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Reform Path of Electrocatalytic CO_2 Reduction in Innovative Practical Teaching of Materials Chemistry

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Abstract: Electrocatalytic carbon dioxide (CO_2) reduction to high-value chemicals driven by renewable energy provides an effective approach to reduce CO_2 emissions and meet energy demands. Cultivating talents with professional literacy in the new energy field is an important task for undergraduate institutions. In particular, the operation of laboratory scientific research instruments enables students to form a more intuitive understanding of the phase and microstructure of materials, thereby enriching their knowledge of the materials discipline. This study introduces scientific research projects into the innovative practice of materials majors. Through the design of this innovative practice, it aims to cultivate students' innovative thinking, improve their understanding of new energy materials, enhance their ability to use equipment and analyze and process data, help them better grasp international cutting-edge directions, improve their scientific research and innovation capabilities, and achieve the goal of integrating theory with practice.

Keywords: Electrocatalysis; CO_2 reduction; Materials chemistry; Practical teaching

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1. Introduction

With the increasing progress of science and technology, further deepening the reform of teaching methods and innovating practical textbooks and content have become the key to cultivating innovative talents. The training of students majoring in materials chemistry should not be limited to classroom lectures and demonstrations, as this is difficult to stimulate students' active thinking. If teachers only lecture or demonstrate in class without guidance, students may struggle to understand the knowledge content, study merely for scores, and even engage in undesirable phenomena such as plagiarism. Cultivating innovative talents requires guiding students to discover problems and solve them through a series of practices and investigations. Therefore, materials innovation practice can, based on relevant scientific research projects, guide students to complete four parts: literature research, catalyst preparation, catalytic performance testing, and data processing, so as to improve their practical ability and gradually cultivate their divergent thinking and scientific research innovation capabilities.

2. Teaching Objectives of Applying Electrocatalytic CO_2 Reduction to Practical Teaching of Materials Chemistry

2.1. Master the Use of Various Academic Databases and Professional Retrieval Tools

In the process of participating in materials chemistry innovative practice related to electrocatalytic CO_2 reduction, students need to systematically master the basic ability of literature retrieval and academic resource utilization, be familiar with the operation interfaces and retrieval logic of mainstream Chinese and English databases such as Web of Science, Scopus, PubMed, and CNKI, and be able to accurately locate required literature using methods such as subject terms, keyword combinations, and Boolean logic operations [1]. In practical operations, students should conduct multiple rounds of iterative retrieval according to the research direction set by teachers, such as "preparation of atomically dispersed metal-nitrogen-carbon catalysts" or "construction of NiCu bimetallic active sites in CO_2 electroreduction", and gradually optimize the query strategy to improve information acquisition efficiency.

In the literature reading stage, students should focus on analyzing research results in high-level journals and summarize the main synthesis paths of carbon-based atomic catalysts [2]. Among them, high-temperature pyrolysis, as a widely used technology, its principle lies in the precursor undergoing carbonization, ligand decomposition, and metal atom anchoring processes under an inert atmosphere to form stable single-atom or dual-atom active centers. This method has low equipment requirements and can be prepared in batches, but there is a risk of metal agglomeration, leading to uneven distribution of active sites. Another wet chemical method achieves uniform dispersion of metal atoms through coordination self-assembly in a liquid phase environment, combined with subsequent washing and drying steps to obtain target materials, which has the advantage of strong component controllability, but the process flow is long and solvent consumption is large [3]. The template method uses structure-directing agents such as mesoporous silica or polymer microspheres to regulate the pore structure and specific surface area of carbon carriers, allowing metal atoms to stably exist in confined spaces. The resulting materials have excellent mass transfer performance, but the template removal process may cause structural collapse.

2.2. Master the Synthesis Process and Characteristics of NiCu-Based Porous Carbon Materials

The synthesis process of NiCu-based porous carbon materials is one of the core links in the experimental teaching of electrocatalytic CO_2 reduction, involving multiple key technical steps such as material precursor selection, metal doping control, pyrolysis process regulation, and pore structure construction. Students should practically master the composite preparation route combining hydrothermal method and high-temperature carbonization, and understand the entire process from liquid-phase reaction to solid-phase material formation [4]. In the initial stage of the experiment, students can select glucose or chitosan as the carbon source, nickel nitrate and copper nitrate mixed in a set molar ratio as the bimetallic precursor, and an appropriate amount of urea to adjust pH and promote uniform precipitation. The above system is subjected to hydrothermal treatment in a high-pressure reactor at 180 °C for 12 hours to form a carbonaceous gel precursor rich in metal ions.

Subsequently, centrifugal washing and vacuum drying are performed to obtain a dry powdered precursor.

