Proceedings of the 4th International Conference on Functional Materials and Civil Engineering
DOI: 10.54254/2755-2721/2026.33575

Research Progress on the Application of Molybdenum Disulfide Materials in Electrocatalysis

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Abstract. The development of efficient electrocatalytic technologies is an important topic in the energy field as the world energy structure increasingly leans towards cleanliness. Electrocatalysis plays a crucial role in water electrolysis and fuel cells due to its ability to operate at normal temperature and pressure, easy reaction control and high efficiency. The electrode material is crucial to electrocatalysis as it determines the performance of the entire electrocatalysis. Molybdenum disulfide (MoS_2), a typical two-dimensional transition metal sulfide, is a promising non-noble metal electrocatalyst due to its unique layered structure and adjustable electronic properties. However, pure-phase MoS_2 has problems such as lower conductivity, surface passivation and fewer active sites. Therefore, this paper first summarizes the basic concepts of electrocatalysis and the composition of electrocatalytic devices, and then summarizes the preparation methods of pure phase MoS_2 such as hydrothermal/solvothermal method, chemical vapor deposition, and rapid thermal annealing method, And the effects of different modification methods, such as introducing heteroatoms, constructing heterostructures, or combining with carbon materials, on the structure of MoS_2 and its electrocatalytic activity are described. Studies have shown that the use of zero-valent cobalt intercalation doping, MoS_2@Mo_2C heterojunctions, and MoS_2/MWCNT composites can significantly reduce the overpotential of the hydrogen evolution reaction, improve the hydrogen adsorption free energy, and enhance its stability. By summarizing, analyzing and comparing the current related work, the mechanism of the effects of different modification methods on the electrocatalytic activity of MoS_2 was obtained, thus laying the foundation for the further development of efficient non-precious metal electrocatalysts.

Keywords: Electrocatalysis, Molybdenum disulfide, Non-precious metal catalysts, Doping modification, Heterojunction

1. Introduction

The development of efficient and clean energy conversion technologies is currently a hot topic in the energy field as the global energy structure moves towards a more environmentally friendly and low-carbon direction. The extensive use of fossil fuels has led to resource shortages and serious pollution problems, prompting humanity to accelerate the development and utilization of renewable energy. Electrocatalysis is a method that promotes chemical changes on electrodes by applying voltage under normal temperature and pressure conditions. It is characterized by controllable reactions,

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DOI: 10.54254/2755-2721/2026.33575

environmental friendliness, low carbon emissions, and high efficiency, and has good application prospects in green energy conversion such as water electrolysis, CO_2 reduction, nitrogen reduction, and fuel cells. The electrolyzer is the main equipment for electrocatalytic reactions, and its internal composition and the materials used will affect the effectiveness and durability of the electrocatalytic process. The electrode material is an important factor in electrocatalysis, and the microstructure and electronic properties of the electrode material have a significant impact on the activity and selectivity of electrocatalysis. At present, although noble metal-based catalysts have good electrocatalytic effects, their large-scale application is limited due to their high cost and limited resources [1]. In recent years, non-noble metal catalysts represented by molybdenum disulfide (MoS_2) have received extensive attention in electrocatalysis due to their special two-dimensional layered structure, large specific surface area and a large number of edge active sites [2]. However, pure-phase MoS_2 (mainly 2H semiconductor phase) itself has problems such as poor electrical conductivity, surface passivation and few active sites. Therefore, researchers have developed a series of modifications including heteroatom doping,heterostructure construction, and conductive carbon composites to improve their electronic properties, enhance charge transport capacity, and increase active sites. Based on this, this paper reviews the basic devices and processes of electrocatalysis, as well as the preparation methods, hoping to provide some guidance for the design of excellent non-noble metal electrocatalysts [3,4].

