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# *Preparation of Molybdenum Disulfide Nanomaterials and Their Applications in Catalysis and Energy Storage*

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***Abstract.*** Nowadays, against the backdrop of increasing global energy crises and environmental pollution issues, the development of novel and highly efficient materials for energy conversion and storage has emerged as a prominent research focus. Molybdenum disulfide ($MoS_2$), as a typical layered transition metal dichalcogenide, is regarded as possessing immense application potential due to its unique graphene-like structure and tunable bandgap. This review systematically summarizes the preparation methods and structural modulation strategies of $MoS_2$ nanomaterials, along with their applications in photocatalytic degradation, electrocatalytic hydrogen evolution, and electrochemical energy storage. The crystal structures (1T, 2H, and 3R phases) and fundamental properties of $MoS_2$ are initially introduced. Subsequently, the advantages and disadvantages of top-down exfoliation methods and bottom-up synthesis approaches, as well as their influence on the morphology of the resulting products, are discussed. Emphasis is placed on the mechanisms underlying performance enhancement through strategies such as doping, heterojunction construction, interlayer expansion, and compositing with conductive materials. Research indicates that modulating the crystal phase and interlayer spacing of $MoS_2$ can significantly optimize its photocatalytic degradation efficiency, hydrogen evolution reaction activity, and lithium/sodium-ion storage capacity. Finally, the challenges and future research directions for $MoS_2$-based materials are prospectively outlined.

***Keywords:*** Molybdenum disulfide, Nanomaterials, Photocatalytic degradation, Heterojunction, Electrochemical hydrogen evolution

## 1. Introduction

Due to the intensifying global energy crisis and the increasingly severe environmental pollution, the development of efficient energy conversion and storage materials has become an urgent need. The discovery of graphene has sparked a research craze on two-dimensional materials. However, the zero band gap of graphene limits its application in fields such as photocatalysis [1]. Molybdenum disulfide ($MoS_2$), as a typical layered transition metal sulfide, possesses a tunable band gap and a graphene-like structure and has become a research hotspot [2, 3]. However, the mechanisms by which different crystal phases (1T, 2H, and 3R) and interlayer spacings affect the photocatalytic, electrocatalytic, and energy storage properties of $MoS_2$ are not yet very clear. In particular, the

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synergistic effect of the 1T/2H heterojunction and the interlayer expansion has not been systematically studied [1, 4].

This article focuses on $MoS_2$ nanomaterials as the research object, systematically reviewing its preparation methods, crystal phase regulation, and interlayer engineering strategies. It particularly explores the influence patterns of different crystal phases and interlayer spacings on the photodegradation of organic pollutants, electrocatalytic hydrogen evolution, and lithium/sodium ion storage performance [5-7]. This research can provide a theoretical basis for the development of efficient and stable $MoS_2$-based catalysts and electrode materials and promote their practical applications in environmental governance and the field of new energy.

## 2. The structure and basic properties of $MoS_2$

### 2.1. The crystal structure of $MoS_2$

$MoS_2$ is a typical layered transition metal sulfide. Its crystal structure is formed by a layer of molybdenum atoms and two layers of sulfur atoms through strong covalent bonds, resulting in a "sandwich" type single-layer structure (S-Mo-S). The thickness of the single layer is approximately 0.65 nm, and the layers are stacked together by weak van der Waals forces [8].

Due to the different coordination modes between sulfur atoms and molybdenum atoms, $MoS_2$ mainly exists in three crystal phases: the 2H phase (hexagonal structure, trigonal prism coordination), the 1T phase (tetragonal structure, octahedral coordination), and the 3R phase (orthorhombic structure, metastable state) [9]. Among them, the 2H phase is the most stable crystal form in nature and exhibits semiconductor properties; the 1T phase has metallic characteristics and excellent conductivity, but it is metastable and is prone to transforming into the 2H phase upon heating or prolonged storage; the 3R phase is relatively rare during hydrothermal synthesis [1, 8]. The main phase that can be stably present under normal conditions is 2H-$MoS_2$, with lattice parameters of $a = b = 0.3160$ nm and $c = 1.2294$ nm [8]. The $MoS_2$ with different crystal phases exhibits significant differences in electronic structure, the number of surface active sites, and chemical stability. These differences directly determine its performance in fields such as catalysis and energy storage [1, 9].

