Materials Advances

Accepted Manuscript

This article can be cited before page numbers have been issued, to do this please use: P. Hlophe, M. Tawalbeh, A. Al-Othman and P. F. Msomi, *Mater. Adv.*, 2026, DOI: 10.1039/D6MA00429F.

ROYAL SOCIETY OF CHEMISTRY

rsc.li/materials-advances

View Article Online
DOI: 10.1039/D6MA00429F

Autonomous AI Agents in ORR Electrocatalyst Discovery: A Review of Closed-Loop Workflows, Materials Bottlenecks, and Energy System Translation

Philani V. Hlophe[a], Muhammad Tawalbeh[b,c], Amani Al-Othman[d,e], Phumlani F. Msomi[a*]

[a]Institute for Catalysis and Energy Solutions (ICES), College of Science Engineering and Technology, University of South Africa, South Africa

[b] Sustainable and Renewable Energy Engineering Department, University of Sharjah, PO. Box 27272, Sharjah, United Arab Emirates

[c]Sustainable Energy & Power Systems Research Centre, RISE, University of Sharjah, P.O. Box 27272, Sharjah, United Arab Emirates

[d]Department of Chemical and Biological Engineering, American University of Sharjah, PO. Box 26666, Sharjah, United Arab Emirates

[e]Energy, Water and Sustainable Environment Research Centre, American University of Sharjah, PO. Box 26666, Sharjah, United Arab Emirates

[*]Corresponding author: msomipf@unisa.ac.za

Abstract

The commercialization of fuel cells and metal-air batteries is hindered by the slow kinetics of the oxygen reduction reaction (ORR) at the cathode and the high cost of the platinum-supported carbon (Pt/C) catalyst. Although Pt/C catalysts remain the benchmark, their reliance on scarce, expensive platinum and limited durability under acidic conditions underscores the urgent need for alternative materials. Promising candidates, including 2D materials (e.g., MXenes and graphene derivatives), M-N-C single-atom catalysts, and high-entropy alloys, occupy vast and complex compositional spaces, making traditional trial-and-error approaches too slow to fully explore them. While machine learning has accelerated the identification of potential catalysts, challenges, such as long-term stability, practical synthesizability, and experimental validation, remain unresolved. In this Perspective, we highlight the

emerging role of Autonomous AI Agents in ORR catalyst discovery. By integrating large language models, structured knowledge graphs, and robotic high-throughput experimentation, these systems can autonomously design, execute, analyze, and refine experiments with minimal human input. We emphasize that the success of such closed-loop systems depends on moving beyond simple automation toward high-fidelity discovery. This requires integrating physics-informed machine learning (PIML) frameworks with robust surrogate models that use uncertainty quantification and physically meaningful representations to avoid extrapolation errors.

This review examines key challenges to realizing this vision, including the need for standardized operando datasets, minimum reporting checklists to ensure reproducibility, explainable AI frameworks that provide chemical insight, and robust strategies for synthesizing complex materials such as 2D sheets. Finally, we propose a roadmap for developing and implementing these Autonomous AI Agents, aiming to accelerate the discovery of the next generation of ORR electrocatalysts through a synergy of rapid execution and deep theoretical rigor.

Keywords: Autonomous AI Agents; Oxygen Reduction Reaction; Electrocatalysis; Self-Driving Laboratories; Hydrogen Energy

1. Introduction: The Grand Challenge of ORR and the Limits of Current ML

1.1. The ORR Bottleneck: Beyond

The transition to a global hydrogen economy critically depends on high-efficiency electrochemical energy devices, including proton exchange membrane fuel cells (PEMFCs) and metal–air batteries. In these systems, the oxygen reduction reaction (ORR) at the cathode remains the primary performance-limiting step due to its sluggish kinetics [1-2]. Platinum-on-carbon (Pt/C) catalysts are the commercial benchmark thanks to their high intrinsic activity; however, their widespread deployment is constrained by three interrelated challenges: high cost, limited availability, and insufficient durability [3-4]. Platinum alone accounts for roughly half the cost of a PEMFC stack, highlighting the urgent need for cost-effective alternatives. Moreover, Pt/C catalysts undergo degradation pathways, such as Ostwald ripening (particle aggregation) and carbon corrosion during prolonged operation, which limit their long-

View Article Online
DOI: 10.1039/D6MA00429F

term performance [3]. Together, these issues underscore the necessity of moving "beyond Pt/C" toward electrocatalysts that can combine high activity, stability, and affordability.

1.2. The Material Frontier: Non-Precious Catalysis

The most promising strategies for replacing Pt involve the exploration of non-precious metal (NPM) and low-platinum-group-low-PGM) electrocatalysts. Current research focuses on materials that maximize atomic utilization and tailor active site environments [2, 5]. Figure 1 illustrates the material frontier for non-precious ORR electrocatalysts, highlighting single-atom M-N-C catalysts, high-entropy alloys, and Pt-alloy systems. Beyond these, emerging 2D materials such as MXenes and functionalized graphene derivatives offer unique properties due to their high surface-to-volume ratios and tunable electronic properties. Autonomous AI agents are increasingly being used to navigate the complex exfoliation and surface-functionalization parameters of these 2D systems to optimize their ORR activity.

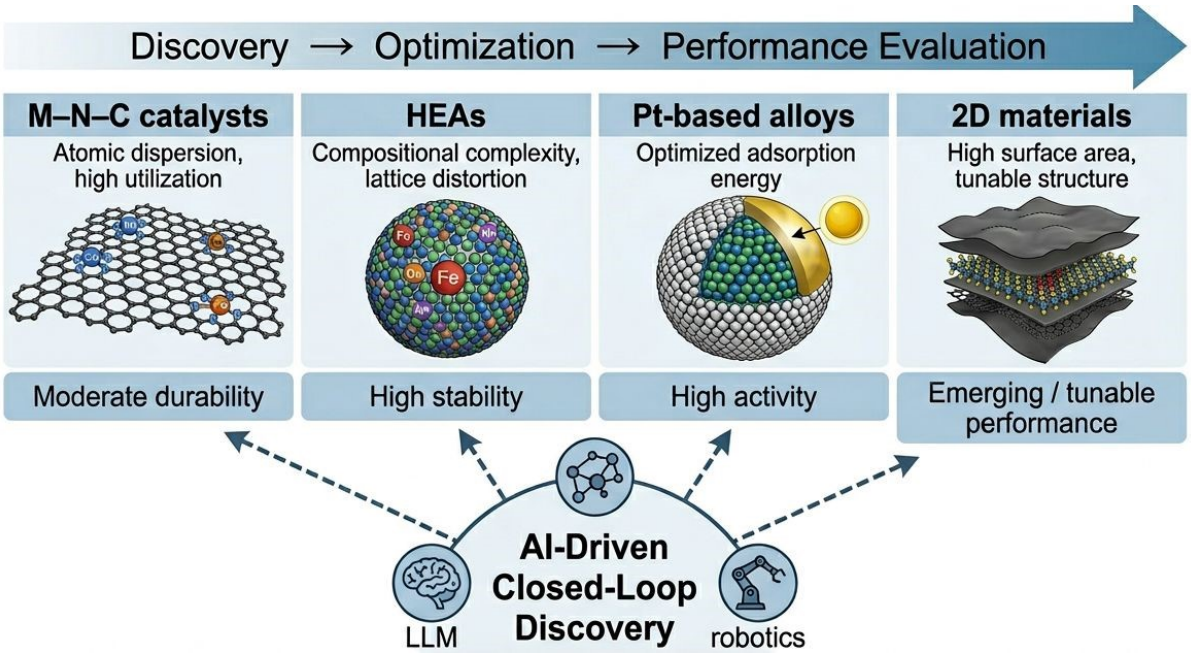


Figure 1. Schematic representative classes of ORR catalysts, including M–N–C single-atom catalysts, high-entropy alloys, Pt-based alloys, and two-

View Article Online
DOI: 10.1039/D6MA00429F

dimensional (2D) materials. The diversity and increasing complexity of these materials underscore the need for advanced AI-driven discovery strategies.

