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Research Article

Electrochemical Activation in Alcohol Oxidation Reaction and Hydrogen Evolution Reaction: A Mechanistic Mini Review

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ABSTRACT

Alcohol oxidation reactions (AORs) and the hydrogen evolution reaction (HER) are central to electrochemical energy conversion technologies. However, their practical implementation remains limited by sluggish kinetics, surface poisoning, and catalyst instability. In recent years, electrochemical activation has emerged as an effective strategy to enhance catalytic performance by inducing dynamic interfacial transformations under operating conditions. Rather than acting as a simple pretreatment, electrochemical activation generates operando-stabilized surface states characterized by altered adsorption behavior and increased availability of oxygenated intermediates. This mini-review summarizes the fundamental principles governing AOR and HER, with particular emphasis on interfacial reaction pathways and shared surface intermediates. The mechanistic role of electrochemical activation is critically examined as a bridge between AOR and HER, highlighting the contribution of HER-assisted water activation and hydroxyl species formation to the oxidative removal of poisoning intermediates. In addition, catalyst-dependent manifestations of activation, protocol sensitivity, and implications for stability are discussed. Finally, key challenges and emerging opportunities are outlined, underscoring the need for operando characterization and activation-aware catalyst design to enable predictive control of electrochemical reactivity.

1. Introduction

The transition toward sustainable energy conversion technologies has intensified interest in electrochemical reactions that enable the efficient utilization of small organic molecules and the production of clean hydrogen [1-3]. Among these, alcohol oxidation reactions (AORs), including methanol and ethanol oxidation, play a central role in direct alcohol fuel cells. In contrast, the hydrogen evolution reaction (HER) remains indispensable for water electrolysis and emerging hybrid electrochemical systems. Despite decades of investigation, both reactions continue to face kinetic limitations, catalyst poisoning, and stability challenges, particularly under practical operating conditions. Addressing these issues requires not only advanced catalyst design but also a deeper mechanistic understanding of how electrochemical environments dynamically alter catalytic interfaces [4-7].

In recent years, electrochemical activation has emerged as a powerful strategy to markedly enhance the activity of electrocatalysts toward alcohol oxidation, often accompanied by notable changes in HER behavior. Rather than serving as a simple surface-cleaning or conditioning step, electrochemical activation has been shown to induce profound interfacial transformations under applied potential, generating catalytically competent surface states that differ substantially from those of the pristine materials. These activated states are commonly associated with the formation of oxygenated intermediates, modified adsorption energetics, and altered reaction pathways, leading to substantial improvements in oxidation kinetics and tolerance toward poisoning species. Importantly, activation effects have been reported across a wide range of catalyst systems, spanning noble-metal, non-noble, and hybrid architectures, and under both acidic and alkaline environments [8-13].

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A recurring mechanistic theme linking these observations is the coupling between electrochemical activation and hydrogen evolution–related processes. Under activation conditions, HER and/or water electrolysis can occur concurrently, generating adsorbed water and hydroxyl species at the electrode–electrolyte interface. These oxygen-containing intermediates are widely proposed to facilitate the rate-determining steps of alcohol oxidation by promoting the oxidative removal of strongly adsorbed carbonaceous species. In bifunctional and hybrid catalysts, additional contributions from metal hydroxides, oxide supports, or redox-active stabilizers further amplify this effect by enhancing water dissociation and stabilizing reactive OH-containing surface ensembles. Nevertheless, the precise mechanistic role of HER during activation—whether as a beneficial facilitator, a surface modifier, or a parasitic side reaction—remains incompletely resolved [14-20]. In general, HER tends to act synergistically under alkaline conditions and moderate overpotentials where surface water dissociation provides additional OH* for alcohol oxidation, while it becomes competitive under strongly cathodic potentials or in acidic media where hydrogen adsorption dominates and suppresses OH* stability. These boundaries define the transition between cooperative interfacial water activetion and parasitic hydrogen evolution, providing a clearer mechanistic framework for evaluating activation outcomes.