### 2.2. Structural properties and optical characteristics

The energy band structure of $MoS_2$ exhibits significant layer-dependent characteristics, which is one of the important differences between it and graphene. The bulk $MoS_2$ is an indirect bandgap semiconductor with a bandgap width of approximately 1.2 eV; when the number of layers decreases to a single layer, it transforms into a direct bandgap semiconductor, with the bandgap increasing to approximately 1.8 eV. This transition from an indirect bandgap to a direct bandgap significantly enhances the photoluminescence efficiency and light absorption capacity of the material, giving it unique advantages in optoelectronic devices and photocatalysis fields [1, 7].

The band gap of $MoS_2$ can be adjusted within the range of 1.20 to 1.90 eV, corresponding to an upper limit of light absorption wavelength of 690 to 1030 nm. This is highly compatible with the main energy range of the solar spectrum and theoretically has a relatively high utilization rate of solar light [8]. Through crystal phase regulation, the energy band structure of it can be further optimized. For example, in the 1T/2H mixed-phase $MoS_2$, due to the formation of a heterostructure between the metallic phase and the semiconductor phase, its effective band gap can be slightly lower

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than that of the pure 2H phase, thereby significantly broadening the absorption range of visible light [1].

Furthermore, interlayer expansion can also affect the electronic structure of $MoS_2$: when the interlayer spacing expands by approximately 7%, the electronic coupling between adjacent monolayers significantly weakens, causing the behavior of each monolayer to approach that of a free-state monolayer of $MoS_2$, thereby optimizing its light absorption and photocatalytic performance [3].

### 2.3. Layered structure and electrochemical properties

The layered structure of $MoS_2$ provides an important foundation for its application in the field of electrochemical energy storage. Its natural layer spacing (0.615 nm) can support the reversible insertion and extraction of $Li^+$ (with a radius of approximately 0.076 nm). However, for ions with larger radii or multiple charges such as $Na^+$ (with a radius of approximately 0.102 nm but high charge density) and $Mg^{2+}$ (with a radius of approximately 0.072 nm but high charge density), the diffusion barrier is high, resulting in slow intercalation kinetics [3].

The expansion of interlayer spacing through ion or molecule intercalation can effectively reduce the ion diffusion barrier and improve the kinetic performance. Theoretical calculations (such as density functional theory) indicate that an 11% expansion of the interlayer spacing is optimal for $Li^+$ and $Mg^{2+}$ intercalation, while a 23% expansion is optimal for $Na^+$ intercalation. The corresponding intercalation energies are -2.57 eV, -1.50 eV, and -1.30 eV, respectively [3]. The expanded interlayer spacing not only improves the ion migration kinetics but also buffers the volume expansion during charging and discharging cycles, thereby enhancing the cycling stability and rate performance of the electrode material [3, 10].

Furthermore, the unsaturated sulfur atoms at the edges of $MoS_2$ are regarded as the active centers for electrocatalytic hydrogen evolution reactions. The expansion of the layer spacing can regulate the electronic states of the edge atoms and reduce the Gibbs free energy of hydrogen adsorption, thereby enhancing the intrinsic catalytic activity [3, 11, 12]. Therefore, in-depth study of the layered structure of $MoS_2$ and its interactions with ions/molecules is of great significance for the design of high-performance energy storage and electrocatalytic materials.