2. Electrocatalysis and its apparatus

2.1. Introduction to electrocatalysis

Electrocatalysis is an important means of the interaction of electrochemistry and catalysis science. Under the effect of an external electric field, the catalyst promotes electron migration at the electrode-electrolyte interface, reduces the activation energy of the reaction, controls the reaction pathway and the selectivity of the product. Unlike traditional thermal catalysis, electrocatalysis can occur at normal temperature and pressure, and it has many advantages such as adjustable voltage, energy conservation and high efficiency, and is widely used in water electrolysis, CO_2 reduction, N_2 reduction, fuel cells, etc.

2.2. Composition and structure of the electrocatalytic apparatus

Electrolytic cells are important equipment for electrocatalysis and play a crucial role in the process of converting electrical energy into chemical energy. They are also essential tools for electrocatalysis. As shown in Figure 1, the basic electrocatalytic apparatus consists of the anode, cathode, electrolyte, diaphragm, electrolytic cell, etc. Oxidation occurs at the anode, releasing electrons to form a current loop and completing the oxidation half-reaction; Reduction occurs at the cathode, which is where the product is produced; The function of the electrolyte is to conduct ions and maintain the charge balance on the electrode surface; The separator is used to separate the anode and cathode products and to prevent short circuits while allowing ions to flow freely; The electrolytic cell acts as a seal to prevent gas or liquid leakage, which is conducive to the stability of the experiment.

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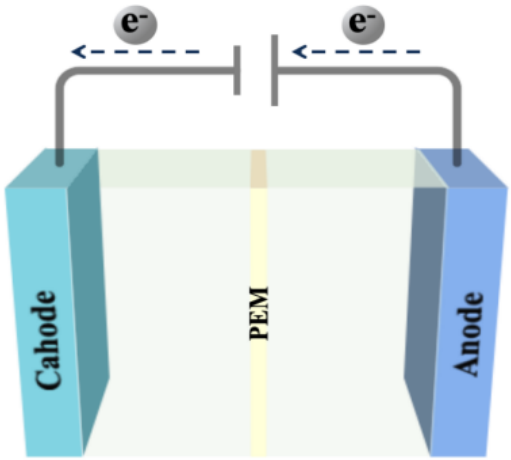

Figure 1. Electrocatalytic device

2.3. Typical electrocatalytic apparatus types

The most commonly used electrocatalytic apparatus in laboratories and industries are H-type electrolytic cells and membrane electrode (MEA) electrolytic cells. As shown in Figure 2, the H-type electrolytic cell is a relatively simple basic laboratory instrument with two chambers separated by an ion-exchange membrane. It is widely used in the study of catalyst activity, impedance and stability, but has poor mass transfer and cannot operate at high current [5,6].

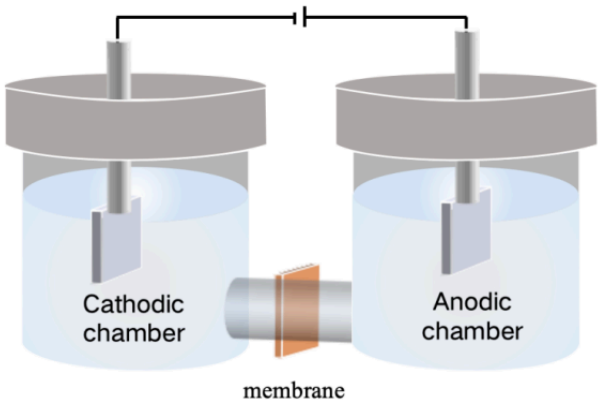

Figure 2. H-type electrolytic cell electrocatalytic device

As shown in Figure 3, the membrane electrode electrolytic cell is formed by hot-pressing five layers: the anode gas diffusion layer, the anode catalyst layer, the proton exchange membrane, the cathode catalyst layer, and the cathode gas diffusion layer. The proton exchange membrane is the core part, allowing only protons to pass through, with a fast mass transfer rate, low energy consumption and high energy utilization rate. It is closest to industrial scale-up among all electrocatalysis and can be applied in CO_2 reduction, fuel cells and other fields [7].

