Application Note

Evaluation and Stability Testing of Electrocatalysts for Water Electrolysis

Application Note: 2024/04

The stability of electrocatalysts employed in water electrolysis is not merely a consideration, but a critical determinant of the efficiency and durability of hydrogen production systems. Drawing insights from the article "A Guide to Electrocatalyst Stability Using Lab-Scale Alkaline Water Electrolyzers" (DOI: 10.1021/acsenergylett.3c02758)[1], this application note offers guidelines for performing electrochemical stability tests. It underscores the importance of electrocatalyst stability in advancing your research and applications in hydrogen production. Understanding and optimizing catalyst stability is essential for enhancing the reliability and longevity of electrolysis systems, ultimately contributing to the widespread adoption of clean energy technologies.[2]

Flow cells provide a practical and effective method for identifying and evaluating promising electrocatalysts. Unlike traditional three-electrode cells, flow cells can accommodate larger electrodes and support higher current densities, offering a more accurate testing environment for catalyst performance.[3] A key challenge in designing flow electrolyzers is minimizing ohmic cell resistance. This is achieved by compressing two porous electrodes against an ionic conductive separator, creating a zero-gap configuration.[4] During assembly, a typical flow electrolyzer uses stainless steel bolts tightened uniformly at low torque, with close monitoring of electrolyte temperature to ensure consistent conditions.

Electrochemical conditioning steps are essential before conducting performance and stability evaluations. For example, applying chronopotentiometry at 10 mA cm^{-2} followed by multistep chronopotentiometry involving sequential current steps to generate polarization curves. Electrochemical impedance spectroscopy (EIS) is conducted at each current level to assess high-frequency resistance (HFR), which reflects combined ion and electronic resistances. The HFR data is used for iR correction of the polarization curves, ensuring accurate characterization of the electrocatalyst's behavior under operational conditions. These meticulous steps are crucial for reliable and informative assessments of electrocatalytic performance in flow electrolyzer systems.

Electrode stability is a critical factor impacting the longevity and operational costs of water electrolysis systems, particularly when considering intermittent renewable power sources. Testing protocols must replicate actual industrial electrolyzer conditions by utilizing shorter timeframes and lower current densities. Accelerated degradation tests are essential to accurately simulate degradation mechanisms and develop robust electrocatalysts. These tests mimic realistic operating conditions over appropriate time scales, facilitating the identification of crucial degradation pathways.

An initial step towards benchmarking involves proposing a protocol for evaluating electrocatalytic material stability using lab-scale electrolyzers, as depicted in Figure 1. This protocol draws from benchmarking efforts in alkaline water electrolysis (AWE), Proton Exchange Membrane (PEM) electrolysis, and fuel cell research communities.

Six critical factors should be considered for performance evaluation:

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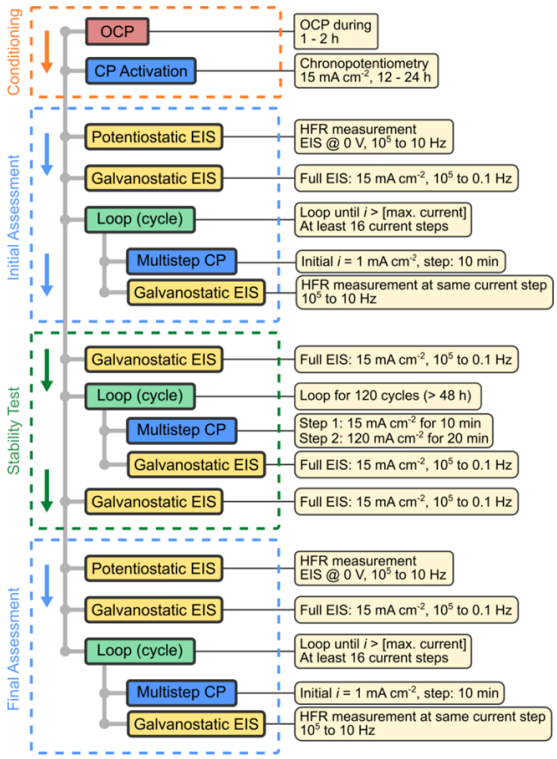
1. Focus on deactivation rates rather than stability duration or activity, utilizing metrics such as the activity-stability factor, S-number, or reverse-current stability factor.
2. Report reaction environments and setups in detail, utilizing reference materials for comparison.
3. Emphasize understanding catalyst deactivation and failure mechanisms over merely reporting stabilities.
4. Subject electrocatalysts to challenging environments, particularly under claims of "exceptional" stability, where intermittent operation truly tests catalyst resilience.
5. Ensure reproducibility and evaluate uncertainty in experimental results.
6. Use iR-free polarization curves to compare electrocatalytic activity and high-frequency resistance (HFR) to assess and monitor cell resistance, akin to iR compensation measurements in three-electrode cells.



Scheme 1. The recommended workflow for conducting electrochemical measurements is to evaluate electrocatalytic stability in lab-scale water electrolyzers under fluctuating current conditions. Reproduced from Ref. [1] by permission of the American Chemical Society.

By adhering to these critical factors, researchers can implement comprehensive and informative evaluations of electrocatalytic stability, facilitating the development of durable and efficient materials for industrial water electrolysis applications.

