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A Review on the Preparation of Nickel Molybdate Electrode Materials and Their Application in Supercapacitors

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Abstract. In the context of the continuous transformation of global energy structure towards a green and low-carbon direction, the development of high-performance and sustainable energy storage technologies is particularly important. Supercapacitors, due to their high power density and rapid charging and discharging capabilities, have shown promising application prospects in the field of new energy. The electrode material, which determines the performance of the device, directly affects the energy density and cycle life of the device. Molybdate nickel ($NiMoO_4$), as a bimetallic oxide, not only has abundant redox active sites and high theoretical specific capacity, but also exhibits excellent electrochemical reversibility. In recent years, it has gradually become a popular choice for constructing high-performance pseudocapacitive electrode materials. This paper, based on current research progress, systematically reviews the basic structure and working principle of supercapacitors, and analyzes the potential and main challenges of molybdate nickel-based materials in improving the comprehensive performance of energy storage devices.

Keywords: Preparation, Nickel Molybdate Electrode Materials, Application in Supercapacitors

1. Preface

With the increasingly severe global energy crisis and environmental issues, the development of efficient and clean energy storage technologies has become an important research direction in the current energy field. The excessive use of fossil energy not only exacerbates resource shortages but also triggers serious environmental problems, making the transformation of the energy system towards renewable energy sources an inevitable trend [1-4]. Supercapacitors, as a new type of energy storage device between traditional capacitors and chemical batteries, possess high power density, rapid charging and discharging capabilities, and excellent cycle stability, and have shown broad application prospects in smart grids, electric vehicles, and portable electronic devices, making it one of the key technologies for achieving sustainable energy utilization. Electrode materials determine the performance of supercapacitors, and their microstructure and electrochemical properties directly affect the energy density and power output of the devices. Currently, carbon materials, metal oxides, and conductive polymers are the main research directions. However,

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traditional carbon materials have low energy density, and single metal oxides have problems such as poor conductivity and insufficient cycle stability [5-8]. In recent years, double metal oxides such as nickel molybdate ($NiMoO_4$), which utilize the synergistic effect between the two metals, abundant redox active sites, and controllable nanostructures, have gradually become the focus of research on pseudocapacitive electrode materials. Recent studies have systematically explored phase regulation, synthesis routes, hydrated structures, biomass-derived composites, homogeneous and heterogeneous core-shell architectures, multidimensional heterostructures, metastable β-$NiMoO_4$, MXene interfaces, Ni-Co DH systems, and NiMn-LDH core-shell electrodes [9-11]. In 2013, Cai et al. successfully prepared $NiMoO_4$ nanospheres through the hydrothermal method, with a specific capacitance of 974 F/g and an energy density of 20.1 Wh/kg, demonstrating excellent electrochemical performance [12]. Moreover, different preparation methods have a significant impact on the crystal structure, morphological characteristics, and surface chemical state of the materials, thereby determining their energy storage mechanism and device performance. Currently, nickel molybdate-based materials have been widely used to construct core-shell structures, one-dimensional nanofibers, and carbon-based composite electrodes for high-performance energy storage systems. Based on the above background, this paper systematically introduces the basic working principle of supercapacitors and summarizes and analyzes the classification, preparation methods, and electrochemical properties of nickel molybdate-based electrode materials, with the aim of providing certain references for the design and application of high-performance double metal oxide energy storage materials [13].

2. Supercapacitors and their energy storage devices

Supercapacitors, also known as electrochemical capacitors, are devices that achieve energy storage through the double-layer or pseudo-capacitive mechanism. They have extremely fast charging and discharging speeds, high power density, and long cycle life, and have become one of the important technologies for connecting renewable energy sources with the power grid.