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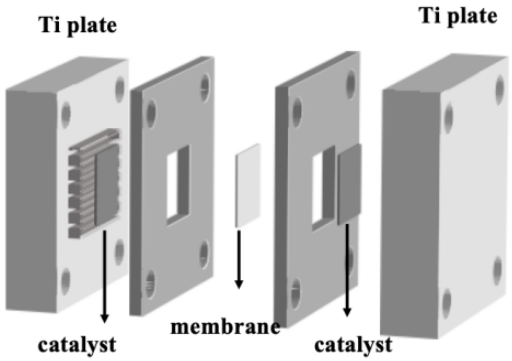

Figure 3. Membrane electrode assembly (MEA) electrolyzer

3. Principles of electrocatalysis

The essence of electrocatalysis is that at the electrode-electrolyte interface, under the action of an external electric field, the catalyst reduces the activation energy of the reaction, stabilizes the reaction intermediates, and facilitates electron transfer, thereby increasing the rate of electrochemical reactions and the selectivity of the products [8]. From a thermodynamic perspective, electrocatalysis does not change the overall Gibbs free energy of the chemical reaction; it merely speeds up the rate. From a microscopic perspective, a complete electrocatalytic process typically consists of several basic processes as shown in Figure 4: reactants or solvated ions move from the inside of the solution to a lower concentration near the electrode surface; The reactants are adsorbed by active sites attached to the electrode surface. Under the influence of an external electric field,electrons are rapidly transferred between the electrode and the adsorbed species, generating unstable reaction intermediates [9]; The intermediates bind, rearrange or transform at the active site to achieve the desired electrochemical reaction; The reaction products desorb from the catalyst surface and diffuse away from the electrode surface to return to the main body of the electrolyte [10]. During this process, the electrocatalyst can provide a large number of active sites that facilitate the adsorption of reactants and intermediates, shorten the electron transfer path, significantly reduce the overpotential required for the reaction, save energy, and accelerate the reaction rate.

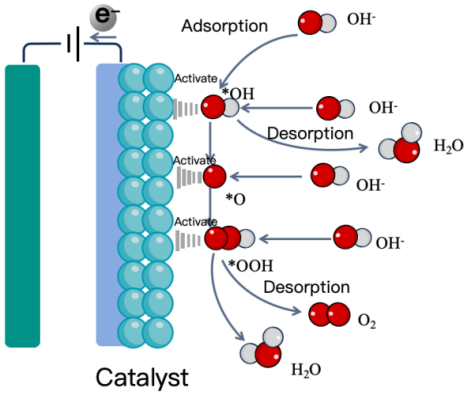

Figure 4. Schematic diagram of electrocatalytic principle

DOI: 10.54254/2755-2721/2026.33575

4. Types of electrocatalytic materials

Electrocatalytic materials are important factors that affect the rate and selectivity of electrochemical reactions. Generally speaking, electrocatalytic materials can be roughly classified into several types such as carbon-based materials, precious metal-based materials, transition metal oxides or sulfides, and other conductive polymers.

Materials that contain carbon are very commonly used in electrocatalysis because they have a large specific surface area, good electrical conductivity, high chemical stability and low cost. But pure carbon materials lack sufficient active sites and require enhancement by adding heteroatoms or metal nanoparticles [11]. Materials based on noble metals (platinum, iridium, and ruthenium) have outstanding hydrogen evolution, oxygen evolution, and oxygen reduction properties, yet are constrained by high costs and limited availability [1]. At present, the content of noble metals is minimized by alloying, core-shell structures or single-atom dispersion methods. Conductive polymers (e.g., polyaniline and polypyrrole) have high electrical conductivity and flexibility, which may be controlled by doping or functional group substitution [12]. They are frequently integrated with carbon materials or transition metal oxides to enhance their electrochemical performance. The compounds of transition metals (sulfides, phosphides, nitrides, and oxides) have gained popularity as catalysts of non-noble metals because of the vast amounts of resources, low cost, and modifiable electronic structure. In this group, transition metal phosphides are highly effective in hydrogen and oxygen evolution reactions and nickel, cobalt and iron-based oxides or hydroxides have great influence on oxygen evolution reactions. The electrocatalytic capability can be further improved by multi-component composites [13].