- **Single-Atom Catalysts:** Atomically dispersed metal-nitrogen-carbon (M-N-C), particularly Fe-N-C materials, have emerged as the leading PGM-free alternative. They feature well-defined sites and offer high atomic efficiency and intrinsic activity comparable to M-N_4. However, a critical bottleneck remains their long-term durability in acidic environments, which often leads to metal demetallation and structural degradation [1,6].
- **Alloys and High-Entropy Systems:** Binary, ternary, and quaternary Pt-alloy catalysts (e.g., PtCo, PtNi) and High-Entropy Alloy (HEA) electrocatalysts are being investigated to tune the electronic structure and stability of the active surface sites [6,7]. The challenge here is the vast and complex compositional space, which makes their optimal design and synthesis control highly intractable via traditional methods.
- **Two-Dimensional (2D) Materials:** Two-dimensional materials, including graphene derivatives, MXenes, and transition metal dichalcogenides (TMDs), have emerged as promising platforms for ORR electrocatalysis due to their high surface area, tunable electronic structure, and capacity for heteroatom doping and defect engineering. These materials offer flexible pathways to tailor active sites and optimize adsorption energetics. However, their intrinsic ORR activity often remains lower than that of Pt-based systems, and their performance is highly dependent on surface functionalization, hybridization, and structural modification. In addition, challenges related to long-term stability, restacking, and scalable synthesis limit their practical deployment, highlighting the need for advanced design strategies and controlled fabrication approaches [5,7].

1.3. The Shortcomings of the Human-in-the-Loop Status Quo

Traditional catalyst discovery follows a slow, sequential cycle: Design → Synthesis → Test → Analyze. The recent integration of machine learning (ML) with high-throughput screening (HTS) has accelerated the prediction step, enabling rapid filtering of promising candidates from large computational databases, such as density functional

View Article Online
DOI: 10.1039/D6MA00429F

theory (DFT)-based datasets [8]. Despite these advances, current approaches remain constrained by a human-in-the-loop bottleneck:

- **Limited Scope:** ML models excel at predicting catalyst activity (e.g., binding energies) but often fail to capture critical challenges, such as long-term stability and complex synthesis optimization [1].
- **Data Quality:** The accuracy of predictions is limited by heterogeneous, low-quality, and non-standardized experimental datasets, creating a persistent "data bottleneck" [1].
- **Lack of Agency:** Even with high-fidelity predictions, the iterative synthesis and testing steps remain slow, prone to human error, and constrained by the throughput limits of human intuition [8].

These limitations highlight the need for approaches that go beyond conventional human-guided workflows, paving the way for autonomous systems capable of accelerating catalyst discovery at scale.

1.4. Introducing the Paradigm Shift: Autonomous Discovery

To overcome the limitations of current approaches and meet the stability and activity targets required for commercial viability (e.g., U.S. DOE 2025 mass activity and durability goals), the field must move beyond human-guided, data-driven predictions toward fully autonomous, closed-loop discovery. The idea of this Perspective is that Autonomous AI Agents-intelligent robotic laboratories integrated with advanced reasoning AI, including large language models (LLMs) and structured Knowledge Graphs (KGs)-represent the next transformative step in electrocatalyst development [9-10].

Critically, this shift is not merely about increasing experimental throughput but about enhancing model fidelity. By integrating physics-informed machine learning (PIML) and intrinsic uncertainty quantification, these agents can navigate vast chemical spaces with first-principles accuracy. By prioritizing structural descriptors such as coordination environments and electronic d-band centers over mere statistical correlations, Autonomous AI Agents transform the discovery loop (Predict → Make → Measure → Learn) from a high-throughput automation task into a high-fidelity scientific engine capable of solving the long-standing challenges of catalyst activity and durability.

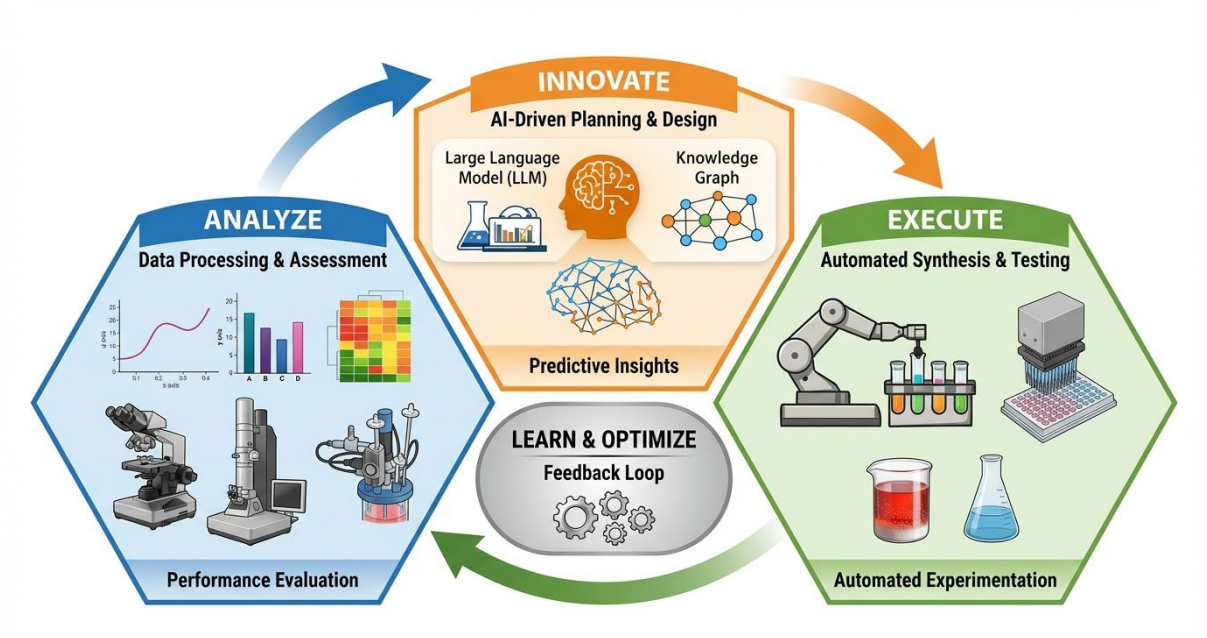
View Article Online
DOI: 10.1039/D6MA00429F

2. The Mechanics of the Autonomous AI Agent

The shift from conventional human-in-the-loop ML to fully autonomous discovery is enabled by Closed-Loop Self-Driving Laboratories (SDLs), orchestrated by an Autonomous Agent. Unlike a traditional program executing optimization algorithms, the agent is an intelligent, embodied system capable of reasoning, testing hypotheses, and self-correction [8]. By integrating experimental execution, data analysis, and iterative learning, such agents can navigate complex chemical spaces far beyond the reach of manual workflows.

2.1. The Closed-Loop Architecture:

The efficiency of an Autonomous Agent depends on an intelligent feedback system that explicitly closes the Predict → Make → Measure → Learn loop. This loop can be understood as a continuous Analyze → Innovate → Execute (A–I–E) cycle, as per Figure 2. In Figure 2, the Analyze stage ingests experimental and computational data; the Innovate stage generates hypotheses via LLM reasoning and Knowledge Graph (KG) contextualization; and the Execute stage implements these via robotic high-throughput synthesis and testing.



View Article Online
DOI: 10.1039/D6MA00429F

Figure 2: Closed-Loop Architecture. Autonomous agent workflow integrating Analyze (data/ML insights), Innovate (LLM + KG), and Execute (robotic synthesis and testing). Arrows indicate iterative learning.

