Chalcogenide Nanocomposites for Energy Materials

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ABSTRACT: This conversation explores various aspects of chalcogenide nanocomposites for energy applications, delving into synthesis methods, environmental concerns, and performance optimization strategies. Discussions highlight the significance of sustainable synthesis techniques like hydrothermal and UV-assisted methods, emphasizing their advantages in terms of economic feasibility and environmental impact. Additionally, attention is drawn to challenges such as limited cycle life and suboptimal efficiency, prompting exploration of solutions including enhanced conductivity, electrode design optimization, and understanding degradation mechanisms. The conversation underscores the importance of interdisciplinary research efforts in addressing these challenges and advancing the field of chalcogenide nanocomposites for sustainable energy technologies. Through collaboration and innovation, researchers aim to harness the full potential of these materials, paving the way towards a greener and more efficient energy future.

KEYWORDS: Chalcogenide nanocomposites, Energy applications, Synthesis methods, Sustainability, Performance optimization.

1. INTRODUCTION

In recent years, the global landscape of energy consumption has undergone a transformative shift, driven by the growing awareness of environmental sustainability and the need for efficient, clean energy sources. This paradigm shift has propelled the demand for advanced materials that can address the intricate challenges associated with energy production, storage, and conversion [1]–[3]. As traditional energy resources face limitations and environmental concerns, there is an urgent need for innovative materials capable of revolutionizing the way we harness and utilize energy. The relentless pursuit of cleaner and more sustainable energy solutions has spurred a wave of research and development in materials science. Advanced materials, characterized by tailored properties at the nanoscale and multi-functionality, have emerged as pivotal components in the quest for enhanced energy technologies [4], [5]. These materials not only offer improved efficiency and performance but also exhibit the potential to mitigate environmental impacts associated with conventional energy sources.

The field of energy materials has become a focal point for scientific inquiry and technological innovation, encompassing a diverse array of applications such as energy storage devices (batteries, supercapacitors), energy conversion technologies (solar cells, thermoelectric devices), and catalysts for sustainable fuel production [6]–[9]. The integration of advanced materials into these applications holds the promise of unlocking unprecedented levels of efficiency, durability, and environmental sustainability.

In this dynamic and evolving landscape, chalcogenide nanocomposites have emerged as particularly promising candidates for addressing the intricate challenges in energy materials. Chalcogenides, characterized by their unique electronic and optical properties, present an exciting avenue for research and development in the pursuit of efficient and sustainable energy solutions. As the demand for energy continues to escalate, harnessing the full potential of chalcogenide nanocomposites becomes imperative for advancing the frontiers of energy science and technology.

This increasing demand for advanced materials in the field of energy sets the stage for the exploration and investigation proposed in this review – the synthesis and characterization of chalcogenide nanocomposites. By delving into this innovative realm, the research aims to contribute to the ongoing global efforts to reshape the energy landscape, addressing challenges and unlocking new possibilities for a sustainable and energy-efficient future.

1.1. Chalcogenides

In recent years, the pursuit of sustainable and efficient energy solutions has driven intensive research into advanced materials tailored for energy applications. Among these materials, chalcogenide nanocomposites have emerged as particularly promising candidates. Chalcogenides are compounds that contain elements from the chalcogen group, which includes oxygen (O), sulphur (S), selenium (Se), tellurium (Te), and polonium (Po). These compounds are characterized by the presence of chalcogen atoms, often in combination with other elements [10], [11].

Chalcogenides are known for their diverse range of properties, including electronic, optical, and thermal characteristics. These properties make chalcogenides valuable in various applications, such as semiconductor

devices, photovoltaics, thermoelectric materials, and as components in energy storage systems [12]–[16]. Chalcogenides has tunable electronical properties. The nanoscale dimensionality of these materials further enhances their electronic characteristics, enabling precise control over conductivity and band structures. Chalcogenides shows high charge carrier mobility and chemical stability which is high required for improved energy applications. Chalcogenides, including oxygen, are not only highly useful, but they are also abundantly available in the Earth's crust, alleviating concerns about resource scarcity. This abundance not only renders chalcogenide nanocomposites economically viable but also aligns with the principles of sustainability in the development of energy materials.

Chalcogenides are particularly well-suited to form compounds with transition metals due to the variable oxidation state of transition metals, allowing them to form a variety of compounds with chalcogenides. This versatility is crucial for the development of materials with diverse properties and applications [17]. Transition metals can form coordination complexes with chalcogenides, leading to the creation of well-defined structures. Transition metals have partially filled d-orbitals, providing numerous vacant and accessible orbitals for bonding with chalcogen atoms [18]. This electronic configuration allows for the formation of stable compounds with chalcogenides. The interaction between transition metals and chalcogenides can lead to materials with desirable electronic and optical properties [19], [20]. This makes them suitable for applications in electronics, sensors, and optoelectronic devices [21], [22].

1.2. Transition Metal Chalcogenides
Transition metal chalcogenides are compounds composed of transition metals (elements from the d-block of the periodic table) and chalcogens, which are elements from Group 16 (also known as Group VI-A or the oxygen group). Transition metal chalcogenides can form a wide range of compounds with various stoichiometries, and they often exhibit interesting electronic, magnetic, and catalytic properties. Common transition metals that form chalcogenides include zinc (Zn), cadmium (Cd), mercury (Hg), and nickel (Ni), among others.

These compounds exhibit diverse properties depending on the specific combination of transition metal and chalcogen, and they have applications in various fields, including catalysis, electronics, and energy storage [23]–[25]. The properties and applications can vary widely, making transition metal chalcogenides an intriguing area of study in materials science and chemistry.

2. UNIQUE PROPERTIES OF TRANSITION METAL CHALCOGENIDE NANOCOMPOSITES
The justification for research in transition metal chalcogenide nanocomposites stems from their unique properties and the promising potential they hold for applications in energy storage and conversion.

2.1. Tunable Electronic Properties
Transition metal chalcogenides exhibit tunable electronic properties, a characteristic that underpins their versatility and appeal in various technological applications. This tunability arises from the unique combination of transition metals and chalcogens in their composition. One of the defining features is the ability to manipulate the band gap, the energy range where electrons can exist or not, making these materials suitable for applications in electronics and optoelectronics. For instance, compounds like molybdenum disulfide (MoS_2) and zinc sulphide (ZnS) are well-known for their tunable band gaps, enabling the development of transistors and sensors with tailored electronic characteristics [26], [27]. This property is crucial in designing semiconductor devices, where precise control over the flow of electrons is essential. The tunable electronic properties of transition metal chalcogenides extend beyond traditional semiconductors, with applications in catalysis, energy storage, and conversion devices. Researchers leverage this tunability to optimize performance in specific applications, offering a platform for advancements in materials science and technology.

2.2. High Surface Area
The high surface area of transition metal chalcogenides is a distinctive feature that plays a crucial role in determining their reactivity and suitability for a range of applications. Transition metal chalcogenides often exhibit nanostructured forms, such as nanosheets, nanotubes, or nanoparticle ensembles, which contribute to their increased surface area compared to bulk materials [28]–[31]. This enhanced surface area is a result of the unique arrangements and morphologies that arise during the synthesis processes.

