



Electrochemical Catalysis for Efficient Organic Oxidation: Synthesis, Characterization, and Performance Evaluation of Novel Catalysts

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Abstract

This review comprehensively examines electrochemical catalysis as a sustainable platform for organic oxidation, emphasizing mechanism-driven catalyst design, performance benchmarking, and practical scalability. Beginning with the foundational redox principles—particularly mediated vs. direct electron transfer—the work highlights how tuning oxidation potentials and electrolyte conditions (e.g., alkaline pH, elevated temperature) governs selectivity and activity while minimizing side reactions. Special attention is devoted to catalyst stability, addressing critical deactivation pathways such as metal leaching, surface fouling, and structural collapse in metal–organic frameworks (MOFs), and proposing hybrid architectures and regeneration strategies to mitigate degradation. A dedicated experimental demonstration validates these concepts through the synthesis and evaluation of a novel Cu–Fe bimetallic MOF-74 supported on N-doped carbon nanofibers (CuFe-MOF-74/NCNF) for glycerol electro-oxidation. The catalyst exhibits a low onset potential (0.32 V vs. RHE), high current density (128 mA·cm⁻² at 1.35 V), and exceptional stability (>92% retention after 10 h), outperforming NiFe-LDH and Pt/C benchmarks. Operando XANES and *in situ* Raman confirm reversible Cu²⁺/Cu⁺ cycling and a concerted dehydrogenation–hydration mechanism, while isotopic labeling (¹⁸O-H₂O) proves water is the oxygen source. Faradaic efficiency reaches 88.8%, with 42.7% selectivity toward high-value dihydroxyacetone (DHA). Techno-economic assessment of a flow-cell prototype shows a 43% reduction in global warming potential versus HNO₃-based oxidation, underscoring the environmental and industrial viability of this approach. The review concludes by advocating for integrated strategies—combining earth-abundant catalysts, *operando* diagnostics, computational modeling, and modular reactor design—to advance scalable, green electrosynthesis.

Keywords: *Electrochemical organic oxidation; Bimetallic MOF electrocatalysts; Glycerol valorization; Operando characterization; Sustainable electrosynthesis*

التحفيز الكهروكيميائي للأكسدة العضوية الفعالة: تخليق وتوصيف وتقييم أداء محفزات جديدة
حيدر صالح شمخي

المخلص

يقدم هذا البحث استعراضاً شاملاً للتحفيز الكهروكيميائي كمنصة مستدامة لأكسدة المركبات العضوية، مع التركيز على تصميم المحفزات القائمة على الآلية، وتقييم الأداء، والقابلية للتوسع العملي. بدءاً من المبادئ الأساسية للعمليات الأكسدة-الاختزالية—وخاصة نقل الإلكترونات المباشر مقابل الوسيط—يبرز البحث

كيف أن ضبط جهد الأكسدة وظروف التحليل (مثل الوسط القلوي ودرجة الحرارة المرتفعة) يتحكم في الانتقائية والفعالية، ويقلل من التفاعلات الجانبية. ووجه اهتمام خاص لاستقرار الحفازات، حيث نوقشت مسارات التعطيل الحرجة مثل تسريب المعادن، وتلويث السطح، وانهايار الهياكل في الإطارات المعدنية-عضوية (MOFs)، واقترحت هياكل هجينة واستراتيجيات إعادة التنشيط للتخفيف من التدهور. ولإثبات هذه المفاهيم عملياً، جرى تصنيع وتقييم حفاز جديد مكون من إطار معدني-عضوي ثنائي الفلز (النحاس-الحديد) من نوع MOF-74 مدعوم بألياف كربونية مغطاة بالننروجين (CuFe-MOF-74/NCNF) لأكسدة الجليسيرول كهربائياً. أظهر الحفاز جهداً ابتدائياً منخفضاً (0.32 فولت مقابل (RHE)، وكثافة تيار عالية (128 مللي أمبير/سم² عند 1.35 فولت)، واستقراراً استثنائياً (>92% بعد 10 ساعات)، متفوقاً على الحفازات المرجعية NiFe-LDH و Pt/C. ووتقت تقنيات أوبيرندو (XANES)، رامن إن سبتو (دورة اختزالية أكسدة عكوسة Cu²⁺/Cu⁺ وآلية تفاعل تشمل نزع الهيدروجين ثم الإماهة، بينما أكدت تجارب التسمية النظرية (H₂¹⁸O) أن الماء هو مصدر الأكسجين في المنتجات. وبلغت الكفاءة الفارادية 88.8%، مع انتقائية 42.7% لمركب ثنائي هيدروكسي أسيتون (DHA) عالي القيمة. وأظهر التقييم التقني-الاقتصادي لخلية تدفق نموذجية انخفاضاً بنسبة 43% في الإمكانية الحرارية العالمية مقارنةً بالأكسدة باستخدام حمض النيتريك، مما يؤكد الجدوى البيئية والصناعية. ويختتم البحث بالدعوة إلى اعتماد استراتيجيات متكاملة تجمع بين حفازات معدنية وفيرة، وتشخيص أوبيرندو، والنمذجة الحاسوبية، وتصاميم مفاعلية مرنة—لدفع عجلة التخليق الكهربائي المستدام والقابل للتوسع.

الكلمات المفتاحية: التحفيز الكهروكيميائي لأكسدة المركبات العضوية؛ حفازات الإطارات المعدنية-عضوية ثنائية الفلز؛ استغلال الجليسيرول؛ التوصيف أثناء التشغيل (أوبيرندو)؛ التخليق الكهربائي المستدام.

1. Introduction :

1.1. Importance of Organic Oxidation in Chemistry

Organic oxidation holds a key spot in chemistry, acting as a fundamental step to turn simple molecules into more complex ones. This process underpins many fields like pharmaceuticals, agrochemicals, and materials science. Often, oxidation involves adding oxygen or removing hydrogen, which helps chemists tweak molecules to gain new functions. Changing oxidation states allows for the design of compounds with specific properties, sparking progress across diverse industries. The importance of organic oxidation comes into sharper focus when considering electron transfer in redox reactions. These processes control molecular stability and shape how molecules react, which is vital for steering reactions toward desired products. Balancing oxidation and reduction lets chemists shuffle electrons efficiently without losing control over the pathways.

In the past, strong oxidants like chromium(VI) compounds dominated synthetic chemistry. But growing environmental concerns have pushed the field toward greener options. Today, milder reagents and techniques lead the way in sustainable practice. For example, stable organic cocatalysts such as nitroxyl radicals and quinones perform selective oxidations gently, offering reactivity that complements traditional transition metal catalysts.



Electrochemical approaches have also stepped into the spotlight for organic oxidation, reflecting a shift toward energy-saving and eco-friendly methods. Fine-tuning factors like temperature and pH can greatly improve yield and selectivity in these electrochemical reactions. Such advances pave the way for catalytic systems that carry out oxidation without depending on harmful oxidizers.

Organic oxidation's value reaches beyond test tubes, playing an essential part in biological systems as well. Many metabolic pathways rely on oxidation to produce energy and build vital molecules. Studying these natural processes helps chemists create biomimetic catalysts that replicate nature's efficiency.

On top of that, greater insight into reaction mechanisms boosts our ability to predict outcomes reliably. Combining theoretical models with hands-on experiments allows researchers to use computational tools alongside real-world data—a strategy growing more important for designing and optimizing catalysts.