- **Analyze (Learn/Predict):** The agent ingests experimental results, such as ORR performance and stability data, as well as characterization data (e.g., XPS, XAS) from the robotic platform. This information is integrated into its internal knowledge structure, allowing the agent to learn structure–property relationships and predict the most promising next experiment.
- **Innovate (Design):** Leveraging its reasoning core, the agent generates chemically valid hypotheses. For example: *"Synthesize Fe–Co–N–C with a 1:2 Fe:Co ratio annealed at 850 ºC to maximize Fe–N_4 site stability."*
- **Execute (Make/Measure):** The agent translates these hypotheses into precise, codified instructions, such as robotic arm movements, pump flow rates, and temperature protocols, which are directly executed by the automated hardware [10].

This closed-loop approach dramatically accelerates catalyst discovery, enabling the system to perform hundreds of iterative experiments in the time it would take human researchers' years to complete [8].

2.1.1 Physics-Informed ML Frameworks and Uncertainty Aware Discovery

The integration of Autonomous AI Agents into ORR discovery necessitates a shift from a simple workflow automation to the deployment of physics-informed machine learning (PIML) frameworks. A fundamental challenge in autonomous discovery is the model fidelity vs. computational efficiency trade-off; while Density Functional Theory (DFT) provides quantum-level accuracy, its computational cost is prohibitive for screening vast chemical spaces.

To bridge this gap, modern agents utilize surrogate models that incorporate physical constraints directly into the learning architecture. Graph-based structural learning and equivariant neural networks allow the agent to represent complex catalyst surfaces such as the strained lattices of high-entropy alloys or the defective planes of MXenes, while maintaining rotational and translational invariance [54].

View Article Online
DOI: 10.1039/D6MA00429F

Furthermore, high-throughput discovery is not merely a matter of volume but of navigating uncertainty. Advanced frameworks now integrate Sparse Gaussian Process Regression (SGPR) coupled with committee-machine scaling (e.g., Robust Bayesian Committee Machine (RBCM)) [55]. These methods enable unified, scalable, and uncertainty-aware materials discovery by providing intrinsic uncertainty quantification (UQ). In a closed-loop system, UQ serves two critical functions:

1. Active Learning Guidance: It identifies regions of the chemical space where the model's confidence is low, directing the robotic hardware to perform experiments that maximize information gain.
2. Risk Mitigation: It prevents the agent from falling into "extrapolation traps", where the model might predict high performance in chemically incoherent regions of the configuration space.

By prioritizing models that retain near-DFT accuracy while enabling large-scale screening, the Autonomous AI Agent transforms from a high-throughput automation tool into a high-fidelity discovery engine capable of identifying non-intuitive catalyst architectures [55].

2.2. The Reasoning Core: Large Language Models and Knowledge Graphs

The agent's ability to achieve true autonomy, moving beyond simple Bayesian optimization to chemically informed decision-making, resides in its reasoning core.

A. Large Language Models for Chemical Intuition

Recent advances have shown that LLMs are powerful tools for high-level, human-interpretable reasoning and planning [9]. Acting as the agent's "chemical intuition," the LLM performs tasks that traditional algorithms cannot:

- **Hypothesis Generation:** Translating abstract goals, such as "*Improve Fe–N_4 stability by 10%"*, into concrete, literature-backed synthetic strategies.
- **Protocol Codification:** Producing and debugging the software code required to operate complex laboratory instruments, removing manual programming bottlenecks [9].
- **Literature Mining:** Integrating unstructured textual knowledge, including papers, patents, and lab notebooks, directly into the decision-making process, a capability inaccessible to purely numerical ML models [8].

View Article Online
DOI: 10.1039/D6MA00429F

B. Structured Knowledge Graph

To ensure the LLM's chemical intuition is accurate and grounded, it is paired with a structured KG. Serving as the single source of truth, the KG aggregates heterogeneous data, DFT calculations, synthesis protocols, characterization spectra, and electrochemical performance metrics into a machine-readable, relational format.

- **The KG plays a critical role by:**

Contextualizing Decisions: Enabling the agent to search for relationships across different data types, e.g., *"Which synthesis conditions minimize Fe leaching for a given M–N–C structure?"*

Preventing Redundancy: Systematically recording all prior experiments and outcomes, preventing the agent from revisiting previously explored dead ends [8].

2.3. The Action Layer: Robotics and High-Throughput Screening Integration

The physical implementation of the agent's plans relies on robust, integrated hardware, commonly referred to as an SDL or robotic platform.

- **Modular Synthesis:** The platform must support flexible, modular synthesis methods, such as hydrothermal, pyrolysis, or solution-phase techniques, allowing rapid preparation of materials, including M–N–C precursors [10]. To enhance reproducibility and modularity, these systems rely on specific software frameworks; communication between the AI reasoning core and the hardware is typically facilitated through Python-based APIs or the Robot Operating System (ROS), ensuring seamless execution of synthesis protocols.
- **Real-Time Electrocatalysis:** High-throughput testing is conducted using automated systems or micro-fuel cell setups, which feed performance and durability data directly back to the reasoning core in real time. This immediate feedback is essential for enabling the iterative learning cycle.

View Article Online
DOI: 10.1039/D6MA00429F

By coupling LLM-driven reasoning with robotic execution via a structured KG, the Autonomous AI Agent transforms electrocatalyst discovery from a slow, human-mediated workflow into a rapid, self-optimizing, closed-loop process.

3. Agent-Driven Solutions to Bottlenecks

The true value of an Autonomous AI Agent lies not only in speed but in its ability to simultaneously optimize parameters that remain intractable for human researchers, such as durability and precise synthesis control [10]. By closing the loop between prediction, synthesis, measurement, and learning, these systems are uniquely positioned to overcome the most critical limitations of non-precious metal catalysts, particularly the leading M–N–C materials [11]. Table 1 summarizes the major bottlenecks in ORR catalyst development and the corresponding AI-driven strategies enabled by autonomous discovery platforms.

Table 1. Autonomous AI-driven strategies for overcoming key bottlenecks in ORR catalyst discovery.

Bottleneck in Catalyst Development	Autonomous AI-Agent Strategy	Key Technologies	Impact on Catalyst Discovery	Ref
Catalyst durability and degradation in acidic PEMFC environments	AI-guided accelerated stress test (AST) design that dynamically tunes degradation conditions based on knowledge graph learning	Machine learning models, adaptive AST protocols, degradation monitoring	Enables targeted identification of degradation mechanisms and accelerates durability evaluation from long-term testing to shorter predictive experiments	11, 13, 14
Limited ability to predict long-term catalyst stability	Identification of predictive durability descriptors through real-time monitoring of degradation markers such as CO_2 or H_2O_2 production	Real-time gas analysis, machine learning models, predictive stability descriptors	Early detection of unstable catalysts allows rapid elimination of poor candidates and focuses testing resources on stable materials	11, 15, 16
Complex synthesis parameter space for M–N–C catalysts	Closed-loop synthesis optimization (CSO) linking experimental feedback to machine learning models that update synthesis parameters	Robotics-driven synthesis, ML-guided optimization, automated experimentation	Rapid convergence toward optimal catalyst compositions and synthesis conditions	17, 18, 19
Difficulty in identifying active-site structures	Integration of operando characterization techniques to monitor catalyst structure during synthesis and operation	X-ray absorption spectroscopy (XAS), Raman spectroscopy, in-situ characterization methods	Enables real-time identification of active-site formation and correlation with catalytic performance	20, 21, 22
Exploration of complex catalyst architectures (e.g., HEAs or multi-metal systems)	AI-driven decoupling of structure–activity–synthesis relationships through data-driven modelling and optimization	Machine learning models trained on DFT and experimental data	Facilitates discovery of complex catalyst compositions that balance activity and stability	23, 24
Translation of catalyst discovery to energy systems	Autonomous discovery platforms integrating synthesis, testing, and optimization for hydrogen technologies	Autonomous laboratories, AI-driven materials discovery frameworks	Accelerates development of durable catalysts that improve power density and efficiency in hydrogen production and energy conversion systems	10, 25

View Article Online
DOI: 10.1039/D6MA00429F

3.1. Cracking the Durability Problem via Autonomous Discovery

The Achilles' heel of state-of-the-art Fe–N–C catalysts is their poor long-term stability in the harsh acidic environment of PEMFCs [12]. Degradation arises from complex, coupled mechanisms, including metal demetallation (active-site leaching), carbon corrosion (support breakdown), and attack by reactive oxygen species, such as H_2O_2 [11]. Traditional catalyst evaluation relies on slow, standardized Accelerated Stress Tests (ASTs) (e.g., DOE protocols) [13]. Autonomous AI Agents transform this process in two keyways:

- **AI-Guided Accelerated Stress Test Protocol Design**

Rather than following fixed protocols, the Autonomous AI Agent leverages its KG to learn degradation patterns from prior materials. It dynamically tunes AST parameters, such as potential cycling range, dwell time, temperature, and humidity, to selectively accelerate specific degradation mechanisms (e.g., demetallation vs. carbon corrosion) [14]. A central advantage of this approach is the integration of intrinsic uncertainty quantification (UQ). In active learning loops, the agent uses UQ to guide experiment selection, prioritizing conditions where the model's confidence is lowest, thereby avoiding extrapolation errors when navigating previously unexplored chemical spaces. This targeted approach improves both representativeness and the acceleration factor, reducing human-level 1000 h tests to potentially tens of hours.

