

Integrated Comprehensive Experimental Design for Preparation, Characterization and Performance of NiFeOx Catalysts for Water Electrolysis Hydrogen Production: Teaching Reform Exploration Based on Renewable Energy Background in Xinjiang

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Abstract: To meet the requirements for cultivating students' comprehensive practical and innovative abilities under the background of the Emerging Engineering Education, this paper designs an integrated comprehensive experiment for water electrolysis hydrogen production covering catalyst preparation, structural characterization and performance testing. Taking the development of NiFeOx electrocatalysts as the carrier, the experiment integrates three modules: combustion synthesis, electrochemical measurement and Raman spectroscopy characterization. Guided by the strategic background of hydrogen energy, students systematically master the methods of catalyst design, performance evaluation and structural analysis, and deeply understand the "structure-performance" relationship. The experiment content is closely integrated with cutting-edge scientific research and the regional characteristics of abundant renewable energy in Xinjiang, emphasizing the deep integration of theory and practice. Through task-driven and group cooperation, it is suitable for undergraduate comprehensive experimental teaching of materials, chemistry, energy and other related majors. It effectively improves students' material design ability, instrument operation skills and data analysis literacy, and provides an operable teaching case for cultivating high-quality material talents with innovative thinking and the ability to solve complex engineering problems.

Keywords: Water electrolysis; Green hydrogen; NiFeOx electrocatalyst; Comprehensive experiment

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1. Introduction

Against the background of global energy structure transformation and the promotion of the "dual carbon"

strategy, hydrogen energy, as a clean and efficient renewable energy carrier, has become an important part of the future energy system ^[1]. Water electrolysis hydrogen production technology has attracted extensive attention due to its green and efficient characteristics. Especially in Xinjiang, an important clean energy base in China, its unique solar and wind energy resources provide superior natural conditions for large-scale green hydrogen production.

Using abundant renewable energy power for water electrolysis hydrogen production is not only a strategic path to realize efficient local energy conversion and build a green and low-carbon circular industrial system, but also points out a research direction with distinct regional characteristics for relevant material and technology research and development. However, the anodic oxygen evolution reaction (OER) in the water electrolysis process has become a bottleneck restricting the overall energy efficiency due to its slow four-electron transfer kinetics. Developing non-noble metal OER catalysts with high activity and stability is one of the core topics to reduce the energy consumption of electrolyzers and promote the development of the green hydrogen industry ^[2,3].

In recent years, nickel-iron (NiFe)-based materials have become one of the most promising catalyst systems due to their excellent alkaline OER activity and abundant resource reserves. Studies have shown that the intrinsic activity and stability can be significantly improved by constructing heterostructures, regulating electronic structures, introducing defects or using conductive carriers. In-depth understanding and practicing the internal “composition-structure-performance” relationship of such high-performance materials is an indispensable part of cultivating innovative talents in the field of new energy materials ^[4-6].

However, the current experimental teaching of related majors in colleges and universities generally has the problem of “focusing on unit operation, neglecting system integration”. Most experiments only focus on a single preparation method of materials (such as the hydrothermal method, electrodeposition), basic performance testing (such as LSV curves) or conventional structural characterization (such as XRD, SEM), lacking comprehensive training integrating material design, precise and controllable synthesis, multi-dimensional structural analysis, systematic performance evaluation and even preliminary exploration of reaction mechanism.

This teaching mode is difficult for students to establish a complete scientific research cognitive chain and systematic thinking to solve complex technical problems. Therefore, promoting the transformation of cutting-edge scientific research achievements into high-level experimental teaching resources and designing comprehensive and research-oriented experiments covering the whole process of material research and development has become an important direction of higher engineering education reform in the new era.

For example, Duan Junxin et al. prepared multi-morphology nickel-based electrodes by electrodeposition and explored their hydrogen evolution performance, improving students’ understanding of the relationship between material morphology and performance ^[7]; Li Yang et al. designed experiments on transition metal sulfide catalysts, strengthening students’ mastery of electrochemical testing methods ^[8]; Li Songrui et al. applied copper nanocluster composites to pesticide residue detection, reflecting the whole-process training of “material preparation-characterization-application” ^[9].

However, most existing experiments focus on a certain link (such as preparation or performance testing), but are still insufficient in systematically integrating material preparation, multi-scale characterization and performance correlation analysis, which makes it difficult to comprehensively cultivate students’ engineering thinking and scientific research literacy.