2.1. Overview of supercapacitors

Supercapacitors are a new type of energy storage device that lies between traditional capacitors and chemical batteries. They possess both the rapid response capability of capacitors and the large-capacity energy storage feature of batteries, and occupy an important position in the modern efficient and green energy technology system. Unlike batteries that rely on internal chemical reactions for energy storage, the energy storage process of supercapacitors mainly relies on physical mechanisms and does not involve deep chemical reactions. Therefore, they exhibit excellent cycle stability and reversibility. Their working principle mainly relies on physical adsorption and surface chemical reactions to store electrical energy, namely the double layer effect and pseudocapacitive effect. This energy storage mechanism, which is mainly based on physical processes and supplemented by chemical processes, gives supercapacitors the advantages of fast charging and discharging, long service life, high power density, wide application range, high safety, and environmental friendliness [14]. According to the different energy storage mechanisms, electrochemical capacitors can be divided into double-layer capacitors and Faraday pseudocapacitors; by electrode material type, they can be classified as carbon electrode capacitors, metal oxide electrode capacitors, and conductive polymer capacitors.

DOI: 10.54254/2755-2721/2026.AD35143

2.2. The energy storage structure of supercapacitors

Supercapacitors, as an efficient electrochemical energy storage component, their energy storage and release depend on their physical structure and electrochemical properties. The energy storage structure of supercapacitors is not a single form but is composed of a multi-level system jointly formed by micro-material design and macro-system integration, covering the entire chain from materials to devices.

The energy storage mechanism of supercapacitors mainly consists of two types: electrochemical double-layer capacitance and pseudocapacitance. Electrochemical double-layer capacitance is the most classic working principle, which stores charges through the double layer formed between the electrode material (usually activated carbon) and the electrolyte. Due to the absence of chemical reactions, the energy conversion efficiency is high, and the cycle life can typically reach tens of thousands of times. Pseudocapacitance utilizes rapid reversible redox reactions on the surface of the electrode material to store energy. Compared with double-layer capacitance, it has higher energy density but slightly inferior cycle life. Supercapacitors usually consist of four key components: electrode materials, electrolytes, separators, and current collectors. The selection of electrode materials and electrolytes plays a decisive role in the performance of the device.

2.3. The composition, conducting mechanism and energy storage principle of supercapacitors

Supercapacitors are high-power energy storage devices based on the electrochemical double-layer principle. Their core consists of high specific surface area electrodes, ionic conductor electrolytes, and insulating separators. The conducting mechanism stems from the physical separation of charges: when a voltage is applied, the electrode surface attracts ions with opposite charges, while the interior of the electrode forms opposite charge layers through electron redistribution, and these two are separated at the electrode/electrolyte interface to form a "double layer" for the physical storage of charges. The energy storage principle is based on the classical capacitance formula:

$$E = \frac{1}{2} CV^2 \tag{1}$$

Electrode materials with high porosity can significantly increase the contact area between the electrode and the electrolyte, thereby enhancing the capacitance value. Meanwhile, the type of electrolyte determines the operating voltage of the device, which in turn affects the upper limit of energy storage, enabling a balance between high power density and rapid charging and discharging.

3. Energy storage principle

Figure 1 presents a typical structural schematic of a supercapacitor, including the four core components: electrode materials, electrolyte, separator, and current collector. Charge is stored through the double layer formed at the electrode/electrolyte interface or the pseudo-capacitive reaction on the electrode surface. This figure clearly illustrates the basic structure of a supercapacitor, facilitating the understanding of its energy storage mechanism.

