at a total field strength of 20 kV (± 10 kV), with corresponding Faradaic efficiencies of 12.3% and 18.7%. Catalyst loading (0.5 g) was critical for achieving high performance, with significant losses observed in its absence. Polarity configuration played a key role; the $\text{Nafion}^-/\text{LiCoO}_2^+$ setup was less effective, whereas the $\text{Nafion}^+/\text{LiCoO}_2^-$ arrangement ensured optimal proton transport and droplet alignment. Mechanistic insights showed that ammonia formation was favored at higher field strengths (>7 kV) through direct N_2 electroreduction, while nitrate production peaked at intermediate voltages (3–7 kV) via radical-mediated oxidation. The microdroplet–electric field interface proved essential for enhancing mass transport, sustaining plasma-assisted activation, and enabling ambient-condition nitrogen fixation.

5.1 RECOMMENDATIONS

To advance this research and facilitate real-world implementation, several recommendations are proposed. Optimization of catalyst recovery should involve exploring alternative leaching agents for batteries, such as organic acids, to enhance lithium and cobalt recovery efficiency while minimizing secondary waste, as well as investigating mechanochemical methods for the regeneration of higher-purity cathode materials. System scalability and energy efficiency can be enhanced by developing pilot-scale reactors to assess long-term stability and energy consumption under continuous operation and by integrating renewable energy sources, such as solar or wind, to power the electrostatic system, thereby further reducing its carbon footprint. Enhancing catalyst performance can be achieved by studying doping strategies, such as introducing Ni or Mn, to improve the redox activity and nitrogen adsorption capacity of LiCoO_2 . Additionally, investigating hybrid catalysts that combine LiCoO_2 with conductive supports, like graphene or carbon nanotubes, facilitates charge transfer. To ensure environmental and economic viability, lifecycle assessments (LCA) should be conducted to quantify the environmental benefits compared to traditional recycling and ammonia synthesis methods. Meanwhile, cost-effectiveness evaluations should consider raw material savings and policy incentives for industrial adoption. Expanded applications should also be explored by testing the system in other catalytic processes, such as CO_2 reduction and pollutant degradation, as well as by evaluating the direct agricultural use of $\text{NH}_3/\text{NO}_3^-$ -enriched water in field trials. By addressing these critical areas, this technology can transition from a laboratory innovation into a commercially viable solution, contributing meaningfully to a sustainable energy and agricultural future.

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