The significance of high surface area lies in its direct impact on the material's reactivity and its interactions with other substances. In the context of catalysis, a high surface area provides more active sites for chemical reactions to take place, leading to improved catalytic efficiency. This property is particularly advantageous in applications such as fuel cells, where transition metal chalcogenides can serve as catalysts for various electrochemical reactions [32], [33].

Moreover, in energy storage applications, the high surface area facilitates greater electrode-electrolyte interactions in batteries and supercapacitors. This characteristic is pivotal for achieving high capacitance and rapid charge-discharge rates [34], [35]. The increased surface area also enhances the accessibility of active sites, influencing the overall performance of these materials in energy storage devices.

In the field of nanoelectronics, the high surface area of transition metal chalcogenides contributes to increased contact interfaces, which can positively affect charge transport and electronic conductivity. This property is particularly relevant in the development of electronic devices, such as transistors and sensors [32], [36].

2.3. Chemical Stability

The chemical stability of transition metal chalcogenides is a crucial aspect that underlies their utility in various applications. Generally, these compounds exhibit notable resistance to chemical degradation under specific environmental conditions. This stability arises from the strong covalent bonds formed between the transition metal atoms and the chalcogen atoms, creating robust crystal structures. For example, in molybdenum disulfide (MoS2) and tungsten disulphide (WS$_2$), the metal-chalcogen bonds contribute to the materials' resilience against oxidation and corrosion [37], [38]. The layered structures of many transition metal chalcogenides also play a role in protecting the material from external influences. This chemical stability is particularly advantageous in energy-related applications, where materials are exposed to harsh conditions, such as those encountered in batteries and catalysts. The ability of transition metal chalcogenides to maintain their integrity under such conditions enhances the durability and longevity of devices, contributing to the overall effectiveness of these materials in technological advancements. Researchers continue to explore and optimize the chemical stability of transition metal chalcogenides to further expand their applications in emerging fields of materials science and nanotechnology.

2.4. Catalytic Activity

The catalytic activity of TMCs has garnered significant attention in the field of catalysis due to their unique electronic and structural properties. These materials, which include compounds such as nickel sulphide (NiS), cadmium sulphide (CdS), and zinc oxide (ZnO), exhibit a diverse range of catalytic behaviors that make them promising candidates for various applications. One key aspect contributing to their catalytic prowess is the presence of active sites on their surfaces, often associated with exposed edges and defects in their layered structures. These sites facilitate interactions with reactant molecules, promoting efficient catalytic processes. TMCs have shown remarkable performance in hydrogen evolution reactions (HER), oxygen reduction reactions (ORR), and other electrocatalytic reactions relevant to energy conversion technologies [39]–[44]. Additionally, the tunability of their electronic properties allows for the optimization of catalytic activity, opening avenues for designing catalysts with enhanced performance. The exploration of transition metal chalcogenides in catalysis holds promise not only for energy-related applications but also for environmental remediation and industrial processes, positioning them as valuable contributors to the advancement of sustainable technologies.

3. POTENTIAL APPLICATIONS OF CHALCOGENIDES IN ENERGY STORAGE AND CONVERSION

3.1. Battery Technologies

Transition metal chalcogenides have emerged as promising materials for battery applications, offering a range of unique properties that address key challenges in energy storage. Among these materials, CdO, CdS, NiO, NiS and other TMCs have garnered significant attention [45]–[48]. One of the key advantages lies in their tunable electronic properties, allowing for the optimization of conductivity and band gaps, crucial for enhancing battery performance. The layered structure of these chalcogenides provides a high surface area, facilitating efficient charge transfer and storage [49]–[51]. Additionally, the chemical stability of many transition metal chalcogenides contributes to the longevity and reliability of batteries. As electrode materials, these compounds show promise in improving energy density, cycling stability, and overall efficiency in various battery technologies [52],[53]. The exploration of transition metal chalcogenides for batteries reflects a concerted effort to overcome limitations in current energy storage systems, paving the way for the development of next-generation batteries with enhanced capabilities and sustainability.

3.2. Supercapacitors

Transition metal chalcogenides, particularly compounds like ZnS, HgS and cadmium based materials, have emerged as promising candidates for supercapacitor applications [54]–[56]. The unique electronic and structural properties of these materials make them well-suited for addressing key challenges in energy storage. The high surface area and conductivity of transition metal chalcogenides contribute to their superior electrochemical performance, enabling efficient charge storage and rapid charge-discharge cycles. Additionally, the tunable band gaps of certain chalcogenides enhance their capacitive behaviour, providing a means to optimize energy storage capacity.

Furthermore, the layered structures and two-dimensional nature of many transition metal chalcogenides facilitate ion diffusion, a critical factor for achieving high power density in supercapacitors. The ability to tailor the composition and morphology of these materials allows researchers to optimize their electrochemical properties for specific supercapacitor applications. The transition metal chalcogenides also exhibit excellent stability over numerous charge-discharge cycles, addressing one of the key challenges in supercapacitor longevity.

The environmental sustainability of certain chalcogenides adds an additional advantage, aligning with the increasing demand for green and sustainable energy technologies. Research in this area focuses not only on synthesizing transition metal chalcogenides with enhanced electrochemical properties but also on exploring scalable and cost-effective manufacturing processes to facilitate their integration into practical supercapacitor devices.