DOI: 10.54254/2755-2721/2026.AD35143

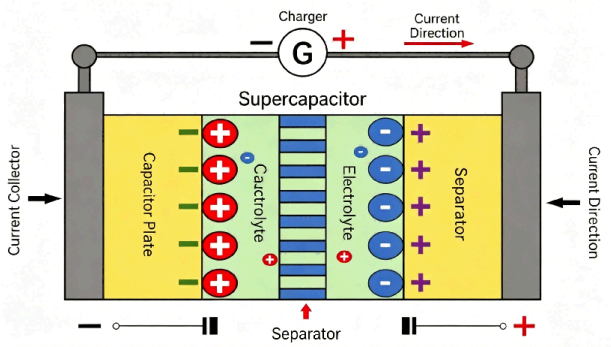

Figure 1. Schematic diagram of supercapacitor

3.1. The principle of double-layer energy storage

Figure 2 presents a typical schematic diagram of the double-layer energy storage principle.Double-layer energy storage is the most fundamental and earliest applied energy storage mechanism in supercapacitors. The key lies in utilizing the electrostatic interaction between the high specific surface area electrode materials (such as activated carbon, graphene) and the electrolyte ions to achieve physical energy storage. The uniqueness of this mechanism lies in its purity of the energy storage process, which solely relies on the physical adsorption of ions on the electrode surface and does not involve any chemical reactions.Due to the extremely short distance between the ionic layer and the electrode surface, this microstructure can generate a large capacitance per unit area. Electrical energy is stored in the form of an electrostatic field between the two layers of charges. In recent years, the research focus has gradually shifted to the regulation of the microstructure. By using biomass or polymer materials to construct carbon materials with microporous structures and high porosity, these materials can not only further increase the specific surface area, thereby enhancing the capacitance, but also improve the ion migration path by optimizing the pore size distribution and surface functionalization. This allows for an increase in energy density without sacrificing power density.

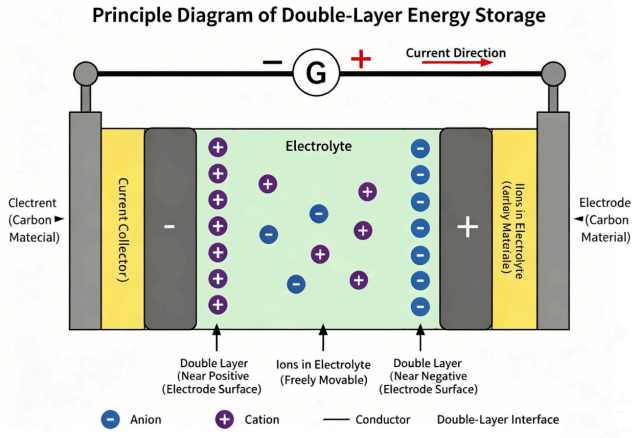

Figure 2. Schematic diagram of double-layer energy storage

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3.2. The principle of pseudo-capacitor energy storage

Figure 3 presents a typical schematic diagram of the principle of pseudo-capacitor energy storage.Pseudo-capacitor energy storage is an important breakthrough in the research of supercapacitors in recent years. Its energy storage mechanism lies between traditional capacitors and batteries. Unlike the physical adsorption of double-layer capacitors, pseudo-capacitance mainly achieves energy storage through rapid reversible oxidation-reduction reactions occurring on or near the surface of the electrode materials. Typical pseudo-capacitive materials include metal oxides and conductive polymers. During charging and discharging, these materials achieve rapid storage and release of charges through electron transfer and ion insertion/deletion processes. The advantage of pseudo-capacitance lies in its significantly higher capacity compared to double-layer capacitors, typically reaching tens to hundreds of times that of the latter. It can improve energy density while maintaining a relatively high power density. However, due to the ion diffusion within the electrode during this process, its power density and cycle life are slightly inferior to those of double-layer capacitors, and the stability of some materials at high voltages still needs to be improved. In recent years, research has gradually shifted to composite materials based on bimetallic oxides or metal-organic frameworks, in an attempt to further enhance cycle life while maintaining high energy density.