Based on this, the teaching team, relying on long-term scientific research accumulation in the field of

MXene-based energy materials, designed this comprehensive experiment. Taking the new two-dimensional material MXene (Ti_3C_2) as the structure-directing agent and conductive bridge, $\text{Fe}_2\text{O}_3/\text{Ni}(\text{OH})_2$ heterojunction nanoarrays were controllably constructed on three-dimensional nickel foam skeletons through step-by-step electrodeposition and hydrothermal method.

Students will personally practice the complete research process from substrate treatment, layer-by-layer material assembly, morphology/structure characterization, to comprehensive evaluation of electrochemical performance and even in-situ Raman observation (**Figure 1**). This experiment not only covers interdisciplinary knowledge of materials, chemistry, physics and other disciplines, but also focuses on guiding students to think about the core scientific issue of “how to regulate catalytic performance through material design and interface engineering”, aiming to comprehensively improve students’ innovative practical ability and comprehensive scientific research literacy.

This experiment transforms the cutting-edge research of green hydrogen catalytic materials into experimental teaching content with local characteristics. It not only pays attention to scientific research feeding teaching, closely connects with regional energy development needs, stimulates students’ learning interest and scientific research enthusiasm, but also guides them to cultivate a sense of responsibility and mission to serve major national strategies and regional economic and social development in the process of solving practical problems, providing a new idea for local colleges and universities to carry out comprehensive experimental teaching with regional characteristics.

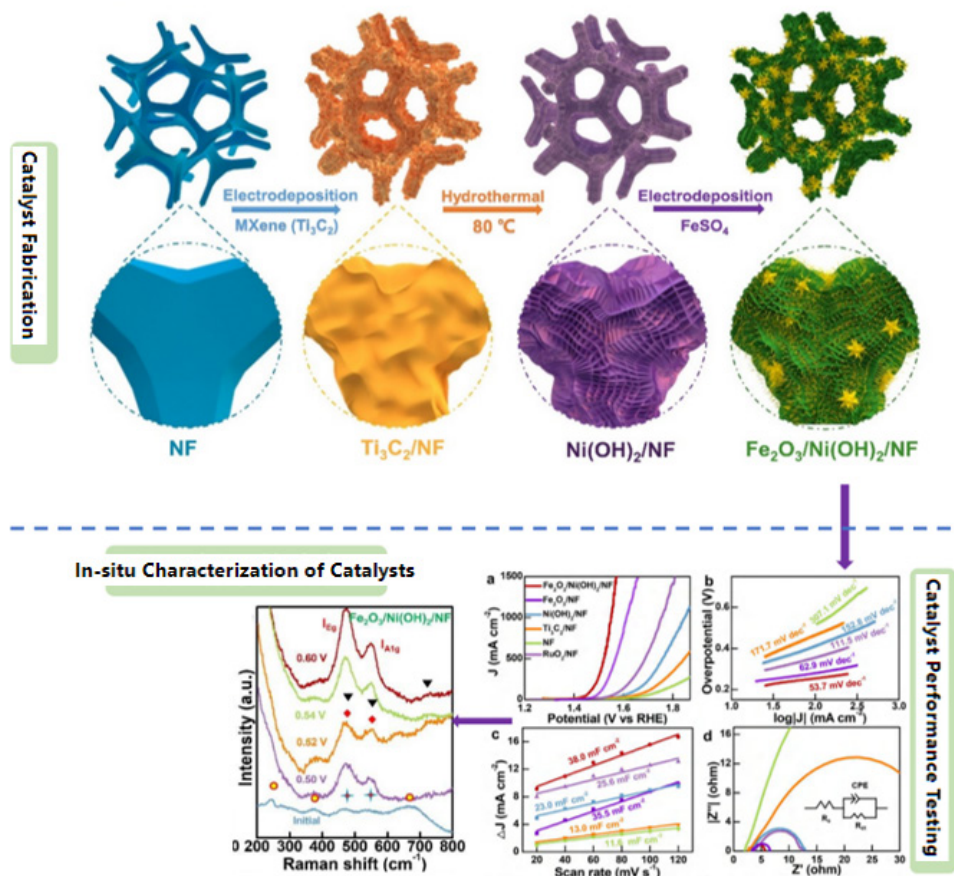


Figure 1. Integrated experimental route of “preparation-structure-performance” of NiFeOx catalyst.