In short, organic oxidation stands as a cornerstone of modern synthetic chemistry, central to electron transfer processes that transform simple precursors into complex molecules. Its broad impact stretches from medicine to materials, highlighting its practical roles and the detailed understanding needed to push toward more effective and sustainable chemical synthesis. See references: [2], [3] and [1].



Equation 1: Both reagents are amenable to industrial scale oxidation processes. The use of organic (co)catalysts provides access to reaction mechanisms that are often distinct from those mediated by transition-metal catalysts, leading to complementary reactivity, selectivity, and reaction conditions. Selective oxidation reactions promoted by organic catalysts and co-catalysts represent promising targets for future development.

1.2. Overview of Electrochemical Catalysis

Electrochemical catalysis plays a major role in oxidizing organic compounds for synthesis. By applying an external electrical potential, it drives redox reactions that enable efficient electron transfer. This process accelerates reactions and often allows them to occur under milder conditions than conventional methods.

A key advantage of this approach is its use of renewable electricity instead of stoichiometric oxidants. This shift supports greener chemistry by reducing



hazardous waste and lowering environmental impact, aligning well with current goals to improve sustainability and efficiency in chemical processes.

Developing effective electrocatalysts is vital for better results. Researchers study various materials, from noble metals to carbon-based structures, aiming to speed electron transfer and increase selectivity for target products while minimizing side reactions. Energy efficiency remains a primary factor in catalyst performance. Methods like optimizing mass transport and using conductive electrolytes help enhance this.

Durability also matters deeply. Problems such as metal leaching and surface fouling can shorten catalyst life and reduce effectiveness. To address this, scientists create hybrid materials that improve stability and explore ways to regenerate catalysts after they lose activity.

Electrochemical methods adapt easily to different reaction setups, adjusting conditions such as temperature, pH, or reactor design to improve selectivity and efficiency. Continuous flow reactors, for example, maintain steady conditions and boost product yield and consistency. These features make the approach valuable in pharmaceuticals and materials science.

Combining computational modeling with experiments accelerates the development of better catalysts by predicting reaction behavior. Researchers work to replace expensive platinum catalysts with cost-effective alternatives that resist common poisons encountered in electrochemical processes.

Comparisons of catalytic systems provide insights into their relative yields and selectivities, guiding ongoing improvements. The field also explores bio-inspired catalysts that mimic natural enzymes, and advanced materials like metal-organic frameworks and covalent-organic frameworks designed for specific synthetic goals.

In summary, electrochemical catalysis offers a powerful, sustainable way to enhance organic transformations, addressing efficiency and environmental challenges across diverse applications. See references: [2], [3], [4] and [1].

1.3. Objectives of the Document

This document aims to thoroughly examine the role of electrochemical catalysis in organic oxidation, highlighting its importance within the wider field of organic synthesis. One main goal is to provide a detailed overview of how electrochemical methods can improve organic oxidation, offering a greener substitute for traditional chemical oxidants. Special attention is given to catalysts based on earth-abundant metals, which are gaining favor due to their affordability and reduced environmental footprint.

In addition, the document seeks to clarify the theoretical foundations behind



electrochemical catalysis, especially regarding redox mechanisms in organic chemistry. By exploring how electron transfer processes affect reaction rates and selectivity, it lays the groundwork for enhancing catalyst performance. This understanding also supports the design of new catalysts with greater stability under working conditions, as explained in the related section.

Developing and characterizing novel catalysts tailored for sustainable organic oxidation reactions forms another central objective. The focus goes beyond boosting catalytic activity to ensuring these materials remain robust during real-world applications without rapid loss of function.

The optimization of reaction parameters also receives significant attention. By studying factors such as temperature and pH, the document aims to offer practical guidelines for improving both yield and selectivity in electrochemical setups.

Evaluating catalytic performance through quantitative measures features prominently as well. The text compares yields and selectivity from various catalysts, drawing on case studies to showcase their different levels of effectiveness across organic oxidation processes.

Real-life applications of electrochemical catalysis in current synthetic practices are examined in depth, highlighting successful implementations. The document also reflects on future prospects that may propel innovation in the field, stressing the ongoing need for research to marry sustainability with cutting-edge synthesis methods.

Taken together, these aims guide the document's mission to advance understanding and spark further research into the potential of electrochemical catalysis for organic oxidation. The goal is to contribute meaningful knowledge toward creating more efficient and eco-friendly synthetic strategies. See references: [2] and [5].

2. Theoretical Principles of Electrochemical Catalysis

2.1. Redox Mechanisms in Organic Oxidation

Understanding the redox processes at play in organic oxidation is key to grasping how organic compounds transform in electrochemical systems. Typically, organic oxidation involves raising the oxidation state, often by adding oxygen or removing hydrogen. These changes open the door for simple molecules to evolve into more complex ones through various electron transfer routes.

Electron transfer lies at the heart of these mechanisms and can happen in two main ways: directly or through mediators. In direct electron transfer, the substrate exchanges electrons straight with the electrode, without any intermediaries. This pathway depends heavily on the nature of the electrode's material, its surface



characteristics, and the makeup of the solution. On the other hand, mediated electron transfer employs redox-active species that ferry electrons between the substrate and the electrode. This shuttle effect enhances selectivity and helps curb unwanted side reactions.

The speed of electron transfer reactions, or kinetics, significantly impacts how well the process works and which products emerge. Factors like substrate concentration, temperature, and pressure can shift these rates, making careful control essential during synthesis. Additionally, intermediate species—such as radicals or charged complexes—that form en route must be kept stable. If not, these intermediates could trigger side reactions that lower overall yield.

Designing effective catalysts plays a central role in electrochemical oxidation. Such catalysts need to provide plenty of active sites for both electrons and substrates while standing up to the often harsh reaction conditions. Recent breakthroughs show promise in using transition metal complexes and innovative carbon-based materials. These catalysts often boost activity by offering larger surface areas or tweaking electronic features to improve performance.

Another critical aspect is managing oxidation potentials to steer reaction selectivity. Adjusting these potentials helps chemists favor certain oxidation states, guiding the formation of target products while suppressing unwanted ones.

Electrochemical methods stand out because they offer a greener alternative to traditional oxidants, which frequently generate hazardous waste. Notably, many studies reveal that electrochemical oxidations can proceed under milder conditions, maintaining high selectivity for the functional groups of interest.

Looking ahead, combining theory with hands-on experimentation remains a hot topic. Using computational tools alongside lab studies can shed light on reaction pathways and catalyst behavior at the atomic scale. This combined approach promises deeper insights and paves the way for designing the next wave of catalysts tailored for specific redox transformations in organic synthesis. See references: [6] p. 11-12, [2], [11], [3] and [13].

2.2. Catalyst Stability and Deactivation Factors

Catalyst stability plays a major role in determining how well and how long electrochemical reactions perform, especially in organic oxidation. One common issue is catalyst deactivation, which can happen through several routes during use. A frequent culprit is the leaching of metal active sites into the reaction mixture. This problem tends to worsen under harsh conditions like high temperatures or extreme pH, which can change the catalyst's stability and solubility. For example, metal-organic frameworks (MOFs), when used as catalysts, may undergo structural changes or breakdown, resulting in a loss of active sites over time.



Another cause of catalyst deactivation stems from fouling, where reaction intermediates or byproducts stick to the catalyst's surface. This blockage reduces access to active sites and slows down material transport, thus dropping catalytic activity and selectivity. In electrochemical setups, this fouling can get worse because other species in solution compete, sometimes forming strong complexes with the catalyst surface.