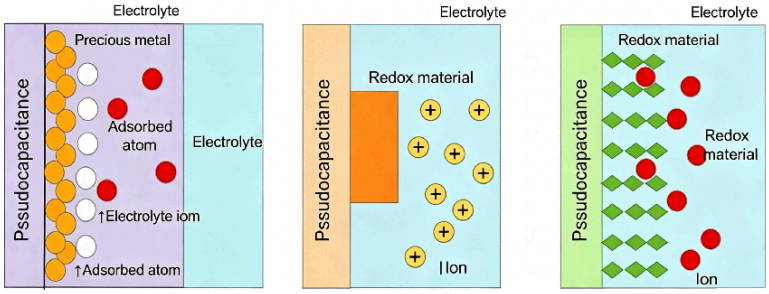

Figure 3. Schematic diagram of supercapacitor energy storage

4. Classification of electrode materials for supercapacitors

4.1. Carbon materials

Carbon materials are the most widely used electrode materials in supercapacitors, mainly storing energy through the double-layer capacitance mechanism. The advantages of these materials lie in their good chemical stability, high conductivity, and large specific surface area. Currently, the research trend is shifting from the traditional "activated carbon" to "high specific surface area carbon" and "functionalized carbon". In recent years, the research focus has been on constructing hierarchical pore structures to facilitate the rapid diffusion of ions within the electrode. Carbon materials prepared using biomass or metal-organic framework precursors can form multi-level pore structures consisting of micropores, mesopores, and macropores. However, pure carbon materials have a relatively low energy density and are difficult to meet the requirements of high energy density applications. Therefore, researchers have attempted to combine carbon materials with metal oxides to construct multifunctional composite materials. Carbon materials provide rapid electron transmission channels and ion diffusion paths, while the metal components introduce additional redox active sites. This "carbon + metal" dual-function design helps to enhance the energy density and power density of supercapacitors.

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4.2. Metal oxide

Metal oxides are the key materials for achieving high energy density in supercapacitors, mainly relying on the pseudocapacitive mechanism for energy storage. In recent years, research has focused on improving the electrical conductivity, structural stability, and multivalent activation of the materials. Studies have shown that by constructing bimetallic oxides (such as $NiMoO_4$, $CoMoO_4$, etc.), the synergistic effect between the bimetallic elements can induce phase transitions or enhance structural stability, thereby significantly improving the electrochemical performance. Nickel molybdate ($NiMoO_4$), as a typical bimetallic oxide, has the multivalent transition characteristics of Ni^{2+}/Ni^{3+} and Mo^{4+}/Mo^{6+}, and can provide abundant redox active sites, with a theoretical capacity far exceeding that of single-metal oxides. Additionally, by regulating the nanostructure (such as nanospheres, nanorods, nanosheets, etc.), the specific surface area can be further increased, and the ion diffusion path can be shortened, thereby enhancing the electrochemical performance [1, 8].

Figure 4 shows the schematic diagram of a supercapacitor device using $NiMoO_4$ as the electrode material. This device adopts an asymmetric structure, with the active material as the positive electrode and activated carbon as the negative electrode, fully leveraging the synergistic advantages of the high specific capacity of $NiMoO_4$ pseudocapacitive materials and the high power characteristics of activated carbon double-layer materials.

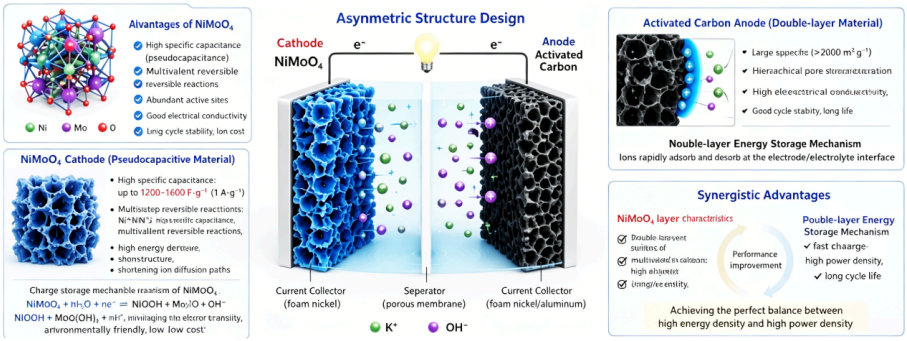

Figure 4. Schematic diagram of nickel molybdate capacitor device

4.3. Conductive polymer

Conductive polymers possess the high specific capacity of metal oxides and the flexibility of organic materials, making them particularly suitable for flexible electronic devices. Compared to metal oxides, conductive polymers have higher electrical conductivity and better flexibility, demonstrating excellent performance under high current density. However, there are still some issues in practical applications, such as the polymer chains may break due to volume expansion during long-term cycling, resulting in capacity degradation. To address this problem, the research trend in recent years is to combine conductive polymers with carbon materials or metal oxides. This not only enhances mechanical strength but also prevents the polymer layer from falling off through the supporting effect of carbon-based materials, thereby significantly improving cycling stability [7].