2. Experimental design concept and purpose

2.1. Design concept

This experiment follows the concept of “scientific research feeding teaching, theory connecting with practice, focusing on process exploration” and constructs a miniature “material research and development project”:

- (1) Simulate the real scientific research process, covering the complete chain of “literature research and scheme design → controllable material synthesis → structural characterization → systematic electrochemical performance test → structure-activity relationship analysis and mechanism discussion”.
- (2) Integrate modern material research methods such as electrochemical deposition, low-temperature hydrothermal synthesis, Raman spectroscopy and a variety of electrochemical testing techniques.
- (3) Focus on key questions such as “How does MXene assist the growth of ordered nanostructures?”, “Why can the introduction of Fe significantly improve the OER activity of Ni(OH)₂?”, “What is the real active center of the catalyst during the reaction?”, guiding students from phenomenon to essence.
- (4) Place the experimental theme in the context of renewable energy hydrogen production in Xinjiang, and guide students to think about the special requirements of intermittent renewable energy power supply for catalyst performance (such as response speed, stability) in the introduction and discussion sections.

2.2. Experimental purpose

- (1) Enhance students’ understanding of the “dual carbon” strategy and establish a sense of mission to serve national energy needs through material innovation;
- (2) Understand the basic principle of water electrolysis hydrogen production, the mechanism of electrocatalysts and the structure-activity relationship of NiFeOx materials;
- (3) Master electrodeposition-hydrothermal synthesis technology, operation of electrochemical workstation, use of Raman spectrometer and data fitting analysis methods;
- (4) Cultivate comprehensive scientific research literacy such as material design, experimental planning, team cooperation, data interpretation and scientific report writing.

3. Experimental content

3.1. Reagents and instruments

3.1.1. Experimental chemicals

Ti₃C₂ dispersion (3 mg mL⁻¹), nickel foam (NF, 1 mm thick), deionized water, ferrous sulfate (FeSO₄), etc.

3.1.2. Experimental instruments

Constant temperature water bath, vacuum drying oven, platinum electrode clip, carbon rod electrode, Hg/HgO reference electrode, DC stabilized power supply, electrochemical workstation (CHI760E), field emission scanning electron microscope (FESEM, Apreo S LoVac), transmission electron microscope (TEM), X-ray photoelectron spectrometer (XPS, K-Alpha+), laser confocal Raman spectrometer (LabRAM HR Evolution).

3.2. Experimental steps

- (1) Preparation of Fe₂O₃/Ni(OH)₂ catalyst

A step-by-step electrodeposition-water bath heating method was adopted. Cut nickel foam (2.5 cm × 3.0 cm as anode, 2.5 cm × 3.5 cm as cathode) was used as a substrate. Ti₃C₂ was electrodeposited at a constant current of 5 mA for 10 min, and Ti₃C₂/NF was obtained after drying. It was treated in a water

bath at 80 °C for 3 h to grow Ni(OH)₂ nanoarrays in situ, and Ni(OH)₂/NF was obtained after drying. Using Ni(OH)₂/NF as cathode and nickel foam as anode, constant current electrodeposition (10 mA cm⁻²) was carried out in FeSO₄ solution (0.02–0.06 mol L⁻¹). After oxidation at room temperature and drying, Fe₂O₃/Ni(OH)₂/NF composite electrode was obtained.

(2) Performance test of Fe₂O₃/Ni(OH)₂ catalyst

A three-electrode system was adopted, with 1.0 mol·L⁻¹ KOH as electrolyte, the prepared electrode as working electrode, Hg/HgO as reference electrode and graphite rod as counter electrode. LSV, CV, EIS and i-t stability tests were carried out through an electrochemical workstation.

(3) Structural characterization of Fe₂O₃/Ni(OH)₂ catalyst

SEM and TEM were used to observe the morphology, and Raman spectroscopy was used to analyze the phase and active species evolution.

4. Results and discussion

4.1. Morphology and structural characterization of catalysts

SEM images (**Figure 2a**) show that Ni(OH)₂ nanosheets grow vertically on the NF skeleton after hydrothermal treatment; after Fe₂O₃ electrodeposition, acicular/petal-like particles are uniformly distributed on the surface and gaps of nanosheets (**Figure 2b**). HRTEM confirms the coexistence of Ni(OH)₂ (101), TiO₂ (101) and Fe₂O₃ (311) crystal planes (**Figure 2c**).