- **Predictive Durability Descriptors**

The Autonomous AI Agent moves beyond measuring only final performance, focusing on identifying early-stage descriptors predictive of long-term stability [15]. By moving beyond descriptive summarization, the agent uses robust surrogate models that generalize to physically meaningful representations, ensuring the system maintains high-fidelity discovery rather than merely rapid automation. By integrating real-time gas analysis (e.g., CO_2 or H_2O_2 production rates) with its ML models during ASTs, the Agent correlates instantaneous chemical markers with long-term voltage decay [11,16]. This predictive loop allows rapid elimination of unstable candidates, concentrating costly testing resources on materials with the highest potential for intrinsic stability.

3.2. Synthesis Optimization via Atomic-Level Feedback

The synthesis of highly active M–N–C materials involves a vast and non-linear parameter space, including metal precursor concentration, ligand type, pyrolysis temperature, atmosphere, and cooling rate [17,18]. Conventional approaches struggle because they are typically decoupled from the actual synthesis process and rely on post-mortem characterization. However, the core success of closed-loop discovery hinges on integrating robust surrogate models rather than on rapid automation alone. Without physically meaningful representations, such as those that capture the electronic d-band center or the local coordination environment, the framework risks becoming a high-throughput but low-fidelity system that overlooks the underlying physics of the ORR process.

The closed-loop durability and synthesis optimization process illustrated in Figure 3 highlights the predictive descriptors and in-situ feedback utilized by the Autonomous AI Agent. Specifically, the agent defines and tracks predictive descriptors, such as the metal-center coordination number and oxidation state, to clarify how these atomic-level features feed back into the AI decision-making loop.

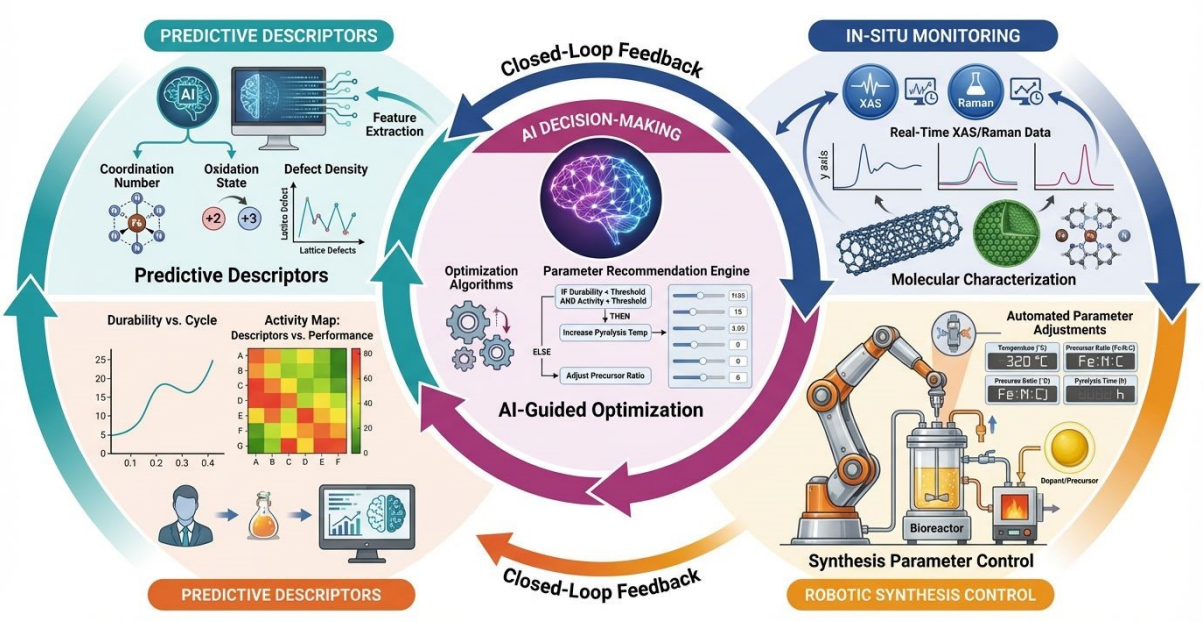


Figure 3. Durability and Synthesis Optimization. AI-guided closed-loop system integrating predictive descriptors and in-situ characterization to optimize catalyst performance and stability.

View Article Online
DOI: 10.1039/D6MA00429F

The Autonomous AI Agent extends conventional catalyst discovery strategies through closed-loop synthesis optimization (CSO), in which experimental observations obtained from in-situ or operando characterization are continuously fed into machine-learning models that update synthesis parameters and guide subsequent robotic experiments. By refining these models iteratively, the agent ensures that predicted configurations are grounded in structural accuracy rather than mere statistical correlation, enabling robust generalization even when exploring novel chemical spaces with sparse experimental data. Such iterative experimentation–learning cycles, as demonstrated in robotic nanoparticle synthesis platforms, enable rapid convergence toward optimal materials configurations by autonomously refining reaction conditions and precursor compositions [19].

A. Characterization Integration

The Agent's most powerful sensor inputs are advanced characterization techniques, particularly X-ray absorption spectroscopy (XAS) and Raman spectroscopy [20-21].

- **Active Site Fingerprinting:** During pyrolysis or activation steps, the Agent monitors the formation and coordination of metal sites, distinguishing desired Fe–N_4 centers from inactive Fe nanoparticles [22].

- **Real-Time Optimization:** Spectral signatures ("fingerprints") are continuously correlated with electrochemical performance. If XAS indicates a sub-optimal coordination (e.g., M–N_3O), the Agent leverages its reasoning core (LLM) to adjust pyrolysis conditions, for instance: *"Increase temperature by 50 ºC to promote C–N restructuring and enhance Fe–N_4 site density."* [22].

B. Decoupling Synthesis and Structure Descriptors

The AI system can handle the synthesis of complex, next-generation catalysts, including diatomic M_1–N–M_2–C systems and high-entropy alloys (HEAs), which are intractable via trial-and-error [23]. By training ML models on its own DFT and synthesis data, the Agent systematically decouples geometric and electronic effects, generating a structure–activity–synthesis map beyond human intuition [24]. This CSO enables

View Article Online
DOI: 10.1039/D6MA00429F

rapid discovery of previously inaccessible coordination environments that balance activity and stability.

By tightly integrating iterative synthesis with real-time operando characterization and adaptive durability-testing feedback loops, autonomous AI agents can rapidly navigate the vast design space of next-generation electrocatalysts, including emerging 2D materials and multi-component architectures. Such closed-loop platforms enable the systematic identification of active-site configurations that simultaneously optimize catalytic activity, stability, and resistance to degradation. Beyond accelerating catalyst discovery, this approach has direct implications for energy technologies: the rapid development of durable, low-cost catalysts could significantly enhance power density and operational lifetime in hydrogen production and electrochemical energy conversion systems. In this way, autonomous discovery platforms may play a pivotal role in bridging the gap between catalyst innovation and scalable deployment within the hydrogen economy. [25].