The above points show that different types of electrocatalytic materials have their own characteristics, but there is also room for improvement: the intrinsic electrocatalytic activity of carbon-based materials is relatively low; Precious metal-based materials are expensive and scarce; Conductive polymers are less durable;The conductivity and number of active sites of transition metal compounds also need to be improved. In this context, molybdenum disulfide (MoS_2), as a representative transition metal sulfide, exhibits good electrocatalytic performance due to its unique two-dimensional layered structure, large specific surface area, good chemical stability, and abundant marginal active sites. In numerous reports, it has been found that molybdenum disulfide (MoS_2) edge sites have a hydrogen adsorption free energy close to platinum for hydrogen evolution reactions, making it a promising non-noble metal electrocatalyst [14].

5. Preparation methods of molybdenum disulfide and its composites/doped materials

5.1. Preparation methods of molybdenum disulfide (MoS_2)

The preparation of pure-phase MoS_2 is a prerequisite for subsequent doping and recombination. Due to the differences in the expected product morphology, crystal form, number of layers, and application fields, there are currently multiple methods available for the preparation of pure-phase MoS_2, mainly including hydrothermal/solvothermal methods, chemical vapor deposition methods, and rapid thermal annealing methods, etc.

Hydrothermal/solvent thermal is the preparation technique that is most often applied in laboratories. The reaction between ammonium molybdate or sodium molybdate as the molybdenum source and thiourea, thioacetamide or L-cysteine as the sulfur source is performed at 180 -240 ℃ over 12-48 hours. Depending on the temperature, time, pH level and precursor ratio, different morphologies including nanoflowers, nanosheets and nanospheres may be produced. This approach

DOI: 10.54254/2755-2721/2026.33575

is straightforward, morphology is controlled and yield is good, but the product crystal quality is low and the layer thickness cannot be accurately controlled [3]. Chemical vapor deposition method is capable of producing high quality, continuous and homogeneous single-layer or multi-layer MoS_2 films on SiO_2/Si, sapphire or graphene substrates. With MoO_3 or $(NH_4)_2MoS_4$ as the starting material and sulfur powder or H_2S as the sulfur source, the reaction is conducted in an inert atmosphere at 600-800 ℃ . It is applicable to scientific research and planar device research but it is costly and has low yield [4]. Fast thermal annealing is a novel method that involves using carbon fibers as the heat source which can produce multi-layer MoS_2 films within a very short period. In comparison with CVD, it has the benefits of greater process window, less synthesis time and increased energy efficiency allowing it to be applied to mass production and application [2].

The raw materials, conditions, product features, advantages and disadvantages used in the above pure-phase MoS_2 preparation methods are shown in Table 1 Comparison of Preparation Methods for molybdenum disulfide (MoS_2).

Table 1. Comparison of preparation methods for molybdenum disulfide

Serial Numbers	Preparation method	Raw materials	Reactionconditions	Advantages	Disadvantages	References
1	Rapid thermal annealing method	$(NH_4)_2MoS_4$	300-500℃, flash pyrolysis	Wide process window and highefficiency	Medium crystallinity	[2]
2	Hydrothermal/ solvothermal methods	Ammonium molybdate/sodium molybdate+thiourea/L-cysteine	180-240 ℃,12-48h	Simple operation,controllable morphology,high yield	Medium crystallinity, inaccurate layer count	[3]
3	Chemical vapor deposition (CVD)	MoO_3/$(NH_4)_2MoS_4$+ Sulfur powder / H_2S	600-800 ℃, Ar/H_2 atmosphereerial	High crystallinity and precise controllability of layer thickness	Expensive equipment and low output	[4]

