World Journal of Advanced Research and Reviews
eISSN: 2581-9615 CODEN (USA): WJARAI
Cross Ref DOI: 10.30574/wjarr
Journal homepage: https://wjarr.com/

(Review Article)

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A comprehensive review of molybdenum nanomaterials

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World Journal of Advanced Research and Reviews, 2024, 23(02), 1624–1632

Publication history: Received on 27 June 2024; revised on 11 August 2024; accepted on 13 August 2024

Article DOI: https://doi.org/10.30574/wjarr.2024.23.2.2377

Abstract

In the past few decades, there have been significant developments in nanotechnology in various areas. The present review focuses on the Molybdenum-based nanomaterials which have shown promising applications in the field of electronic and energy storage devices due to their tunable bandgaps, strong interaction with light. Molybdenum nanomaterials have characteristics next to graphene nanomaterials. Nano-scale forms of molybdenum oxide and molybdenum sulfide are excellent materials for supercapacitor electrodes. They exhibit high carrier mobility and optical transparency, which are considered to be the development of photodetectors. The review indicates different methods preferred for the synthesis of molybdenum-based nanomaterials and focuses on Molybdenum nanomaterials for electronic and energy storage devices.

Keywords: Molybdenum nanomaterials; Electronic; Energy; Photodetectors; Molybdenum oxide; Molybdenum sulfide; Supercapacitor

1. Introduction

The demand and the advance in the development of flexible, portable electronic and energy storage devices are based on the energy storage devices such as fuel cells and supercapacitors. Supercapacitors are electrochemical capacitors, which act as a linking bridge between batteries and conventional capacitors. Batteries have higher specific energy for lower specific power and capacitors usually have low specific energy over the high specific power, whereas, supercapacitor shows higher power densities, short charging time, and long discharging time compared to batteries and an excellent specific capacitance and high energy densities than the traditional capacitors [1].

Electrochemical double-layer Capacitors and Pseudocapacitors are the two types of supercapacitors. Electrochemical double-layer capacitors employ electrostatic adsorption at the electrode and electrolyte interface to store energy. These include nanoporous carbonaceous materials which are defined with high specific surface area, high conductivity, and high mechanical conductivity. Pseudocapacitors prefer transition metal oxides and conducting polymers, as quick and reversible redox reaction takes place nearby / on to the surface of the electrodes in the process of the storage of charge [2,3].

Higher capacitances and energy densities are exhibited by pseudocapacitors, cost-effective over the electrochemical double-layer capacitors. Examples of transition metal oxides electrodes for pseudocapacitors are ruthenium oxide, manganese dioxide, and cobalt oxide. Conducting polymers such as polyaniline, polypyrrole and polythiophene can be used as pseudo capacitor electrodes [4,5].

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However, the growing demand for energy storage devices led to the development of new types of electrode materials. Therefore, the research of nanometer-scale metal oxide and sulfide as the material of supercapacitor electrodes has become a new field. For example, cobalt sulfide (CoS, CoS_2), nickel sulfide (NiS, NiS_2, Ni_3S_2), molybdenum sulfide (MoS_2), copper sulfide (CuS, Cu2S), and vanadium sulfide (VS, VS_2) have been used as supercapacitors electrode materials [6 - 8].

Transition metal dichalcogenides (TMDs) are of great potential for the use of next-generation electronic devices. Of these, semiconducting TMDs can be produced by combining the metals (M)W and Mo with ore-forming chalcogens (X) S or Se in the form MX_2. These materials have a structure that consists of strong in-plane covalent bonds (X–M–X) that create isolated atomic layers resulting in a bulk crystal when they interact with one another through weak van der Waals forces [9-11].