This protocol serves as an initial framework and requires further refinement for comprehensive electrocatalyst evaluation. Figure 1 illustrates the importance of conducting *ex-situ* characterization post-assessment to identify the most stable phase after preactivation. This step enables comparisons of structural and chemical compositions before and after stability testing. It's worth noting that destructive *ex-*

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situ characterization methods necessitate separate electrocatalyst samples. Characterizing hydrodynamics and mass transport is crucial for custom cell construction, as this will optimize performance and ensure reproducibility. Additional protocols are essential for specific electrolyzer evaluations, such as assessing separator stability and studying degradation mechanisms.

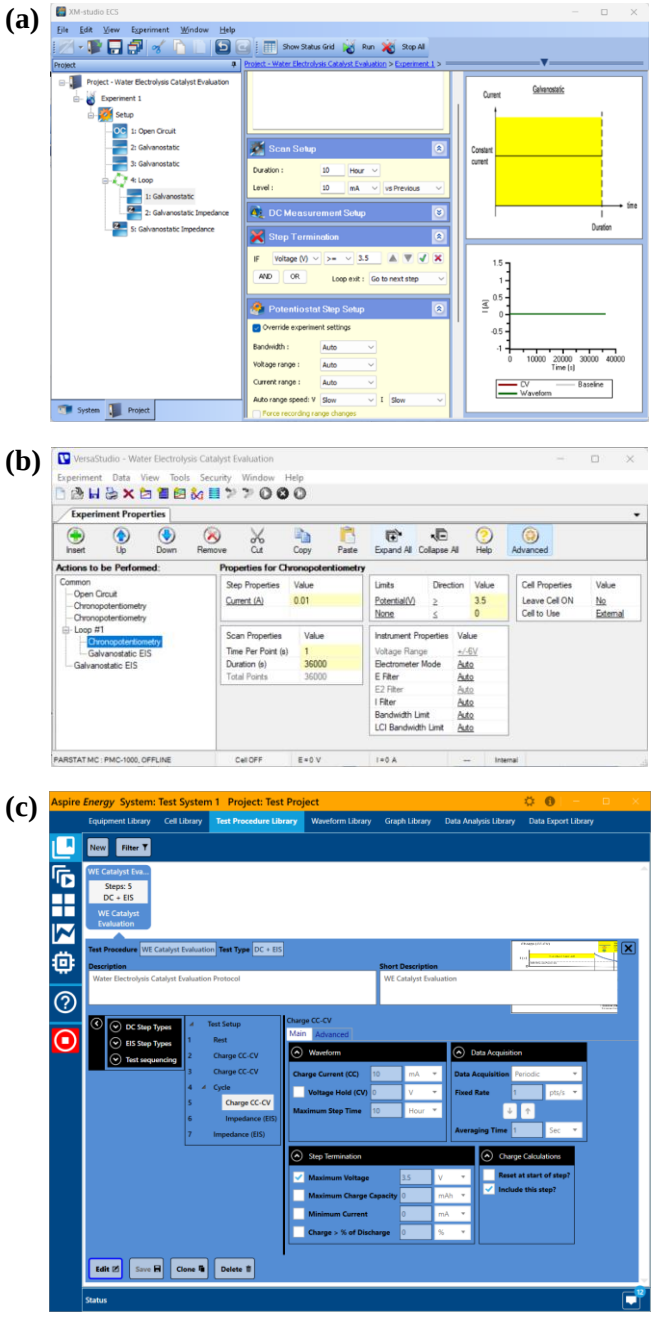

Figure 1. Electrolysis electrocatalyst evaluation and stability test procedure with (a) VersaStudio, (b) XM-studio, and (c) Aspire software. Test procedure files corresponding to these instruments and software are available upon request.

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The entire product line of AMETEK SI, which includes Princeton Applied Research and Solartron instruments with VersaStudio, XM-studio, and Aspire software (as depicted in Figure 1), is suitable for conducting water electrolysis electrocatalyst evaluations and stability tests. Test procedure files corresponding to these instruments are available upon request, providing a robust toolkit for comprehensive electrochemical research and development.

Reference

[1] Marquez, R. A., Espinosa, M., Kalokowski, E., Son, Y. J., Kawashima, K., Le, T. V., Chukwuneke, C. E., Mullins, C. B. A Guide to Electrocatalyst Stability Using Lab-Scale Alkaline Water Electrolyzers. *ACS Energy Lett.* **2024,** 9, 547–555.

[2] Edgington, J.; Seitz, L. C. Advancing the Rigor and Reproducibility of Electrocatalyst Stability Benchmarking and Intrinsic Material Degradation Analysis for Water Oxidation. *ACS Catalys*is **2023,** 13, 3379-3394.

[3] Domalanta, M. R., Bamba, J. N., Matienzo, D. D., del Rosario-Paraggua, J. A., Ocon, J. Pathways towards Achieving High Current Density Water Electrolysis: From Material Perspective to System Configuration. *ChemSusChem* **2023,** 16, e202300310.

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Summary – This application note outlines a comprehensive approach to evaluate the stability of electrocatalysts for alkaline water electrolysis based on the principles discussed in the referenced article. By employing these guidelines, researchers can assess electrocatalyst performance and develop strategies to enhance stability, ultimately advancing the development of efficient and durable hydrogen production technologies

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