3.3. Solar Cells

Transition metal chalcogenides have emerged as promising materials for solar cell applications, showcasing unique properties that make them attractive for harnessing solar energy. Among these, ZnS, NiO, HgS and WO_3, etc has garnered significant attention [57]–[61]. One of the key advantages lies in their tunable band gaps, allowing for efficient absorption of sunlight in the visible and sometimes even near-infrared regions of the spectrum. This property is crucial for enhancing the efficiency of solar cells. Additionally, transition metal chalcogenides often exhibit excellent charge carrier mobility, facilitating the efficient transport of electrons and holes within the material [62]–[64]. The layered structures of these materials also offer the possibility of creating thin films or two-dimensional heterostructures, enabling the design of lightweight and flexible solar cell devices. Furthermore, the chemical stability of certain chalcogenides contributes to the long-term durability of solar cells, a critical factor for practical applications. Researchers are actively exploring novel synthesis methods and engineering approaches to optimize the performance of transition metal chalcogenides in solar cells, aiming to contribute to the development of cost-effective and high-efficiency photovoltaic technologies. As solar energy continues to play a central role in the pursuit of sustainable energy sources, transition metal chalcogenides stand as promising candidates to drive advancements in solar cell technology (figure-1).
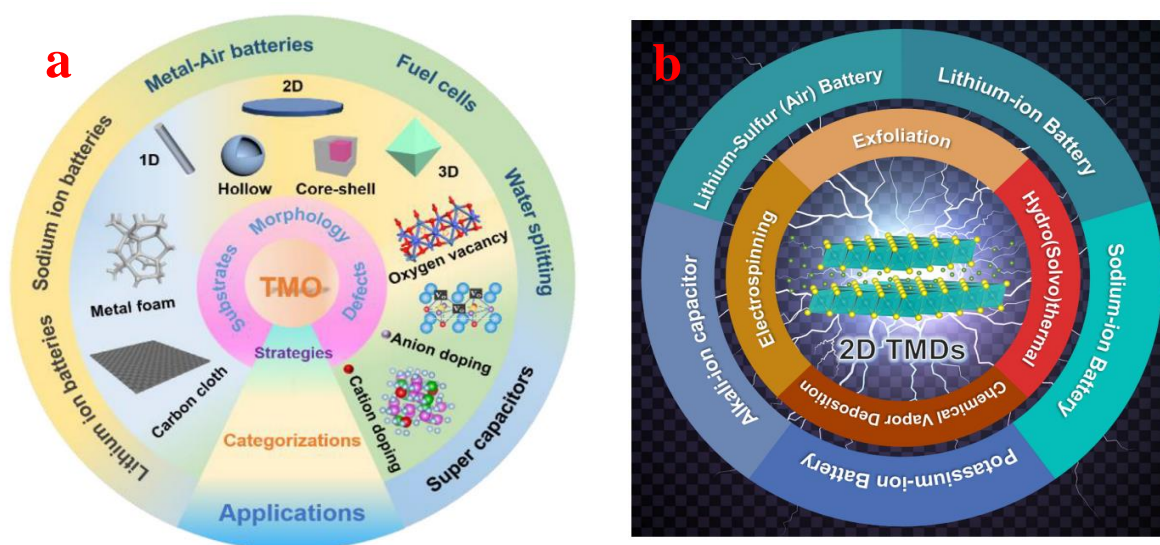
Figure-1. (a) Modification strategies, corresponding means and electrochemical applications of TMOs reproduced with permission from ref [2] copyright © 2021 Elsevier B.V., (b) Synthesis strategies of TMDs and their applications in batteries and emerging batteries reproduced with permission from ref [9] copyright © 1999-2023 John Wiley & Sons, Inc.

4. CURRENT NEED OF ENERGY MATERIALS

In the realm of energy materials, several challenges persist that hinder the seamless integration of novel technologies into practical applications. The following are key current challenges in energy materials:

1.1. **Energy Storage Capacity:** One major challenge is the limited energy storage capacity of existing materials used in batteries and supercapacitors. Enhancing the energy density while maintaining stability is essential for extending the capabilities of energy storage devices.

1.2. **Charge/Discharge Efficiency:** The efficiency of charge and discharge processes in energy storage systems remains a critical issue. Improving the kinetics of these processes is essential to reduce energy losses and enhance overall performance.

1.3. **Cycling Stability:** Many energy materials face degradation over repeated charge-discharge cycles, impacting the longevity and reliability of energy storage devices. Developing materials with enhanced cycling stability is vital for prolonged and sustainable use.

1.4. **Cost-effectiveness:** The high cost of materials and fabrication processes poses a significant obstacle to the widespread adoption of energy technologies. Addressing cost-effectiveness without compromising performance is a key challenge in the field.

1.5. **Environmental Impact:** The environmental impact of energy materials, including extraction, production, and disposal, needs careful consideration. Developing sustainable and eco-friendly materials and manufacturing processes is critical for minimizing the environmental footprint of energy technologies.

1.6. **Materials Abundance:** Some energy materials rely on scarce or geopolitically sensitive elements. Finding alternatives or developing technologies that use more abundant elements is important for ensuring a stable and sustainable supply chain.

1.7. **Integration with Renewable Sources:** Efficiently integrating energy storage materials with renewable energy sources, such as solar and wind, remains a challenge. Matching the intermittent nature of renewable energy production with reliable and scalable energy storage solutions is crucial for the widespread adoption of clean energy.

1.8. **Heat Dissipation:** Thermal management is a significant challenge, especially in high-performance energy devices. Ensuring efficient heat dissipation is vital for maintaining the stability and longevity of materials used in energy applications.

Addressing these challenges requires a multidisciplinary approach, combining materials science, chemistry, physics, and engineering to develop innovative solutions. **The synthesis and characterization of chalcogenide nanocomposites, as proposed in the review, offer a promising avenue to tackle some of these challenges and contribute to the advancement of energy materials. The primary objective of this review is to synthesize and systematically characterize chalcogenide nanocomposites with the aim of advancing their application in the field of energy materials.** Through a comprehensive investigation of synthesis methods and detailed characterization techniques, this research endeavours to contribute to the development of high-performance materials for energy storage and conversion applications. By addressing key challenges and exploring the unique properties of chalcogenide nanocomposites, the review seeks to provide valuable insights that can potentially enhance the efficiency, durability, and sustainability of energy-related technologies.

5. SCOPE OF THE PRESENT RESEARCH

The present research explores the synthesis and characterization of chalcogenide nanocomposites with a specific focus on their application in energy materials. The scope encompasses a broad spectrum of chalcogenides and nanocomposites, targeting their unique properties for diverse energy-related applications.

5.1. Types of Chalcogenides

The present study investigates a range of chalcogenides, including but not limited to oxides and sulphides, with future prospects extending to selenides and tellurides materials. The review discusses energy materials such as ZnO, ZnS, CdO, CdS, NiO, MoO_3 and HgS.

5.1.1. Oxides

Oxides are compounds that contain oxygen and another element. They are among the most prevalent types of chalcogenides due to oxygen's abundance in the Earth's crust and its high reactivity. Oxides can be classified based on the nature of the other element they are combined with. Metal oxides, such as magnesium oxide (MgO) and aluminum oxide (Al_2O_3), are typically ionic compounds with high melting points and are often used in ceramics, refractories, and as catalysts. Non-metal oxides, like carbon dioxide (CO_2) and sulfur dioxide (SO_2), are covalent compounds with significant roles in environmental chemistry, such as in the greenhouse effect and acid rain formation. Oxides can also be amphoteric, like aluminum oxide, showing both acidic and basic properties depending on the reacting substances.

5.1.2. Sulfides

Sulfides are compounds that include sulfur and another element. They are prominently found in minerals and play crucial roles in various industrial processes. Metal sulfides, such as iron sulfide (FeS_2) and zinc sulfide (ZnS), are key components in ore processing and metallurgy. Iron sulfide, also known as pyrite, is used in the production of sulfuric acid, while zinc sulfide is essential in phosphorescent applications. Non-metal sulfides, such as hydrogen sulfide (H_2S), are characterized by their distinct, often unpleasant odor and are important in biological systems and industrial processes. Hydrogen sulfide is produced naturally by decaying organic matter and is used in the manufacture of chemicals.

5.1.3. Selenides

Selenides are compounds composed of selenium and another element. While less common than oxides and sulfides, selenides are significant in various technological applications. Metal selenides, such as copper selenide (Cu_2Se) and cadmium selenide (CdSe), are notable for their use in semiconductors, photovoltaic cells, and as pigments in glass and ceramics. Non-metal selenides like hydrogen selenide (H_2Se) are less frequently encountered but have important uses in chemical synthesis and research. The unique properties of selenides, such as their electrical conductivity and photoresponsive behavior, make them valuable in advanced material science.