4. Challenges and Ethical Considerations in the Era of Autonomous Discovery

The rise of SDLs and AI-driven materials discovery in electrocatalysis introduces significant scientific, economic, and ethical challenges that must be addressed to ensure successful industrial translation [26].

4.1. Scientific Challenges: Reproducibility and Data Infrastructure

The primary scientific barriers to widespread adoption revolve around reproducibility and the establishment of robust, standardized data infrastructures [27].

4.1.1 The Reproducibility Crisis in Electrocatalysis

The electrocatalysis community is actively confronting a reproducibility crisis, which poses a major hurdle for autonomous laboratories that rely on high-quality data. Variations in synthesis conditions, electrochemical testing protocols, and reporting practices can lead to significant discrepancies in reported catalyst performance across laboratories. Table 2 summarizes the major sources of irreproducibility in electrocatalysis and highlights their implications for AI-driven catalyst discovery platforms. [28-29].

Table 2. Sources of irreproducibility in electrocatalysis and implications for autonomous discovery platforms.

Source of Irreproducibility	Description	Comments (Impact on Reported Performance / Recommended Practices)	Ref
Incomplete reporting of synthesis parameters	Variations in catalyst synthesis conditions such as precursor composition, pyrolysis temperature, and gas environment are often not fully documented.	Small differences in synthesis conditions can significantly alter active-site density and catalytic activity, leading to inconsistent performance across laboratories. Researchers should follow a "Checklist for Autonomous Synthesis," reporting at minimum: precursor molarity, ramp rates, atmosphere flow velocity, and software configuration for reproducibility.	28, 31, 35
Variability in electrochemical testing protocols	Differences in electrolyte composition, catalyst loading, electrode preparation, and measurement conditions (e.g., rotating disk electrode parameters) can influence measured catalytic activity.	Inconsistent testing methodologies can lead to large discrepancies in reported ORR performance metrics. Standardized testing protocols and benchmarking practices are recommended to ensure meaningful comparison of catalyst performance across studies.	13, 30, 35
Difficulty in active-site quantification	Determining the exact number and nature of active sites in heterogeneous catalysts remains challenging due to structural complexity.	Uncertainty in active-site density complicates the determination of intrinsic catalytic activity (e.g., turnover frequency). Advanced operando spectroscopy and improved characterization methods are needed to accurately quantify active sites.	11, 20, 21, 22
Fragmented experimental knowledge in literature	Critical experimental details are often embedded in unstructured text across numerous publications, making systematic data extraction difficult.	Fragmented knowledge limits the ability of machine-learning models to access reliable datasets. Knowledge graphs and structured data frameworks can help organize experimental information and improve data interoperability for autonomous discovery platforms.	37, 38, 40, 42
Limited availability of high-quality datasets for AI models	Machine-learning models require large, high-quality datasets for training and validation, but such datasets are scarce in catalysis research.	Autonomous laboratories and standardized data infrastructures can generate consistent high-throughput datasets, enabling more reliable AI-driven materials discovery.	27, 34, 45

View Article Online
DOI: 10.1039/D6MA00429F

Addressing these challenges is essential for building reliable data infrastructures capable of supporting autonomous discovery systems. Standardized experimental protocols, improved reporting practices, and structured data frameworks will be critical for enabling robust machine-learning models and self-driving laboratories.

Interlaboratory studies have shown that variability in heterogeneous electrocatalysis often arises from undescribed but critical process parameters in catalyst synthesis and testing protocols, which can lead to substantial discrepancies in reported activity metrics such as overpotential, current density, and stability across laboratories [30]. To address this systematically at the architectural level, integrating a 'Marker-Checker' protocol into the autonomous workflow is a crucial piece of the puzzle. In this framework, the generative 'Marker' (the Autonomous AI Agent) proposes experimental parameters, while a physics-informed 'Checker' (a validation module) audits the proposal against known chemical constraints and safety boundaries before execution. This dual-layer oversight prevents the propagation of 'hallucinated' or physically inconsistent data into shared infrastructure.

The inherently multidisciplinary nature of electrocatalytic systems, spanning electrochemistry, materials science, surface science, and spectroscopy, makes it difficult to accurately determine fundamental quantities such as active-site density and intrinsic catalytic activity [31,32]. These challenges have prompted growing community efforts to improve rigor and reproducibility, including workshops and collaborative initiatives to establish standardized experimental practices and reporting guidelines for catalysis research [33]. Implementing a standardized 'Checklist for Autonomous Synthesis' (see Table 2) within the Marker-Checker loop ensures that every data point, including both the intended parameters and the verified execution, is accompanied by the necessary metadata. By capturing undocumented process parameters (e.g., precise ramp rates or atmospheric moisture levels) as mandatory fields in the Autonomous AI Agents reporting loop, the community can move toward a reliable data infrastructure capable of supporting robust cross-laboratory benchmarking.

View Article Online
DOI: 10.1039/D6MA00429F

4.1.2 Data Standardization and Quality Control

The SDLs generate massive volumes of data, making standardization and quality control critical for effective AI-driven discovery [34].

- **Standardized Metrics:** To ensure fair comparisons and provide reliable training data for AI, the field is moving toward community-wide standardized metrics for evaluating catalyst performance. For example, benchmarks have been established for Ru-based ammonia decomposition catalysts, providing reproducible performance indicators [35].

- **Quality Control Tools:** Developing robust quality control tools and in-line or end-of-line diagnostics is essential, particularly for enhancing the manufacturability and recyclability of fuel cell components [36]. These tools enable system-level integration and ensure that data feeding the AI agents is both accurate and actionable.

4.1.3 Leveraging Knowledge Graphs

Managing complex and fragmented experimental knowledge is a major challenge for AI-driven discovery. Structured data formats, such as KGs, offer a solution [37,38].

- **Fragmented Knowledge:** Currently, most experimental details and synthesis pathways are “buried in unstructured prose” across countless research articles, limiting their accessibility and utility for AI models [39].

- **Knowledge Graph Frameworks:** Emerging open-source frameworks, including CATDA (Corpus-aware Automated Text-to-Graph Catalyst Discovery Agent) and CatKG (Catalyst Knowledge Graphs), leverage LLMs and text-mining techniques on vast literature corpora to:
    - Extract and structure information on synthesis pathways, precursor properties, catalytic performance, and experimental conditions into queryable graph representations [40-41].
    - Enable natural-language queries for knowledge discovery, allowing researchers and AI agents to interrogate complex datasets intuitively [42].

View Article Online
DOI: 10.1039/D5MA00429F

- Generate machine-learning-ready datasets with near-human fidelity, bridging the gap between literature knowledge and autonomous experimental planning [43].

By integrating KGs into autonomous systems, AI agents gain a structured, holistic view of experimental knowledge, enabling more informed decision-making and dramatically reducing redundant experimentation.

4.2. Economic and Scale-up Challenges

Transitioning from laboratory-scale prototypes to industrial-scale applications presents significant economic hurdles [44].

Cost of Self-Driving Laboratories

The initial investment required to establish a fully functional, multi-purpose SDL is substantial [45].

- High Capital Requirements: Major institutions have secured multi-million-dollar grants to build SDL networks, for instance, a $2 million NSF grant for collaborative SDL efforts and a $200 million Canadian CFREF grant for materials discovery, highlighting the significant financial commitment needed for state-of-the-art facilities [46].
- Towards Cost-Effective Solutions: Parallel initiatives are exploring more affordable SDLs. Examples include the Bayesian Robotic Investigator of Novel Electrolytes (BRINE), which integrates open-source robotics with standard electrochemical equipment to autonomously test formulations [47]. Such systems suggest a path toward more accessible, purpose-built autonomous platforms that could democratize AI-driven materials discovery.

4.3. Ethical Considerations in AI-Driven Discovery

The deployment of AI and autonomous systems in materials science introduces a range of ethical considerations that must be proactively addressed [48].