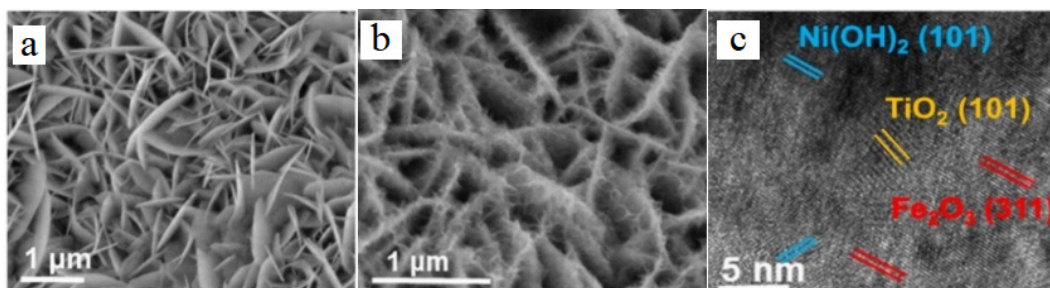


Figure 2. SEM images of Ni(OH)₂/NF electrode prepared by the hydrothermal method of Ti₃C₂/NF (a), SEM image (b), and TEM image (c) of Fe₂O₃/Ni(OH)₂/NF electrode.

In the Raman spectrum (**Figure 3**), characteristic peaks of Fe₂O₃ (240, 373, 674 cm⁻¹) appear after Fe deposition, and no TiO₂ signal is detected, indicating that Ti₃C₂ is partially oxidized but with low content.

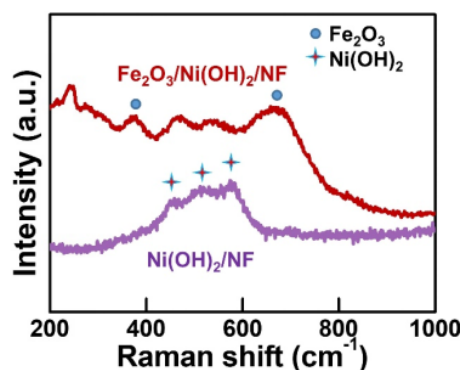


Figure 3. Raman spectra of Ni(OH)₂/NF and Fe₂O₃/Ni(OH)₂/NF electrodes.

4.2. Electrochemical performance study of Fe₂O₃/Ni(OH)₂/NF catalyst

The LSV curve (Figure 4a) shows that the overpotentials of Fe₂O₃/Ni(OH)₂/NF at 500, 1000, 1500 mA cm⁻² are 291, 319, 341 mV, respectively, which are significantly better than commercial RuO₂ and each control group, indicating that Fe doping effectively optimizes the electronic structure of Ni(OH)₂ and enhances OER activity.

In the i-t test (Figure 4b), the electrode runs stably at 1300 mA cm⁻² for 17 h with no obvious attenuation of current density, showing excellent high-current stability. The above results indicate that the introduction of Fe₂O₃ not only improves the OER catalytic activity of the catalytic electrode at high current density.

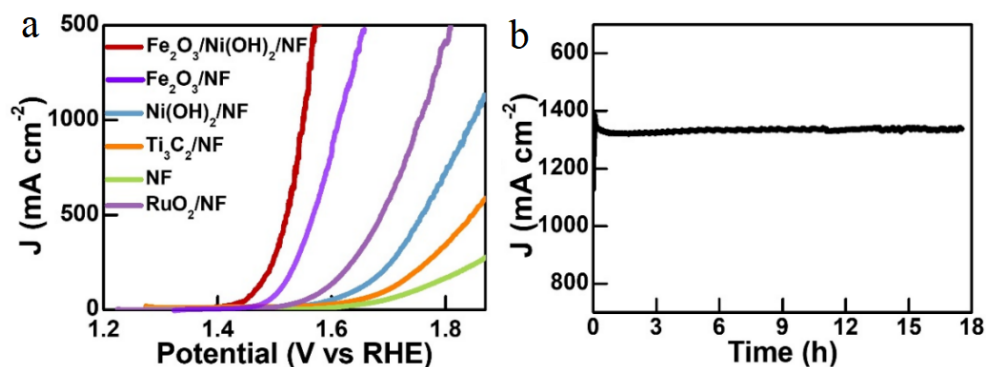


Figure 4. LSV polarization curves of NF, Ti₃C₂/NF, TiO₂/Ni(OH)₂/NF, RuO₂/NF, Fe₂O₃/NF and Fe₂O₃/Ni(OH)₂/NF electrodes (a); i-t stability test of Fe₂O₃/Ni(OH)₂/NF electrode (b).