5.1.4. Tellurides

Tellurides are compounds containing tellurium and another element. These compounds are especially relevant in the fields of electronics and materials science due to their semiconducting properties. Metal tellurides, such as bismuth telluride (Bi_2Te_3) and lead telluride (PbTe), are extensively used in thermoelectric devices, which convert temperature differences into electric voltage and vice versa. These materials are crucial for applications in power generation and refrigeration. Non-metal tellurides like hydrogen telluride (H_2Te) are rarer and typically studied for their chemical properties and potential uses in synthesis.

5.1.5. Polychalcogenides

Polychalcogenides are compounds that contain multiple chalcogen elements within the same molecule. These compounds exhibit diverse structures and properties. Metal polychalcogenides, such as sodium polysulfide (Na_2S_4) and potassium polyselenide (K_2Se_4), are known for their unique electronic and magnetic properties, making them interesting for research in advanced materials and battery technology.

Non-metal polychalcogenides like sulfur dioxide diselenium (SSe₂) combine different chalcogen atoms and find use in specialized chemical applications, including as reagents in synthetic chemistry.

5.1.6. Chalcogenide Glasses

Chalcogenide glasses are amorphous solids made from chalcogen elements, often combined with other elements like arsenic, germanium, or gallium. These materials are distinguished by their unique optical and electrical properties, making them highly valuable in the field of photonics and infrared optics. Binary chalcogenide glasses, such as arsenic selenide (As_2Se_3), and ternary chalcogenide glasses, like gallium lanthanum sulfide ($Ga_2La_2S_3$), are used in the production of optical fibers, lenses, and infrared sensors. Their ability to transmit infrared light and their non-crystalline structure provide advantages in specific technological applications, including telecommunications and thermal imaging.

5.1.7. Mixed Chalcogenides

Mixed chalcogenides contain two or more different chalcogen elements combined with other elements, creating compounds with tailored properties for various applications. Sulfoselenides, such as cadmium sulfoselenide (CdS_xSe_{1-x}), are used in optoelectronics and as pigments due to their tunable bandgap and vibrant colors. Tellurosulfides, such as tellurium sulfide (TeS_2), are studied for their potential in photovoltaic and thermoelectric applications. These mixed chalcogenides often exhibit unique electronic, optical, and chemical properties that make them suitable for advanced technological applications.

5.1.8. Organic Chalcogenides

Organic chalcogenides are compounds where chalcogen elements are bonded to carbon within organic molecules. These compounds play significant roles in biological systems and industrial applications. Thiols, like methanethiol (CH_3SH), are characterized by their strong odors and are important in biochemistry, particularly in the structure and function of proteins. Selenols, such as benzeneselenol (C_6H_5SeH), have applications in organic synthesis and pharmaceuticals due to their unique reactivity and biological activity. Organic chalcogenides often exhibit interesting chemical behavior, making them valuable in both scientific research and practical applications.

6. LITERATURE SURVEY

6.1. Chalcogenide Nanocomposites

TMCs, primarily oxides, sulphides, and selenides, have received increasing research interest as potential electrode materials for energy storage and conversion over the past decades due to their tunable stoichiometric compositions, unique crystal structures, and rich redox sites, as well as relatively higher electrical conductivity in comparison to their transition metal oxide counterparts [109]–[111]. TMCs, for example, have greater theoretical special capacity than standard anode materials (graphite) in lithium ion batteries (LIBs) based on the insertion/de-insertion process [110], [112], which can be mainly attributed to the conversional mechanism that can be described as [113]–[115]

$$MS(Se)_n + 2nLi^+ + 2ne^- \leftrightarrow nLi_2S(Se) + M$$
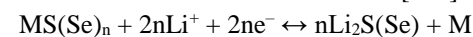
Furthermore, further alloying operations (M = Zn, Ni, Sb, and Bi) or insertion procedures (layered structure, M = Mo, W, and V) may contribute to electrode capacity in some circumstances. When used as polar hosts in lithium-sulphur batteries (LSBs), nanostructured TMCs can provide stronger affinity with soluble polysulphides, resulting in a serious "shuttle effect" due to the polar sulphurphilic surface of TMCs, providing LSBs with higher sulphur utilization and long cycling life [113]–[115]. Benefited from the reversible redox reaction of TMCs in alkaline medium ($MS(Se) + OH^- \leftrightarrow MS(Se)OH + e^-$), supercapacitors with TMC electrodes also demonstrate excellent energy density. In the case of dye-sensitized solar cells (DSSCs), an important type of energy conversion technology, TMCs employed as counter electrodes typically outperform cells with Pt electrodes in terms of energy conversion efficiency.

6.1.1. TMC's for Batteries

In general, the interaction of metal sulphides (e.g., CdS, NiS) with Li ions can contribute significantly to lithium storage. Mahmood et al. [116] reported a hydrothermal method followed by thermal treatement for manufacturing nickel sulphides (Ni3S4 and NiS1.03) sheets composites with good reversibility. To resolve electron/ion transport difficulties in big metal sulphides crystals and remove strain caused by volume variation during cycle, Wu and co-workers revealed zero-dimensional (0D) metal sulfides (CoS, NiS, MnS)/one-dimensional (1D) porous carbon nanowires/three-dimensional (3D) graphene network nanocomposites with novel multiscale, multidimensional, and hierarchically structured architecture [117].

Due to its high theoretical specific capacity (1675 mAh/g) and theoretical energy density (2600 Wh/kg), rechargeable Li-S batteries have drawn unprecedented interest in order to address the need for high energy/power density batteries in energy markets [118], [119]. Furthermore, elemental S is low cost, earth rich, and environmentally benign, making Li-S batteries more suitable for high-end applications [120]–[122]. In general, a typical Li-S battery is made up of four major components: Organic electrolyte, separator, and Li metal anode with S/C composite cathode. Because of their high theoretical specific energy (3500 Wh/kg), lithium-air (Li-air or Li-O₂) batteries have received remarkable interest in recent decades [123], [124]. A typical Li-O₂ cell consists of a Li metal anode, a lithium salt electrolyte, and a porous cathode [125], [126]. The electrochemical process of Li-O₂ is, in theory, dependent on the battery operating patterns. During discharging, Li metal is oxidized: $Li = Li^+ + e^-$; whereas the cathode undergoes oxidation reduction reaction with electro-catalysts: $O_2 + 2Li^+ + 2e^- = Li_2O_2$. During charging, Li ion

deposition occurs at the Li anode surface: $Li^+ + e^- = Li$, whereas the OER comes from the porous cathode: $Li_2O_2 = O_2 + 2Li+ + 2e^-$ [127]. Although high energy/power density is appealing, some unresolved issues of $Li-O_2$ batteries remain, particularly the sluggish electrochemical reaction kinetics for ORR and OER, as well as the subsequent passivation and block for the porous electrode surface caused by insulating Li_2O_2 products. This often results in low round-trip efficiency, a short cycle life, and voltage degradation [128], [129].

6.1.2. TMC's for Supercapacitors

Supercapacitors (electrochemical capacitors) are another key form of advanced energy storage technology that has received a lot of interest due to its high power density and long cycle life [130]–[132].