Electronic properties of the material change with the number of layers. Monolayer exhibits a direct bandgap, when the layer increases, materials show an indirect bandgap with its bulk structure. this fact is identified by spectroscopic and electronic studies in the mechanically exfoliated MoS2 and other semiconducting TMDs [12, 13]. Mineral crystals are used for the isolation of monolayers of MoS_2 by exfoliation through different top-down approaches, including mechanical exfoliation [10,14], chemical exfoliation [15], and ultrasonic treatments [16].

Molybdenum trioxide (MoO3) is an n-type semiconductor metal oxide and an important transition metal oxide and an important transition metal oxide due to its high melting point, high chemical stability and thermal stability. Therefore catalysis, lubricants, solar cells, lithium-ion batteries, equipment etc., have applications in the industry. But, the effectiveness depends on the synthetic process and the experimental conditions such as reaction time, temperature etc.

In recent years, the contribution of Molybdenum oxide (MoO_3) and molybdenum sulfide (MoS_2) related materials has been identified [6]. As it stimulated interest among other transition metal sulfides due to its layered structure and inherent conductivity,[17] and it is considered to be a suitable replacement for graphene and carbon nanotubes in energy storage applications. In addition, molybdenum-based materials (such as MoO_3, MoO_2, and MoS_2) exhibit various valences and rich chemical properties, making them viable candidate materials for electrochemical applications [18].

2. Synthesis Of Molybdenum Sulfide Nanoparticles

MoS_2 is a transition metal sulfide with a layered structure, where a metal molybdenum layer is sandwiched between two sulfur layers with weak van der Waals forces, and the interlayer S–Mo–S atoms are strongly linked with covalently [19-21]. MoS_2 possesses unique physicochemical properties due to its unique atomic and electronic structure. It has application in solid lubricants, catalysts, supercapacitors, and lithium-ion batteries [22-24]. Among these, the research on the application of MoS_2 as a supercapacitor electrode material is the most extensive.

Soon *et al.* found that the MoS_2 nano-film presented an electric double layer capacitance behaviour [25].

Ma *et al.* reported that nano-MoS_2 intercalated in polypyrrole could improve its capacitance performance [26].

Cao *et al.* fabricated micro-supercapacitors using coated MoS2 nanofilms and showed that MoS_2 has excellent electrochemical performance in aqueous electrolytes due to its structure. Usually, two-dimensional electrochemical electrodes face inadequate contact with the electrolyte and hence low surface area utilization efficiency. Numerous efforts have been made to design three-dimensional (3D) electrodes, such as MoS_2/mesoporous carbon spheres. Recently, there have been some reports related to $NiCo_2S_4$ and graphene oxide composites applied in supercapacitors [27].

Krishnamoorthy *et al.* reported specific capacitance of chemically prepared MoS_2 nanostructure as 92.85 F/g [28].

Huang *et al.* reported polyaniline/MoS_2 composites as supercapacitor electrodes with the specific capacitance of 575 F/g. With the hydrothermal method, flower-like molybdenum disulfide microspheres were synthesized. It can be used as a supercapacitor electrode and exhibited high specific capacitance (518.7 F/ g) and excellent cycling performance (88.2% retention after the completion of 2500 cycles). In addition, a high-performance symmetric supercapacitor was successfully fabricated by using MoS2 as both positive electrode and negative electrode, which exhibited a high energy density of 12.46 W h/kg at a power density of 70 W/ kg [29].

Molybdenum disulfide (MoS_2) is a material having fascinating properties, like high surface area, higher ionic conductivity than metal oxides [30], and good mechanical flexibility [31]. Hence, its application is extended towards in the field of electronic appliances, gas sensors, supercapacitors, batteries, hydrogen evolution reactions [28, 32-35]. The MoS_2 nanosheets are synthesized either by bottom-up approaches, such as hydrothermal, chemical vapor deposition (CVD), or by top-down approaches, like, ball milling, mechanical exfoliation, and liquid phase exfoliation [28, 36-39].