4.3. Oxygen evolution reaction mechanism of Fe₂O₃/Ni(OH)₂/NF catalyst

In-situ Raman spectroscopy (Figure 5) shows that the characteristic peaks of Fe₂O₃ and Ni(OH)₂ disappear at OER potential, while overlapping peaks belonging to FeOOH (474, 728 cm⁻¹) and NiOOH (549 cm⁻¹) appear, and the intensity increases with the increase of potential. It confirms that the catalyst surface is transformed into hydroxy oxide active phase in situ, and FeOOH and NiOOH synergistically drive the OER process.

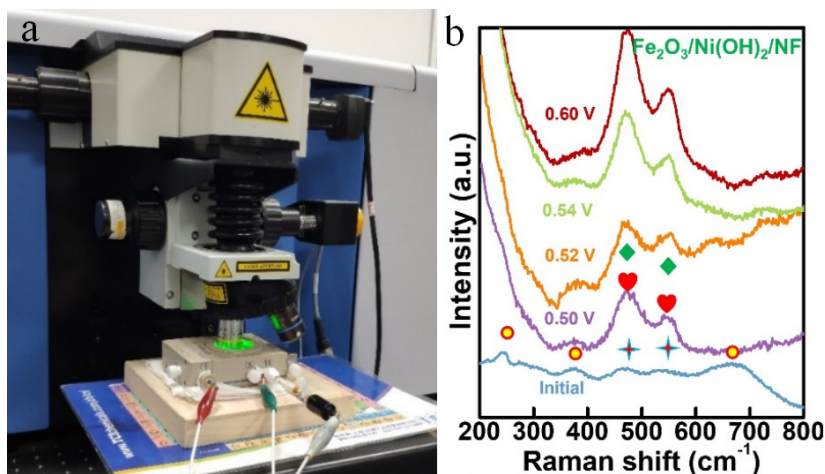


Figure 5. In-situ Raman spectroscopy characterization device of Fe₂O₃/Ni(OH)₂/NF catalyst and corresponding Raman spectra at different voltages.

5. Discussion

During the experiment, the following matters should be noted to ensure safety and improve the experimental effect:

- (1) Experimental operation specifications: keep the electrode spacing constant and current stable during electrodeposition to avoid uneven material growth caused by uneven current density; prevent scalding during water bath heating reaction;
- (2) KOH used in the electrochemical test is a strong alkali, so be careful during operation. If it splashes on skin or eyes accidentally, rinse with plenty of water immediately and seek medical advice in time. Check whether the connection of each electrode is firm and whether the reference electrode solution is sufficient before using the electrochemical workstation;
- (3) Before the Raman spectroscopy test, ensure that the sample surface is clean and dry to avoid interference from impurity peaks;
- (4) RHE potential should be uniformly used for calibration during data processing to ensure the comparability of results.
- (5) After the experiment, waste electrolyte, chemical reagents and samples should be collected by classification and harmlessly treated according to laboratory regulations.

6. Experimental implementation plan and teaching suggestions

6.1. Experimental implementation plan

The total class hours of this experiment are recommended to be 12 class hours, implemented in three stages, with specific arrangements as follows:

- (1) Stage 1 (4 class hours): Material preparation
Students prepare $\text{Ti}_3\text{C}_2/\text{NF}$ carrier, hydrothermal growth of $\text{Ni}(\text{OH})_2$ and electrodeposition of Fe_2O_3 in groups. Teachers explain the structural characteristics of MXene, the principle of electrodeposition and the hydrothermal synthesis mechanism, and guide students to complete material preparation.
- (2) Stage 2 (4 class hours): Electrochemical performance test
Students conduct LSV, Tafel, EIS and stability tests, and learn the operation of the electrochemical workstation and data analysis methods. Teachers guide students to compare the performance differences of different catalyst samples and understand the “composition-structure-performance” relationship.
- (3) Stage 3 (4 class hours): Material structural characterization and data analysis
Combined with Raman characterization results, group discussions are carried out to analyze the evolution of active centers of $\text{Fe}_2\text{O}_3/\text{Ni}(\text{OH})_2$ oxygen evolution reaction. Students write experimental reports, including data charts, result analysis and thinking questions.