There are primarily three types of supercapacitors shown in figure-2:

Electrostatic Double-Layer Capacitors (EDLCs): EDLCs, often referred to as electric double-layer capacitors, operate based on the electrostatic charge separation at the interface between a high-surface-area electrode and an electrolyte. The energy storage mechanism is primarily electrostatic, and ions in the electrolyte are adsorbed onto the electrode surface. Carbon-based materials, such as activated carbon, are commonly used for their high specific surface area in EDLCs.

These capacitors are known for their high power density, rapid charge and discharge rates, and long cycle life.

Pseudocapacitors: Pseudocapacitors store energy through a redox (reduction-oxidation) reaction at the electrode-electrolyte interface. Unlike EDLCs, pseudocapacitors involve a reversible faradaic process, where charge is stored through fast and reversible redox reactions. Transition metal oxides, conducting polymers, and other materials with high specific capacitance are commonly used as electrodes in pseudocapacitors. Pseudocapacitors offer higher energy density compared to EDLCs, making them suitable for applications where a balance between energy and power density is essential.

Hybrid Supercapacitors: Hybrid supercapacitors combine the features of both EDLCs and pseudocapacitors to achieve an optimal balance between high energy density and high power density. These devices typically consist of an EDLC electrode and a pseudocapacitor electrode, allowing for a synergistic combination of the advantages of both types. By integrating the strengths of electrostatic and faradaic charge storage mechanisms, hybrid supercapacitors aim to offer improved overall performance. Common configurations include combining a carbon-based electrode with a metal oxide or conducting polymer electrode.
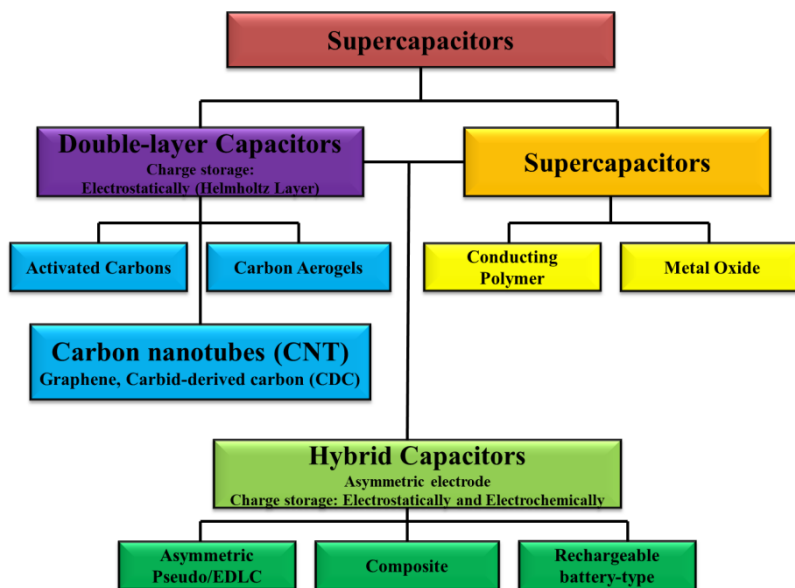

Figure-2. Types of supercapacitors and materials used for their particular applications.

Because of their higher redox reactivity than metal oxide equivalents, metal sulphides, particularly cobalt sulphides, nickel oxide, and nickel sulphides, are projected to match the criteria for supercapacitors [130], [133]. Nonetheless, the relative poor electrical conductivity of these materials has hampered efforts to improve supercapacitor performance [134]. Qu et al. created a cobalt sulphide (CoS1.097) nanoparticle-decorated conductive graphene nanocomposite with a superior specific capacitance of 1535 F/g at a current density of 2.0 A/g and a high capacitance of 725 F/g at an extremely high current density of 40 A/g, corresponding to a high power density of 11.98 kW/kg [134]. Wang et al. created a homogenous dispersed NiS nanoparticles supported on the GO film, which demonstrated a high specific capacitance of 800 F/g at 1.0 A/g [135]. The hybrids revealed increased electron transport and capacitance by the incorporation of conductive rGO into sulphides. However, the electrical conductivity of rGO is limited due to a lack of conjugate

electrons on the rGO panel. Yan et al. effectively produced NiS supported rGO and single-walled CNT (SWCNT) nano hybrids using a one-step solvothermal technique [136]. They demonstrated that the typically improved electrochemical performance of NiS/SWCNTs over NiS/rGO was attributed to the higher electrical conductivity of SWCNTs and its more effective inhibition of NiS aggregation. The rate of electrolyte absorption into electrode materials can also have an effect on the electrochemical performance of the electrode. Because of their higher redox activity than comparable single metal sulphides, mixed metal sulphides, particularly ternary nickel cobalt sulphides, have a high potential to improve the electrochemical performance of supercapacitors [137]. Nonetheless, the comparatively poor conductivity is an impediment to future activity development. Peng et al. originally reported ultrathin $NiCo_2S_4$ nanosheets anchored on rGO sheets as supercapacitor electrode materials with greater specific capacitance, better rate performance, and longer cycle life than bare $NiCo_2S_4$ [138]. Even with a simple physical mixing, the inclusion of graphene (as little as 5 wt%) in $CoNi_2S_4$/graphene composite resulted in a remarkable increase in specific capacitance [139]. Yang et al. recently used an in situ anion exchange technique to create edge site-enriched nickel-cobalt sulphide (Ni-Co-S) nanoparticles loaded on graphene frameworks [140]. They believed that the etching-like activity caused by the S^{2-} ions was the key contributor to the presence of sufficient edge active sites on Ni-Co-S nanoparticles; moreover, the edge sites were confirmed to allow robust interaction with OH^-. As a result, the synergistic impact of edge sites and graphene significantly aided electrochemical reaction kinetics, yielding a high specific capacitance of 1492 F/g at 1.0 A/g current density and an even higher rate performance of 96% at 50 A/g. Furthermore, Zn0.76Co0.24S nanosheets modified nitrogen-doped graphene/carbon nanotube film [141] and CuCo2S4 attached onto nitrogen-doped rGO nanosheets composites displayed good electrochemical performance in supercapacitors.

6.1.3. TMC's for Solar Cells

Researchers have been exploring the use of TMD heterostructures and composites to improve the efficiency and stability of solar cells. Combining different TMDs with complementary properties or integrating TMDs with other materials can lead to improved light absorption, charge separation, and overall device performance. Jiang et al. demonstrated that the $ZnO@MoS_2$ heterostructure, modified to have a controllable photoelectric structure, exhibits outstanding photoelectric characteristics. These features make it highly suitable for applications in nonlinear optics, photoelectric functional devices, energy environments, and various other fields [142]. In a separate study conducted by Maruthasalamoorthy et al., the composite heterostructure of La-supported $Bi_2S_3@g\text{-}C_3N_4$/rGO demonstrated enhanced cyclic stability and superior electrochemical performance [143].

