Perspective

Carbon Future

Electrocatalytic glycerol valorization: From catalyst design to integrated systems

Kaiwei Meng[1,2], Lin Dong[1,2], Wenjun Zhang[1,2] ✉, Yanqin Wang[3], and Zupeng Chen[1,2] ✉

[1] *State Key Laboratory for Development and Utilization of Forest Food Resources, Nanjing Forestry University, Nanjing 210037, China*
[2] *Jiangsu Co-Innovation Center of Efficient Processing and Utilization of Forest Resources, International Innovation Center for Forest Chemicals and Materials, Nanjing Forestry University, Nanjing 210037, China*
[3] *Key Laboratory for Advanced Materials and Joint International Research Laboratory of Precision Chemistry and Molecular Engineering, Feringa Nobel Prize Scientist Joint Research Center, Research Institute of Industrial Catalysis, School of Chemistry and Molecular Engineering, East China University of Science and Technology, Shanghai 200237, China*

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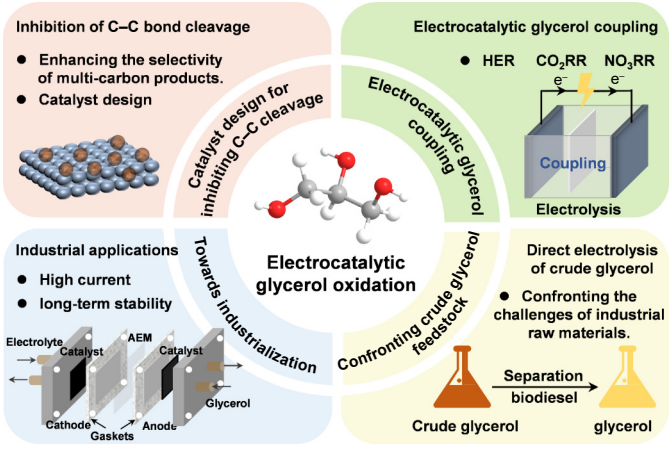
ABSTRACT: The electrochemical glycerol oxidation reaction (GOR) has emerged as a sustainable pathway for transforming biodiesel byproducts into valuable resources, while addressing the growing demand for renewable energy and green chemical production. This perspective provides a comprehensive examination of recent advances in GOR technology, with a particular emphasis on catalyst design, reaction mechanisms, and system integration strategies. It highlights key challenges related to selectivity, stability, and scalability, which are critical for advancing the technology toward industrial applications. We explore how both noble metals (e.g., Pt, Au, and Pd) and non-noble metal alternatives (e.g., Ni, Co, and Cu) can be engineered through various methods, such as facet control, single-atom incorporation, and dynamic potential modulation, to selectively preserve C–C bonds and direct selectivity toward valuable multi-carbon products. Beyond standalone GOR processes, the integration with cathodic reactions presents new opportunities for system-level optimization. We discuss the benefits of coupling GOR with cathodic reactions, such as hydrogen evolution reaction (HER), carbon dioxide reduction reaction (CO_2RR), and nitrate reduction reaction (NO_3RR), which not only reduce the energy consumption but also enable the co-production of high-value chemicals and clean fuels. Despite the significant progress in GOR technology, several critical challenges remain for its industrial implementation, including mass transfer limitations, tolerance to crude glycerol, and long-term stability. This perspective provides a roadmap for addressing these challenges, proposing targeted solutions ranging from advanced membrane-electrode assemblies to integrated techno-economic assessments. Ultimately, this work aims to guide the field beyond a focus on catalyst activity, toward a holistic paradigm that prioritizes system-level integration and economic viability, thereby accelerating the industrialization of GOR technology.



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1 Introduction

The growing depletion of fossil fuel resources has highlighted the urgency of identifying sustainable alternatives, with biomass emerging as a pivotal renewable carbon source due to its abundance and environmental benefits[1]. Among various biomass-derived molecules, glycerol stands out as a major byproduct of biodiesel production, representing approximately 10 wt.%–15 wt.% of the total output. The rapid expansion of the biodiesel industry has led to an overabundance of glycerol, making its high-value conversion a critical priority for advancing biomass refining and establishing circular economy systems[2].

As a polyhydric small molecular compound, glycerol can be electrochemically upgraded under mild conditions using renewable electricity to produce a spectrum of high-value chemicals, significantly increasing its market value compared to raw glycerol (~ \$0.6/kg). These value-added products include: dihydroxyacetone

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✉Address correspondence to Wenjun Zhang, zhangwj@njfu.edu.cn; Zupeng Chen, czp@njfu.edu.cn

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(DHA, ~ $15/kg), glyceric acid (GLA, ~ $25/kg), lactic acid (LA, ~ $3/kg), tartaric acid (TA, ~ $6/kg), glycolic acid (GA, ~ $5/kg), and formic acid (FA, ~ $1.5/kg)[3]. Importantly, the glycerol oxidation reaction (GOR) offers distinct advantages over conventional anodic oxygen evolution reaction (OER), featuring lower thermodynamic overpotential and the unique ability to couple with valuable cathodic processes such as hydrogen evolution reaction (HER)[4], carbon dioxide reduction reaction (CO_2RR)[5], and nitrate reduction reaction (NO_3RR)[6]. This dual-functionality enables an integrated system for simultaneous energy conversion and resource utilization.

Despite the notable advancements in electrocatalytic GOR research, its industrial implementation faces persistent challenges across multiple scales. At the mechanistic level, a critical limitation lies in the inherent selectivity bias of existing catalytic systems toward C–C bond cleavage, which predominantly generates low-value C_1 products (e.g., FA) while leaving the selective pathways for high-value multi-carbon products insufficiently understood[7]. Recent studies have shed light on this challenge: For instance, Titirici and colleagues demonstrated that in PtNi bimetallic catalysts, a high Ni content electronically modifies the d-band center to weaken C–C bonds, significantly enhancing their cleavage[8]. Similarly, Kim et al. revealed that in non-precious CuCo oxides, the strong oxygen affinity of active sites directly facilitates C–C bond scission, achieving a remarkable formic acid selectivity of 90% through density functional theory (DFT)-verified pathways[9]. These findings across both noble and non-noble catalytic systems reveal a fundamental thermodynamic preference for C–C bond cleavage, which currently limits the selective production of more valuable multi-carbon products. On the process development side, there are notable deficiencies in system energy efficiency optimization and product separation/purification technologies, particularly in handling industrial-grade crude glycerol containing complex impurities (e.g., salts, water, and organic byproducts)[10]. Engineering validation also requires a deeper investigation into critical industrialization parameters, including catalyst inactivation mechanisms and long-term stability under high-current-density operation.

This perspective provides a comprehensive examination across four critical dimensions: (i) fundamental molecular mechanisms governing selective C–C bond preservation and corresponding rational catalyst design principles, (ii) innovative system integration strategies for reaction coupling and energy efficiency maximization, (iii) practical industrial implementation pathways enhanced by rigorous techno-economic assessments, and (iv) development and large-scale validation of direct crude glycerol conversion technologies. By analyzing current research advancements, fundamental limitations, and transformative opportunities in electrocatalytic GOR, this work establishes a foundational framework to bridge fundamental research with industrial application. It offers both theoretical insights and practical guidelines for developing sustainable biomass refining platforms, ultimately contributing to the advancement of circular biorefineries and sustainable chemical manufacturing.