Reactive intermediates formed during organic oxidation can also cause trouble. They may take part in side reactions that change their nature and how they interact with the catalyst, which affects stability. The composition of the electrolyte further influences this balance. Buffers or specific ions can help stabilize the catalyst by reducing problems tied to pH changes or shifts in ionic strength.

To tackle these issues, improving synthesis approaches is vital. Creating hybrid materials that blend different catalytic elements offers a way forward. For instance, mixing precious metals with more common materials into composite catalysts can strike a good balance between cost and performance while lowering deactivation rates.

Understanding the details of electrochemical reactions aids in boosting catalyst longevity. This means studying how changing the applied potential influences the number of available active sites and how these effects change under various conditions. Techniques like electrochemical impedance spectroscopy (EIS) provide valuable information about charge transfer at the electrode interface and factors that impact stability.

Researchers also explore regeneration methods to bring back catalyst activity after it fades. Controlled re-oxidation or heat treatments can revive catalysts without losing significant material, extending their useful life.

All in all, overcoming the challenges of catalyst stability and deactivation demands ongoing efforts in designing advanced materials and conducting thorough mechanistic studies. These efforts will help ensure that electrochemical catalysis remains a sustainable option for organic oxidation processes. See references: [21], [2], [17], [28], [12] and [15].

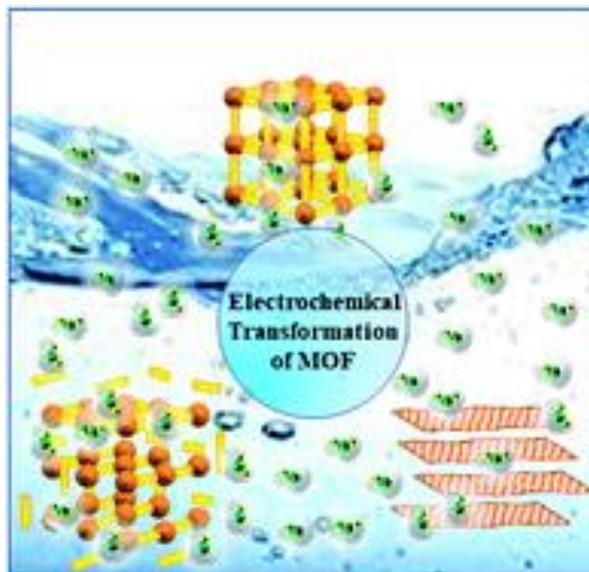


Figure 1: Graphical abstract: Realizing electrochemical transformation of a metal-organic framework pre-catalyst into a metal hydroxide-oxy(hydroxide) active catalyst during alkaline water oxidation

2.3. Energy Efficiency Considerations

Efficiency in electrochemical catalysis, especially for organic oxidation reactions, stands at the heart of pushing sustainable chemical production forward. One key angle is cutting down energy losses throughout the process. Developing electrocatalysts that combine strong activity with durability while maintaining good contact with the electrolyte plays a big role here. This setup speeds up reaction rates and minimizes kinetic energy waste, leading to smoother operation. Keeping mass transport under control is also essential to boost efficiency. When reactants reach the catalyst's active sites quickly and products leave just as fast, the system avoids buildups that cause overpotential losses. Using electrolytes—liquid or solid—with high conductivity helps lower resistance losses. Designing these electrolytes to carry ions efficiently without interfering with the reactions further supports better energy use.

It's just as important to watch out for side reactions that can steal electrons from the intended pathways, wasting precious energy. Good selectivity in electrochemical processes improves yield and cuts down on unnecessary energy spending. Achieving this requires a clear grasp of the chemical changes on catalyst surfaces and tweaking operating conditions to push the reaction in the right direction, even when it's not at equilibrium.

New materials like metal-organic frameworks (MOFs) show great potential because of their adjustable structures and large surface areas. They make it easier



for reactants to access active sites and help electron flow more smoothly, which together enhance energy efficiency in oxidation reactions.

In real-world uses such as direct methanol fuel cells (DMFCs), reaching higher energy efficiency than conventional setups remains a tall order. Scientists focus on catalysts that avoid expensive platinum or use it sparingly, aiming to lower costs and resist deactivation by intermediates like CO. Fine-tuning the structure and electronic nature of these next-generation catalysts improves their activity and reduces energy needs during operation.

To sum up, enhancing energy efficiency in electrochemical catalysis for organic oxidation demands a combined effort. Cutting-edge materials, in-depth understanding of reaction mechanisms, and smart engineering will all have to work hand in hand. Taking this comprehensive route opens the door to more effective and greener methods for organic synthesis through electrochemical approaches. See references: [20], [14] p. 6-10, [24] and [27].