In this mini-review, the fundamental electrochemical principles governing alcohol oxidation and hydrogen evolution reactions are first outlined, with emphasis placed on interfacial reaction pathways and key surface intermediates that control reaction kinetics. Electrochemical activation is then critically analyzed as a unifying mechanistic framework, highlighting how activation-induced interfacial transformations and HER-assisted oxygen chemistry regulate catalytic performance across diverse material systems. Finally, the key challenges and emerging opportunities associated with electrochemical activation are discussed, with particular attention to mechanistic uncertainties, protocol dependence, and stability considerations, to identify directions toward predictive and activation-aware electrocatalyst design.

2. Fundamentals of AOR and HER

2.1 Fundamental aspects of AOR

AORs are the anodic processes in direct alcohol fuel cells (DAFCs), where alcohols (typically C1 and C2 alcohols such as methanol and ethanol) are electrochemically oxidized to generate electrical energy. Although DAFCs are conceptually attractive, the overall performance is strongly limited by the sluggish kinetics of AOR and the difficulty of activating key bond-breaking steps at practical potentials [21].

Thermodynamically, complete electrochemical oxidation of low-carbon alcohols is highly favorable, and key thermodynamic metrics (e.g., ΔG° and E°_{cell}) are commonly used to compare fuels and rationalize their theoretical energy densities [22].

Using methanol as a model fuel, complete methanol oxidation involves multi-electron transfer and proceeds differently depending on electrolyte pH. The overall stoichiometries are commonly expressed as [21, 23]:

$CH_3OH + H_2O \rightarrow CO_2 + 6H^+ + 6e^-$ (acidic medium)

$CH_3OH + 6OH^- \rightarrow CO_2 + 5H_2O + 6e^-$ (alkaline medium)

At the mechanistic level, AOR generally proceeds through the adsorption of the alcohol molecule, followed by stepwise dehydrogenation and the formation of surface-bound intermediates. The strong adsorption of carbonaceous intermediates (often CO-like species) can block active sites, making their oxidative removal a central kinetic bottleneck for sustained activity. Consequently, the generation and utilization of oxygenated surface species—particularly OH*—is widely regarded as critical for oxidizing poisoning intermediates and regenerating free catalytic sites during operation [16, 21, 23].

2.2 Fundamental aspects of HER

The HER is the cathodic half-reaction in water electrolysis and proceeds via elementary steps involving hydrogen adsorption and molecular hydrogen formation. The widely accepted mechanistic framework describes HER using the Volmer (electrochemical adsorption), Heyrovsky (electrochemical desorption), and Tafel (chemical recombination) steps. In acidic electrolytes, protons serve as the hydrogen source. In contrast, in alkaline electrolytes, water dissociation supplies adsorbed hydrogen (H*) and produces OH^-, introducing an additional kinetic barrier associated with water activation (Figure 1) [24, 25].

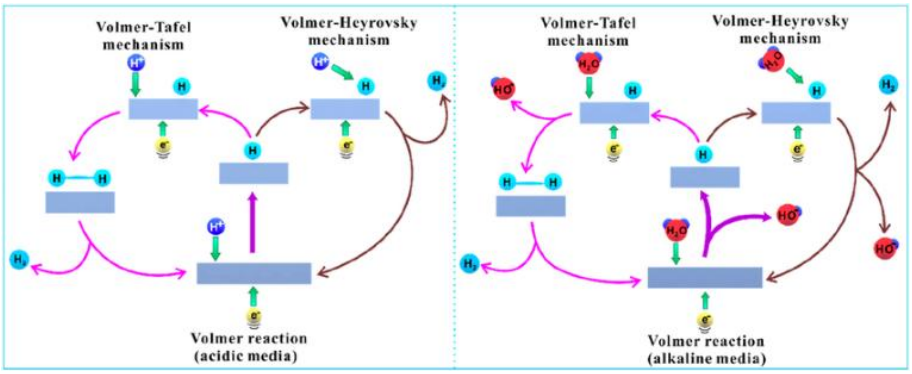

Fig.1. HER pathway under acidic and alkaline media [28]

A key descriptor governing HER kinetics is the balance between hydrogen adsorption and desorption. If hydrogen binding is too weak, surface coverage of reactive H* becomes insufficient; if too strong, active sites are blocked by persistent H* coverage. The thermodynamic descriptor ΔGH* is therefore used to rationalize optimal catalytic behavior, where ΔGH* near zero corresponds to the best balance between adsorption and release of H_2. In alkaline media, HER is often slower because the Volmer step requires water dissociation; thus, catalysts that improve interfacial adsorption/desorption processes and facilitate water activation are generally necessary to accelerate HER kinetics [25-27].