2 GOR for value-added chemical products

Glycerol, a trihydroxy alcohol, exhibits exceptional chemical versatility due to the unique spatial arrangement of three hydroxyl groups within the molecular structure. Through controlled electrochemical oxidation, glycerol can be selectively converted into a diverse range of high-value platform chemicals with tunable carbon chain lengths: Partial oxidation preserves the carbon backbone to yield valuable C_3 compounds, while complete oxidation induces C–C bond cleavage to generate C_1 and C_2 molecules (**Fig. 1**). Remarkably, these oxidation products demonstrate substantial commercial value, with market prices

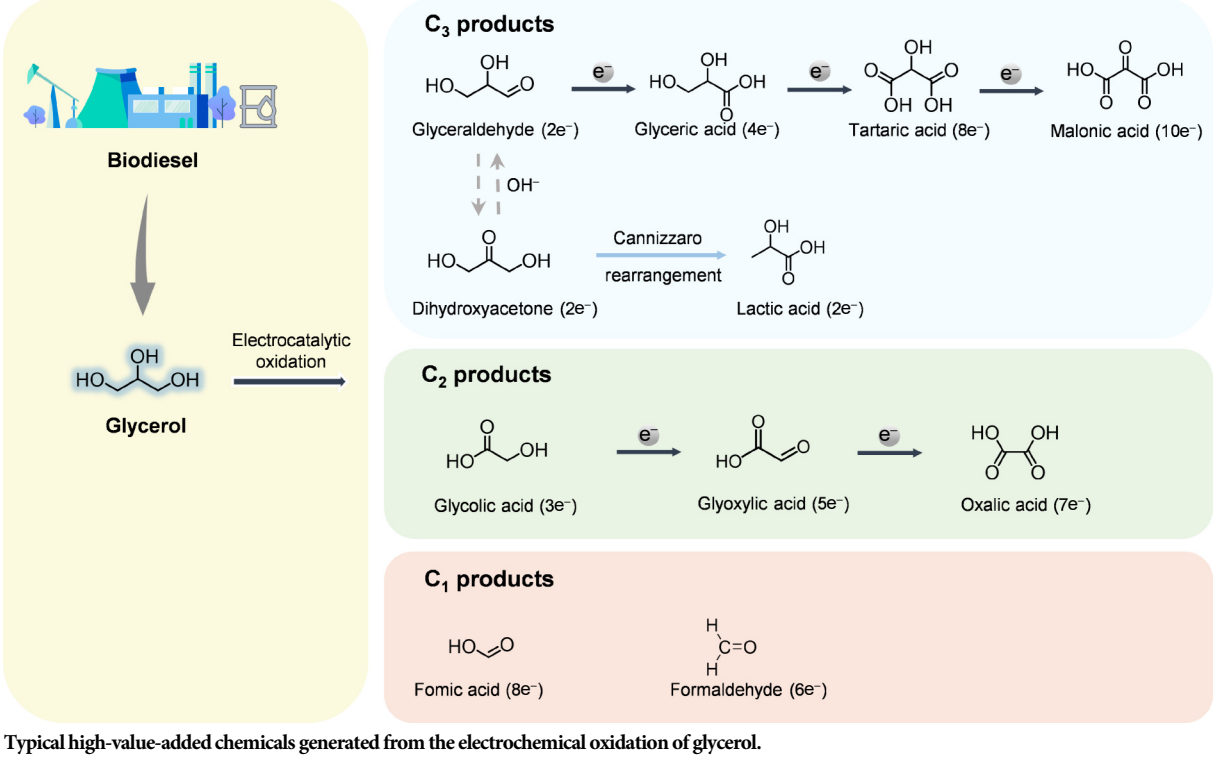

Fig. 1. Typical high-value-added chemicals generated from the electrochemical oxidation of glycerol.

typically 20–4000 times higher than raw glycerol[11]. This versatile conversion network enables the production of multiple valuable products from a single renewable feedstock, thereby significantly enhancing glycerol's economic value while establishing a sustainable carbon source for bio-based fine chemicals, pharmaceutical intermediates, and environmentally friendly materials[12].

Among C_3 products, glyceraldehyde (GLAD) is obtained through the two-electron oxidation of the primary hydroxyl group, while DHA is generated via the oxidation of the secondary hydroxyl group with the transfer of two electrons[13, 14]. GLAD and DHA are isomers that can be interconverted under alkaline conditions. DHA serves as a key component in tanning agents and is an important precursor for bio-based 1,3-propanediol[14]. Further oxidation of GLAD yields GLA ($4e^-$ oxidation product)[15], which can be continuously oxidized to form TA ($8e^-$ oxidation product)[16]. Tartaric acid can then be oxidized to unstable mesoxalic acid (MA $10e^-$ oxidation product)[17]. Lactic acid (LA), a two-electron oxidation product, is obtained primarily through the Cannizzaro rearrangement reaction of DHA and is a raw material for bio-based polylactic acid (PLA)[18].

In terms of C_2 products, GA is formed by C–C bond cleavage of glycerol, with the number of electrons transferred generally recognized as three[19]. As an important pharmaceutical intermediate, GA can be used in the synthesis of various drugs and as a raw material for preparing polyglycolic acid (PGA). Further oxidation of GA produces glyoxylic acid (GXA, a five-electron oxidation product), which is easily oxidized in air and widely used in the cosmetics industry due to its whitening effect[20]. In addition, oxalic acid (OA), formed by deeper oxidation of glycerol (a seven-electron oxidation product), plays an important role in rare earth element separation and functions as a key auxiliary for bleaching and dyeing in the textile industry[21].

In the glycerol oxidation pathway, complete cleavage of the C–C bonds can generate FA. Each molecule of glycerol can yield up to three molecules of FA, with the transfer of eight electrons. FA has important applications in the rubber, pharmaceutical, dye, leather industries, and the hydrogen fuel cell field[22]. Glycerol can be catalytically converted to formaldehyde (HCHO, a six-electron oxidation product). However, the practical application of formaldehyde is significantly limited due to its high toxicity[23]. The economic feasibility and environmental sustainability of GOR are fundamentally dependent on the selective formation of multi-carbon products. Market valuation analysis demonstrates that C_3 products, such as DHA and GLA, possess substantially higher economic value compared to C_1 products like FA[24]. Atom economy considerations further highlight that preserving the carbon skeleton significantly enhances carbon utilization efficiency[25]. However, achieving selective C–C bond cleavage remains a key challenge. DFT calculations and experimental studies have collectively demonstrated that during the electrocatalytic oxidation of glycerol, the activation barrier for C–C bond cleavage is significantly lower than that for C–O bond cleavage (0.48 vs. 0.96 eV). This kinetic advantage leads to a preferential pathway for C–C bond cleavage. Notably, although the thermodynamic barrier for oxidation via stable pathways of key intermediates, such as GLAD, is relatively low (ΔG = −0.26 eV), the activation barrier for further deep oxidation to form C_1 products like FA is even lower. This competition between kinetic and thermodynamic factors explains the intrinsic mechanism underlying the selective formation of C_1 products[26, 27]. Furthermore, the strong adsorption of deeply oxidized intermediates such as *CHO and *COOH on catalyst surfaces exacerbates over-oxidation phenomena. These dual effects, the synergistic action between low-barrier C–C cleavage pathways and strongly adsorbed intermediates, readily drive the reaction toward excessive oxidation[28]. Therefore, precisely modulating the electronic structure and surface properties of catalysts to balance intermediate adsorption strength with reaction barriers is a critical design principle for achieving efficient conversion to high-value-added products.

3 Taming C–C bond cleavage: Atomic-level design strategies for selective glycerol oxidation

Noble metals such as Pt, Au, and Pd have proven to be highly effective in the GOR, particularly for the selective production of high-value multi-carbon products[28]. Their superior catalytic performance stems from their unique electronic configurations and surface characteristics, which provide several key advantages: (i) excellent structural stability, ensuring prolonged catalytic activity[29]; (ii) remarkably low reaction overpotentials, which enable energy-efficient conversions[30]; (iii) precisely tunable surface electronic states, allowing for controlled reaction selectivity[31]; and (iv) robust tolerance to varying reaction conditions, ensuring consistent performance across a range of operational parameters[30–32]. The distinctive d-electron properties of these noble metal surfaces facilitate optimal interactions with glycerol molecules in electrochemical environments. By carefully controlling intermediate adsorption geometries and electron transfer dynamics, these catalysts can achieve highly selective transformations, even under relatively mild oxidation potentials, ensuring the preservation of valuable C–C bonds[29]. This combination of electronic and surface properties positions noble metals as an exceptional platform for steering glycerol conversion toward desired multi-carbon products with enhanced economic value. The ability to precisely control these fundamental interactions at the molecular level represents a critical advantage in developing efficient GOR systems for sustainable chemical production.