Among them, liquid-phase exfoliation is the simple and high-yielding route to prepare the MoS_2 nanosheets. The solvents such as ethanol, dimethylformamide (DMF), N-Methyl-2-pyrrolidone (NMP) are used to exfoliate the bulk MoS_2 particles into nanosheets [39,40]. The MoS_2 nanosheets are proven to improve the electrochemical performance of materials like Co_3O_4, polyethylene dioxythiophene (PEDOT), polyaniline (PANI), and Mn_3O_4 [29, 41-43]. The mixture of MoS_2 and n-butyllithium (2.5 M in hexanes) was autoclaved at 90^0C for 12h with stirring, and the product formed was filtered and washed with anhydrous hexane. It was vacuum-dried and subjected to ultrasonication. The obtained suspension was then neutralized with 1 M HCl and the product obtained was washed with distilled water-methanol, finally, it was subjected to freeze-drying [44].

Three-dimensional flower-like molybdenum disulfide microspheres composed of nanosheets were prepared by a hydrothermal method using ammonium molybdate as the molybdenum source and thiourea as the sulfur source. Structural and morphological characterizations were performed by X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), energy-dispersive X-ray (EDX) spectroscopy, and X-ray photoelectron spectroscopy (XPS).

The electrochemical properties of MoS_2 electrode were studied by performing cyclic voltammetry (CV), galvanostatic charge-discharge analysis, and electrochemical impedance spectroscopy (EIS). When used as an electrode material for supercapacitor, the hybrid MoS2 showed a high specific capacity of 518.7 F/g at a current density of 1 A/g and 275 F/g at a high discharge current density of 10 A /g. In addition, a proportional supercapacitor composed of MoS_2 showed high energy density. The outstanding performance of the MoS2 electrode material indicates its great potential for applications in the high-performance energy storage system [45].

Molybdenum Oxide (MoO_3) is a prominent transition metal oxide with rich polymorphism and structural litheness [7], with many valences and unique structure with outstanding specific capacitance [46]. The electrochromic and catalytic properties are important in storage media, gas sensors, and organic solar cells. It possesses greater electrochromic and catalytic properties [17] that can potentially be functional in storage media [47], gas sensors [48] and humidity sensors [49], organic solar cells [50]. The oxidation states of molybdenum oxide range from + 2 to + 6 and predominantly exist in two primary forms, viz., molybdenum (IV) oxide and molybdenum (VI) oxide [51]. Solution combustion [52, 53], sol-gel [54], microwave [55], and green synthesis methods [56] are the common techniques preferred for the synthesis of molybdenum oxide nanoparticles. Of which, the hydrothermal procedure [57] is preferred for the preparation of molybdenum oxide nanoparticles. Molybdenum oxide nanoparticles are obtained in different morphologies such as nanobelts [58], nanoflowers [59], nanowires [60], and nanocubes [61] can be obtained. These morphologies are important in knowing the specific capacitance of the substantial. With the hydrothermal method, the effect of parameters such as temperature, pressure, and reaction time on the physiochemical performance of the material can be determined [62].

3. Synthesis of molybdenum oxide nanoparticles

Miao et al. reported the synthesis of Molybdenum Trioxide nanostructures by cost-effective metal-assisted chemical wet etching method, with this method material indicated a specific capacitance of 30.85 F/g in 0.5M Na_2SO_4 electrolyte solution [63].

Wang et al. synthesized α-MoO_3 nanorods through the hydrothermal method and observed that annealed α-MoO3 nanorods demonstrated excellent specific capacitance compared to hydrothermally obtain ones [64].

Shakir *et al.* reported the preparation of orthorhombic molybdenum trioxide nanowires using a hydrothermal method which yielded a specific capacitance of 168 F /g at 0.5/ Ag current density and 97% cyclic retention in 1M H_2SO_4 electrolyte solution [65].