2.3 Interfacial commonalities between AOR and HER

Although AOR and HER proceed in different directions and involve different reactants, both are governed by interfacial adsorption-controlled elementary steps and are sensitive to surface coverage of key intermediates. In AOR, oxygenated species such as OH* are central to oxidizing poisoning intermediates and maintaining free active sites [21]. In HER—especially under alkaline conditions—OH^-/OH* species arise from water dissociation and influence interfacial kinetics through their impact on adsorption/desorption and water-activation energetics [25-27]. This shared dependence on surface intermediates provides the mechanistic basis for understanding how electrochemical conditions that alter interfacial water and hydroxyl chemistry can simultaneously influence alcohol oxidation and hydrogen evolution. An overview of the fundamental mechanistic aspects associated with AOR and HER is presented in Table 1.

3. Electrochemical activation as a mechanistic bridge between alcohol oxidation and hydrogen evolution

3.1 Interfacial meaning of electrochemical activation

Electrochemical activation is increasingly recognized as an operando-generated catalytic state rather than a mere pretreatment or surface cleaning step. Across diverse catalyst classes and electrolytic environments, activation is shown to induce profound changes in interfacial chemistry, leading to markedly enhanced alcohol oxidation activity and, in many cases, modified hydrogen evolution behavior. Rather than altering bulk structure, activation predominantly reshapes the chemical identity and population of surface intermediates, particularly oxygenated species, which govern the kinetics of multi-electron oxidation reactions [29-34].

Despite differences in catalyst composition and activation protocols, a unifying observation is that catalytic performance measured after activation does not reflect the pristine material but a dynamically evolved surface state stabilized under electrochemical bias. This insight challenges static structure–activity correlations and underscores the need to treat electrocatalysts as adaptive systems.

3.2 HER-assisted generation of oxygenated surface species

A recurring mechanistic motif is the coupling between activation-induced hydrogen evolution and the formation of reactive oxygen-containing intermediates. During activation at sufficiently negative or positive potentials, HER and/or water electrolysis occur concurrently with alcohol oxidation environments. This process is proposed to generate adsorbed water (H_2O*ads) and hydroxyl species (OH*ads) at the electrode–electrolyte interface, which subsequently facilitate the oxidative removal of poisoning intermediates such as CO-like fragments [30, 32, 35].

In alkaline systems, this coupling is mechanistically straightforward: water dissociation during HER produces surface-bound hydroxide species that directly participate in the rate-determining oxidation step of alcohols [31, 34]. In acidic media, where HER consumes protons rather than producing OH^-, enhanced alcohol oxidation is instead attributed to local interfacial pH modulation and transient water activation, leading to OH* formation despite the bulk acidity [32].

While electrochemical signatures—such as increased anodic peak currents, negative shifts in onset potentials, and improved stability—support this interpretation, the mechanistic link between HER intensity and steady-state OH* coverage remains largely inferred rather than directly quantified. Consequently, although HER-assisted activation provides a compelling framework, it should be regarded as chemically plausible but not yet conclusively resolved.

3.3 Catalyst-dependent manifestations of activation

The expression of electrochemical activation depends strongly on catalyst composition and support chemistry. In non-noble bimetallic systems, activation leads to dramatic increases in methanol oxidation activity, often exceeding an order of magnitude. These improvements are attributed to enhanced formation of oxygenated species and improved tolerance to poisoning intermediates. However, direct evidence for surface oxidation-state evolution or alloy segregation during activation is typically absent [29, 30, 33].