## 3. Preparation of $MoS_2$ nanomaterials

### 3.1. The "top-down" divestiture method

The "top-down" approach mainly includes the mechanical exfoliation method, lithium-ion intercalation method, and liquid-phase ultrasonic method. The mechanical exfoliation method uses tape to overcome the interlayer van der Waals forces and can obtain high-quality monolayer $MoS_2$, but the yield is relatively low and it is not suitable for large-scale production [8].The lithium-ion intercalation method (such as using n-butyl lithium) involves inserting lithium ions into the interlayer, and during the subsequent reaction with water, hydrogen gas is produced to achieve interlayer delamination, resulting in a high proportion of the 1T phase structure, which is suitable for applications such as electrocatalysis [4, 8]. The liquid-phase ultrasonic method involves dispersing the bulk $MoS_2$ in a specific solvent (such as dodecylthiol or sodium alginate aqueous solution) and then using ultrasonic assistance to peel off to obtain few-layer nanosheets. The operation is simple and suitable for large-scale production [8].

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### 3.2. "Bottom-up" synthesis method

The hydrothermal/solvent thermal method is one of the most commonly used approaches for preparing $MoS_2$ nanomaterials. By adjusting the reaction temperature and time, as well as the proportions and types of precursors, various morphologies such as nanosheets, nanoflowers, and hollow spheres can be synthesized [5, 6, 8]. For instance, using ammonium molybdate and thiourea as raw materials, and reacting at 220°C for 18 hours, flower-shaped microspheres of $MoS_2$ with a diameter of 2 μm and a thickness of 10 nm can be prepared [8]. Chemical vapor deposition is suitable for growing large-sized and high-quality monolayer $MoS_2$ films on substrates and is widely applied in the research of electronic devices [7]. For instance, using sulfur powder and molybdenum trioxide as raw materials, triangular $MoS_2$ films with a thickness of less than 5 nm can be fabricated on a $Si/SiO_2$ substrate [8].

### 3.3. Specific synthesis of interlayer expansion

Interlayer-expanded $MoS_2$ can be achieved through the "top-down" method of intercalation/deposition followed by re-deposition, or the "bottom-up" method of hydrothermal/solvothermal synthesis [3]. In the hydrothermal method, by controlling the Mo/S molar ratio and the filling degree of the reaction vessel, different crystal phases and layer spacings of the products can be obtained. For example, Han Gaiying et al. by adjusting the Mo:S molar ratio (1:10 and 1:25) and the filling degree (45% and 75%), respectively, obtained the mixed phases of 1T/2H-$MoS_2$ and 1T/3R-$MoS_2$. Among them, the layer spacing of 1T/2H-$MoS_2$ was 0.95 nm (the 002 peak shifted from 14.1° to 9.3°) [1]. Furthermore, the microwave-assisted solvothermal method using $(NH_4)_2MoS_4$ as the precursor and DMF as the solvent can synthesize IE-$MoS_2$ with a layer spacing of 0.94 nm at 240°C. This is attributed to the insertion of DMF molecules [3].

## 4. Strategies for controlling the properties of $MoS_2$ and its applications in energy and environment

### 4.1. Crystal phase of $MoS_2$ and interlayer engineering control

By means of lithium-ion intercalation and other techniques, the 2H phase can be induced to transform into the 1T phase, thereby leveraging the high conductivity of the 1T phase to enhance the performance of electrocatalytic hydrogen evolution and electrochemical energy storage [11, 12]. Studies have shown that the 1T/2H-$MoS_2$ heterostructure prepared by hydrothermal method reduces the band gap from 1.84 eV of the 2H phase to 1.72 eV, thereby broadening the visible light absorption range to a certain extent [1]. Additionally, by ion or molecule intercalation to expand the interlayer spacing, the ion diffusion barrier can be reduced, and the rate performance and capacity in sodium/magnesium ion batteries can be enhanced [3, 10]. For instance, intercalating polyethylene oxide into the interlayer of $MoS_2$ can expand the interlayer spacing to 1.45 nm, significantly improving the diffusion ability of $Mg^{2+}$ [3]. In summary, controlling the crystal phase and interlayer engineering are important strategies for improving the comprehensive performance of $MoS_2$.