Researchers explore integrating TMDs with perovskites in solar cells to boost stability and performance. TMDs serve as versatile layers, addressing hysteresis effects and enhancing device efficiency. They are also investigated in tandem solar cells, aiming for higher overall efficiencies by combining with materials like silicon or perovskites. In tandem architectures, TMDs act as intermediate absorbers or charge transport layers, improving solar spectrum utilization. Studies report the preparation of TMD/perovskite composites for enhanced properties. For instance, Sathiyamoorthy et. al. has synthesized $LaCoO_3/MoS_2$ hybrid nanostructures showcasing outstanding photodegradation performance against Alizarin Red S (ARS) and Rhodamine B (RhB). This suggests promising potential for their utilization as effective photocatalysts in environmental remediation applications [144]. A numerical analysis of a double perovskite solar cell (PSC) model featuring an environmentally friendly $Cs_2AgBiBr_6$ compound has been reported by Jaiswal et. al [145]. The cell follows a standard n-i-p architecture with the structure $FTO/C_{60}/Cs_2AgBiBr_6/MoS_2/Pt$ and achieved PCE of 23.49%.

TMC's, such as molybdenum disulfide (MoS_2) and tungsten diselenide (WSe_2), are notable 2D materials with unique electronic properties [38], [63], [146]. Their preparation methods, elements and structures, electronics and optoelectronics are briefly discussed in fig. 3. TMCs exhibit a direct bandgap at the monolayer level, facilitating efficient light absorption and emission as discussed by Zheng et. al. [147]. The exploration of TMCs with energy bandgaps spanning the visible and near-infrared spectral range has been a subject of discussion. These materials offer significant benefits in a range of photonic and optoelectronic applications, including but not limited to light-emitting devices, transistors, photovoltaic devices, photodetectors, and nanocavity lasers. An essential aspect lies in comprehending the mechanisms and effectively controlling the optical properties of 2D TMDs, as this knowledge is pivotal for advancing their applications in integrated photonics.
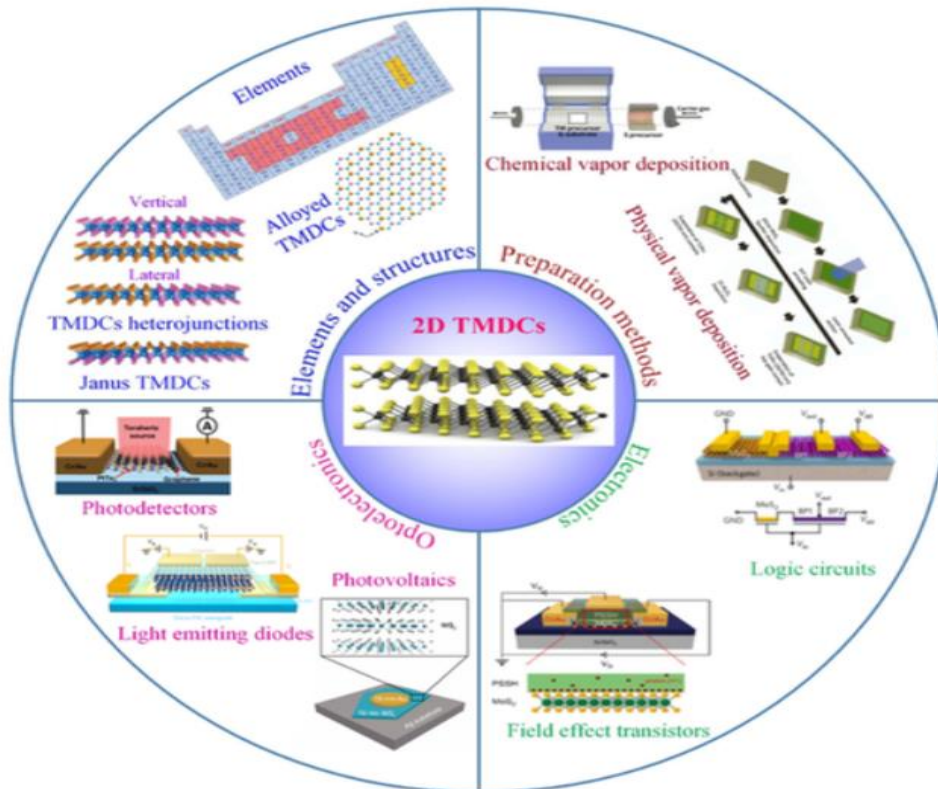
Figure-3 Brief details of 2D TMDs including preparation methods, structure and properties reproduced with permission from ref [148] copyright © 1999-2023 John Wiley & Sons, Inc.

7. DIFFERENT TECHNIQUE FOR SYNTHESIS OF TMD'S

Synthesizing transition metal chalcogenides involves a variety of methods to tailor their structure, morphology, and properties for specific applications. Some of them are hot-plate method, one-pot heat-up method, hydrothermal or solvothermal process, electrospinning, chemical vapour deposition (CVD), sol-gel method, microwave-assisted synthesis, template-assisted synthesis, chemical precipitation and mechanical alloying as shown in figure-4.
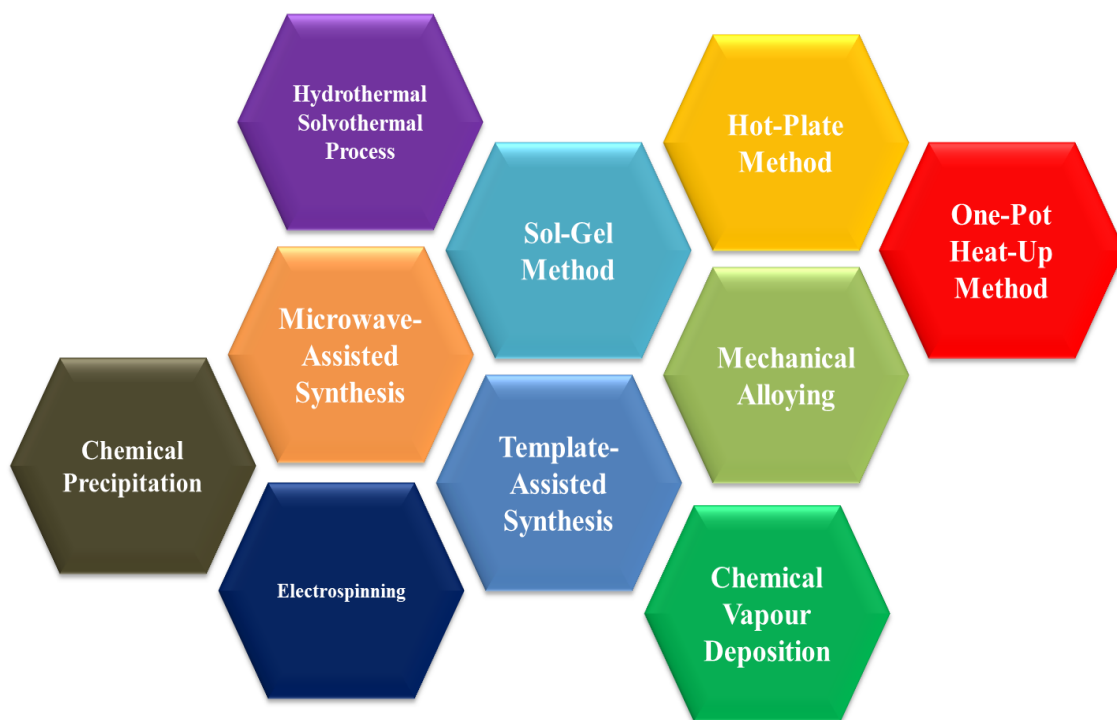
Figure-4 Different Technique for Synthesis of TMD's

7.1. Hot-Plate Method:

The hot-plate method is a relatively simple and commonly used technique for the synthesis of transition metal chalcogenides. This method is suitable for producing thin films or coatings of chalcogenide materials and involves the heating of precursor materials on a hot plate to induce chemical reactions leading to the formation of the desired compound [149], [150]. Here is a general description of the hot-plate method for synthesizing transition metal chalcogenides.