Table 1. Fundamental mechanistic features of AOR and HER

Aspect	AOR	HER
Reaction role	Anodic oxidation	Cathodic reduction
Typical systems	DAFC anodes	Water electrolysis cathodes
Main reactant	Alcohol (e.g., CH_3OH, C_2H_5OH)	H^+ (acidic) / H_2O (alkaline)
Electron transfer	Multi-electron (e.g., $6e^-$ for MOR)	$2e^-$ per H_2
Key intermediates	*CO, *CHO, *COH, OH*	H*, OH* (alkaline)
Rate-limiting step	Oxidative removal of adsorbed carbon species	H* formation or desorption
Role of OH*	Oxidizes poisoning intermediates	Product of water dissociation
pH sensitivity	Strong (acidic vs alkaline pathways)	Robust (water activation in alkaline)
Thermodynamic descriptor	ΔG°, E°cell	ΔG_H*
Major kinetic limitation	Catalyst poisoning	Water dissociation (alkaline)

In contrast, noble-metal-based and hybrid catalysts exhibit a more explicit bifunctional mechanism, where adjacent phases—such as metal hydroxides, oxide supports, or polyoxometalates—actively promote water dissociation and stabilize OH* near active metal sites. In these systems, activation is frequently interpreted as a process that pre-populates the surface with catalytically competent OH*-metal ensembles, thereby lowering the kinetic barrier of the rate-determining oxidation step [34].

However, evidence also indicates that activation may not always be purely chemical or reversible. In systems employing redox-active stabilizers or oxide-containing supports, activetion can induce irreversible chemical transformations, generating new metal–oxygen species that persist beyond the activetion step. This observation complicates the interpretation of activation as simple surface conditioning and raises important questions regarding catalyst evolution and long-term stability [34].

3.4 Dependence on activation protocol and electrolyte environment

Another critical insight is that electrochemical activation is highly protocol-dependent. Variations in applied potential, duration, electrolyte composition, and pH result in qualitatively similar performance enhancements but may arise from distinct underlying physicochemical processes. Cathodic activation in buffered electrolytes, anodic polarization in acidic media, and in situ activation during alcohol oxidation all lead to improved activity, yet the interfacial states generated under these conditions are unlikely to be identical [29-36].

This variability cautions against treating electrochemical activation as a universal or transferable procedure. Without shared operando descriptors—such as quantified OH* coverage, interfacial pH gradients, or reconstructed phase fractions—claims of generality should be interpreted as phenomenological rather than mechanistically unified.

3.5 Critical synthesis and mechanistic outlook

Taken together, the literature establishes electrochemical activation as a powerful means of unlocking latent catalytic activity in alcohol oxidation systems, often with concomitant effects on hydrogen evolution. The prevailing mechanistic picture emphasizes interfacial oxygen chemistry, in which activation increases the availability of OH* and related species required for the oxidative removal of adsorbed intermediates. This framework is internally consistent and supported by electrochemical trends across a wide range of catalyst architectures [29-36].

Throughout this discussion, mechanistic interpretations are derived from collective electrochemical trends and operando-accessible proxies rather than direct atomic-scale observation. The boundaries between experimentally established behavior and interpretative inference are therefore indicated where relevant to distinguish robust evidence from proposed mechanistic models.

Nevertheless, several unresolved issues remain central to advancing the field:

I. the precise origin, lifetime, and spatial distribution of activation-generated oxygenated species;
II. the balance between reversible surface conditioning and irreversible chemical evolution; and
III. the conditions under which HER acts as a beneficial facilitator rather than a parasitic competitor.

Addressing these challenges will require moving beyond ex situ characterization and voltammetric inference toward operando spectroscopic and spatially resolved electrochemical techniques, such as operando X-ray absorption spectroscopy, vibrational spectroscopy, and scanning probe methods capable of resolving local pH and surface reconstruction. Only then can electrochemical activation be transformed from a powerful empirical strategy into a predictively controllable mechanistic tool. An overview of electrochemical activation-driven mechanisms linking alcohol oxidation and hydrogen evolution is provided in Table 2.

Collectively, these observations indicate that certain mechanistic features—such as the involvement of interfacial OH* species and the role of hydrogen-assisted water activation—are general across diverse catalyst classes. In contrast, the extent of irreversibility, surface reconstruction, and bifunctional coupling is strongly dependent on the specific catalyst architecture and activation protocol employed.