3.1 Pt-based catalysts in GOR research: Enhancing performance and overcoming challenges

Pt-based catalysts have established themselves as benchmark systems in GOR due to their outstanding catalytic activity and broad pH tolerance[33]. The foundational work by Koper et al. employed experimental and theoretical approaches to uncover facet-dependent reaction mechanisms on Pt electrodes in acidic media. Their systematic investigation demonstrated that although Pt (111) surfaces produced a variety of products (e.g., GLAD, GLA, and DHA), Pt (100) facets exhibited remarkable selectivity for GLAD production[34]. DFT calculations provided molecular-level insights, showing that different adsorption configurations of dehydrogenated intermediates, such as enediol formation via dual Pt–C single bonds on Pt (111) versus selective glyceraldehyde production through Pt=C double bonds on Pt (100), underlie the observed structure–activity relationships. Despite these advancements, conventional Pt catalysts encounter significant challenges, including susceptibility to poisoning by strongly adsorbed intermediates (particularly CO) and deteriorating C_3 selectivity at industrially relevant current densities[35]. To overcome these limitations, innovative strategies such as bimetallic modifications have shown remarkable effectiveness. For instance, Koper et al. demonstrated that Bi adatoms could not only mitigate CO poisoning but also

promote glyceraldehyde isomerization to DHA through specific interactions with Pt (111)-stabilized enediol intermediates[36]. Similar improvements have been achieved via the use of Sn and Sb modification, where secondary metals modulate Pt's electronic structure to optimize glycerol adsorption and suppress C–C cleavage[37, 38]. Alloying strategies have yielded equally impressive results, exemplified by Shi et al.'s development of self-supported honeycomb-like PtAu alloy catalysts on Ni foam (hp-PtAu/NF). These advanced catalysts demonstrated exceptional performance, achieving a lactic acid selectivity of 70% at 0.4 V vs. reversible hydrogen electrode (RHE), with total C_3 selectivity reaching 95%. Remarkably, they maintained a C_3 selectivity of 62% under industrial conditions (921.5 mA·cm^{-2} at 1.6 V vs. RHE), with performance attributed to Au-enhanced hydroxyl species adsorption and favorable mono-carbon binding modes that minimized C–C scission (Fig. 2a)[39]. A particularly innovative approach was developed by Zou et al., who introduced a pulsed potential electrolysis strategy to address noble metal catalyst deactivation. Comprehensive characterization using *in situ* Fourier transform infrared (FTIR) spectrometer, quasi-*in situ* X-ray photoelectron spectroscopy (XPS), and finite element simulations revealed that periodic potential modulation precisely controls catalyst oxidation states and surface microenvironments. This strategy prevents toxic intermediate accumulation while maintaining optimal OH adsorbate and glycerol coverage, thereby increasing the GLA selectivity from 37.8% to 81.8% at 0.7 V vs. RHE compared to constant potential operation (Figs. 2b and 2c)[40]. These advancements collectively establish a comprehensive framework for next-generation GOR catalyst design, where the facet control, atomic modification, alloying strategies, and innovative potential modulation techniques synergistically address stability, selectivity, and scalability challenges. The integration of pulsed potential approaches with advanced material designs represents a particularly promising direction, offering dynamic control over catalyst surface states while maintaining structural advantages. Such rationally designed catalytic systems provide a reliable pathway toward the practical implementation of glycerol electrocatalytic oxidation technology, integrating fundamental mechanistic understanding with engineering solutions for sustainable chemical production. Continued progress in this field will focus on optimizing these combined approaches for industrial-scale applications while further elucidating structure-performance relationships under realistic operating conditions.

3.2 Au-based catalysts in GOR research: Enhancing performance and overcoming challenges

Au-based catalysts have emerged as a promising class of materials for GOR due to their distinctive advantages: exceptional C–C bond preservation capability, superior selectivity toward multi-carbon products, optimal hydroxyl adsorption energy for intermediate stabilization, and remarkable corrosion resistance[41]. Recent advancements in Au-based catalyst design have yielded significant performance improvements through innovative structural and compositional engineering[42].

Fundamental studies have elucidated critical structure–performance relationships in Au catalysts. The Marshall's group established a clear correlation between particle size and facet effects, demonstrating that small-sized (≤ 4.7 nm) catalysts enriched with (110) facets exhibit enhanced activity[43]. Building on this

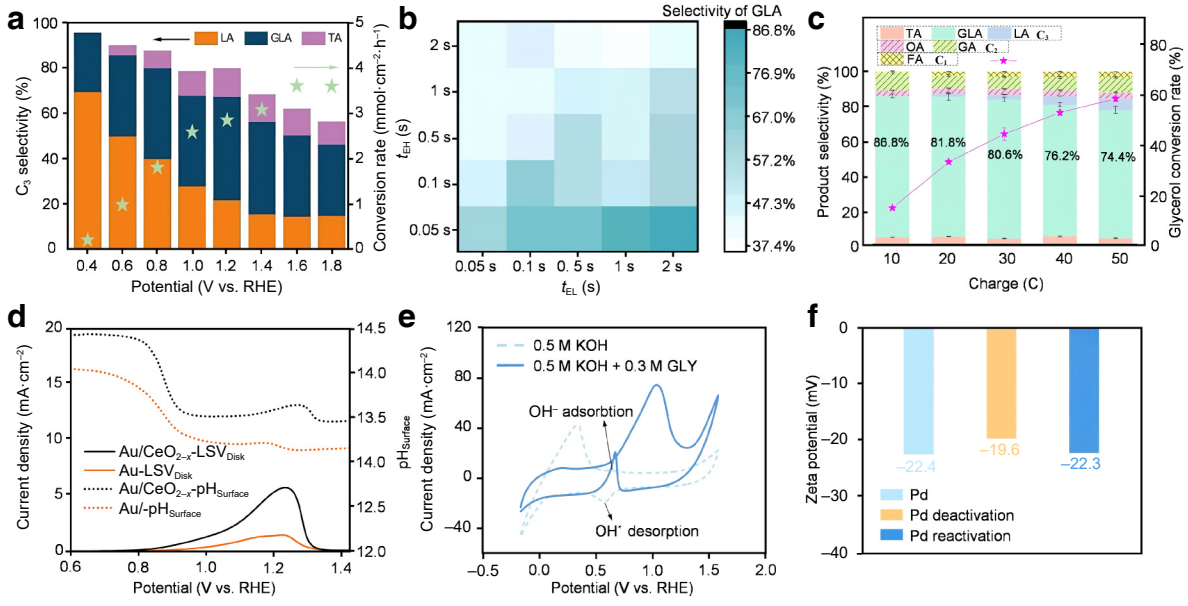

Fig. 2. GOR performance of noble metal catalysts. a, Glycerol conversion and C_3 product selectivity of the hp-PtAu/NF material at different potentials. Reproduced with permission from Ref.[39], © The Royal Society of Chemistry 2024. **b,** GLA selectivity under different pulse time conditions (PE1-PE5). All experiments were conducted in 1 M KOH with 50 mM glycerol for a total electrolysis time of 1 h. The high-potential (E_H) and low-potential (E_L) steps were fixed at 0.7 and 0.3 V vs. RHE, respectively. The duration of E_H was maintained at 0.05 s, whereas the duration of E_L (t_{EL}) was systematically varied from 2.0 to 0.05 s. **c,** Performance evaluation under optimal pulse conditions. Glycerol conversion and product distribution were achieved using the PE3 pulse mode (E_L: 0.3 V vs. RHE for 0.5 s; and E_H: 0.7 V vs. RHE for 0.05 s). Reaction conditions: 20 mM glycerol in 1 M KOH. Black percentages denote the corresponding GLA selectivity. Reproduced with permission from Ref.[40], © Chen, W. et al. 2024. **d,** Synchronous local pH changes on the surfaces of Au and Au/CeO_{2-x} according to the linear sweep voltammetry (LSV) curve in 1 M KOH with 0.5 M GLY measured by rotating ring-disk electrode (RRDE) at 1600 rpm. Reproduced with permission from Ref.[48], © Wiley-VCH GmbH 2024. **e,** Cyclic voltammetry (CV) curves of glassy carbon electrode supported Pd in 0.5 M KOH with or without 0.3 M GLY **f,** Zeta potential of Pd, deactivated Pd, and reactivated Pd in 0.5 M KOH solution. Reproduced with permission from Ref.[55], © Wiley-VCH GmbH 2024.

foundation, Jihun Oh et al. developed an electrochemical reduction-anodic treatment technique, successfully regulating the ratio of (100) to (110) facets on the Au surface. This approach increased the number of active sites by 16 times and achieved a GA selectivity of 47% at 1.0 V vs. RHE[44]. Further enhancement was achieved by Chen et al. through the surface modification with polyethyleneimine (PEI) molecules, where interfacial electronic effects and steric hindrance synergistically improved the GLA selectivity by 20%[45]. Alloying strategies have proven particularly effective in optimizing catalytic performance. Graham et al. designed hollow spherical Au_1Cu_1 bimetallic catalysts that achieved a glycerol conversion of 90% with a GA selectivity of 45% [46]. Additionally, Luo et al. developed core–shell PdAu@Ag trimetallic catalysts that delivered a current density of 3.94 mA·cm^{-2} with a DHA selectivity of 70.1% under alkaline conditions[47].