- **Bias and Fairness:** AI models are inherently susceptible to data biases, which can lead to algorithms that inadvertently prioritize or overlook certain materials.

This may hinder the discovery of globally accessible or cost-effective solutions [49].

- **Transparency and Explainability (T&E):** Autonomous systems often operate as "black boxes." Ethical deployment requires that AI decision-making in material selection and optimization remains transparent and interpretable to human researchers, balancing explainability with other considerations such as privacy [50].
- **Accountability and Responsibility:** Clear lines of responsibility must be defined for outcomes, defects, and potential harms associated with autonomously discovered materials, ensuring that human oversight is maintained [51].
- **Safety and Security:** Systems must be designed to mitigate unintended harms and minimize vulnerabilities to malicious attacks, particularly given the sensitive nature of energy-related materials research [52].
- **Sustainability and "Do No Harm":** Ethical principles dictate that AI development and deployment should align with legitimate objectives while minimizing negative environmental and societal impacts, including resource consumption, waste generation, and broader sustainability considerations [53].

5. Future Perspectives and Strategic Roadmap

The next decade (2025–2035) will be pivotal for autonomous materials discovery in electrocatalysis, as SDLs and AI-driven workflows mature. To fully realize their potential, a coordinated roadmap spanning technical development, data infrastructure, and industrial translation is essential.

A. Technical Milestones and Research Priorities

Advanced AI Reasoning:

- Develop domain-specific LLMs capable of predictive synthesis planning, multi-step reaction reasoning, and hypothesis generation across multi-metal systems (e.g., HEAs, diatomic M_1–N–M_2–C catalysts).

View Article Online
DOI: 10.1039/D6MA00429F

- Integrate Explainable AI (XAI) approaches to ensure interpretability of predictions for human researchers.

Closed-Loop Synthesis and Characterization:

- Expand in-situ and operando characterization (XAS, Raman, electron microscopy) with real-time feedback for CSO.
- Implement multi-modal data fusion, correlating structural, electronic, and electrochemical performance metrics to guide autonomous experiments.

Durability and Performance Targets:

- Establish predictive descriptors of stability for acidic ORR environments and other electrochemical reactions (CO_2RR, OER).
- Focus on designing next-generation PGM-free catalysts that balance activity, stability, and scalability, aiming to meet U.S. DOE 2025–2030 benchmarks.

B. Data and Knowledge Infrastructure

Standardization and FAIR Data Principles:

- Adopt community-wide standards for reporting synthesis, characterization, and electrochemical testing.
- Ensure datasets are Findable, Accessible, Interoperable, and Reusable (FAIR) to maximize AI utility.

Knowledge Graph Expansion:

- Leverage structured KGs to integrate literature, experimental data, and simulation results.
- Enable AI-guided hypothesis generation, cross-experiment reasoning, and machine-learning-ready datasets for broader materials discovery applications.

C. Industrial Translation and Policy Alignment

Scalable SDL Platforms:

- Develop cost-effective, modular SDLs for industrial adoption, balancing flexibility with affordability.

- Promote public-private partnerships to accelerate adoption in fuel cell, battery, and electrochemical industries.

Workforce Development:

- Establish training programs to upskill researchers in AI, robotics, and advanced materials science, ensuring human oversight and ethical governance of autonomous systems.

Sustainability and Ethics:

- Incorporate life-cycle analysis, sustainability metrics, and ethical guidelines into all autonomous workflows.
- Ensure AI-guided discovery minimizes environmental impact, maximizes resource efficiency, and adheres to principles of fairness, accountability, and transparency.

Strategic Outlook

By following this roadmap, autonomous discovery will accelerate the identification of high-performance, durable, and scalable electrocatalysts, compressing research timelines from decades to years. Success hinges on interdisciplinary collaboration, robust data infrastructures, and proactive policy frameworks that align scientific innovation with societal needs. The roadmap positions SDLs and autonomous agents not just as research tools, but as transformational platforms that will define the future of sustainable energy materials discovery. We propose a strategic roadmap for the development and deployment of autonomous agent systems (Figure 4), highlighting key milestones in laboratory automation, AI integration, and industrial translation between 2025 and 2030.

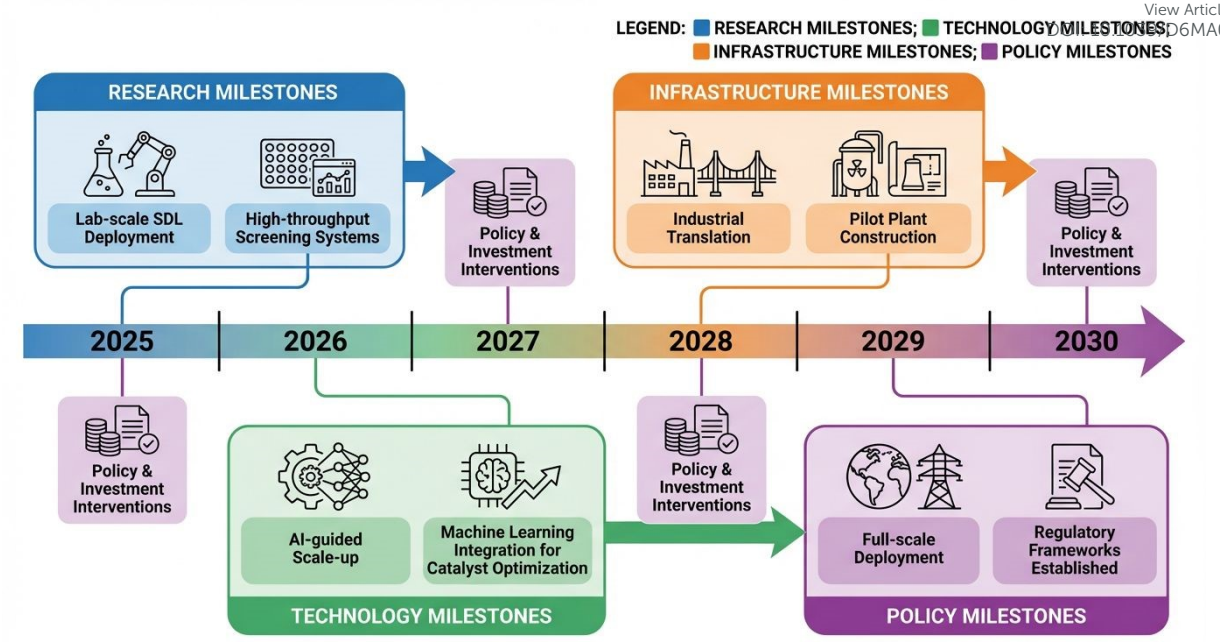


Figure 4. Strategic roadmap for autonomous ORR electrocatalyst discovery from 2025–2030, showing key milestones in self-driving laboratory deployment, AI integration, industrial translation, and policy/investment interventions.

6. Conclusions

The transition to a sustainable energy and chemical economy critically depends on autonomous materials discovery, particularly in electrocatalysis. Over the next decade, the deployment of self-driving laboratories (SDLs) and Autonomous AI Agent workflows is poised to accelerate the search for next-generation catalysts, fundamentally transforming the energy materials landscape.

Autonomous systems address the key challenge in electrocatalysis, simultaneous optimization of selectivity, efficiency, and stability, by enabling rapid, high-throughput exploration of complex material parameters, including composition, morphology, and surface structure. This capability allows the design of catalysts – ranging from M-N-C systems to 2D MXenes and high-entropy alloys - with high Faradaic efficiency for high-

View Article Online
DOI: 10.1039/D6MA00429F

value products such as CO, formate, and multi-carbon fuels, compressing traditional R&D timelines from decades to years.

However, the core success of these closed-loop workflows hinges on the integration of robust surrogate models. While robotics and LLMs enable experiments, the system requires physically meaningful representations that generalize across diverse catalyst classes. Future efforts must prioritize physics-informed machine learning frameworks that bridge first-principles accuracy and scalable computational efficiency, ensuring these platforms function as high-fidelity discovery engines rather than mere high-throughput automation tools.