An overview of electrochemical activation-driven mechanisms linking alcohol oxidation and hydrogen evolution is provided in Table 2, which summarizes representative catalyst systems selected to illustrate diverse catalyst classes, electrolyte environments, and activation protocols rather than providing an exhaustive survey.

4. Challenges and Opportunities

4.1 Key challenges

Despite the clear effectiveness of electrochemical activation in enhancing alcohol oxidation and modulating hydrogen evolution, several fundamental challenges remain unresolved. A primary challenge lies in the incomplete mechanistic understanding of activation-generated interfacial states. While increased activity is consistently correlated with the presence of oxygenated surface species, the precise origins, lifetimes, and spatial distributions of these species under operating conditions remain poorly constrained. As a result, current mechanistic interpretations often rely on indirect electrochemical signatures (e.g., activity trends, onset shifts, and voltammetric features) rather than direct observation of the active interface, and should therefore be interpreted with appropriate caution.

Table 2. Mechanistic interpretation of electrochemical activation in alcohol oxidation and its coupling with hydrogen evolution across representative catalyst systems

Catalyst system	Medium	Activation protocol	Dominant activation effect	Role of HER during activation	Mechanistic limitation / open question	Ref
Co–Sn bimetallic nanoalloys	Acidic	In situ anodic / cathodic polarization	Enhanced OH* availability via interfacial water activation	Local pH modulation and transient H_2O*ads generation	OH* formation inferred indirectly; interfacial pH not quantified	[32]
Co–Sn bimetallic nanoalloys	Alkaline	Electrochemical pre-activation	Accelerated removal of CO-like intermediates	Direct water dissociation during HER supplies OH^- / OH*	Stability of the activated surface under prolonged cycling	[31]
Pd/Ni$(OH)_2$/N-rGO hybrids	Alkaline	In situ electrochemical activation	Pre-formation of Pd–OH* sites (bifunctional effect)	HER facilitates water dissociation at Ni$(OH)_2$ domains	Persistence of OH* coverage during steady-state AOR	[33]
PdPt–ZrO_2/MWCNT composites	Alkaline	Electrochemical activation step	Improved alcohol adsorption–oxidation kinetics	HER assumed to assist surface oxygen chemistry	Structural vs purely chemical contribution unclear	[30]
PdNPs@polyoxometalate	Alkaline / neutral	High-potential activation in buffer	Combined Pd–OH* formation and support chemical evolution	HER generates OH* and alters POM redox structure	Irreversibility of support transformation	[36]
Au@Pd/POM systems	Buffered media	Cathodic activation	Interfacial reconstruction enhancing AOR and HER	HER treated as synergistic surface modifier	Generality of mechanism across pH regimes	[29]

Another major challenge concerns the coupling between hydrogen evolution and alcohol oxidation. Although HER-assisted generation of oxygenated species can facilitate the oxidative removal of poisoning intermediates, HER may also act as a competing reaction, consuming charge carriers and altering mass transport at the electrode surface. Distinguishing conditions under which HER acts as a beneficial interfacial modifier from those where it becomes parasitic remains an open question, particularly under prolonged operation and high current densities.

The dynamic and protocol-dependent nature of electrochemical activation further complicates its rational application. Similar enhancements in catalytic performance can be achieved using markedly different activation potentials, durations, and electrolyte compositions, suggesting that distinct physicochemical pathways may converge toward superficially similar electrochemical outcomes. This lack of a universal descriptor for the activated state limits the transferability of activation strategies across catalyst systems and operating environments.

From a materials perspective, activation-induced surface evolution poses stability concerns. In some catalyst architectures, electrochemical activation may involve irreversible chemical transformations or support restructuring that initially enhance activity but compromise long-term durability. Differentiating reversible surface conditioning from permanent catalyst evolution remains a critical challenge for translating activation concepts into practical energy-conversion devices.