5. The preparation method of nickel molybdate material

Nickel molybdate ($NiMoO_4$), as a dual-metal oxide with diverse structures and excellent electrochemical activity, has become an important choice for preparing high-performance pseudocapacitive electrode materials due to its dual-metal synergy effect and controllable

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nanostructure. Based on its conversion pathway and performance requirements, various preparation methods have been developed, including nanostructure regulation, core-shell structure design, and composite electrode optimization, etc.

In the aspect of electrode material preparation, the research mainly focuses on three directions: Firstly, through hydrothermal or solvothermal methods, the nickel source and the molybdenum source react directly to form nanostructured molybdenum acid nickel, which utilizes its high specific surface area and abundant active sites to enhance the capacitance performance; Secondly, constructing core-shell structure or heterostructure (such as $NiMoO_4$@$NiMoO_4$, $MnMoO_4$@$NiMoO_4$, etc.) to compensate for the problems of poor conductivity and insufficient cycle stability of a single material; Thirdly, combining molybdenum acid nickel with carbon materials (such as graphene, carbon nanotubes, etc.) to prepare composite electrodes with high energy density and excellent cycle stability. These strategies aim to balance the conductivity, ion transport rate and structural stability of the materials, thereby optimizing the overall performance of supercapacitors. To obtain nanostructured materials with high specific surface area, hydrothermal methods have been widely used. Using nitric acid nickel and sodium molybdate as raw materials, in a mixed solvent of water and ethanol at a specific temperature (120–180°C) and pH conditions, a hydrothermal reaction can form different morphologies (nano spheres, nano rods, nano sheets, etc.) of molybdenum acid nickel, thereby significantly improving the ion transport ability and charge storage capacity. In 2013, Cai et al. prepared $NiMoO_4$ nanorods with a diameter of approximately 80 nm and a length ranging from 300 nm to 1 μm at 150°C through hydrothermal methods, and prepared hierarchical nano-spheres composed of nanosheets at 180°C with a diameter of approximately 2.5 μm and a nanosheet thickness of 10–20 nm . In the construction of core-shell structure, the two-step hydrothermal method is suitable for preparing molybdenum acid nickel-based core-shell heterostructures. This method first synthesizes the molybdenum acid nickel core material, and then grows the shell layer material through a secondary hydrothermal process on its surface to form a closely combined core-shell interface. The obtained material has both high specific capacitance and excellent cycle stability, suitable for high-performance supercapacitors. To address the issue of insufficient conductivity of a single molybdenum acid nickel material, in situ growth method can achieve effective composites of molybdenum acid nickel with carbon materials (such as reduced graphene oxide, carbon nanotubes, etc.), fully leveraging the synergistic effect of the two components, and improving specific capacitance and cycle stability [15].

Furthermore, by adjusting the synthesis parameters (such as temperature, time, solvent ratio, etc.), the crystal structure and morphology of nickel molybdate can be precisely controlled. Studies have shown that when the hydrothermal temperature is below 150°C, it is conducive to the formation of nanorod arrays, while at 180°C, it transforms into nanosheet arrays. Among them, the nanorod arrays exhibit superior electrochemical performance due to their larger active area and better electronic conductivity [16].

Table 1 summarizes the research results of Wang et al. [1] on the controllable synthesis of $NiMoO_4$ crystal phase and morphology by regulating the urea addition amount. As shown in Table 1, under the condition without urea, the hydrothermal synthesis yields thermodynamically stable α-$NiMoO_4$ nanorods with a specific capacitance of 252.6 C/g; while after introducing urea, the metastable β-$NiMoO_4$ exists in the form of nanosheets, with the specific capacitance increasing to 332.8 C/g. This result indicates that the urea-assisted synthesis can effectively stabilize the β phase and optimize the morphology, thereby significantly improving the electrochemical performance.