5.2. Modified preparation methods of doped/composite molybdenum disulfide (MoS_2)

Due to the inherent defects of pure-phase MoS_2, such as low conductivity and few active sites on the basal plane, researchers have proposed two improved methods, namely element doping and material compounding. Element doping is achieved by introducing different heteroatoms, such as transition metals or oxygen, to change the electronic properties and hydrogen adsorption free energy of MoS_2 ; Material recombination, on the other hand, uses carbon-based materials with high electrical conductivity or other active components to form heterojunctions to enhance the charge transport capacity and synergy of the entire system.

Element doping modification is mainly co-doping by hydrothermal/solvothermal methods, during which transition metal salts (such as cobalt nitrate, iron nitrate) are added to the molybdenum and sulfur sources or oxygen atoms are introduced by competitive reduction. Ma et al. prepared Fe-atom-doped MoS_2egg-shell nanoflower microspheres using a one-step hydrothermal method, and different ratios of 1T/2H phase transitions [15]. An et al. used the "intercalation - sulfidation" method to introduce zero-valent Co into MoS_2 to form Co(0) -MOS_2. Compared with the traditional Co(II) doping, zero-valent Co has more d electrons resulting in more "CO-S-Mo" interatomic electron perturbation, thereby improving the electron properties [16].

The heterostructure is based on the combination of MoS_2 with other transition metal compounds to obtain a heterojunction catalyst with interfacial synergistic effect. Fan et al. prepared a

DOI: 10.54254/2755-2721/2026.33575

MoS_2@Mo_2C heterojunction catalyst using the hydrothermal method. They reported that electron injection at the heterointerface caused a phase transition from 2H semiconductor to 1T metal in MoS_2, which effectively reduced the hydrogen adsorption free [4].

Carbon material composite modification can improve the conductivity of MoS_2. When conductive carbon materials such as carbon nanotubes (CNT) and graphene are mixed with molybdenum and sulfur sources and subjected to hydrothermal reactions under certain conditions, MoS_2/CNT or MoS_2/ graphene composites can be obtained; When MoS_2 is combined with MWCNT, hexagonal phase MoS_2 is uniformly distributed on the surface structure of CNT, and the carbon material provides support to enhance the conductivity of the entire system and prevent the MoS_2 nanosheets from clustering together, thereby exposing more edge active sites [17].

The raw materials, conditions, modification methods and final product characteristicsof the doping/composite modification preparation method are shown in Table 2.

Table 2. Comparison of modified preparation methods for doped/composite molybdenum disulfide (MoS_2)

Serial Numbers	Material type	Raw materials	Preparation method	Modification strategies	References
1	MoS_2@Mo_2C	Ammonium molybdate+ Mo_2C precursor	Hydrothermal method +annealing	Phase transition induced by heterojunction interface	[4]
2	Fe-MoS_2	Ammonium molybdate + thiourea + ferric nitrate	One-step hydrothermal method	Fe doping +1T/2H mixed phase regulation	[15]
3	Co(0)-MoS_2	Ammonium molybdate + Zero-valent Co	Interlayer-sulfidation method	Zero-valent transition metal doping	[16]
4	MoS_2/MWCNT	Ammonium molybdate+thiourea+ MWCNT	hydrothermal method	CNT composite	[17]

6. Study on the performance of pure phase and modified MoS_2 in electrocatalysis

Molybdenum disulfide (MoS_2), a typical two-dimensional transition metal sulfide, shows great potential [14]. However, pure-phase MoS_2(mainly 2H semiconductor phase) has poor conductivity, lower surface activity and fewer active sites, which makes its electrocatalytic activity far inferior to that of the noble metal Pt as a standard reference. To improve its electrocatalytic performance, a series of methods have been proposed to modify it, such as doping with different types of heteroatoms, constructing heterostructures, or combining with conductive carbon materials to change the electronic structure of MoS_2, increase the charge migration rate, and add more active sites. This chapter provides a detailed summary of the main characteristics of pure-phase MoS_2 and

DOI: 10.54254/2755-2721/2026.33575

various modified MoS_2 materials when used as electrocatalysts for electrocatalytic HER, with the aim of studying the effects of different modification methods on catalyst activity.