Despite these advances, Au-based catalysts still face several critical challenges, including: (i) performance degradation in acidic media, (ii) active site poisoning due to local acidification, and (iii) insufficient understanding of reactive oxygen species (ROS) regulation. Innovative solutions have emerged to address these limitations. Shi et al. developed an anode–electrolyte interface acidity regulation strategy using CeO_{2-x}-supported Au nanoparticles. The Lewis acid component in this design promoted the OH^- enrichment at the electrode surface, achieving a LA selectivity of 81% and a current density of 693 mA·cm^{-2} while mitigating local acidification (Fig. 2d)[48]. Duan et al. systematically investigated the key role of ROS species (e.g., OH* and O*) in GOR and their regulation rules, finding that OH* on Au-based catalysts exhibited optimal glycerol oxidation activity[49]. Through the microkinetic analysis, they constructed a "volcano-type" relationship curve, indicating that the glycerol oxidation activity was synergistically regulated by ΔG_{OH^*} and ΔG_{ads}. This provides new insights for overcoming bottlenecks in achieving both high activity and selectivity in biomass electrocatalytic conversion.

Based on these findings, future development of Au-based catalysts should focus on: (i) multi-scale regulation: atomically precise design of active centers, nano-scale optimization of morphological structures, and macro-scale improvement of electrode assembly; (ii) operating condition adaptability: development of new catalysts with wide pH windows and anti-poisoning properties; (iii) mechanistic research: combination of *in situ* characterization and theoretical calculations to clarify the dynamic action mechanism of ROS; and (iv) system integration: design of membrane electrode reactors to address mass transfer limitations. The catalyst design should prioritize balancing the atomic economy, energy efficiency, and process feasibility, ultimately achieving the economic viability and environmental sustainability of high-value glycerol conversion.

3.3 Pd-based catalysts in GOR research: Enhancing performance and overcoming challenges

Pd-based catalysts have emerged as a cornerstone catalytic system for GOR, demonstrating exceptional performance in alkaline media[50]. Recent advances in catalyst design have significantly enhanced both activity and selectivity toward valuable multi-carbon products while effectively suppressing undesirable C–C bond cleavage. Structural engineering approaches have yielded particularly impressive results. Shen et al. developed core–shell Pt@Pd nanocubes that achieved a remarkable current density of 3.22 mA·cm^{-2}, representing a 3.5–4.8 fold improvement over commercial catalysts[51]. Their work established clear structure–selectivity relationships, with pure Pd nanocubes exhibiting a exceptional selectivity of 61.2% toward GLAD. Furthermore, research by Wang et al. demonstrated the effectiveness of electronic modulation through nitrogen-doped carbon/graphene supports (Pd-CN_x/G). The nitrogen doping optimized Pd's electronic structure, achieving a GLA selectivity of 70% and a 10%–15% improvement in C_3 product yields[52]. Surface modification strategies have proven equally valuable. Christophe et al.'s study on Bi-modified Pd nanocubes showed simultaneous improvements in both activity and pathway selectivity[53]. Additionally, Ali et al.'s bimetallic Pt–Pd nanoporous electrode achieved impressive performance metrics, including a low onset potential of −0.54 V vs. saturated calomel electrode (SCE) and 4.2-fold current density enhancement[52]. From an application perspective, Martin et al.'s innovative three-dimensional (3D)-printed micro-electrolyzer (production cost < $5) demonstrated the industrial viability of Pd nanocubes, achieving a TA selectivity of 99% and confirming the critical role of Pd (100) facets[54]. To address the inherent instability of Pd-based catalysts, which significantly limits their practical application value, Duan et al. developed a groundbreaking asymmetric pulse potential strategy. This strategy enables long-term stable operation (> 2800 h) for glycerol electrooxidation while maintaining a GLA selectivity > 70%. This represents a dramatic improvement over conventional potentiostatic operation, where the deactivation typically occurs within 6 h[55]. Mechanistic studies revealed that the periodic potential modulation of the potential between 0.8 and 0 V vs. RHE prevents the irreversible oxidation of active Pd-OH* species to inactive PdO_x, while maintaining optimal catalytic activity. This approach has been successfully validated in flow electrolyzers, demonstrating its industrial applicability (Figs. 2e and 2f).

Looking forward, the development of Pd-based catalysts should focus on three key areas: (i) enhanced poison resistance through co-catalyst incorporation (e.g., Bi) and advanced potential modulation strategies; (ii) precise facet engineering and nanostructure design to optimize both selectivity and stability; and (iii) improved performance under industrially relevant conditions, particularly at high current densities. The integration of pulse potential techniques with advanced material designs represents a particularly promising approach to overcome current limitations. These innovations, combined with systematic reactor engineering, will be crucial for advancing GOR technology toward practical and large-scale implementation in sustainable chemical production.

3.4 Non-noble metal catalysts

Ni-, Co-, and Cu-based catalysts are widely employed in the GOR, yet they often face challenges related to over-oxidation. This phenomenon occurs because their active species are typically identified as oxyhydroxides (MOOH)[56–58], which generally require high oxidation potentials. These high potentials tend to drive excessive oxidation of glycerol towards C–C bond cleavage, resulting in the formation of C_1 products, such as FA[7]. Despite these limitations, non-noble metal catalysts offer significant cost advantages compared to noble metals like Pd, Au, and Pt, while also demonstrating superior stability and minimal issues with catalyst poisoning[59]. To address the challenge of C–C bond scission and enhance the selectivity towards high-value multi-carbon products, researchers developed various strategies. A critical aspect of these strategies is to understand structure–activity relationships, as

exemplified by Zhao et al. on MnO_2-CuO/copper foam (CF) composites. The study demonstrated that the bimetallic synergy and electron density modulation could achieve a DHA selectivity of 60% at 1.3 V vs. RHE while effectively suppressing over-oxidation[60]. The importance of crystallographic control is further highlighted by Chiang et al.'s study on Co_3O_4 (111) facets. Their study revealed that the (111) facets of Co_3O_4, which were rich in Co^{2+} active sites, improved the DHA yield by 3.5-fold compared to conventional catalysts. This highlighted the deterministic influence of surface atomic arrangement on product selectivity[61]. Additionally, their study showed that the solution pH played a significant role in product distribution, achieving both high DHA selectivity (~ 60%) and current density (3 $mA{\cdot}cm^{-2}$) under mild alkaline conditions (pH 9)[62]. Meanwhile, CuO was found to exhibit unique catalytic activity towards secondary hydroxyl oxidation of glycerol, which promoted the spontaneous conversion of DHA to GLAD and subsequently oxidation into C_2 and C_1 byproducts in strongly alkaline conditions (pH 13). Operating under moderately alkaline conditions (pH ~ 9–10) help stabilize the MOOH active phase while preventing complete oxidation.

Recent advancements in catalyst architectures have further enhanced performance. Gong et al.'s ligand engineering approach used functionalized phenanthroline. This method allowed precise regulation of C–C cleavage through Hammett parameter optimization, achieving a FA selectivity up to 92.7% and a OA selectivity of 45.3% (**Figs. 3a** and 3b)[63]. Shi et al.'s innovative single-atom Ru doping strategy in Co_3O_4, for example, achieved remarkable activity (1.16 V vs. RHE at 10 $mA{\cdot}cm^{-2}$) and selectivity (60% for GLAD, and 90% for total C_3 products) in neutral media (1 M $KHCO_3$) through the Ru-induced lattice activation and generation of electrophilic Co^{3+}-$(OH)_{ads}$ species. These species preferentially oriented the glycerol's primary hydroxyl groups (C_α-OH) toward the desired oxidation pathway (Figs. 3c and 3d)[64]. Additionally, Zheng et al.'s Ni_2Sn intermetallic system maintained a GLA selectivity of 62% for 150 h through Sn-mediated electronic modulation, showcasing the tremendous potential of atomic-level ordered design (Figs. 3e and 3f)[65].