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5.1. Research on phase regulation and morphology control of nickel molybdate

Table 1. Comparison of synthesis conditions, morphology and electrochemical performance of different crystalline phases of $NiMoO_4$

Type of connection	Synthesis conditions	Appearance	Capacitance（C/g）	Characteristics	Source of literature
α-$NiMoO_4$	Without urea, hydrothermal treatment	Nanorods	252.6	Thermodynamically stable phase	[1]
β-$NiMoO_4$	With urea assistance, hydrothermal treatment	Nanosheets	332.8	Metastable phase, better electrochemical performance	[1]
Mixed phase	Intermediate condition	Mixed morphology	Between the two	Intermediate state during phase transition	[1]

To overcome the problems of insufficient conductivity and poor cycling stability of the single $NiMoO_4$ material, researchers have developed various composite and modification strategies. Table 2 summarizes the main modification methods reported in the literature and their performance characteristics. By combining with carbon materials, conductive polymers, or other metal oxides, the synergistic effects of each component can be fully exploited, improving the specific capacitance, energy density, and cycling life of the material. For example, Wang et al. [1] combined β-$NiMoO_4$ nanosheets with reduced graphene oxide hydrogel (rGH) to construct an asymmetric supercapacitor with an energy density of 36.1 Wh/kg; Zhang et al. [7] prepared a multi-dimensional $NiMoO_4$/$ZnMoO_4$/NF heterostructure that exhibited an ultra-high specific capacitance of 2064.07 F/g and excellent cycling stability.

5.2. The composite/multi-modified strategies reported in the literature

Table 2. Modification strategies and performance characteristics of $NiMoO_4$-based composite materials

Modification strategy	Composite materials	Preparation method	Performance improvement	Source of literature
Carbon composite	$NiMoO_4$ /rGO	Hydrothermal method	Improve conductivity	[7]
Conductive polymer composite	$NiMoO_4$ /rGO/PANI	Three-step synthesis	High specific capacitance	[7]
Heterogeneous structure	$NiMoO_4$ @$Co(OH)_2$	Electrospinning + Hydrothermal treatment	One-D core-shell structure	[1]
Bimetallic molybdate	$NiCoMoO_4$	Hydrothermal method	Synergistic effect	[8]
MXene composite	$NiMoO_4$ /MXene	Interface Engineering	Flexible supercapacitor	[9]
Rare earth doping	Nd-doping $NiMoO_4$	Sol-gel method	Introduce defects to enhance activity	[7]

Different preparation methods have significant effects on the crystal structure, morphology and electrochemical performance of $NiMoO_4$. Table 3 compares the main preparation methods reported in the literature and the characteristics of their products. As shown in Table 3, Sakthivel et al. [2]

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compared the co-precipitation method and the microwave combustion method. The former had a specific capacitance of 168 F/g at 1 A/g, while the latter could reach 224 F/g. Kuri et al. [3] systematically compared the hydrothermal method and the sol-gel method, and found that the pure α phase was obtained by hydrothermal synthesis at 180°C, while an α+β mixed phase was formed at 220°C. Dhandapani et al. [13] used the soft template method to finely control the morphology of $NiMoO_4$. These methods provide important references for designing $NiMoO_4$ nanostructures on demand.

5.3. Comparison table of preparation methods for nickel molybdate ($NiMoO_4$) materials

Table 3. Effects of different preparation methods on the morphology and properties of $NiMoO_4$

Preparation method	Specific process parameters	Appearance	Capacitance（C/g）	Test conditions	Source of literature
Co-precipitation method	temperature - 80°C, pH regulation, precipitant $NaOH/NH_3 \cdot H_2O$	Nanoparticles	168	1 A/g	[2]
Microwave combustion method	Microwave power: 300 - 800 W. Urea is used as the fuel. The reaction lasts for several minutes.	Nanoparticles	224	1 A/g	[2]
Hydrothermal method（180°C/24h）	180°C，24 h，650°C calcination	α-phase nanoparticles	-	-	[3]
Hydrothermal method（220°C/24h）	220°C，24 h，650°Ccalcination	α+β-phase nanoparticles	-	-	[3]
CTAB-assisted hydrothermal method	With the assistance of CTAB surfactant, the morphology can be controlled.	Nanoflakes / Nanorods	-	-	[14]
Electrospinning method	Voltage: 10 - 30 kV, Calcination: 400 - 600°C	Nanofibers	-	-	[15]
Solvothermal method	Different synthesis conditions	Shape and size adjustable	-	-	[3]