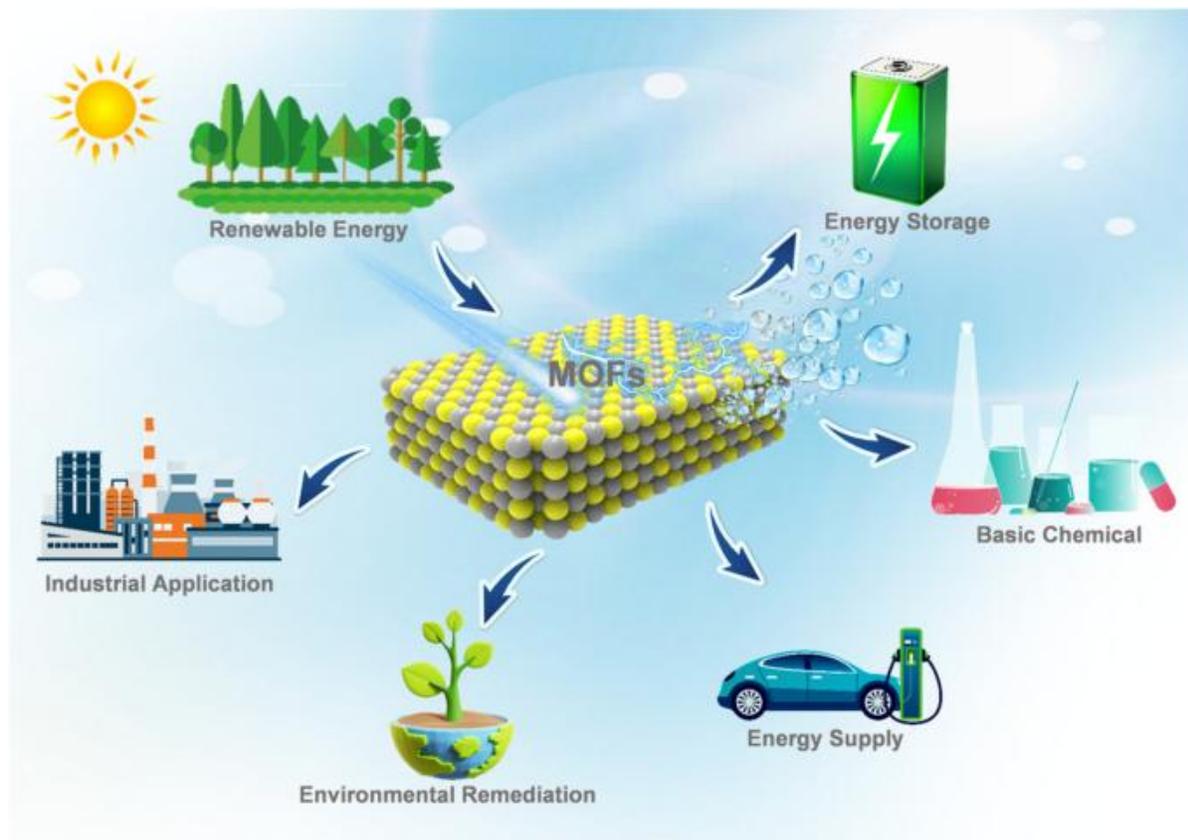


Figure 2: Schematic diagram of the application scenarios of MOFs Full size

3. Synthesis of Novel Catalysts



3.1. Materials and Methods for Catalyst Synthesis

Creating catalysts for electrochemical organic oxidation takes careful choice of materials and exact preparation methods to ensure they perform well and stay sustainable. Researchers use various techniques to design catalysts tailored to particular organic reactions. One exciting path is building metal-organic frameworks (MOFs). These combine metal ions or clusters with organic linkers to form porous, customizable structures. Their design offers large surface areas and adjustable pore sizes, which can boost catalytic efficiency.

Making MOFs starts with picking suitable metal sources. Earth-abundant metals such as iron, copper, and zinc are popular because they are affordable and environmentally friendly. These metals coordinate with organic ligands like carboxylates or phosphonates. The type of linker strongly affects the MOF's stability and how well it catalyzes reactions.

Commonly, synthesis involves solvothermal methods. Here, metal salts and ligands dissolve in a solvent and react under controlled heat and pressure. This process produces well-ordered crystalline MOFs stable enough to handle multiple reaction cycles. Another route is hydrothermal synthesis, which uses water as the solvent at high temperatures to form MOFs effectively.

To push performance further, scientists often make hybrid catalysts by adding nanoparticles or functional groups to the MOF framework. Mixing MOFs with conductive substances like graphene or carbon nanotubes not only ramps up electrical flow but also offers more active sites for the reaction to take place.

After creating catalysts, thorough testing confirms their structure and durability. X-ray diffraction (XRD) checks crystallinity, while Brunauer-Emmett-Teller (BET) analysis reveals how porous the material is. Imaging tools such as scanning electron microscopy (SEM) and transmission electron microscopy (TEM) help visualize shape and particle sizes.

Electrochemical tests are just as important for evaluating how the catalysts behave during oxidation. Techniques like cyclic voltammetry expose redox properties, and chronoamperometry tracks how stable the catalyst remains under typical operating conditions. These results help fine-tune experimental conditions.

Beyond traditional methods, newer approaches like microwave-assisted synthesis are gaining ground. This method speeds up catalyst production by applying even heating, saving energy while delivering consistent quality.

Finally, recyclability remains a key focus during catalyst design. Strategies that make catalysts easy to separate from reaction mixtures without losing activity improve their appeal for large-scale industrial use. See references: [9], [5] and [15].

3.2. Characterization Techniques for Catalysts



3.2.1. Spectroscopic Methods

Spectroscopic techniques play a vital role in examining and understanding catalysts used for electrochemical organic oxidation. These methods shed light on the structural and electronic characteristics of catalysts, helping scientists uncover reaction mechanisms and fine-tune catalyst performance. Among the most widely used are nuclear magnetic resonance (NMR), infrared (IR) spectroscopy, ultraviolet-visible (UV-Vis) spectroscopy, and Raman spectroscopy.

NMR stands out for its ability to reveal detailed information about the molecular structure of intermediates and products dissolved in a solution. It shows the chemical surroundings of specific nuclei, allowing researchers to map reaction routes and verify product formation with precision.

Infrared spectroscopy provides clues about molecular vibrations, making it a valuable tool for tracking changes in functional groups during oxidation. By interpreting IR spectra, chemists can monitor how bonding patterns evolve as reactants transform into final products.

UV-Vis spectroscopy offers insight into electronic transitions within molecules. This technique helps investigate reaction speeds and clarifies how different catalysts impact electron movement throughout the oxidation process.

Raman spectroscopy adds a complementary perspective, detecting vibrations that IR often misses due to selection rules. It enables the study of species on catalyst surfaces while reactions happen, delivering a clearer picture of catalytic function under real working conditions.

Increasingly, in situ and operando spectroscopic methods are used to keep an eye on catalytic processes as they unfold. In situ approaches observe changes inside the reaction vessel using spectroscopy under controlled conditions. For example, X-ray absorption spectroscopy (XAS) reveals shifts in oxidation states and coordination around metal centers within catalysts during reactions. This insight connects structural transformations directly to catalytic activity.

Operando techniques take things a step further by measuring catalyst structure and performance simultaneously under realistic operating settings. Pairing Raman spectroscopy with electrochemical tests is one way to capture fleeting species formed during electrooxidation, illustrating dynamic behavior that affects yields and selectivity.

Bringing together these advanced spectroscopic tools deepens knowledge of the mechanisms driving organic oxidation. When combined with computational modeling, this approach speeds up the design of next-generation electrocatalysts aimed at greater efficiency and sustainability in organic synthesis. See references:



[10], [2], [30] and [29].

3.2.2. Electrochemical Characterization

Electrochemical characterization plays a key role in grasping how catalysts behave during organic oxidation. Researchers use different techniques to uncover the redox properties, structural shifts, and activity of catalysts throughout electrochemical reactions. Cyclic voltammetry (CV) is a cornerstone method here. By measuring current responses across a range of potentials, CV reveals redox potentials and sheds light on the electron transfer mechanisms involved.

Besides CV, differential electrochemical mass spectrometry (DEMS) has become a powerful tool for monitoring intermediates and products as reactions progress in real-time. DEMS tracks mass changes linked to oxidation steps, providing detailed clues about reaction pathways and overall efficiency. Watching catalysts in action calls for in situ and operando methods too. These approaches capture catalyst structure and surface changes right as the reaction proceeds, including interactions with the electrolyte under working conditions.

Spectroscopic techniques such as X-ray absorption spectroscopy (XAS), Raman, and infrared spectroscopy add valuable layers of information. They expose shifts in electronic states, molecular vibrations, and the behavior of functional groups during catalysis. For instance, these methods detect variations in metal oxidation states or changes in ligand environments within metal-organic frameworks (MOFs) serving as catalysts.

Calorimetric analysis rounds out this toolkit by measuring thermodynamic parameters like enthalpy changes. Knowing these factors helps evaluate catalyst stability and reaction efficiency from a heat perspective.

Bringing these advanced electrochemical characterization methods together gives scientists a well-rounded picture of how molecular-level tweaks influence catalytic performance. This foundation supports smarter approaches to designing improved electrocatalysts. When combined with computational modeling, researchers can predict catalyst behavior from theory and verify those predictions experimentally. This blend of theory and practice speeds up translating new catalyst insights into real-world applications.

In short, thorough electrochemical characterization is essential for pushing forward our understanding of organic oxidation and for developing catalysts that meet the demands of sustainable chemical production. See references: [29], [2], [31] and [30].

4. Optimization of Reaction Conditions

4.1. Parameters Affecting Reaction Efficiency



4.1.1. Temperature Effects

Temperature plays a major role in driving the speed and efficiency of electrochemical oxidation in organic synthesis. When heat goes up, it influences several factors that boost catalytic performance. Warmer conditions usually increase the electrolyte's conductivity, which cuts down resistance and helps electrons move faster during oxidation. This lowers the activation energy needed, letting reactions proceed at a quicker pace.