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## 4.2. The application of $MoS_2$ in the fields of energy and environment

### 4.2.1. Photocatalytic degradation

$MoS_2$ and its composite materials exhibit excellent performance in the photocatalytic degradation of organic pollutants. Han et al. compared the degradation effects of different crystal phases of $MoS_2$ on hydrochloric acid tetracycline, and methyl orange: After 1 hour of visible light irradiation, the degradation rate of TCH by 1T/2H-$MoS_2$ reached 79.62%, with a reaction rate constant of 0.02624 $min^{-1}$, which was much higher than that of 1T/3R-$MoS_2$ (71.54%, 0.02136 $min^{-1}$) and 2H-$MoS_2$ (8.73%, 0.00127 $min^{-1}$). The degradation rates of MO were 28.31%, 25.32%, and 21.54%, respectively. Free radical capture experiments confirmed that $\cdot O_2^-$ was the main active species [1].

Furthermore, the $MoS_2$/ZnO heterojunction can degrade methylene blue by 99% within 60 minutes under simulated sunlight, which is 10% higher than that of pure ZnO. The $Na^+$-MMT/$MoS_2$ composite material can degrade 96% of rhodamine B within 80 minutes and maintains good performance after 5 cycles [8].

### 4.2.2. Electrochemical hydrogen evolution

Interlayer-expanded $MoS_2$ exhibits significant advantages in the hydrogen evolution reaction. The microwave-synthesized IE-$MoS_2$ (with a layer spacing of 0.94 nm) has an initial overpotential of only 0.10 V (vs RHE), which is much lower than that of bulk $MoS_2$. DFT calculations indicate that when the layer spacing expands from 0.62 nm to 0.95 nm, the hydrogen adsorption Gibbs free energy ΔG_H decreases by 0.149 eV, which is conducive to HER catalysis. After combining IE-$MoS_2$ with nitrogen-doped reduced graphene oxide, the initial overpotential is further reduced to 55 mV, the Tafel slope is 41.3 mV $dec^{-1}$, and the exchange current density reaches 0.72 mA $cm^{-2}$ [3].

### 4.2.3. Lithium/sodium/magnesium ion batteries

Interlayer-expanded $MoS_2$ as the negative electrode material for ion batteries can significantly enhance capacity and cycle stability. For instance, $MoS_2$ with graphene quantum dot intercalation (layer spacing of 0.78 - 0.80 nm) has a discharge capacity of 1031 mAh $g^{-1}$ after 80 cycles at 0.10 A $g^{-1}$. For sodium-ion batteries, $MoS_2$ nanocylinders (layer spacing of 0.66 - 0.69 nm) maintain a capacity of 350 mAh $g^{-1}$ after 1500 cycles at 0.05 A $g^{-1}$. For magnesium-ion batteries, $MoS_2$ with PEO intercalation (layer spacing of 1.45 nm) has a capacity of 75 mAh $g^{-1}$ at 0.005 A $g^{-1}$, which is 3.4 times that of the bulk $MoS_2$ [3]. These performance improvements are attributed to the expanded layer spacing, which reduces the ion diffusion barrier and buffers volume changes.

## 4.3. Doping with non-atomic substances and composite engineering

Doping with metals (such as Co and Ni) and non-metals (such as Se and O) can regulate the electronic structure of $MoS_2$, optimize the hydrogen evolution reaction pathway, and optimize the light absorption range [6, 11]. For instance, oxygen-doped $MoS_2$ (with a layer spacing of 0.95 nm) has a band gap reduced to 1.30 eV, enhancing the intrinsic conductivity [3]. Moreover, by combining $MoS_2$ with graphene, carbon nanotubes, polypyrrole, etc., a three-dimensional conductive network can be constructed, improving the electron transmission rate and structural stability [2, 10, 11]. For the purpose of facilitating comparison, the main preparation methods, typical conditions, and product characteristics of $MoS_2$ nanomaterials are summarized in Table 1.