These collective advancements underscore the importance of multiscale catalyst design, where the atomic-level electronic structure manipulation, nanoscale morphological control, and macroscopic electrode engineering work synergistically to optimize intermediate adsorption and reaction pathways. The field is evolving toward more sophisticated systems that combine fundamental mechanistic understanding with practical considerations for industrial implementation, representing a promising pathway for sustainable biomass valorization through electrocatalytic conversion. Future developments will likely focus on further elucidating structure–performance relationships under operational conditions and addressing scalability challenges for commercial applications (**Fig. 4**).

4 Synergistic coupling strategies for energy-efficient glycerol valorization

The development of GOR systems coupled with cathodic processes has brought about a transformative advancement in electrocatalytic technology. This approach addresses the inherent limitations of conventional anodic processes in terms of both energy efficiency and economic viability. The fundamental thermodynamic advantage of this approach lies in the significantly lower standard oxidation potential of GOR (−0.003 V vs. RHE) compared to the OER (1.23 V vs. RHE), as demonstrated by recent studies[66]. This substantial potential difference provides a compelling rationale for coupling GOR with various cathodic reduction reactions, such as

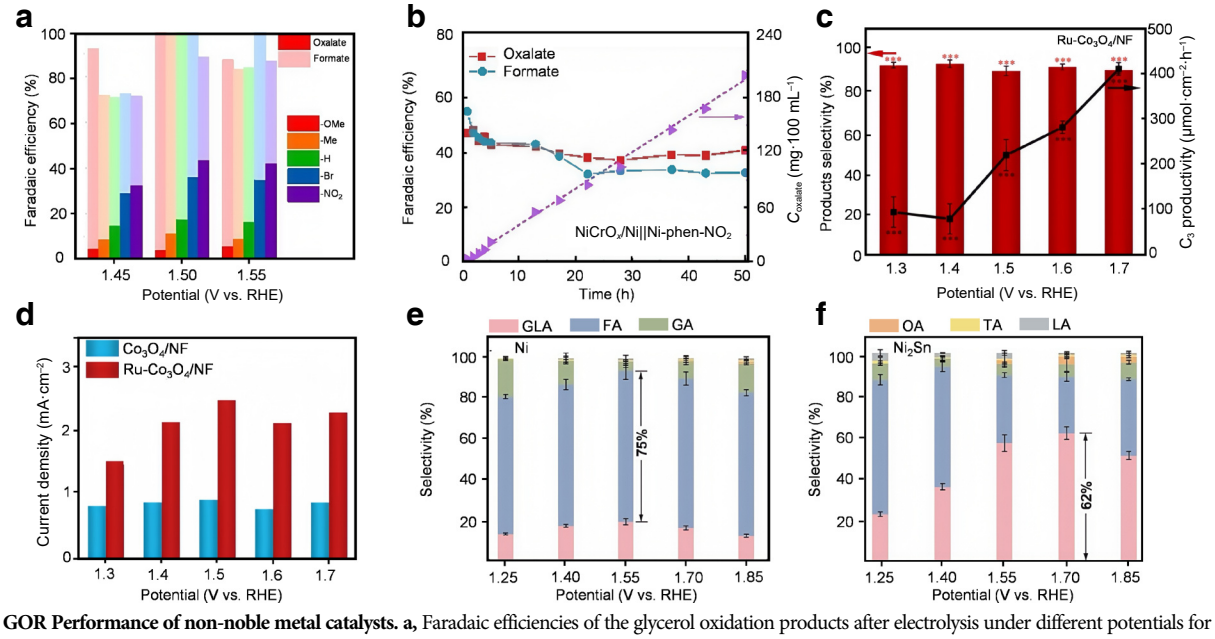

Fig. 3. GOR Performance of non-noble metal catalysts. a, Faradaic efficiencies of the glycerol oxidation products after electrolysis under different potentials for 30 min. **b,** The FEs of OA and FA and the total oxalate concentration after 1.65 V electrolysis in a 500 mL reactor with 2 M KOH + 0.1 M crude glycerol. Reproduced with permission from Ref.[63], © Wiley-VCH GmbH 2023. **c,** The total C_3 product selectivity and C_3 product productivity of Ru-Co_3O_4 /NF. Data are presented as mean ± standard deviation (SD). Error bars (SD) represent the standard deviation of three separate measurements. **d,** The ratios of path C_α-OH/C_β-OH obtained from the selectivity of Ru-Co_3O_4/NF and Co_3O_4/NF. Reproduced with permission from Ref.[64], © Wiley-VCH GmbH 2025. All electrochemical tests did not undergone *iR*-correction. **e, f,** GOR product selectivity in 0.5 M KOH and 0.5 M glycerol for Ni (e) and Ni_3Sn (f). No *iR*-correction was applied. Reproduced with permission from Ref.[65], © Wiley-VCH GmbH 2024.

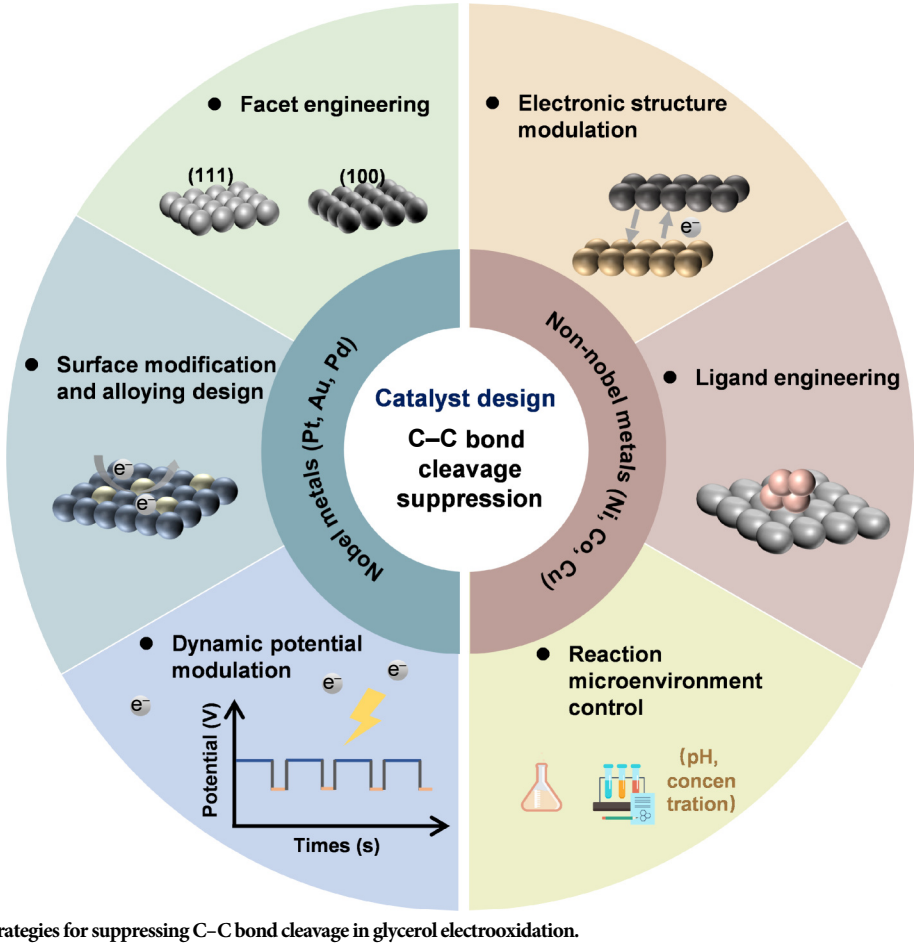

Fig. 4. Catalyst design strategies for suppressing C–C bond cleavage in glycerol electrooxidation.

HER, CO_2RR, and NO_3RR (Fig. 5). Such integrated systems offer multiple synergistic benefits. Perhaps most notably, they lead to a dramatic reduction in the overall reaction overpotential and improve energy utilization efficiency. At the same time, these systems enable the co-production of valuable chemicals at both electrodes. Specifically, at the anode, glycerol is oxidized to produce GLA, LA, and FA. Simultaneously, at the cathode, processes such as HER, CO_2RR, and NO_3RR generate H_2, FA, and NH_3 (Table 1).