6.1. Electrocatalytic performance of pure-phase MoS_2

Pure-phase MoS_2(2H phase) shows weak catalytic performance when used for electrocatalytic HER, mainly due to its semiconductor nature that makes charge flow difficult and there are only a few active sites on the surface. It has been reported that in an alkaline environment, pure-phase MoS_2 requires a large voltage to make the HER current density above 10 $mA \cdot cm^{-2}$ and a large overpotential of 181 mV [16]. From the perspective of reaction kinetics, its Tafel slope is 330 $mV \cdot dec^{-1}$, which is much greater than that of commercial Pt/C catalysts (about 55 $mV \cdot dec^{-1}$). This indicates that the pure-phase MoS_2 HER is mainly due to the high energy barrier of the Volmer step (H_2O dissociation), which leads to a slower kinetics and is not suitable for efficient electrocatalysis.

6.2. Performance of heteroatom doping modified MoS_2

Heteroatom doping can alter the electronic properties of MoS_2 and enhance its adsorption/desorption capacity for hydrogen intermediates. An et al. Zero-valent Co was introduced between the layers of MoS_2 by the "Interlayer-sulfidation method" to obtain Co(0)-MoS_2 [16].XPS and XAFS analysis revealed that charge transfer from Co(0) to MoS_2 occurred after the introduction of zero-valent Co, resulting in an increase in electron cloud density on Mo and S atoms. Due to this improvement in electronic structure, Co(0)-MoS_2 exhibits good electrocatalytic activity in the basic HER, achieving a current density of 10$mA \cdot cm^{-2}$ with just 28mV overpotential and a Tafel slope of 47$mV \cdot dec^{-1}$, which is already comparable to commercial Pt/C catalysts; Conventional Co (II) -doped MoS_2 requires a 92mV overpotential and a Tafel slope of 115$mV \cdot dec^{-1}$, which is far lower than that of zero-valent co-based catalysts. And the reasons for the increased activity were also clarified by DFT calculations: The additional d electron provided by Co(0) increase electron transfer between "CO-S-Mo", reducing the energy required for H_2O dissociation from 1.98 eV to 0.85 eV and optimizing the hydrogen adsorption free energy (ΔGH*) from -0.58 eV to 0.21 eV. In terms of stability, the Co(0)-MoS_2 shows almost no current drop during 24-hour continuous operation and has excellent durability [16].

6.3. Heterostructures build the performance of modified MoS_2

A reasonable heterointerface can not only facilitate charge transfer but also promote favorable phase transitions in MoS_2. Guo et al. obtained MoS_2@Mo_2C heterojunction catalysts XRD, Raman, and XPS characterizations indicated that electron injection at the heterointerface transformed MoS_2 from a 2H semiconductor phase to a 1T metal phase, which enhanced the material's conductivity and increased the number of active sites [4]. Due to this structural advantage, it performs well in the basic HER: a current density of 10 $mA \cdot cm^{-2}$ can be achieved with just 65 mV overpotential, and the Tafel slope is 53 $mV \cdot dec^{-1}$, which is much lower than that of single-phase MoS_2. DFT calculations show that charge redistribution at the interface reduces the hydrogen adsorption free energy (ΔGH) from 1.23 eV to 0.13 eV, significantly enhancing the hydrogen binding capacity and putting it in the optimal adsorption-desorption equilibrium state. In addition, it maintained good activity without significant attenuation after 800 hours of continuous operation, demonstrating excellent durability and being highly advantageous for practical application.