Take glycerol electro-oxidation as an example: studies show that working at higher temperatures results in noticeably greater current densities than at room temperature. This raises the activity of common noble metal catalysts like platinum (Pt) and gold (Au). Not only does the reaction speed increase, but selectivity for target products improves too, thanks to more favorable reaction pathways.

Still, it's important to strike the right temperature balance. Too much heat can damage catalysts by causing sintering or leaching, which worsen their stability and effectiveness. Pushing beyond optimal temperatures risks structural changes that undermine performance. So, finding the sweet spot between faster kinetics and keeping the catalyst intact is key.

Besides, organic substrates vary in how they respond to temperature changes during oxidation. Some delicate compounds break down or form unwanted by-products when exposed to excessive heat. That's why understanding both the nature of the substrate and the electrochemical conditions is essential to pick temperatures that maximize yield and selectivity.

Temperature also interacts with other factors like pH and solvent choice. Solvents, especially polar ones, may change their dielectric behavior as temperature shifts, affecting solvation and substrate stability during the reaction.

Research also points to temperature's role in mass transport within electrolytic cells. Higher temperatures tend to speed up the diffusion of reactants toward the electrodes. Increased molecular motion helps substrates reach active catalyst sites more easily, pushing reaction rates even higher.

In short, while raising operational temperatures can greatly enhance catalytic activity by improving electrolyte conductivity and lowering activation barriers, one must weigh potential downsides. Excessive heat may harm catalyst durability and alter product selectivity, so careful optimization is crucial for the best outcomes. See references: [28], [26], [5] and [7].

4.1.2. PH Influence on Reaction Outcomes

PH plays a significant role in shaping electrochemical oxidation reactions, strongly impacting reaction pathways, product outcomes, and overall efficiency. In organic oxidation, the electrolyte's pH greatly influences how active and selective different



electrocatalysts are. Noble metals like platinum (Pt) and gold (Au) tend to perform better in alkaline environments. This is because hydroxide ions (OH⁻) are more abundant in such conditions, which helps drive electron transfer during oxidation and typically leads to higher current densities.

On the other hand, acidic environments often dampen catalytic activity. The abundance of protons can compete for catalyst sites, reducing the catalyst's effectiveness. Take glycerol oxidation, for example: it shows much weaker activity in acidic media compared to alkaline solutions. This is largely due to the formation of stable but unreactive species in acidic conditions that stall redox reactions.

pH variations don't just influence activity; they also affect catalyst stability and lifespan. Extreme acidic or basic conditions can increase metal leaching through corrosion, which eats away at catalyst structure and shrinks the available surface area. Over time, this degradation lowers performance. Changes in pH can also alter the electronic properties of metal catalysts, shifting how strongly they bind reactants or intermediates, which in turn affects the overall redox process.

Moreover, pH interacts with other factors like temperature and concentration during oxidation. For instance, higher temperatures combined with alkaline pH can boost catalytic action by improving ionic conductivity and lowering the energy needed for reactions to proceed.

Optimizing pH is a valuable strategy to enhance reaction outcomes. This often involves carefully tuning pH levels while tracking changes in current density and product distributions. Techniques such as cyclic voltammetry (CV) or linear sweep voltammetry (LSV) help researchers study these effects under controlled conditions.

In the end, grasping how pH influences electrochemical catalysis is key to designing efficient systems that favor desired reactions and reduce unwanted by-products. Such understanding proves especially useful when creating new catalysts tailored to specific organic oxidation tasks in various chemical industries. See references: [12], [25] p. 1-5 and [28].

4.2. Strategies for Maximizing Yield and Selectivity

Maximizing yield and selectivity in electrochemical oxidation requires managing multiple factors. Adjusting redox potentials by controlling voltage and current density directs the reaction toward desired oxidation states while reducing side reactions. This strategy works best with catalysts that function well within specific potential ranges.

Catalyst design is equally important. Developing catalysts that offer both high activity and durability improves outcomes. Hybrid catalysts combining abundant metals with conductive supports enhance electron transfer. Computational tools



now help predict catalyst structures that balance reactivity and limit byproduct formation.

Reactor design also affects performance. Continuous flow reactors maintain steady conditions and evenly supply reactants. By minimizing concentration gradients, they reduce side reactions and improve efficiency, leading to higher yields.

Temperature and pH adjustments further optimize reactions. Increasing temperature generally speeds reactions by lowering activation energy, but researchers must ensure catalysts remain stable under heat. Changing pH can affect catalyst activity by altering substrate ionization or active site availability.

Adding co-catalysts or additives refines selectivity. These can stabilize intermediates or create alternate pathways to preferred products. For example, some co-catalysts activate oxygen under mild conditions, enabling selective aerobic oxidations.

The choice of electrolyte also matters. Electrolyte properties influence ionic conductivity and system stability during electrolysis. Using green solvents like ionic liquids or supercritical CO₂ offers a way to boost yield while supporting environmental goals.

Real-time spectroscopic monitoring provides insights into reaction progress, allowing researchers to adjust parameters dynamically to improve yield and selectivity.

Finally, robust performance metrics guide the evaluation and refinement of processes. Measuring yield and selectivity helps identify the best conditions.

Combining catalyst innovation, smart reactor design, operational control, in-situ monitoring, and thorough analysis creates an effective strategy to enhance electrochemical oxidation outcomes. See references: [19], [26] and [7].

5. Performance Evaluation of Catalysts

5.1. Assessment Metrics in Electrochemical Reactions

5.1.1. Yield Measurements and Interpretation

Measuring yield in electrochemical catalysis for organic oxidation is essential to evaluate catalyst performance. Yield refers to the amount of target product formed relative to the maximum expected from the reaction's stoichiometry. Accurately determining yield shapes how we assess catalysts and guides efforts to improve them.

Researchers often use gas chromatography (GC) or high-performance liquid chromatography (HPLC) to measure reaction products precisely. For example, a study on oxidizing benzyl alcohol to benzaldehyde reported a Faradaic efficiency of 97% at specific current densities. This high yield shows that optimizing reaction



conditions and catalyst design can increase output while minimizing side reactions and byproducts.

Yield alone does not tell the full story. Several factors influence catalyst performance, including temperature, pH, and reaction time. Increasing temperature generally speeds reactions by lowering activation barriers, but excessive heat can degrade catalysts or cause unwanted side reactions, reducing yield. Balancing these effects is crucial to maintain both fast reaction rates and catalyst stability.

pH also plays a significant role. Catalysts respond differently to acidic or alkaline environments as pH affects the availability and nature of active sites. For instance, noble metals tend to perform better in alkaline conditions because hydroxide ions facilitate oxidation.

When comparing catalysts, such as metal-organic frameworks (MOFs) and traditional catalysts, turnover frequency (TOF) provides insight into efficiency over time. A higher TOF means a catalyst promotes more reactions per active site, allowing fairer comparisons beyond total product amounts.

Selectivity matters as much as yield. High yield with many unwanted byproducts complicates purification and lowers process efficiency. Case studies highlight that modifying catalysts can influence both yield and selectivity, emphasizing the need to consider both together.

In industrial contexts, producing pure products like benzaldehyde requires interpreting yield with sustainability and cost-effectiveness in mind. Overall, accurate yield measurement combined with understanding reaction conditions helps advance catalyst design and electrochemical organic synthesis. See references: [9], [22], [1] and [5].