Table 4 summarizes the electrochemical performance of various $NiMoO_4$ -based electrode materials reported in the literature for supercapacitors in recent years. As shown in Table 4, the specific capacitance of pure $NiMoO_4$ materials ranges from 168 to 974 F/g, and the specific values are strongly dependent on the preparation method and morphology structure. By constructing composite materials and heterostructures, the performance can be significantly improved: Zhang et al. [7] prepared the $NiMoO_4$ /$ZnMoO_4$ /NF electrode with a specific capacitance of up to 2064.07 F/g, and the capacity retention rate reached 99% after 10,000 cycles; Tang et al. [10] constructed the $NiMoO_4$/Ni-Co hierarchical structure and achieved an energy density of 38 Wh/kg and a cycle retention rate of 80% (5,000 cycles) in the hybrid supercapacitor. These results fully demonstrate that reasonable structural design and composite strategies are the key approaches to improving the performance of NiMoO4-based supercapacitors.

DOI: 10.54254/2755-2721/2026.AD35143

5.4. Performance data sheet of nickel molybdate material for use in supercapacitors

Table 4. Summary of supercapacitor performance of $NiMoO_4$-based composite materials

Material system	Preparation method	Capacitance（C/g）	Current density	Cyclic stability	Energy density	Source of literature
Pure $NiMoO_4$ (co-precipitated)	Co-precipitation method	168 F/g	1 A/g	-	-	[2]
Pure $NiMoO_4$ (Microwave Combustion)	Microwave combustion method	224 F/g	1 A/g	-	-	[2]
$NiMoO_4$ nanosheets (β phase)	Hydrothermal method (with urea as an auxiliary agent)	332.8 C/g (~832 F/g)	1 A/g	70.2% (5000)	36.1 Wh/kg	[1]
$NiMoO_4$ nanorods (α phase)	Hydrothermal method (without urea)	252.6 C/g (~631 F/g)	1 A/g	-	-	[1]
$NiMoO_4$ nanorod hydrate	Hydrothermal method (without calcination)	-	-	60(~1100 min)	-	[4]
$NiMoO_4$@Ni-Co DH/CC	Two-step hydrothermal method	-	-	80% (5000)	38 Wh/kg / 7043 W/kg	[10]
$NiMoO_4$ nanosheets @ Ni-Co DH nanoniblock clusters	Two-step hydrothermal method	-	-	80% (5000)	38 Wh/kg / 7043 W/kg	[10]
$NiMoO_4$@MnO_2 nanoflowers	hydrothermal method	-	-	-	-	[5]
$CoMoO_4$-$NiMoO_4$ microspheres	Urea-assisted hydrothermal process	-	-	-	-	[16]
$NiMoO_4$/$CoMoO_4$ nanosheet arrays	hydrothermal method	-	-	-	-	[1]

6. Conclusion

Nickel molybdate ($NiMoO_4$), as a high-performance bimetallic oxide electrode material, can significantly enhance its electrochemical performance through strategies such as method optimization (hydrothermal method, two-step hydrothermal method), phase regulation (α phase → β phase), morphology design (nanosheets, core-shell structure), and composite modification (carbon materials, conductive polymers, heterostructures). In particular, the hierarchical structure of $NiMoO_4$nanosheets@Ni-Co DH nanoneedle clusters prepared by the two-step hydrothermal method exhibits excellent energy density and cycle stability in both hybrid supercapacitors and alkaline zinc batteries, providing an important reference for the design of next-generation high-performance energy storage materials.

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