The economic impact of these advances is equally transformative. The discovery of stable, high-performance, non-precious metal catalysts can drastically reduce the reliance on costly PGMs, lowering fuel cell stack costs and accelerating market adoption. The global fuel cell market is projected to expand to billions of US dollars, and autonomous catalyst innovation will be a critical enabler of this growth.

Realizing this potential requires strategic investment in infrastructure, workforce development, and data standardization, including the adoption of minimum reporting checklists to ensure cross-laboratory reproducibility. Coupled with ethical and sustainable practices, these efforts will ensure that autonomous discovery not only accelerates innovation but also supports safe, responsible, and globally impactful deployment. By integrating advanced AI with automated experimentation, the next decade promises a paradigm shift in materials discovery, where rapid, knowledge-driven innovation becomes the norm and contributes directly to global decarbonization goals.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

Generative AI declaration statement

View Article Online
DOI: 10.1039/D6MA00429F

During the preparation of this work, the authors used Grammarly to polish the language and improve the quality, clarity, and readability of the text. After using this tool/service, the authors reviewed and edited the content as needed and take full responsibility for the content of the publication. The authors also emphasize that no generative AI tools were used to generate scientific text, hypotheses, or to write or rewrite any substantive portions of the introduction, methods, results, or discussion beyond basic grammar and spelling correction.

Data availability

- No data was used for the research described in the article.

References

1. Zhou, Y., Xu, Y., Song, Y., Wang, P., Lin, B., Xu, F., Liu, C. & Zhang, C. Boosting Oxygen Electrocatalytic Activity of Catalysts by Phosphorus Incorporation. *J Am Chem Soc*, 145 (6) 3647–3655 (2023).
2. Wang, A.-L. Catalysts for Electrocatalytic Oxygen Reduction Reaction. in *Electrochemical Energy Conversion via Small Molecule Catalysis* 183–208 (John Wiley & Sons, Ltd, 2025).
3. Pescher, F., Fally, F., Hueso, C., Sorel, C., Valler, M., Wippermann, D., Escribano, B., Siewert, E. & Dubau, L. Pt/C catalysts synthesized in a commercial particle atomic layer deposition system enabling improved durability in fuel cells. *RSC Adv*, 14, 32358–32369 (2024).
4. Alabi, A. S., Popoola, A. P. I., Popoola, O. M., Mathe, N. R. & Abdulwahab, M. Materials for electrocatalysts in proton exchange membrane fuel cell: A brief review. *Front Energy Res, 11, 1091105* (2023).

5. Payattikul, L., Chen, C.-Y., Chen, Y.-S., Raja Pugalenthi, M. & Punyawudho, K. Recent Advances and Synergistic Effects of Non-Precious Carbon-Based Nanomaterials as Electrocatalysts: A Review. *Molecules*, 28(23), 7751 (2023).

6. Lian, Y., Xu, J., Zhou, W., Lin, Y. & Bai, J. Research Progress on Atomically Dispersed Catalysts for the Oxygen Reduction Reaction. *Molecules*, 29(4), 771 (2024).

7. Liang, Z., Yan, K., Zhang, K., Zheng, C., Chen, J., Feng, Q. & Li, Y. High-entropy materials for electrocatalytic oxygen reduction reaction. *J Mater Chem A*, 13, 25195–25212 (2025).

8. Li, J., Ding, C., Liu, D., Chen, L. & Jiang, J. Autonomous laboratories in China: an embodied intelligence-driven platform to accelerate chemical discovery. *Digital Discovery*, 4, 1672–1684 (2025).

9. Xie, Y., He, K. & Castellanos-Gomez, A. Toward Full Autonomous Laboratory Instrumentation Control with Large Language Models. *Small Struct*, 6 (8), 2500173 (2025).

10. Zhou, J., Yin, D., Chen, C., Yan, Z. & Zhang, D. A multi-robot–multi-task scheduling system for autonomous chemistry laboratories. *Digital Discovery*, 4, 636–652 (2025).

11. Kumar, K., Dubau, L., Jaouen, F. & Maillard, F. Review on the Degradation Mechanisms of Catalysts for the Oxygen Reduction Reaction in Acid Electrolyte: Current Understanding and Mitigation Approaches. *Chem Rev*, 123 (15), 9265–9326 (2023).

12. Liu, Q., Liu, H., Zhang, W., Xu, Q. & Su, H. Advanced Electrocatalyst Supports for High-Temperature Proton Exchange Membrane Fuel Cells: A Comprehensive Review of Materials, Degradation Mechanisms, and Performance Metrics. *Catalysts*, 15(9), 871 (2025).

13. Zhang, H., Liu, W., Wang, C., Li, S., Wang, H., Wang, J., Yang, H., Chen, S., Sun, S., Wang, S. & Xia, Z. Standardized protocols for evaluating platinum group metal-free oxygen reduction reaction electrocatalysts in polymer electrolyte fuel cells. *Nat Catal*, 5, 455–462 (2022).

View Article Online
DOI: 10.1039/D6MA00429F

14. Shokhen, V., Strandberg, L., Skoglundh, M. & Wickman, B. Impact of Accelerated Stress Tests on the Cathodic Catalytic Layer in a Proton Exchange Membrane () Fuel Cell Studied by Identical Location Scanning Electron Microscopy. *ACS Appl Energy Mater*, 5 (9), 11200–11212 (2022).

15. Samala, N. R., Friedman, A., Elbaz, L. & Grinberg, I. Identification of a Durability Descriptor for Molecular Oxygen Reduction Reaction Catalysts. *J Phys Chem Lett*, 15 (2), 481–489 (2024).

16. Craig, M. J. & García-Melchor, M. Applying Active Learning to the Screening of Molecular Oxygen Evolution Catalysts. *Molecules*, 26(21), 6362 (2021).

17. Li, A., Qureshi, N. & Maheshwari, V. Synthesis of high-entropy alloys for electrocatalysis. *Nanoscale*, 17, 20020–20043 (2025).

18. Wang, M., Ma, Z., Liu, S., Shi, Z., Liu, K., Su, R., Yang, J., Han, X. & Zhang, J. An Fe–N–C electrocatalyst with dense active sites synthesized by expeditious pyrolysis of a natural macrocyclic complex. *J Mater Chem A Mater*, 10, 23001–23007 (2022).

19. Park, J., Kim, K., Park, H., Lee, S., Heo, H., Han, Y., Choi, J. & Kim, J. Closed-loop optimization of nanoparticle synthesis enabled by robotics and machine learning. *Matter*, 6 (3), 677–690 (2023).

20. Chen, M., Huang, Y., Li, S., He, C., Lin, J., Zhang, Q., Liu, H. & Zhou, Z. In-situ/operando Raman techniques for in-depth understanding on electrocatalysis. *Chemical Engineering Journal*, 461, 141939 (2023).

21. Ren, R., Sun, X., Xiao, B., Tang, J., Liu, B. & Wu, X. Chapter 8 - In situ/operando -ray absorption spectroscopy in small molecule–based electrocatalysis. in *Single Atom Catalysts:* Design, Synthesis, Characterization, and Applications in Energy (eds. Menezes, P. W., Sarkar, D. & Awasthi, K.) 199–214 (Elsevier, 2024).

22. Jia, Q., Cullen, D. A., K . Spectroscopic insights into the nature of active sites in iron–nitrogen–carbon electrocatalysts for oxygen reduction in acid. *Nano Energy*, 29, 65–82 (2016).

23. Xu, W., Diesen, E., He, T., Reuter, K. & Margraf, J. T. Discovering High Entropy Alloy Electrocatalysts in Vast Composition Spaces with Multiobjective Optimization. *J Am Chem Soc*, 146 (11), 7698–7707 (2024).