There are various synthesis methods adopted such as Sol-Gel, Solution Combustion, Infrared Irradiation, Sputtering, Microwave, Hydrothermal, Electrochemical, Sono-chemical, etc. Amongst these, hydrothermal method has unique advantages in terms of high reactivity and easy control of interface reactions and enables to produce stable and condensed phases with low energy consumption and less environmental pollution without emitting any harmful gases. Reports indicated that the synthesis of MoO3 nanoparticles of 100nm via citrate Sol-Gel method at 250 °C /1h followed by calcination at 500 °C. The synthesis of MoO_3 nanoparticles via solution combustion method employing ammonium hepta molybdate (AHM) at 470 °C. Microwave assisted synthesis of MoO3 nanoparticles was involving high power of 70 Watts.

In the above-reported works, very high concentrations of electrolyte were used for determining the electrochemical performance of the synthesized molybdenum oxide nanomaterials. In the synthesis of molybdenum oxide nanorods hydrothermal method is preferred, and its electrochemical studies were based on the low concentration of electrolyte, carrying exceptional rate competence and high specific capacitance.

Ultrasonication-assisted liquid-phase exfoliation method was considered for the preparation of MoS_2 nanosheets [66]. Polyvinylpyrrolidone (PVP, MW ~ 40,000) was dissolved in ethanol to bulk MoS_2 powder. The mixture was ultrasonicated to exfoliate MoS_2 nanosheets. The exfoliated MoS_2 sheets which remained on top of the solution were subjected to post-treatment with isopropyl alcohol (IPA).

The hydrothermal method was followed for the synthesis of Molybdenum oxide nanostructures. Ammonium Heptamolybdate Tetrahydrate solution was treated with 0.01M sodium dodecyl sulfate on stirring and pH was maintained to 3 with the dilute HCl. Hydrothermal treatment was given in the Teflon-lined stainless-steel autoclave 180°C for 24 h. the precipitate formed was washed with water and ethanol and the precipitate was obtained on centrifugation was subjected to drying at 60 °C overnight to get dry powder [1].

There are various synthesis methods adopted such as Sol-Gel, Solution Combustion, Infrared Irradiation, Sputtering, Microwave, Hydrothermal, Electrochemical, Sono-chemical, etc. [28–30]. Amongst these, hydrothermal method has unique advantages in terms of high reactivity and easy control of interface reactions and enables to produce stable and condensed phases with low energy consumption and less environmental pollution

With modified alumina powder in the solution of molybdenum(V) chloride in ethanol, Alumina/molybdenum nanocomposites were obtained. Modified alumina powder was subjected to calcined at low temperature to eliminate the organic residue. It was followed with reduction steps to form obtain 2–10 nm metallic nanoparticles on the surface of the alumina crystals. The method indicated the formation of microstructure with improved mechanical behaviour [67].

So, it is presumed that the electrochemical stability of the SnO2 could be improved by using MoS_2 nanosheets along with the SnO2 nanoparticles. There are very limited studies reported focussing on the supercapacitor application with the combination of MoS2–SnO2 phases as nanocomposite [68]. The hydrothermal method is the widely used technique to prepare MoS_2–SnO_2 nanocomposite with the ligand exchange process [69], SnO_2 nanoparticles are functionalized onto the surface of the MoS_2 nanosheets at room temperature. The route is expected to be energy-saving and produce the MoS_2–SnO_2 nanocomposite which will serve as a good supercapacitor electrode material.

According to Ziying *et al.,* MoS_2-RGO hybrids were prepared by self-assembly of MoS_2 NPs and GO nanosheets, followed by hydrothermal treatment [70].

In the study conducted by Yun *et al.,* for the synthesis of MoS_2/ GO nanocomposites, the bulk MoS_2, was mixed with stock solutions of GO by sonication for 40 h, respectively. As a control, bulk MoS2 was sonicated alone in deionized water. The mixture was subjected to settling and then centrifuged to obtain the final precipitate [71].