Procedure:

Precursor Preparation:

Begin by preparing the precursor materials. These typically include transition metal salts and chalcogenide sources, such as metal chloride or nitrate salts and sulfur or selenium sources.

Precursor Mixing:

Combine the transition metal salt and chalcogenide source in the desired stoichiometric ratio. The mixture can be dissolved in a suitable solvent, depending on the solubility of the precursors shown in figure-5.

Solution Deposition:

Deposit the precursor solution onto a substrate. The choice of substrate can influence the properties of the final chalcogenide material. Common substrates include Glass, silicon, or other materials compatible with the synthesis conditions.

Evaporation and Drying:

Place the substrate with the precursor solution on a hot plate and evaporate the solvent by applying heat. The hot plate provides a controlled and uniform temperature for the drying process.

Thermal Treatment:

After the solvent has evaporated, continue heating the substrate to a higher temperature to initiate the chemical reactions between the metal and chalcogenide precursors. This thermal treatment leads to the formation of the desired transition metal chalcogenide.

Cooling and Collection:

Allow the synthesized material to cool to room temperature. The transition metal chalcogenide, now in the form of a thin film or coating, can be collected from the substrate.

Advantages:

- The hot-plate method is relatively straightforward and does not require complex equipment.
- It is suitable for producing thin films, making it applicable to applications like sensors, optoelectronics, and catalysis.

Limitations:

- The method may not be suitable for large-scale production.
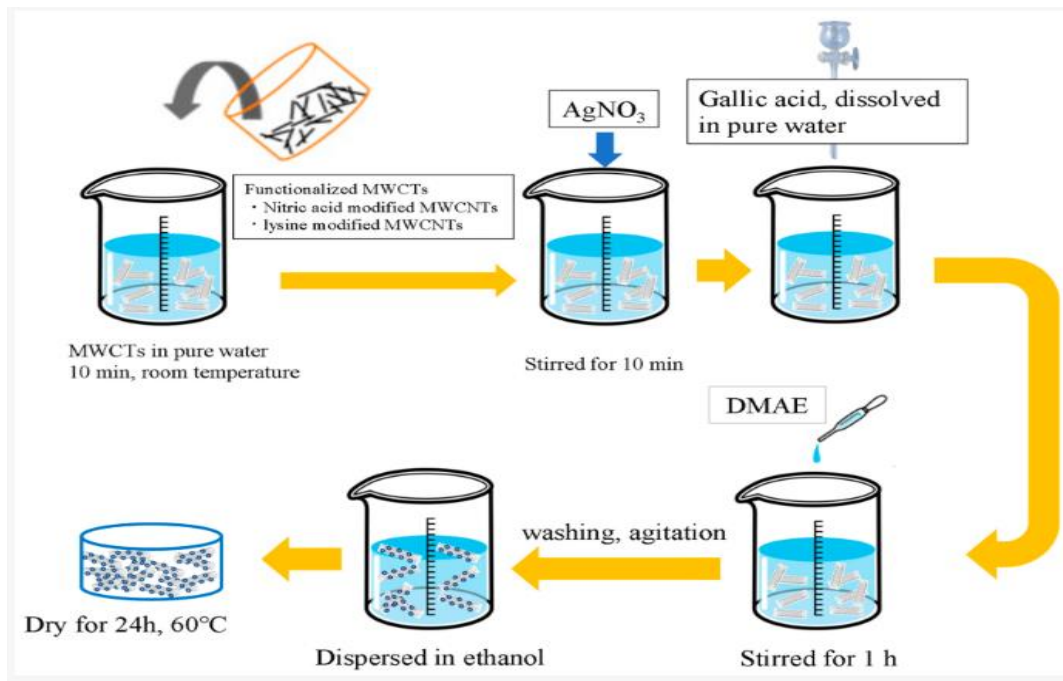- Control over the thickness and uniformity of the films may be challenging.

Figure-5 Stepwise process of hot plate method for TMC's synthesis reproduced with permission from ref [151] copyright © 1996-2024 MDPI (Basel, Switzerland).

7.2. One-Pot Heat-Up Method:

The One-Pot Heat-Up Method is a convenient and efficient approach for the synthesis of transition metal chalcogenides. In this method, all the precursor materials required for the formation of the chalcogenide compound are combined in a single reaction vessel or pot. The reaction mixture is then subjected to controlled heating, typically in a furnace or oven, initiating the chemical reactions that lead to the synthesis of the desired transition metal chalcogenide [152][152].

The key steps of the One-Pot Heat-Up Method are as follows:

Precursor Preparation:

All necessary precursor materials, which typically include transition metal salts and chalcogenide sources, are accurately weighed and mixed in the desired stoichiometric ratios. The choice of precursors depends on the specific chalcogenide compound targeted for synthesis.

Formation of Homogeneous Mixture:

The precursor mixture is thoroughly mixed to ensure homogeneity. The homogeneity of the mixture is crucial for achieving uniform distribution of elements in the resulting chalcogenide product.

Transfer to Reaction Vessel:

The homogeneous precursor mixture is then transferred to a reaction vessel or pot. This vessel is designed to withstand the elevated temperatures required for the synthesis process.

Controlled Heating:

The reaction vessel containing the precursor mixture is placed in a furnace or oven. The temperature is gradually increased, and the mixture is heated under controlled conditions. The heating profile is crucial to control the kinetics of the chemical reactions and the formation of the chalcogenide phase.

Chemical Reactions and Formation:

As the temperature rises, the precursor materials undergo chemical reactions leading to the formation of the desired transition metal chalcogenide compound. These reactions involve the reduction of metal ions and the incorporation of chalcogen atoms into the crystal lattice.

Cooling and Product Recovery:

Once the synthesis is complete, the reaction vessel is allowed to cool to room temperature. The resulting product, now in the form of powders or bulk material, is recovered from the vessel.

The One-Pot Heat-Up Method offers several advantages, including simplicity, cost-effectiveness, and the ability to scale up for bulk production. It minimizes the number of reaction steps and vessels required, reducing the complexity of the synthesis process. Additionally, this method is suitable for producing a wide range of transition metal chalcogenides with varying compositions and properties.