Beyond these performance advantages, the technology also delivers significant environmental benefits by facilitating the conversion of greenhouse gases and pollutants into valuable chemicals. This makes it a promising pathway for achieving both biomass refining and carbon neutrality goals.

4.1 GOR–HER coupling system

Among various coupling configurations, the integration of GOR with HER represents a particularly promising approach in the field of energy-efficient glycerol valorization. This coupling combines the production of clean hydrogen with the synthesis of valuable chemicals, offering a dual benefit in terms of energy production and chemical conversion. One of the key advantages of this system is the low theoretical potential of GOR compared to conventional OER, as demonstrated in earlier work[67]. This lower potential translates into reduced energy requirements, enhancing the overall efficiency of the process. The coupling of GOR with HER thus presents a more efficient alternative to traditional methods of hydrogen production.

Significant advancements have been made in developing efficient catalyst systems for this application. For instance, Shi et al.'s nickel-molybdenum nitride nanosheet catalyst (Ni-Mo-N/CFC) demonstrated exceptional bifunctional performance, achieving a Faradaic efficiency (FE) of 99.7% for HER and a FE of 95.0% for formate production at the anode[4]. This system attains a current density of 10 $mA{\cdot}cm^{-2}$ at a cell voltage of just 1.36 V, corresponding to a reduction of 260 mV relative to conventional alkaline water electrolysis and an enhancement of 20% in energy conversion efficiency. Another notable development comes from Wen et al., who created a high-entropy alloy electrode (HEA-CoNiCuMnMo) that combined with a hybrid alkaline/acidic electrolyzer configuration. This system achieved stable operation for 12 days at industrial-level current densities (50 $mA{\cdot}cm^{-2}$), maintaining high FEs for hydrogen (> 99%) and formate (92%) production[2]. Duan et al.'s work on Au_3Ag_1 catalysts further expanded the possibilities by demonstrating efficient pathways for C_3-products, such as lactate. A stacked electrolyzer using this catalyst achieved significant current output (8A-level) at 1.4 V while simultaneously producing both lactate (68.9 $mmol{\cdot}h^{-1}$) and hydrogen (3.5 $L{\cdot}h^{-1}$) at remarkably low energy consumption (3.1 $kWh{\cdot}Nm^{-3}$ H_2)[68].

Despite these encouraging developments, several challenges remain on the path to industrialization. Key among these are the need for cost-effective and stable HER catalysts, as well as high-performance non-precious or low-precious metal GOR catalysts that offer both high selectivity and stability. The development of durable and high-performance anion exchange membranes

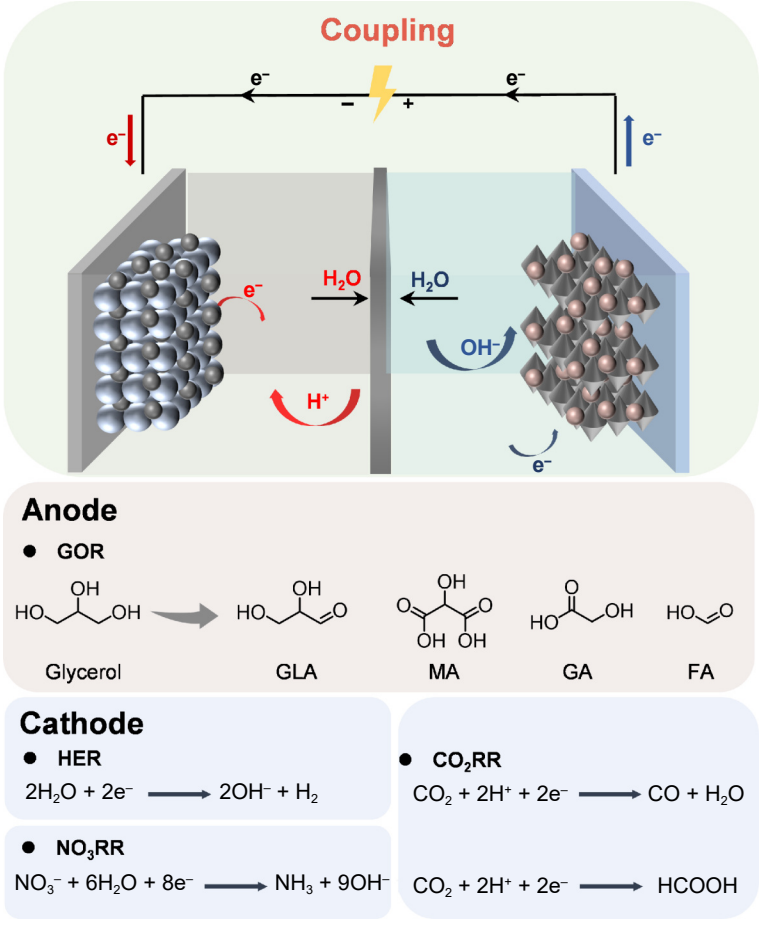

Fig. 5. GOR coupled with HER/CO_2RR/NO_3RR: value-added product generation.

(AEMs) at competitive costs represents another crucial requirement for ensuring the commercial viability of these systems. Additionally, the system validation under industrially relevant conditions, particularly at high current densities (> 500 mA·cm^{-2}) and during extended operation (> 1000 h), remains an important hurdle to overcome. This step is crucial for confirming the reliability and robustness of the technology in real-world applications. Furthermore, the successful scaling and integration of these systems into existing industrial processes will require innovative engineering solutions.

Although the GOR–HER coupling system offers significant potential for efficient energy production and valuable chemical synthesis, addressing the outlined challenges will be essential for translating laboratory-scale successes into practical and economically viable industrial applications. This technology holds the promise of contributing meaningfully to sustainable energy and chemical production infrastructures by effectively integrating hydrogen production with glycerol valorization.

4.2 GOR–CO_2RR coupling system

The integration of GOR with CO_2RR has emerged as an innovative strategy to overcome the fundamental limitations of conventional CO_2 electrolysis systems that couple with OER. The conventional CO_2RR–OER configurations are plagued by significant thermodynamic and kinetic challenges, primarily due to the energy-intensive nature of OER, which accounts for approximately 90% of the total energy consumption while yielding low-value oxygen. In contrast, the integration of GOR with CO_2RR demonstrates notable advantages in both thermodynamic feasibility and practical performance[69]. This alternative approach not only achieves significant energy savings through the much lower oxidation potential of GOR compared to OER, but also enables the simultaneous production of valuable chemicals at both electrodes, creating a synergistic "product pairing" effect that enhances overall atom economy[70]. Recent advancements in this field have demonstrated the technical feasibility and potential of such coupled systems. For instance, Wen et al. developed an integrated electrolyzer that combines atomically dispersed Ni-N single-atom cathodes with three-dimensional interconnected $CoSe_2$ nanostructured anodes. This innovative design has been shown to maintain stable operation for 15 days at 100 mA·cm^{-2} with FE exceeding 90% for products at both electrodes[71]. Similarly, Yu et al. designed a bifunctional catalyst system capable of co-producing formic acid at both the cathode (FE = 84.5%) and anode (FE = 90.2%). This dual-product generation approach maximizes the electron utilization efficiency, further enhancing the system's practicality[72]. Further advancement in catalyst design was achieved by Andreu et al., whose Au-In alloy catalysts reduced the onset potential by 210 mV[73]. These catalysts also achieved an energy consumption as low as 202 kWh·kmol^{-1} while maintaining a FE over 90% at industrially relevant current densities of 200 mA·cm^{-2}. At the system level, Angel et al.'s membrane electrode assembly (MEA) configuration pushed performance boundaries even further, achieving exceptional product concentrations of 359 g·L^{-1} formate at the cathode (FE = 95%) alongside DHA production at 0.434 mmol·m^{-2}·s^{-1} at the anode[5].