As per Bin *et al.,* a mixture of MoS_2 or M-MoS_2 in acetone was subjected to magnetic stirring at room temperature followed by sonication with the addition of epoxy resin. The contents will be subjected to vacuum distillation under stirring with a magnetic stirrer at 80 ^{0}C. On cooling a stoichiometric amount of curing agent (D_{230}) corresponding to 100% of EP resin content was added and stirred for some time. The resulting mixture was cured and post-cured to obtain the final nanocomposite [72].

Amine-functionalized MoS_2 and acyl chloride-coordinated ND, chemically conjugated nanodiamond (ND)/MoS_2 nanocomposite was formed and its structure with morphology were analyzed to know the scattering of MoS_2 on the ND platform. The study revealed that the efficient electron capacity of the ND/MoS_2 nanocomposite was considerably greater than that of the MoS_2 electrode alone. Therefore, the nanophase electrode showed higher electrochemical capacitance than that of the MoS_2 electrode alone [73].

4. Characterization

Characterization of molybdenum nanoparticles is essential for understanding their properties and potential applications. Here's a general review of the characterization techniques commonly used for molybdenum nanoparticles:

- Transmission Electron Microscopy (TEM): It is used to determine the size, shape, and dispersion of molybdenum nanoparticles. It provides high-resolution images, allowing researchers to observe individual nanoparticles.
- Scanning Electron Microscopy (SEM): It is useful for studying the surface morphology and size distribution of molybdenum nanoparticles. It provides 3D images at lower resolution compared to TEM.
- X- ray Diffraction (XRD): It is used to identify the crystal structure and phase composition of molybdenum nanoparticles. It can provide information about the size of nanoparticles using Scherrer equation [74].
- Energy Dispersive X-ray Spectroscopy (EDS) : It is coupled with SEM to determine the elemental composition of molybdenum nanoparticles and to verify their purity.
- Fourier Transform Infrared Spectroscopy (FTIR): FTIR is used to analyze the surface functional groups and chemical bonds on the surface of molybdenum nanoparticles, especially when they are functionalized or coated.
- UV-Visible Spectroscopy: UV-Vis spectroscopy is used to study the optical properties of molybdenum nanoparticles, such as absorption and surface plasmon resonance (SPR) effects.
- Thermal Analysis: Techniques like thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) can provide information about the thermal stability and decomposition behavior of molybdenum nanoparticles.
- Magnetic Measurements: For magnetic nanoparticles, techniques like vibrating sample magnetometry (VSM) can be used to study their magnetic properties.
- Electrochemical Characterization: Techniques like cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) can be used to study the electrochemical behavior of molybdenum nanoparticles, which is important for applications in batteries, sensors, and catalysis.
- Surface Area Analysis: Methods like Brunauer-Emmett-Teller (BET) analysis can be used to determine the surface area and porosity of molybdenum nanoparticles, which is important for catalytic applications.

Overall, a combination of these characterization techniques is often used to fully understand the properties of molybdenum nanoparticles and optimize their synthesis for specific applications. The electrochemical measurement of as-synthesized MoO_3 nanorods was analyzed by cyclic voltammetry, galvanometric charge-discharge, and electrochemical impedance spectroscopy [1].

5. Conclusion

Molybdenum nanomaterials have diverse applications in electronic and energy devices. Various synthesis methods are used to molybdenum based nanomaterials. Molydbenum nanoparticles exhibit tunable bandgaps and strong light interaction. The review highlighted about the synthesis of molybdenum nanomaterials by different approaches, of which it is clear that the hydrothermal method is preferred for the synthesis of molybdenum-based nanomaterials.

Also, the review indicated that the structural and morphological characterization study was done with XRD, SEM, TEM, EDX and XPS. Further study revealed that molybdenum-based nanomaterials have excellent electrochemical properties as it showed high energy density due to which can be considered in the application of high-performance energy storage systems.

Compliance with ethical standards

Disclosure of conflict of interest

There are no conflicts to declare.

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