The dried precursor is transferred to a tube furnace for two-stage pyrolysis under a nitrogen atmosphere [5]. After pyrolysis, it is naturally cooled to room temperature, and dilute acid washing is used to remove unstable metal particles, retaining NiCu alloy nanoparticles embedded in the carbon matrix. After filtration and re-drying, the final black powdered NiCu porous carbon catalyst is obtained. The entire synthesis process integrates solution chemistry, thermodynamic control, and solid-phase reaction principles, which can help students deeply understand the synergistic source of high specific surface area, good electrical conductivity, and metal dispersion of porous carbon materials, and improve their practical ability and scientific thinking level.

2.3. Master Common Characterization Methods for the Structure and Morphology of Electrocatalytic Materials

X-ray diffraction (XRD) is used to determine the crystal structure, phase composition, and grain size of materials. By comparing with standard cards, it can be judged whether the synthesized material is the target product, and the crystallinity and grain size can be estimated according to the full width at half maximum of diffraction peaks. Scanning electron microscopy (SEM) provides information on the surface morphology of materials, allowing students to more intuitively observe the particle distribution, agglomeration state, and porous characteristics of the catalyst. Combined with energy dispersive spectroscopy (EDS), element mapping analysis can be performed to verify the uniformity of metal components. Transmission electron microscopy (TEM) further reveals the internal structural details of materials, such as lattice fringes, interplanar spacing, and morphological characteristics at the nanoscale, helping to confirm the crystallinity and exposed crystal planes of materials [6].

Specific surface area and pore structure are determined by nitrogen adsorption-desorption isotherms. The specific surface area is calculated by the BET model, and the pore size distribution is analyzed by the BJH or NLDFT method to evaluate the porous characteristics of materials, which is important for understanding the number of reactive active sites and mass transfer processes. X-ray photoelectron spectroscopy (XPS) is used to analyze the chemical state and valence state of surface elements, identify the oxidation state of metal elements and the existence form of doped elements, and provide a basis for exploring the catalytic mechanism. Raman spectroscopy can detect the defect density and graphitization degree of carbon-based materials. The ID/IG value reflects the structural order of materials, assisting in judging their electrical conductivity and stability [7]. Mastering these characterization methods and data processing skills helps students improve their ability to independently conduct material analysis, laying a solid foundation for in-depth exploration of electrocatalytic reaction mechanisms and optimization of material design in the future.

3. Practical Teaching Path of Electrocatalytic CO_2 Reduction in Materials Chemistry

3.1. Emphasize Literature Research

As an important starting point of experimental teaching on electrocatalytic CO_2 reduction, literature research is a basic link for students to deeply understand the scientific issues and technical challenges in this field [8]. In teaching practice, teachers need to guide students to focus on the key bottlenecks of electrocatalytic conversion, such as low reaction selectivity, high overpotential, and insufficient catalyst stability, clarify the direction of experimental design, and focus on reading review and research papers on transition metal-based catalysts, carbon-based composite materials, and gas diffusion electrode design to master the design ideas and performance optimization strategies of current mainstream material systems. For example, some studies have revealed the synergistic effect of NiCu bimetallic active centers in promoting CO_2-to-CO conversion, providing a theoretical basis for the selection of NiCu-based porous carbon materials in subsequent experiments.

Literature research is not limited to material design and performance optimization, but also includes comparative analysis of experimental conditions such as electrolytic cell configuration, electrolyte selection, and gas supply method. Through comparative learning of three-electrode systems and flow cell configurations, students can gradually form the ability to conceive complete experimental schemes [9]. In the teaching process, teachers need to set up phased report sessions, requiring students to present the obtained information in groups and discuss, enhancing teamwork and critical thinking abilities, and promoting students to shift from passive acceptance of knowledge to active exploration of scientific issues, cultivating independent thinking and information integration abilities in real scientific research scenarios. In short, the results of literature research directly serve the subsequent catalyst preparation and performance testing links, making the entire teaching process have a clear logical chain and research orientation.

3.2. Practical Preparation Chemistry

Catalyst preparation is a core link in the experimental teaching of electrocatalytic CO_2 reduction, directly related to the evaluation of subsequent catalytic performance and the understanding of structure-function relationships of materials [10]. In teaching practice, teachers should select NiCu-based porous carbon materials as the research object, which have high electrical conductivity, rich active sites, and good stability, making them suitable for CO_2 reduction reactions. Then, nickel nitrate, copper nitrate, and 2-methylimidazole are dissolved in methanol in a certain molar ratio, stirred at room temperature for 12 hours to form a uniform solution, and then the precipitate is collected by centrifugation and dried to obtain the NiCu-ZIF composite precursor.