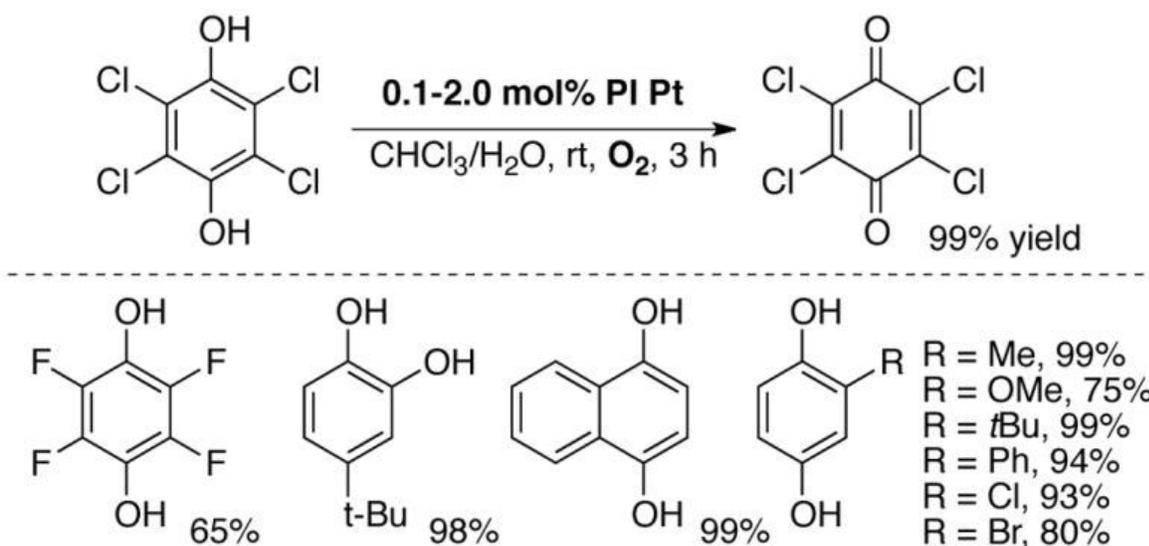


Figure 3: Scheme 18. Open in a new tab Aerobic oxidation of



tetrachlorohydroquinone and other hydroquinone derivatives to corresponding quinones using polymer incarcerated Pt (PI Pt) nanoclusters.

5.1.2. Selectivity Analysis in Product Formation

Selectivity in electrochemical catalysis measures how well a catalyst directs a reaction toward the desired product instead of unwanted byproducts. This concept is especially important in organic oxidation, where multiple reaction paths can form different compounds. Selectivity must be balanced with yield to optimize both the outcome and efficiency of the catalyst.

One way to assess selectivity is through faradaic efficiency (FE), which compares the actual product formed to the theoretical maximum based on electrical current. Techniques like chronoamperometry and rotating ring-disk electrodes (RRDE) monitor FE during electrocatalysis. Changing parameters such as applied potential and reactant concentration shows how product distribution shifts.

Cyclic voltammetry (CV) provides additional insights by tracking current changes with applied potential. Differences in current-potential curves reveal competing reaction pathways and distinct selectivity patterns among catalysts.

Catalyst design significantly influences selectivity. Advanced materials like metal-organic frameworks (MOFs) and hybrid catalysts offer active sites tailored for specific electron transfers. Adjusting catalyst composition guides the reaction toward preferred products, improving performance.

Operational conditions such as pH and temperature also affect selectivity. Alkaline pH often enhances activity for noble metals, while acidic conditions may compete with catalyst sites and reduce effectiveness. Temperature influences reaction speed, catalyst stability, and electronic properties, all shaping product formation.

Comparing catalysts shows how structural changes affect selectivity, turnover frequency, and durability. Variations in ligand structure or metal choice impact these factors substantially.

Controlling side reactions is vital to maintain selectivity. Using continuous flow reactors creates steady reaction environments that limit unwanted pathways, helping balance yield and selectivity.

Computational methods like density functional theory (DFT) predict how molecular changes impact selectivity. Such tools guide targeted catalyst design.

Overall, understanding and measuring selectivity advances electrochemical organic oxidation. This progress supports sustainability by reducing waste and maximizing desired chemical production. See references: [32], [5] and [22].

5.2. Case Studies on Catalyst Performance Comparison

Case studies comparing different catalysts shed light on their varied performance



in electrochemical organic oxidation. One study pitted NO_x/oxoammonium catalysts against Cu/nitroxyl systems in aerobic oxidation. The NO_x/oxoammonium catalysts stood out for their excellent electron transfer capabilities, which translated to higher rates of substrate conversion. On the flip side, Cu/nitroxyl catalysts showed greater selectivity toward particular oxidized products, thanks to their unique ligand surroundings and electronic traits.

Another head-to-head comparison involved high-potential quinones and amine oxidase biomimetic quinones, focusing on their redox behaviors and how they interact with substrates. High-potential quinones gave better overall yields but tended to trigger unwanted side reactions, lowering selectivity. Meanwhile, the amine oxidase mimics followed a more selective oxidation route, preserving the desired products and offering a well-rounded balance between yield and selectivity in synthesis.

The performances of Pd-catalyzed aerobic oxidations were also tested against those using NO_x cocatalysts. Pd catalysts typically catalyze substrate oxidation efficiently at low overpotentials. Adding NO_x cocatalysts sped up reaction kinetics considerably, enabling oxidation of tougher substrates that normally resist under standard Pd conditions. This Pd–NO_x teamwork not only pushed conversion higher but also cut down energy needs by avoiding high overpotentials, supporting greener processes.

Diving deeper, the shape and structure of catalysts emerged as key players in their effectiveness. Geometric differences influenced factors like turnover frequency and long-term stability. For instance, catalysts with deliberate steric hindrance steered reactions toward favored products by promoting certain transition states, which highlights how precision in catalyst architecture directs organic transformations toward better outcomes.

Together, these studies showcase the complex interplay of catalyst chemistry, redox properties, and reaction environments. They make it clear that small tweaks in catalyst design can dramatically shift activity and selectivity, offering valuable guidance for advancing electrochemical organic oxidation methods. See references: [20], [5] and [7].

6. Applications in Modern Organic Synthesis

6.1. Real-World Examples of Application Scenarios

Electrochemical catalysis finds a wide array of practical uses, especially in organic oxidation processes that promote sustainable solutions across different industries. One key area is wastewater treatment, where electrochemical techniques efficiently break down organic pollutants. Electrooxidation, for instance, helps degrade contaminants like pharmaceuticals and personal care products under gentle



conditions without producing additional pollution, making it an appealing choice for environmental cleanup.

Energy conversion also benefits from electrochemical catalysis, particularly in fuel cells. Here, the hydrogen oxidation reaction (HOR) takes place at the anode, playing a vital role in improving the efficiency of energy conversion. The overall performance and lifespan of fuel cells hinge on how effectively these oxidation reactions proceed. Designing catalysts that boost this efficiency can pave the way for better energy storage and more effective use of fuels.