24. Sprueill, H. W., Wang, C., Liu, C., Kim, J. & Coley, C. W. Monte Carlo Thought Search: Large Language Model Querying for Complex Scientific Reasoning in Catalyst Design. In Findings of the Association for Computational Linguistics: EMNLP 2023, 8348–8365, (Singapore, 2023).

25. Motiramani, M., Singh, S. K., Solanki, D., Singh, M. & Saxena, A. AI-ML techniques for green hydrogen: A comprehensive review. *Next Energy*, 8, 100252 (2025).

26. Tobias, A. V & Wahab, A. Autonomous 'self-driving' laboratories: a review of technology and policy implications. *R Soc Open Sci*, 12 (7), 250646 (2025).

27. Yaping Qi, W. Y. From Data to Discovery: How -Driven Materials Databases Are Reshaping Research. *Computers, Materials & Continua*, 83 (2), 1555–1559 (2025).

28. Nwabara, U., Chung, Y., Zhang, X., Zhao, Z., Zhang, S., Liu, M., Zhang, Y., Wang, C., Li, C., Ma, S., Zheng, H. & Chen, S. High throughput computational and experimental methods for accelerated electrochemical materials discovery. *J. Mater. Chem. A*, 13, 26041–26066 (2025).

29. Tu, Z., Stuyver, T. & Coley, C. W. Predictive chemistry: machine learning for reaction deployment, reaction development, and reaction discovery. *Chem. Sci.*, 14, 226–244 (2023).

30. Ehelebe, K. & Senger, A.Benchmarking Fuel Cell Electrocatalysts Using Gas Diffusion Electrodes: Inter-lab Comparison and Best Practices. *ACS Energy Lett*, 7 (2) 816–826 (2022).

31. Keith, J. A., McKone, J. R., Snyder, J. D. & Tang, M. H. Deeper learning in electrocatalysis: realizing opportunities and addressing challenges. *Curr Opin Chem Eng*, 36, 100824 (2022).

32. Hausmann, J. N. & Menezes, P. W. Reproducibility in Electrocatalysis. *ECS Meeting Abstracts MA2024-02*, 2818 (2024).

33. Flaherty, D. W. & Bhan, A. Improving the rigor and reproducibility of catalyst testing and evaluation in the laboratory. *J. Catal.* 431, 115408 (2024).

34. Zhang, L., Liu, M., Yuan, H. & Wang, H. AI-driven new materials development and data standardization . *Chinese Science Bulletin*, 70 (24, 4066–4080 (2025).

35. Voiry, D., Wang, T., Cuisinier, M., Chen, Z. Best Practices for Reporting Electrocatalytic Performance of Nanomaterials. *ACS Nano*, 12 (10), 9635–9638 (2018).

36. Garvie, I. J., Cvijovic, L., Almeida Pazmiño, G. A., Niroumand, A. & Kjeang, E. Fuel Cell Electrochemical Characterization before and after Break-in; Implications for End of Line Manufacturing Diagnostics. *ECS Meeting Abstracts MA2024-02*, 3066 (2024).

37. Zhao, X., Xu, X., Du, S., Yu, K., Liu, B., Wang, Y. & Yao, C. Knowledge Graph-Empowered Materials Discovery. 2021 IEEE International Conference on Big Data (Big Data), Orlando, FL, USA, 4628–4632 (2021).

38. Aggour, K.S. Compound Knowledge Graph-Enabled Assistant for Accelerated Materials Discovery. *Integr Mater Manuf Innov*, 11, 467–478 (2022).

39. Tinnes, C., Ristin, M., Hohenstein, U., Fathi, K. & van de Venn, H. W. From Unstructured Product Descriptions to Structured Data for Industry 4.0 with ChatGPT. 2024 IEEE 7th International Conference on Industrial Cyber-Physical Systems (ICPS), St. Louis, MO, USA, 1–8 (2024).

40. Behr, A. S., Sifri, A., Lee, S., Varma, P. M. Generating knowledge graphs through text mining of catalysis research related literature. *Catal. Sci. Technol.*, 14, 5699–5713 (2024).

41. Suvarna, M., Vaucher, A. C., Mitchell, S., Laino, T. & Pérez-Ramírez, J. Language models and protocol standardization guidelines for accelerating synthesis planning in heterogeneous catalysis. *Nat Commun*, 14, 7964 (2023).

42. Ruehle, B. Natural language processing for automated workflow and knowledge graph generation in self-driving labs. *Digital Discovery*, 4, 1534–1543 (2025).

43. Dwivedi, Y. K., Kshetri, N., Hughes, L., Slade, E., Jeyaraj, A., Kar, A. K., Baabdullah, A. M., Koohang, A., Balakrishnan, J. V., Cadden, D., R. Al-Debei, M.,

View Article Online
DOI: 10.1039/D6MA00429F

S. J. W. A., M. T. A., D. K., L. M., S. B. & P. S. S. Opinion Paper: "So what if wrote it?" Multidisciplinary perspectives on opportunities, challenges and implications of generative conversational for research, practice and policy. *Int J Inf Manage*, 71, 102642 (2023).

44. Cabana, J. Electrochemistry for Decarbonization. *ACS Energy Lett*, 8 (1), 740–747 (2023).

45. Low, A. K. Y., Cheng, J. J. W., Hippalgaonkar, K. & Ng, L. W. T. Self-Driving Laboratories: Translating Materials Science from Laboratory to Factory. *ACS Omega*, 10 (28), 29902–29908 (2025).

46. Seifrid, M., & Jensen, A. K.Autonomous Chemical Experiments: Challenges and Perspectives on Establishing a Self-Driving Lab. *Acc Chem Res*, 55 (17), 2454–2466 (2022).

47. Dave, A., Vachhani, N., Raccuglia, P., Green, W. & Aspuru-Guzik, A. Autonomous Discovery of Battery Electrolytes with Robotic Experimentation and Machine Learning. *Cell Rep Phys Sci*, 1 (12), 100264 (2020).

48. Madika, B. Artificial Intelligence for Materials Discovery, Development, and Optimization. *ACS Nano*, 19 (30) 27116–27158 (2025).

49. Kordzadeh, N. & Ghasemaghaei, M. Algorithmic bias: review, synthesis, and future research directions. *European Journal of Information Systems*, 31(3), 388–409 (2022).

50. Wu, C. Data privacy: From transparency to fairness. *Technol Soc*, 102457 (2024).

51. Tang, X., Zhang, H., Liu, S., Wei, Y. & Hu, Y. Risks of scientists: prioritizing safeguarding over autonomy. *Nat Commun*, 16, 8317 (2025).

52. Findley, M. G., Ghosn, F. & Lowe, S. J. Vulnerability in research ethics: A call for assessing vulnerability and implementing protections. *Proceedings of the National Academy of Sciences*, 121 (34), e2322821121 (2024).

53. Abolhasani, M., Brown, K. A. & Editors, G. Role of AI in experimental materials science. *MRS Bull*, 48, 134–141 (2023).

54. Ha, M., Hajibabaei, A., Kim, D., Singh, A., Yun, J., Myung, C & Kim, K. Al-Doping Driven Suppression of Capacity and Voltage Fadings in 4d-Element Containing Li-Ion-Battery Cathode Materials: Machine Learning and Density Functional Theory. *Adv. Energy Mater*, 30, 2201497 (2022).

55. Ha, M., Pourassd, S., Myung, C & Kim, K. Accelerating Materials Discovery Through Sparse Gaussian Process Machine Learning Potentials. *Acc. Chem. Res*, 59, 10-113 (2026).

View Article Online
DOI: 10.1039/D6MA00429F

Autonomous AI Agents in ORR Electrocatalyst Discovery: A Review of Closed-Loop Workflows, Materials Bottlenecks, and Energy System Translation

Philani V. Hlophe[a], Muhammad Tawalbehi[b,c], Amani Al Othman[d,e], Phumlani F. Msomi[a*]

Data Availability Statement

No new datasets were generated or analysed during this study. All information discussed in this review is derived from previously published literature, which is cited and referenced within the article. Any additional information supporting the conclusions of this work is available from the corresponding author upon reasonable request.