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Table 1. Comparison of main preparation methods for $MoS_2$ nanomaterials

| Preparation method | Representative condition | Product features | Chief application |
|---|---|---|---|
| Mechanical stripping method | Repeatedly peeling off the tape | With high-quality single-layer structure and low yield | For basic research |
| Lithium-ion intercalation method | Tetradecyl lithium, n-hexane | 1T phase enrichment, interlayer spacing expands | For electrocatalysis and energy storage |
| Liquid phase ultrasonic method | Organic solvents or surfactants | Thin nanosheets, which can be produced in large quantities | For composite materials |
| Hydrothermal/Solvent thermal method | Ammonium molybdate + thiourea, 180 - 220°C | The shape is in the form of flowers or spheres, with a layer spacing of 0.62 to 0.95 nm. | For photocatalysis and batteries |
| chemical vapor deposition method | $MoO_3$ + S, Ar atmosphere | With high-quality films and triangular grains | For electronic devices and optoelectronics |
| Microwave-assisted method | $(NH_4)_2MoS_4$+DMF, 240°C | Layer spacing: 0.94 nm, defect controllable | For HER and batteries |

### 4.4. Analysis of performance optimization mechanism

The performance optimization of $MoS_2$ mainly stems from three aspects:

Firstly, the increase in active sites, such as edge defects, sulfur vacancies, etc.; secondly, the regulation of electronic structure, such as phase transformation (2H→1T), doping, and the resulting band changes caused by interlayer expansion; thirdly, the synergistic effect of composite materials, such as the combination with graphene and carbon nanotubes, which enhances conductivity and structural stability [2, 3, 10, 11].

Among them, the 1T/2H heterojunction not only broadens the light absorption range but also promotes the separation and transmission of photogenerated electron-hole pairs [1]. The interlayer extension achieves this by reducing the ion diffusion barrier and regulating the electronic states at the edge atoms, while simultaneously enhancing the storage and catalytic properties [3].

## 5. Conclusion

This article systematically reviews and summarizes the preparation methods, structural regulation, and applications of $MoS_2$ nanomaterials in photocatalysis, electrocatalysis, and energy storage. It can be seen from the research that the performance of $MoS_2$ is closely related to its crystal phase type, layer number, layer spacing, and microscopic morphology. By reasonably regulating these structural parameters, the light absorption capacity, electronic conductivity efficiency, and ion diffusion performance of the material can be effectively improved. In terms of preparation, the "top-down" exfoliation method can obtain high-quality or low-layer $MoS_2$, but the yield is low and is more suitable for basic research; in contrast, the "bottom-up" synthesis methods (such as the hydrothermal method, CVD method, and microwave-assisted method) are more suitable for large-scale production and can obtain diverse morphologies such as nanoflowers and nanosheets, with better controllability and practicability. In terms of performance optimization, there are mainly the following approaches: Firstly, through crystal phase and interlayer engineering, such as constructing 1T/2H heterostructures or expanding the layer spacing, the photocatalytic degradation efficiency, electrocatalytic hydrogen evolution activity, and capacity performance of the ion battery can be significantly improved. Secondly, by combining with graphene, ZnO, etc., a good conductive

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network can be constructed, further improving the cycle stability and electronic transmission ability of the material. In addition, doping of heteroatoms can effectively regulate the electronic structure of $MoS_2$, thereby optimizing its catalytic and energy storage performance.

Although $MoS_2$-based materials show broad application prospects, they still face some challenges at present. For example, the green and large-scale production of high-quality single-layer or specific crystal phase $MoS_2$ is not yet mature. The 1T phase has poor stability under normal conditions, the quantitative relationship between the precise control of layer spacing and the final performance is not yet clear, and the long-term stability in actual devices still needs to be further verified. In the future, efforts should be made to develop more green and efficient synthesis routes, combine in situ characterization techniques and theoretical calculations to deeply reveal its microscopic working mechanism, promote multi-functional integrated design, and explore the heterogeneous integration application of $MoS_2$ with other two-dimensional materials.

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