Table 1 Coupling of GOR with HER, CO_2RR, and NO_3RR

Coupling system	Catalyst	Electrolyte	Potential (V)	Current density ($mA·cm^{-2}$)	Product	Ref.
GOR||HER	Ni-Mo-N/CFC	1 M KOH + 0.1M glycerol||1 M KOH	1.4	10	FA (FE = 99.7%); H_2 (FE = 95.0%)	[4]
GOR||HER	CoNiCuMnMo-NPs/CC||RuIr/Ti	1 M KOH + 0.1M glycerol||0.5 M H_2SO_4	0.89	100	FA (FE = 92.0%); H_2 (FE = 99.0%)	[2]
GOR||HER	Au_3Ag_1||Ni form	1 M KOH + 0.3M glycerol||1 M KOH	1.1	1000	LA (FE = 47%; Sel = 70%); H_2 (—)	[67]
GOR||CO_2RR	$CoSe_2CC$||NiSAs/FN-CNSs/cp	2 M KOH + 2M glycerol||2 M $KHCO_3$	1.97	100	FA (Sel = 90%); CO (Sel = 90%)	[70]
GOR||CO_2RR	$Cu(OH)_2$/CF	1 M KCl + 1 M glycerol||0.1 M $KHCO_3$	1.8	40	FA (FE = 84.5%); FA (FE = 90.2%)	[71]
GOR||CO_2RR	Au||Bi/C	1 M KOH + 1M glycerol||1 M KOH	2.99	90	DHA, LA, GLA, FA (FE = 14%, 30%, 18%, 28%); FA (FE = 81%)	[72]
GOR||CO_2RR	Ni-Co foam||Bi-GDE	1 M KOH + 1M glycerol||H_2O	2.39	45	FA (FE = 95.1%); FA (FE = 95.1%)	[5]
GOR||NO_3RR	$NiCo_2O_4$||Pd@Cu-0.95%	1 M KOH + 0.33 M glycerol||1 M KOH + 0.1 M KNO_3	1.6	100	FA (—); NH^{4+} (FE = 96%)	[75]
GOR||NO_3RR	CNs@CoP	1 M KOH + 1 M glycerol||1 M KOH + 1 M KNO_3	1.6	300	FA (FE = 93.5%); NH^{4+} (FE = 96.4%)	[76]
GOR||NO_3RR	Cu-NiCo/NF	1 M KOH + 1 M glycerol||1 M KOH + 0.1 M KNO_3	1.25	10	FA (yield = 80.6%); NH^{4+} (—)	[77]
GOR||NO_3RR	$NiCo_2O_4$||Cu_2NCN	1 M KOH + 0.33 M glycerol||0.5 M KOH + 0.1 M KNO_3	2.4	400	FA (FE = 96%); NH^{4+} (FE = 94%)	[78]

Despite these significant advances, several critical challenges must be addressed to fully realize the potential of GOR–CO_2RR technology. The catalyst stability remains a primary concern, and potential solutions include the development of core–shell or alloy architectures and the use of self-healing functional materials. Additionally, the optimization of reaction selectivity, particularly for GOR, which tends to produce multiple byproducts, may benefit from advanced catalyst designs, such as single-atom systems combined with *in situ* characterization techniques. At the system level, challenges such as ion-exchange membrane limitations, product crossover, and three-phase boundary mass transfer resistances require innovative reactor designs and improved membrane materials. Practical implementation will need to address long-term operational stability, compatibility with real-world feedstocks containing impurities, and scaling considerations such as thermal management and current distribution uniformity. Overcoming these challenges will be essential for transitioning GOR–CO_2RR technology from laboratory-scale demonstrations to commercially viable processes that can contribute meaningfully to sustainable chemical production and carbon utilization.

4.3 GOR–NO_3RR coupling system

Ammonia synthesis stands at a critical juncture, balancing its pivotal role in global agriculture and industry with the urgent need for sustainable production methods. As a cornerstone of modern chemical industry and food production systems[74], conventional Haber–Bosch ammonia synthesis faces significant sustainability challenges due to its energy-intensive operation (requiring ~ 500 °C and > 100 atm) and its substantial carbon footprint (~ 400 million metric tons CO_2 annually)[75]. These environmental concerns have driven intense research into NO_3RR as a renewable-powered alternative that operates under ambient conditions while addressing both energy and environmental concerns by combining ammonia production with the remediation of nitrate pollution.

The evolution of NO_3RR systems has advanced significantly from traditional configurations that couple with OER to more designs that integrate GOR technology. This strategic coupling leverages the thermodynamic advantages of glycerol's lower oxidation potential[4], resulting in improved energy efficiency and the creation of valuable product streams at both electrodes. At the cathode, ammonia is produced, whereas at the anode, FA and DHA are generated. Recent breakthroughs in catalyst and system design have accelerated progress toward industrial implementation. For instance, Wang et al.'s MEA system, featuring a palladium-doped copper cathode (Pd@Cu-0.95%) cathode and $NiCo_2O_4$ anode, demonstrated significant voltage reduction (450 mV) while maintaining exceptional selectivity (a FE of 96% for NH^{4+} at 1.6 V)[76]. Shao et al.'s work with cobalt phosphide-loaded carbon nanosheets (CNs@CoP) achieved stable ampere-level operation ($300\ mA·cm^{-2}$) with a ammonia efficiency of 88% and a FA yield of 92% over 100 h[77]. Additionally, Wu et al.'s copper-doped nickel-cobalt alloy system exhibited impressive performance, achieving an ammonia FE of 97.9% at −0.15 V and a FA selectivity of 80% during 144 h of operation[78]. Among the most notable advancements is Brinck et al.'s Cu_2NCN-based system, which achieved industrial-scale current densities ($4000\ mA·cm^{-2}$) at just 2.52 V, while maintaining a NH_3 FE of 83% and unprecedented production rates ($97{,}000\ \mu g·h^{-1}·cm^{-2}$) over 100 h[79].

These collective achievements highlight three key technological advantages: (i) substantial voltage reductions (200–450 mV) achieved through the replacement of OER with GOR, (ii) the optimization of synergistic reactions via bifunctional catalysts, and (iii) the development of scalable architectures such as membrane electrode assemblies. The continued development of GOR–NO_3RR coupling systems must focus on the following three areas: (1) the design of multifunctional catalysts with precisely controlled active sites to enhance both activity and selectivity, (2) the optimization of mass transport in reactor configurations to improve reaction

efficiency, and (3) the enhancement of poison resistance to ensure robust performance in real-world wastewater treatment applications. Addressing these challenges requires coordinated advancements in materials science, electrochemical engineering, and system integration.

The successful translation of these laboratory-scale achievements into industrial applications will be crucial for realizing the full potential of sustainable ammonia production pathways. By overcoming the existing technical and operational challenges, GOR–NO_3RR coupling systems have the potential to significantly contribute to both energy sustainability and environmental protection, offering a promising alternative to conventional ammonia synthesis methods.

5 Toward industrial implementation of GOR: Membrane innovations, long-term stability, and crude glycerol adaptation

The industrialization of glycerol electrolysis technology, although representing a promising pathway for the valorization of biodiesel byproducts, is accompanied by a set of interconnected technical challenges that must be systematically addressed to achieve commercial viability. Three critical bottlenecks stand out as primary barriers to large-scale implementation: (i) overcoming mass transfer limitations while maintaining product selectivity at industrial current densities, (ii) ensuring cost-effectiveness through optimized membrane materials and reactor designs that enhance product separation efficiency, and (iii) improving system tolerance to crude glycerol impurities while ensuring long-term operational stability[28]. These challenges become increasingly complex and interdependent during scale-up, necessitating holistic solutions that consider both technical and economic factors[80]. Preliminary techno-economic analyses (TEA) of related systems have highlighted promising commercial prospects and provided essential economic benchmarks for industrialization. For example, Chen et al. demonstrated that the electrochemical conversion of glycerol to FA coupled with HER entails plant-gate processing costs of \$4655–4797 per ton of glycerol, with the resulting products estimated to have a market value of \$6389 per ton at current densities of 100–300 $mA{\cdot}cm^{-2}$ [56]. Similarly, Shao et al. illustrated that coupling value–added anodic oxidation (to glycerol-to-FA/potassium diformate) in the nitrate reduction system could transform an economically unfavorable process (NO_3RR–OER, –\$2629 per ton of NH_3) into a profitable one (NO_3RR–GOR, \$1217–4474 per ton of NH_3)[77]. Additionally, Duan et al. estimated that the direct electro–oxidation of crude glycerol to LA could yield a net profit of \$1311 per ton of LA when the value of co–produced hydrogen is included, underscoring the strong economic incentive for utilizing low-cost biodiesel waste streams[68].