4.2 Emerging opportunities

Notwithstanding these challenges, electrochemical activation offers significant opportunities to advance both alcohol oxidation and hydrogen evolution technologies. One of the most promising directions lies in interfacial-state engineering, where catalysts are intentionally designed to evolve into optimal active configurations under applied potential rather than being optimized solely in their pristine form. This dynamic design philosophy aligns naturally with the adaptive behavior of electrocatalysts under realistic operating conditions.

The shared dependence of AOR and HER on interfacial water and hydroxyl chemistry offers opportunities for synergistic catalyst design. By tailoring catalyst composition and surface architecture to regulate the generation, stabilization, and consumption of oxygenated species, it may be possible to enhance alcohol oxidation kinetics while simultaneously controlling hydrogen evolution behavior. Such strategies are particularly effective for hybrid electrochemical systems in which both reactions coexist.

Another important opportunity arises from optimizing electrolytes and operating conditions. Because electrochemical activation is highly sensitive to pH, buffer composition, and ionic environment, careful control of the electrolyte provides a powerful, materials-independent lever to tune interfacial chemistry and reaction pathways without altering catalyst composition.

Finally, advances in operando and spatially resolved characterization techniques create a pathway toward resolving long-standing mechanistic ambiguities. Direct observation of activation-induced surface states, local pH gradients, and intermediate dynamics under working conditions will enable the development of predictive models that connect activation protocols to catalytic function. Such insights are essential for transforming electrochemical activation from an empirical enhancement strategy into a controllable mechanistic design principle.

The significant challenges and prospective research directions associated with electrochemical activation in coupled AOR–HER systems are summarized schematically in Figure 2.

Fig.2. Schematic roadmap illustrating the key challenges and emerging opportunities associated with electrochemical activation in alcohol oxidation and hydrogen evolution reactions

5. Conclusion

Electrochemical activation has emerged as a central mechanistic element governing the performance of alcohol oxidation reactions and their interplay with the HER; throughout this review, “activation” refers to a dynamic, protocol- and condition-dependent interfacial state formed under applied potential, rather than a fixed pretreatment or static surface modification. Rather than serving as a simple pretreatment or surface-cleaning step, activation induces dynamic interfacial transformations that generate operando-stabilized catalytic states distinct from those of the pristine material. These activated states are characterized by altered adsorption behavior, increased availability of oxygenated intermediates, and altered reaction pathways, collectively enhancing alcohol oxidation kinetics.

A key outcome of this analysis is the recognition that interfacial oxygen chemistry, particularly the generation and utilization of hydroxyl species, plays a decisive role in controlling reaction rates and tolerance toward poisoning intermediates. HER-assisted water activation is shown to contribute to the formation of reactive oxygen-containing species, which facilitate the oxidative removal of strongly adsorbed carbonaceous fragments and enable sustained catalytic activity. While this coupling provides a coherent mechanistic framework, the quantitative relationship between hydrogen evolution, hydroxyl coverage, and alcohol oxidation remains insufficiently resolved.

The manifestations of electrochemical activation depend strongly on catalyst composition, support chemistry, electrolyte environment, and activation protocol: non-noble systems primarily benefit from enhanced oxygenated-species formation and improved poisoning tolerance, whereas noble-metal and hybrid catalysts exhibit more explicit bifunctional behavior involving adjacent hydroxide- or oxide-containing phases.

Collectively, these insights highlight the limitations of static structure–activity relationships and underscore the need to treat electrocatalysts as adaptive interfacial systems. The analysis further reveals that electrochemical activation cannot be considered a universal or transferable procedure, but rather a condition-dependent process whose mechanistic consequences must be evaluated in the context of specific reaction environments.

In conclusion, establishing predictive control over electrochemical activation requires a shift from empirical optimization toward activation-aware catalyst and interface design. Advances in operando characterization and spatially resolved electrochemical techniques will be essential to probe activation-induced surface states directly and to disentangle beneficial interfacial modifications from parasitic processes. Such efforts are expected to enable rational strategies for simultaneously controlling alcohol oxidation and hydrogen evolution, thereby accelerating the development of efficient and durable electrochemical energy-conversion systems.

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Conflict of interest statement

Authors have no conflict of interest to declare.

Data Availability

The data supporting this review's findings are from previously published studies and are available within the article. No new datasets or software were created for this study.

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