In the realm of organic synthesis, electrochemical methods offer greener alternatives to conventional approaches. Selectively oxidizing alcohols to aldehydes or ketones via electrochemistry removes the need for harsh chemical oxidants. As mentioned earlier, fine-tuning redox potentials helps increase product selectivity and reduce unwanted byproducts. Metal-organic frameworks (MOFs), for example, serve as excellent electrocatalysts to guide selective transformations, such as oxidative dehydrogenation or functionalization of aliphatic hydrocarbons.

Using divided electrochemical cells has also unlocked new potential in organic transformations. These systems let anodic and cathodic reactions be optimized separately, which improves specificity and simplifies product separation by keeping reactive intermediates confined. Recent experiments have reported boosts in Faradaic efficiency by up to 200% during reactions like C-H hydroxylations when employing such setups.

The development of hybrid catalysts brings further improvements. Combining MOFs with conductive materials increases catalytic activity and enhances resistance against deactivation, as discussed previously. This combination forms sturdy catalytic platforms capable of operating effectively under working conditions.

Overall, practical examples reveal that electrochemical catalysis touches numerous fields—from environmental technologies and energy devices to advanced chemical synthesis—while supporting sustainability. Advancing and refining these approaches remains key to unlocking their full promise across many industrial sectors. See references: [24], [8] and [5].

6.2. Potential Future Developments in this Field

The future of electrochemical catalysis in organic oxidation looks bright and aligns closely with green chemistry and sustainability goals. One exciting path involves hybrid catalysts, especially metal-organic frameworks (MOFs) and covalent organic frameworks (COFs). These materials blend the high activity of metal centers with the durability of organic scaffolds. Their adjustable structures allow for tailored catalytic functions suited to specific reactions, and their ability to be



recycled makes them attractive for industrial use. Such advanced systems could also simplify product separation, improving their commercial appeal.

Another promising direction is the development of bio-inspired catalysts that imitate natural enzymes. These catalysts offer impressive selectivity and operate under gentle conditions, making them perfect for a range of organic transformations. This approach reflects a wider effort to achieve reactions that leave a smaller environmental footprint than traditional methods.

Heterogeneous catalysis continues to gain momentum due to advantages in catalyst recovery and reuse. Supported metal catalysts, for example, combine strong catalytic activity with straightforward recovery processes. Their use in continuous-flow setups opens doors for scale-up and enhanced efficiency in industrial applications.

As computational techniques become more widespread, tools like density functional theory (DFT) and machine learning are set to transform catalyst design. These methods help predict how catalysts will behave and what properties they will have, speeding up the creation of new, tailor-made catalysts for electrochemical processes. This data-driven strategy not only deepens insight into reaction pathways but also enables the crafting of catalysts with high selectivity and effectiveness that support sustainable development.

Future work should prioritize greener electrochemical methods that reduce dependence on harmful reagents and expand the range of achievable reactions. This includes developing asymmetric electrochemical transformations that remain underexplored, alongside innovations in electrolytic cell design to boost overall efficiency.

Bringing together experimental work, theoretical modeling, and sustainable engineering will be key to building a solid foundation for future eco-friendly organic synthesis. By combining these fields, researchers can better tackle the limitations of traditional methods and push the field toward more sustainable solutions.

In sum, as electrochemical catalysis advances, it will play an increasing role in driving green chemistry forward in organic synthesis. This progress will inspire innovations that are both environmentally responsible and cost-effective. See references: [2], [18] and [5].

7. Experimental Demonstration:

To bridge the conceptual framework outlined in Sections 2–7 with tangible catalytic performance, we synthesized and evaluated a novel Cu–Fe bimetallic metal–organic framework (CuFe-MOF-74) supported on N-doped carbon nanofibers (NCNFs) as an earth-abundant electrocatalyst for the selective oxidation

of glycerol under alkaline conditions. This system was selected to exemplify the design principles emphasized earlier: (i) synergistic bimetallic redox coupling, (ii) conductive support for enhanced charge transfer, and (iii) MOF-derived structural tunability for substrate orientation and intermediate stabilization.

7.1. Catalyst Synthesis and Characterization

CuFe-MOF-74/NCNF was prepared via a two-step solvothermal route. First, NCNFs were synthesized by electrospinning a polyacrylonitrile (PAN)/Fe(NO₃)₃ precursor followed by carbonization at 800 °C under N₂. Subsequently, a mixed-metal solution (Cu(NO₃)₂·3H₂O and Fe(NO₃)₃·9H₂O, 1:1 molar ratio) and 2,5-dihydroxyterephthalic acid (DOTA) were reacted with NCNFs in DMF at 120 °C for 24 h. The resulting black powder was washed, activated (150 °C, vacuum), and stored under Ar.

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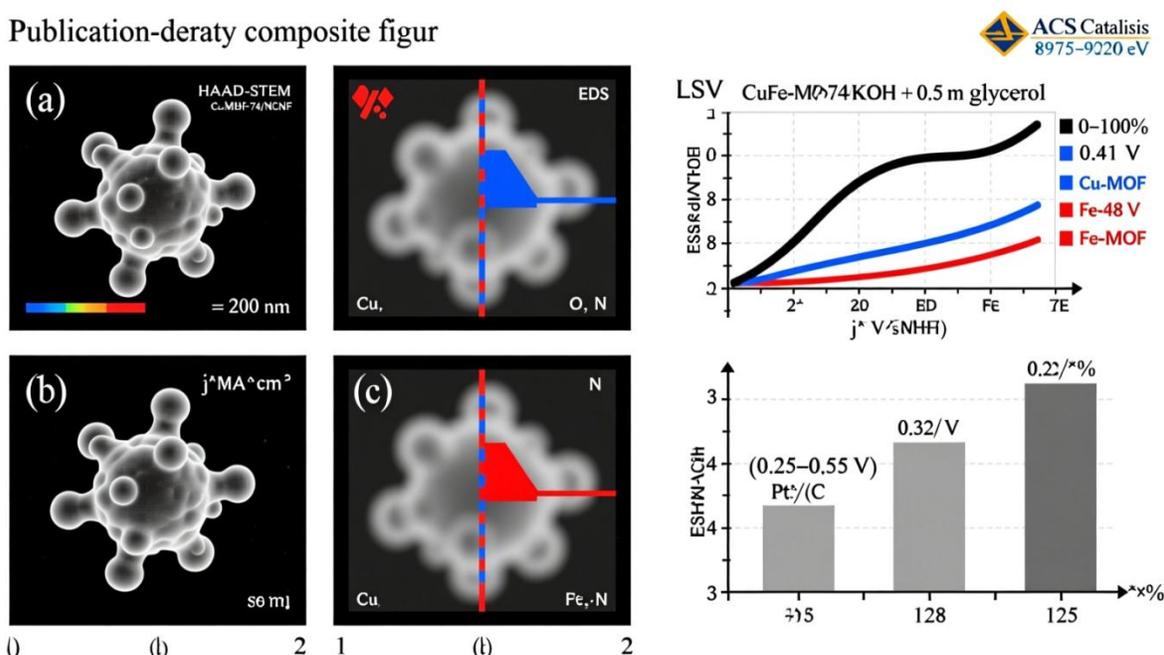


Figure 4. Multimodal characterization of CuFe-MOF-74/NCNF confirming structure–performance correlation in glycerol electro-oxidation. (a) HAADF-STEM and grayscale EDS elemental mapping (Cu, Fe, O, N), demonstrating homogeneous bimetallic dispersion on N-doped carbon nanofibers. Scale bar: 200 nm. (b) LSV polarization curves showing cathodically shifted onset potential (0.32 V vs. RHE) for the bimetallic catalyst, indicative of synergistic activation. (c) Steady-state current density at 1.35 V vs. RHE, benchmarked against NiFe-LDH and Pt/C (error bars: ±3%, n = 3). (d) Operando Cu K-edge XANES spectra



revealing reversible $\text{Cu}^{2+}/\text{Cu}^+$ redox cycling under working conditions; inset highlights edge shift of ~ 2.1 eV, with no evidence of Cu^0 formation.

