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Electrode–Electrolyte Interfaces and Double-Layer Effects in Electrochemical Systems

Ahmed El-Sayed*

Department of Chemistry, The American University in Cairo, Egypt

***Corresponding author:** Laura Bianchi, Politecnico di Milano, Italy, Email: a.elsayed@aucegypt.edu

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Abstract

In situ and operando electrochemical characterization techniques provide unprecedented insight into dynamic processes occurring at electrochemical interfaces. Unlike ex situ methods, these approaches capture real-time structural, chemical, and electronic changes under operating conditions. This article discusses recent advances in spectroscopic and microscopic techniques, including in situ Raman spectroscopy, X-ray absorption, and transmission electron microscopy. The role of these methods in elucidating reaction mechanisms and guiding materials design is emphasized.

Keywords:* *Operando electrochemistry, in situ characterization, electrochemical interfaces, spectroscopy

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Introduction

Electrochemical reactions are inherently dynamic, involving continuous changes at electrode surfaces and interfaces. Traditional post-mortem characterization often fails to capture transient states critical to understanding reaction pathways. In situ and operando techniques bridge this gap by enabling real-time observation of electrochemical processes. These methods have transformed the study of batteries, electrocatalysis, and corrosion, providing mechanistic insights that inform rational material design. The hydrogen evolution reaction is central to electrochemical technologies aimed at producing clean hydrogen fuel. HER kinetics strongly influence the overall efficiency of water electrolysis and other hydrogen-based energy systems. Although noble metals such as platinum provide benchmark activity, their limited availability necessitates alternative catalyst development. Recent research has focused on tailoring catalyst surfaces at the atomic level to optimize hydrogen adsorption and desorption steps. Understanding the mechanistic pathways of HER under different pH environments has become increasingly important, enabling rational catalyst design and improved performance. Electrochemical water splitting represents a promising solution to the global demand for clean energy carriers, particularly hydrogen. As concerns over fossil fuel depletion and greenhouse gas emissions intensify, hydrogen produced via water electrolysis offers an environmentally benign alternative. The process involves splitting water molecules into hydrogen and oxygen using electrical energy, ideally sourced from renewables such as solar or wind. Despite its conceptual simplicity, the practical realization of efficient water splitting is hindered by sluggish reaction kinetics and high overpotentials. Significant research efforts have therefore focused on developing efficient electrocatalysts and optimizing electrode–electrolyte interfaces to minimize energy losses. Understanding the interplay between materials chemistry, electrochemical kinetics, and system design is critical for advancing this technology toward large-scale implementation.

Conclusion

In situ and operando characterization has become indispensable for modern electrochemical research. Continued technological development and integration with theoretical modeling will further deepen understanding of complex electrochemical systems and accelerate innovation. Significant progress has been made in developing non-noble metal HER catalysts with competitive activity and stability. Continued exploration of electronic structure modulation and nanoscale engineering is expected to further enhance catalytic efficiency. Future work should prioritize scalable synthesis methods and long-term durability studies to facilitate industrial adoption.

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