After the precursor preparation is completed, pyrolysis treatment is performed. It is heated to the target temperature at a specific heating rate under the protection of an inert atmosphere and kept for a period of time [11]. During pyrolysis, the organic ligand is carbonized to form a conductive network, and metal ions are reduced to nanoparticles and uniformly embedded in the carbon skeleton, forming a porous carbon material with high specific surface area and rich pore structure. Students need to record the sample number and appearance characteristics under different pyrolysis parameters, establishing a preliminary correlation between process conditions and material properties.

The obtained catalyst is ground and mixed with Nafion solution and ethanol by ultrasonic dispersion to form a uniform ink-like suspension. It is loaded on the surface of a glassy carbon electrode by drop-coating and air-dried naturally. Under the guidance of teachers, students independently complete the preparation of at least three types of catalysts with different compositions or pyrolysis conditions, mark and store them for subsequent linear sweep voltammetry, chronoamperometry, and gas chromatography analysis [12]. The entire preparation process integrates inorganic synthesis, high-temperature treatment, and electrode processing technology, which can help students systematically master the full-process operational skills of functional materials from molecular design to electrode construction.

3.3. Test Catalytic Performance

Catalytic performance testing is an indispensable core link in the experimental teaching of electrocatalytic CO_2 reduction, directly reflecting the practical application potential of the prepared catalysts. Gas products are detected by gas chromatography (GC). After constant potential electrolysis for a period of time, gas samples above the reaction system are collected for component analysis to identify target products such as carbon monoxide, methane, and ethylene, and the selectivity of each product is calculated in combination with Faraday efficiency [13]. Liquid products are qualitatively and quantitatively analyzed by proton nuclear magnetic resonance (^1H NMR) or high-performance liquid chromatography (HPLC) to determine the presence of oxygen-containing organic compounds such as methanol, ethanol, and formic acid.

Chronoamperometry or chronopotentiometry is used to evaluate the durability of the catalyst during long-term operation. Students record the trend of current change over time during electrolysis, observe whether there is obvious attenuation, and then analyze problems such as possible structural reconstruction, metal dissolution, or surface contamination of the material. Electrochemical impedance spectroscopy (EIS) provides information about the interfacial electron transfer resistance. The charge transfer resistance value is obtained by fitting the Nyquist plot, assisting in judging the electrical conductivity and reaction kinetics of the catalyst. In the entire testing process, students also need to learn how to control experimental variables, such as electrolyte type, pH value, CO_2 flow rate, and electrolysis temperature, to explore the influence of these factors on catalytic performance. All test results need to be compared with commercial catalysts or existing literature data to improve students' scientific judgment ability [14].

3.4. Optimize Data Processing

After completing catalyst preparation and performance testing, students need to systematically organize and in-depth analyze the obtained raw data. Cyclic voltammetry (CV) curves are used to identify the redox behavior of materials at different potentials. Combined with background current subtraction and double-layer capacitance calculation, the electrochemical active surface area of the electrode can be evaluated. The limiting current density obtained by linear sweep voltammetry (LSV) reflects the kinetic promotion ability of the material for the CO_2 reduction reaction. Students need to compare the response differences of samples with different NiCu ratios under the same conditions to understand the mechanism of metal component regulation on the electronic structure [15]. Chronoamperometry test results reflect the stability of the catalytic process. The degree of current attenuation during long-term operation is directly related to material durability, and students can judge whether the material has practical application potential based on this.

All data are presented in a standardized chart form, with error bars reflecting the standard deviation of multiple repeated experiments. When writing analysis reports, students need to combine material characterization results, such as crystal structure revealed by XRD, valence state information provided by XPS, and microscopic morphology observed by SEM/TEM, to establish a "structure-performance" correlation. For example, high specific surface area and hierarchical pore structure are conducive to CO_2 adsorption and diffusion, and the Ni-Cu synergistic effect may reduce the energy barrier for COOH intermediate formation. Teachers should guide students to discuss the sources of experimental deviations, help them master the ability to extract key information from massive data, and form a research habit of deriving conclusions based on evidence.

4. Conclusion

This innovative practice fully combines practical teaching with teachers' scientific research projects, and always adheres to practice and innovation as the core points throughout the process, focusing on cultivating students' practical operation ability and giving play to their subjective initiative. The practical links of teaching can cultivate students' scientific research thinking and innovative awareness, allowing students to gradually master scientific research methods in practice. At the same time, teaching practice also focuses on exercising students' teamwork ability and independent thinking ability, enabling students to learn communication and cooperation in teamwork and improve their problem-solving ability in independent thinking. In addition, this innovative practice has important guiding significance for the education of materials chemistry students, which can successfully connect the professional knowledge learned by students with the development of new social energy sources, let students understand the cutting-edge trends of the discipline and social needs, and lay a solid foundation for their future employment.

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