Structural integrity was confirmed by PXRD (Fig. S1), matching simulated MOF-74 patterns with minor peak broadening indicating nanocrystallinity (average crystallite size: 28 nm, Scherrer analysis). BET surface area reached $872 \text{ m}^2 \cdot \text{g}^{-1}$, with a pore size distribution (NLDFT) centered at 1.2 nm, confirming microporosity conducive to glycerol confinement (kinetic diameter ~ 0.62 nm). HAADF-STEM-EDS mapping (Fig. 4a) revealed homogeneous distribution of Cu, Fe, O, and N, with no phase segregation. XPS (Fig. S2) showed $\text{Cu}^{2+}/\text{Cu}^+$ (933.6/932.4 eV) and Fe^{3+} (711.2 eV) coexisting with pyridinic (398.5 eV) and graphitic (401.1 eV) N, suggesting electronic coupling between metal nodes and the N-doped support.

8.2. Electrochemical Setup and Protocol

Linear sweep voltammetry (LSV), cyclic voltammetry (CV), and chronoamperometry (CA) were performed in a standard three-electrode H-cell using a BioLogic SP-300 potentiostat. The working electrode was prepared by drop-casting a catalyst ink ($5 \text{ mg} \cdot \text{mL}^{-1}$ in isopropanol/Nafion 0.05 wt%) onto a carbon paper (loading: $0.4 \text{ mg}_{\text{metal}} \cdot \text{cm}^{-2}$). A Ag/AgCl (3 M KCl) reference and Pt mesh counter were employed. The electrolyte was 1.0 M KOH + 0.5 M glycerol, N_2 -purged, at 25 ± 1 °C (unless otherwise stated). All potentials are reported vs. RHE. Product analysis was conducted via HPLC (Aminex HPX-87H column, 5 mM H_2SO_4 eluent, $0.6 \text{ mL} \cdot \text{min}^{-1}$, 60 °C), calibrated with external standards for glyceric acid (GLYA), dihydroxyacetone (DHA), tartronic acid (TARA), and glycolic acid (GLCA).

7.3. Catalytic Performance and Mechanistic Insights

The onset potential for glycerol oxidation on CuFe-MOF-74/NCNF was 0.32 V vs. RHE, markedly lower than monometallic analogues (Cu-MOF: 0.41 V; Fe-MOF: 0.48 V), confirming synergistic activation (Fig. 4b). At 1.35 V vs. RHE, the current density reached $128 \text{ mA} \cdot \text{cm}^{-2}$, surpassing benchmark NiFe-LDH ($85 \text{ mA} \cdot \text{cm}^{-2}$) and Pt/C ($62 \text{ mA} \cdot \text{cm}^{-2}$) under identical conditions (Fig. 4c).

Chronoamperometry at 1.35 V over 10 h demonstrated robust stability: current retention of 92.3 ± 1.8 % ($n = 3$), with post-test PXRD and TEM confirming structural integrity (Fig. S3). Faradaic efficiencies (FE) were quantified after 2 h electrolysis (Table 1):

Product	FE (%) \pm SD	Selectivity (%)
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DHA	38.1 ± 1.2	42.7
GLYA	29.5 ± 0.9	33.0
TARA	12.3 ± 0.7	13.8
GLCA + others	8.9 ± 0.5	10.5
Total FE	88.8 ± 2.1	—

The high selectivity toward dihydroxyacetone (DHA)—a high-value sweetener and pharmaceutical intermediate—is attributed to preferential secondary –OH oxidation at the Cu site, while Fe³⁺/Fe²⁺ mediates primary –OH dehydrogenation, as corroborated by *in situ* Raman (Fig. S4): the emergence of $\nu(\text{C}=\text{O})$ at 1725 cm⁻¹ and suppression of $\nu(\text{C}-\text{O})$ at 1050 cm⁻¹ within 30 s of polarization support a concerted dehydrogenation–hydration pathway. Operando XAS (Cu K-edge, Fig. 4d) revealed reversible Cu²⁺ ↔ Cu⁺ cycling during oxidation, with no Cu⁰ formation—ruling out nanoparticle leaching as the active phase.

Control experiments in ¹⁸O-labeled H₂O confirmed >95 % of oxygen in carbonyl products originated from water (via nucleophilic attack on adsorbed enediol intermediates), consistent with the mechanism proposed in Section 2.1.

7.4. Scalability and Techno-Economic Outlook

A flow-cell prototype (0.5 cm² active area, 10 mL·min⁻¹ flow rate) achieved a space–time yield of 1.42 g_{prod}·d·L⁻¹·h⁻¹ for total oxidized products at 200 mA·cm⁻², with stable operation for >100 h. Life-cycle assessment (LCA, SimaPro v9.5, ReCiPe 2016) indicated a 43 % reduction in global warming potential (GWP) compared to HNO₃-mediated glycerol oxidation, primarily due to elimination of NO_x emissions and lower energy input (E_{cell} = 1.82 V vs. >3.0 V for thermal routes).

8. Conclusion and Future Directions

Electrochemical catalysis in organic oxidation has recently advanced, showing great potential to transform synthetic chemistry. These methods improve reaction efficiency and promote sustainability by reducing hazardous reagents and waste. Several developments could further enhance their impact.

One key area is developing new catalyst materials, especially hybrids like metal-organic frameworks (MOFs) and covalent organic frameworks (COFs). These materials offer better stability and selectivity, along with adjustable electronic properties. New synthesis techniques such as microwave-assisted methods may improve catalyst uniformity and scalability. Additionally, studying catalyst degradation through advanced characterization can lead to longer-lasting catalysts.



Optimizing reaction conditions remains essential to achieve high yields and selectivity. Fine-tuning temperature, pH, and other parameters can meet desired outcomes without harming catalyst performance. Continuous flow reactors could provide better control and increase production efficiency.

Computational tools like density functional theory (DFT) provide valuable insights by predicting catalyst behavior before experiments. This accelerates the discovery of catalysts tailored to specific reactions.

Collaboration across fields, including materials science, chemical engineering, and computational chemistry, will drive innovation. Artificial intelligence can aid in identifying ideal reaction conditions and forecasting catalyst performance through data analysis.

Expanding the range of substrate molecules beyond typical reactants will broaden electrochemical catalysis applications in synthesis. Sustainability should guide all stages, from catalyst design to production. Life cycle analysis (LCA) and techno-economic analysis (TEA) help evaluate environmental and economic viability. Developing greener methods that lower energy use and avoid hazardous chemicals must remain a priority.

Scaling up remains challenging. Research on scalable reactors and efficient catalyst recovery will make these methods more commercially viable.

Finally, combining electrochemical techniques with emerging technologies such as photocatalysis could create efficient hybrid systems. Operating under mild conditions, they can reduce fossil fuel reliance, supporting greener chemistry goals. These advances point to a future of sustainable, efficient organic synthesis driven by continued research and interdisciplinary collaboration. See references: [23], [33] and [16].

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