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6.4. Performance of carbon composite modified MoS_2

Mixing MoS_2 with a carbon material that has good electrical conductivity can effectively enhance the charge transport capacity in the electrode. Vidhya et al. prepared MoS_2/MWCNT nanocomposites [17].As shown in the figure, MWCNT acts as a conductive support and is uniformly coated on the outside of MoS_2 nanospheres, forming a good three-dimensional conductive structure. This structure is conducive to achieving good electrochemical properties. In the electrochemical impedance spectrum, the composite material has a very low charge transfer resistance (Rct) of 0.20 Ω, which is much lower than 10 Ω of single-phase MoS_2, indicating a significant improvement in charge transport capacity after the composite. From the perspective of energy storage, the material has A large specific capacitance of 848 $F \cdot g^{-1}$ (@1 A·g) and still has 82% remaining capacity after 5000 cycles, indicating good mechanical strength [17].

In order to facilitate the comparison of the effects of various modification methods on the electrocatalytic activity of MoS_2, the relevant parameters of the above-mentioned MoS_2-based electrocatalysts are summarized in Table 3.

Table 3. Comparison of performance of pure phase and modified MoS_2

Material type	Electrolyte	Overpotential ($mV@10mA \cdot cm^{-2}$)	Tafel slope ($mV \cdot dec^{-1}$)	Stability	Main modification strategies	References
$MoS_2@Mo_2C$	1 M KOH	65	53	800 h stable	Heterojunction induces 1T phase transition	[4]
Pure-phase MoS_2	1 M KOH	181	330	-	-	[16]
Co(0)-MoS_2	1 M KOH	28	47	24 h stable	Zero-valent Co intercalation doping	[16]
MoS_2/MWCNT	0.5 M K_2SO_4	-	-	5,000 cycle retention rate 82%	MWCNT conductive network composite	[17]

7. Conclusion

This article reviews the use of molybdenum disulfide (MoS_2) in electrocatalysis, summarizing the research progress as a non-precious metal electrocatalyst and the mechanisms by which different preparation and modification methods affect its structure and catalytic performance. Starting from the basic concepts of electrocatalysis, the characteristics of four types of materials - pure-phase MoS_2, heteroatom doping, heterostructure construction, and carbon material composites - are presented and compared. The results show that pure-phase MoS_2 has disadvantages such as a large overpotential and a low kinetic rate in the hydrogen evolution reaction, which to some extent limit its practical application, and its activity is mainly determined by edge sites rather than basal planes; By zero-valent cobalt intercalation doping, $MoS_2@Mo_2C$ heterojunction and MoS_2/MWCNT recombination, overpotential is effectively reduced, hydrogen adsorption free energy is optimized, charge transport capacity is improved and stability is enhanced in terms of electronic structure regulation, interface engineering and conductive network. Among them, zero-valent Co doping, by adding extra d electrons, intensifies the electron perturbation between "CO-S-Mo" atoms and significantly reduces the H_2O dissociation energy barrier; Heterostructure construction is beneficial

DOI: 10.54254/2755-2721/2026.33575

for conductivity and improvement of hydrogen adsorption free energy because of the synergy at the interface that prompts MoS_2 to change from 2H phase to 1T metallic phase; Carbon composites can form a three-dimensional conductive network that prevents the stacking of MoS_2 nanosheets and adds more edge active sites.The Co(0)-MoS_2 and MoS_2@Mo_2C heterojunction strategies have obvious advantages in reducing overpotential and improving stability, even comparable to commercial Pt/C catalysts. However, due to the complexity of their synthesis methods and the difficulty in large-scale production, MoS_2-based materials still cannot be widely applied in industry. However, due to its low cost and environmental friendliness, it has a promising application prospect in electrocatalysis. By summarizing and comparing the above-mentioned literature, the relationship between structure and performance can be obtained, and certain guidance can be provided for the subsequent development of high-performance stable non-precious metal electrocatalysts.

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