Recent advances have demonstrated promising pathways to address these challenges. In terms of MEA optimization, Xu et al.'s innovative acid-base asymmetric configuration represents a significant breakthrough. This system achieved a FE of 84% for liquid products at industrially relevant current densities of 1000 $mA{\cdot}cm^{-2}$ through a dual mechanism combining electrochemical neutralization energy recovery with precise ion transport regulation[81]. Notably, this system utilized hydroxide ion gradients to effectively occupy anion exchange membrane (AEM) channels, thus mitigating the transmembrane loss of valuable C_3 products such as lactate. Parallel progress has been made in cost reduction through the development of alternative membrane materials. For instance, Ma et al. successfully implemented porous nylon organic membranes that maintained electrochemical performance while significantly lowering costs[82]. These membranes enabled the selective transport of K^+ and OH^-, while physically separating the electrodes in the process. This achievement demonstrated that economical materials could meet the demanding requirements of industrial-scale operation.

The challenge of product separation has also seen innovative solutions, such as Shi's membrane-free electrolyzer system, which addressed the difficulty of separating formate from electrolytes through an innovative neutralization and distillation process[83]. Perhaps most critically for industrial implementation, substantial progress has been made in direct electrolytic conversion of crude glycerol feedstocks. Duan and Shao et al. pushed the boundaries of scale-up using stacked flow electrolyzers with electrode areas of 150 cm^2 and innovative intermittent potential strategies, which achieved a LA yield of 38.4% (11.2 g) alongside 9.3 L of H_2 production from low-grade glycerol esters over 18.6 h[84]. Meanwhile, Shao et al.'s system demonstrated efficient conversion of peanut oil-derived crude glycerol (a yield of 9.3%) with sustained lactate production at 56.9 $mmol{\cdot}h^{-1}$ during 13.2 h of continuous operation[68].

Looking forward, achieving full industrialization requires focused attention on several remaining technical challenges. The development of highly stable anode catalysts and self-healing membrane materials is crucial for extending system lifespans to meet commercial requirements. Equally important is the establishment of comprehensive evaluation frameworks that integrate TEA with life cycle assessment (LCA) to optimize the complex interplay between current density, product selectivity, and energy consumption. This multi-dimensional optimization approach will be essential for transitioning glycerol electrolysis from laboratory-scale demonstrations to commercially viable industrial processes. Through interdisciplinary collaboration, the successful resolution of these challenges will not only advance glycerol valorization technology but also provide critical support for the synergistic development of biomass refining and green hydrogen production. This, in turn, will contribute to a more sustainable chemical manufacturing paradigm (**Fig. 6**).

6 Conclusions

The electrochemical upgrading of glycerol presents a transformative opportunity to integrate biomass valorization with renewable energy utilization, offering a dual solution to address both resource sustainability and carbon emission reduction. This perspective highlights the remarkable progress achieved in understanding and optimizing GOR systems, encompassing advancements from atomic-scale catalyst design to reactor-level engineering. Key insights include: (i) the critical role of noble metal electronic structure and surface geometry on controlling C–C bond cleavage pathways; (ii) the potential of non-noble metal catalysts, which can be engineered through alloying, doping, or ligand engineering to achieve a balanced performance between cost and efficiency; and (iii) the enhanced energy efficiency enabled by coupling GOR with cathodic reactions.

However, the pathway to industrialization necessitates overcoming persistent challenges: improving catalyst stability under

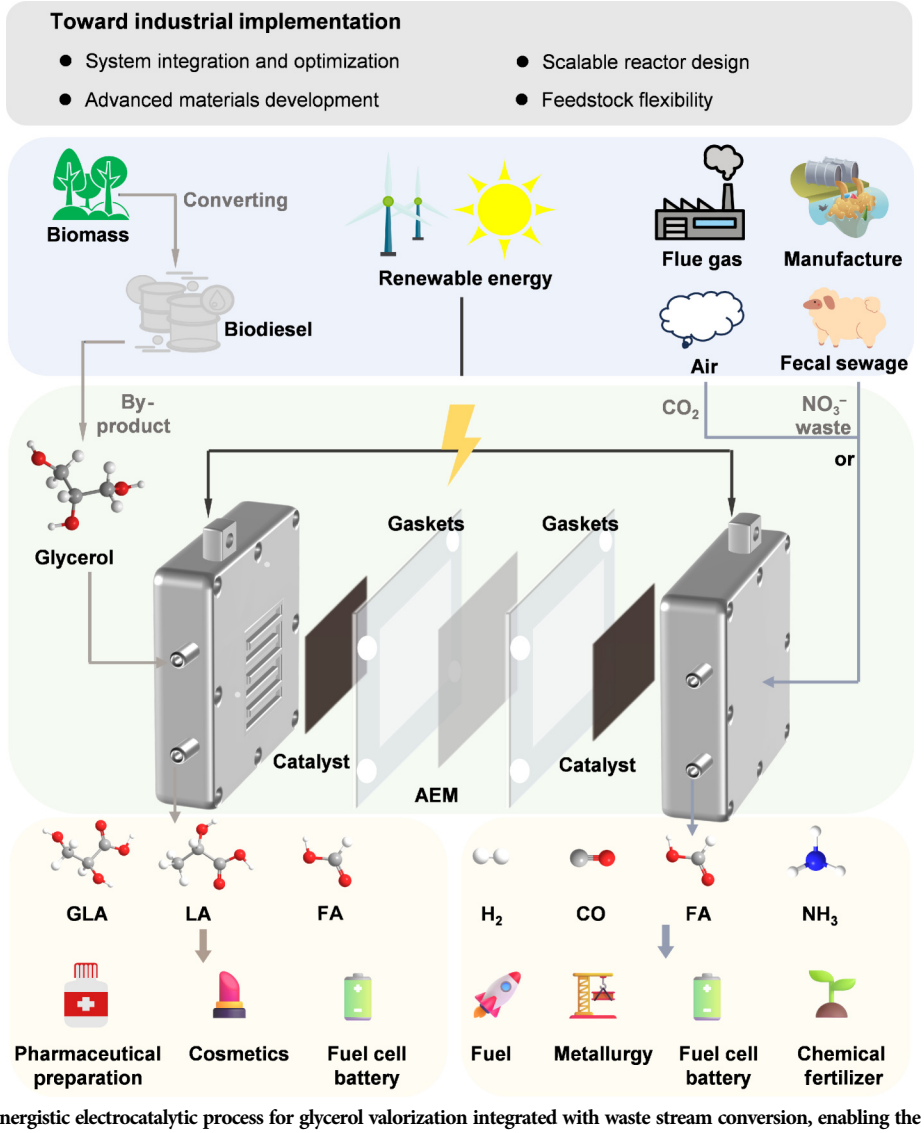

Fig. 6. Schematic of a synergistic electrocatalytic process for glycerol valorization integrated with waste stream conversion, enabling the co-production of diverse valuable products.

high current densities (> 500 mA·cm^{-2}), developing cost-effective separation technologies for complex product streams, and enhancing tolerance to real-world crude glycerol feedstocks. Future research efforts should focus on the development of multifunctional catalyst systems with self-healing capabilities, innovative reactor designs for scalable operation, and integrated assessment frameworks that combine TEA with LCA for comprehensive environmental impact evaluation. Fostering the bridge between fundamental discoveries and commercial deployment will require the integration of *in situ* characterization, machine learning-guided optimization, and circular process design.

As the field continues to evolve, GOR technology is positioned to play a pivotal role in sustainable biorefineries, contributing simultaneously to green hydrogen production, carbon-neutral chemical synthesis, and effective wastewater remediation. Through collaborative efforts spanning materials science, electrochemistry, and chemical engineering, the vision of achieving economically viable and environmentally responsible glycerol valorization can be realized. This advancement represents a significant milestone on the path toward a post-fossil carbon economy.

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Data availability

Not applicable.

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K. W. M.: Writing original draft. L. D.: Review and editing. W. J. Z.: Review and editing, and funding acquisition. Y. Q. W.: Review and editing. Z. P. C.: Review and editing, and funding acquisition.

Competing interests

The authors have no competing interests to declare that are relevant to the content of this article.

Use of AI statement

None.