6.2. Teaching suggestions

- (1) Adopt the “problem-oriented + task-driven” teaching mode, focusing on the core issue of “how to improve the OER performance of NiFe-based catalysts”, guide students to design experimental schemes, analyze data and draw conclusions independently.
- (2) Strengthen scientific research thinking training, encourage students to consult literature before class to understand the latest progress of NiFe catalysts and MXene in the energy field; pay attention to observation and recording of abnormal phenomena during the experiment to cultivate the ability to find

and solve problems.

- (3) Promote “integration of science and education”, encourage outstanding students to enter the scientific research team to participate in relevant research projects, further deepen the experimental data into graduation theses or innovative project content, and realize the positive interaction between teaching and scientific research.
- (4) Focus on process evaluation, assess from multiple dimensions such as standardization of experimental operation, data quality, analysis depth, report integrity and innovation, weaken single result orientation, and strengthen comprehensive literacy training.

7. Conclusion

Based on the “carbon neutrality” strategy and the development of renewable energy in Xinjiang, this experiment transforms the cutting-edge scientific research direction — preparation, characterization and performance research of MXene-based NiFe electrocatalysts into integrated comprehensive experimental teaching content.

Through the whole-process training covering “material design → controllable synthesis → structural characterization → performance evaluation”, students not only master electrodeposition, hydrothermal synthesis, Raman spectroscopy and a variety of electrochemical testing techniques, but also deepen their understanding of the “structure-performance” relationship and catalytic reaction mechanism in practice.

The experimental design pays attention to regional characteristics and scientific research feeding teaching, stimulates students’ sense of responsibility and innovation to serve the national energy strategy, and effectively improves their comprehensive scientific research literacy in material design, instrument operation, data analysis and scientific expression.

This experiment provides a reference case for carrying out high-level, comprehensive and research-oriented experimental teaching for new energy materials-related majors, and helps to cultivate high-quality engineering talents with a solid theoretical foundation, systematic engineering thinking and innovative practical ability.

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Disclosure statement

The authors declare no conflict of interest.

References

- [1] Zheng ZX, Xing GY, Peng S J, et al., 2025, Progress and Perspectives on NiFe Layered Double Hydroxide Catalysts for the Alkaline Oxygen Evolution Reaction. *eChem*, 1(1): 3.
- [2] Liu Y, Wang KL, Huang GT, et al., 2025, Advancing Seawater Electrolysis: NiFe-LDH-Based Electrocatalysts for

- the Oxygen Evolution Reaction. *Advanced Energy Materials*, 2025: e04101.
- [3] Wang ZL, Chen CC, Huang GY, et al., 2026, Precise Phase Regulation of Heterogeneous Structures between Two-Dimensional High Entropy Spinel and LDH for Efficient Oxygen Evolution Reaction. *Applied Catalysis B: Environmental*, 386(5): 126382.
- [4] Jia JZ, Cha YY, Wang ZW, et al., 2025, Boosting OER Performance of NiFe-MOFs via Heterostructure Engineering: Promoted Phase Transformation and Self-optimized Dynamic Interface Electron Structure. *Advanced Functional Materials*, 35: 2500568.
- [5] Li H, Zheng JJ, Bai X, et al., 2025, Synergistic Co and P Co-Doping in MoS₂/NiO Heterostructures for Electronic Modulation Toward High-Performance Alkaline Water Electrolysis. *Advanced Functional Materials*, 2025: e25383.
- [6] Zhao HX, Wang Y, Zhou Z, et al., 2026, Ni, Fe Carbonates/Silicates Heterointerfaces Boosting Oxygen Evolution Reaction. *Carbon Neutralization*, 5: e70110.
- [7] Duan JX, Xu XF, Fan QL, et al., 2025, Preparation of Different Morphologies Ni@Copper Foam (Ni@CF) Electrode Materials and Comprehensive Experimental Design of Electrocatalytic Hydrogen Evolution. *Experimental Technology and Management*, 42(11): 237–243.
- [8] Li JC, Zhao HW, Li Y, 2025, Comprehensive Physical and Chemical Experiment of Hydrogen Production by Water Electrolysis: Preparation, Characterization and Performance of Transition Metal Sulfide Catalysts. *Guangdong Chemical Industry*, 52(11): 171–173.
- [9] Li SR, Jia Q, Zheng HJ, et al., 2026, Comprehensive Experiment of Copper Nanocluster Composites for Fluorescence Detection of Pesticide Residues. *University Chemistry*, 41(X): 1.

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