While the One-Pot Heat-Up Method is versatile, researchers must carefully optimize the reaction conditions, including temperature, reaction time, and precursor ratios, to achieve the desired phase purity, crystallinity, and morphology of the synthesized transition metal chalcogenides shown in figure-6.
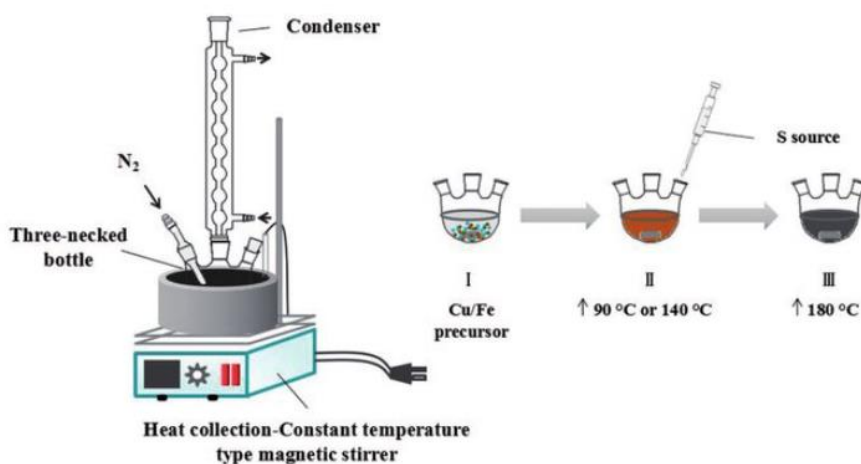

Figure-6 Depiction of one-pot heat up method for TMC's synthesis reproduced with permission from ref [153] Copyright © 2024 Oxford University Press

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7.3. Hydrothermal or Solvothermal Process:

The hydrothermal and solvothermal processes are powerful techniques employed for the synthesis of transition metal chalcogenides, providing control over the composition, morphology, and properties of the resulting materials. Both methods involve the use of high-temperature and high-pressure conditions to facilitate the chemical reactions leading to the formation of chalcogenide nanomaterials [26], [27], [154].

Hydrothermal Process: In the hydrothermal process, the synthesis takes place in an aqueous environment under elevated temperature and pressure. The precursor materials,

typically metal salts and chalcogenide sources, are dissolved in a water-based solution represented in figure-7. The reaction vessel is then sealed and heated above the boiling point of water, creating a high-pressure environment. The elevated pressure allows the reaction to proceed at temperatures higher than the normal boiling point of water [38], [56], [63], [64], [146].

Key steps in the hydrothermal process include:

Precursor Solution Preparation:
Dissolving metal salts and chalcogenide sources in an aqueous solution to form a precursor solution.

Sealing:
The reaction vessel is sealed to create a closed system.

Heating:
Elevating the temperature to initiate the chemical reactions within the precursor solution.

Cooling and Product Recovery:
Allowing the system to cool down after the reaction is complete, followed by recovering the chalcogenide nanomaterials.

Solvothermal Process: The solvothermal process is similar to the hydrothermal process but employs organic solvents instead of water. This method is particularly useful for synthesizing chalcogenide nanomaterials that may not be stable in water or for achieving specific morphologies that are challenging to obtain in aqueous systems [80].

Key steps in the solvothermal process include:

Precursor Solution in Organic Solvent:
Dissolving metal salts and chalcogenide sources in an organic solvent to create a precursor solution.

Sealing:
The reaction vessel is sealed to create a closed system.

Heating:
Elevating the temperature to initiate the chemical reactions within the precursor solution.

Cooling and Product Recovery:
Allowing the system to cool down after the reaction is complete, followed by recovering the chalcogenide nanomaterials.

Both hydrothermal and solvothermal processes offer advantages such as precise control over particle size, morphology, and crystallinity. The high pressure and temperature conditions promote the formation of crystalline phases and often result in nanomaterials with enhanced properties. These methods are widely employed in the synthesis of transition metal chalcogenides for various applications, including catalysis, optoelectronics, and energy storage devices. Researchers often tailor reaction parameters such as temperature, pressure, and precursor concentrations to achieve the desired properties in the synthesized chalcogenide nanomaterials.
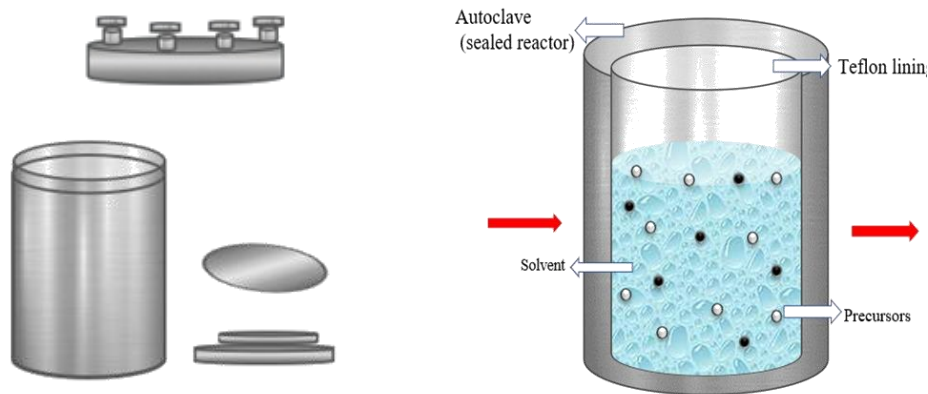
Figure-7 Hydrothermal/Solvothermal route of synthesis of TMC's reproduced with permission from ref [146] copyright © 2023 Springer Nature.

7.4. Electrospinning:

Electrospinning is a versatile and effective technique used in the synthesis of transition metal chalcogenides with controlled morphologies, high surface areas, and enhanced properties. This method is particularly advantageous for producing nanofiber-based structures, providing a unique platform for applications in energy storage, catalysis, and sensors [155], [156].

Process:

Polymer Solution Preparation:
The process begins by preparing a polymer solution that includes the desired transition metal and chalcogenide precursors. The polymer acts as a carrier and support matrix for the formation of nanofibers.

Electrospinning Setup:
The polymer solution is loaded into a syringe connected to a metallic needle, forming a Taylor cone at the needle's tip. A high voltage is applied to

induce charge repulsion, overcoming the surface tension of the solution and initiating the electrospinning process.

Formation of Nanofibers:

As the electric field intensifies, a jet of the polymer solution is ejected from the needle, forming a fine stream. The solvent evaporates during flight, leaving behind solid nanofibers that are collected on a grounded substrate or a rotating drum shown in figure-8.

Heat Treatment:

The collected nanofibers are subjected to heat treatment, typically through calcination or annealing, to remove the polymer matrix and facilitate the conversion of precursor materials into transition metal chalcogenides. This step is crucial for achieving the desired composition and crystal structure.

Advantages:

- **Controlled Morphology:** Electrospinning allows precise control over the morphology of the synthesized transition metal chalcogenides, producing nanofibers with high aspect ratios.
- **High Surface Area:** The nanofibrous structure enhances the specific surface area of the material, crucial for applications such as catalysis and energy storage.
- **Versatility:** The electrospinning technique can be adapted for various transition metal chalcogenide compositions, providing versatility in material design.
- **Scalability:** The process is amenable to scaling up, making it suitable for both laboratory-scale research and potential industrial applications.
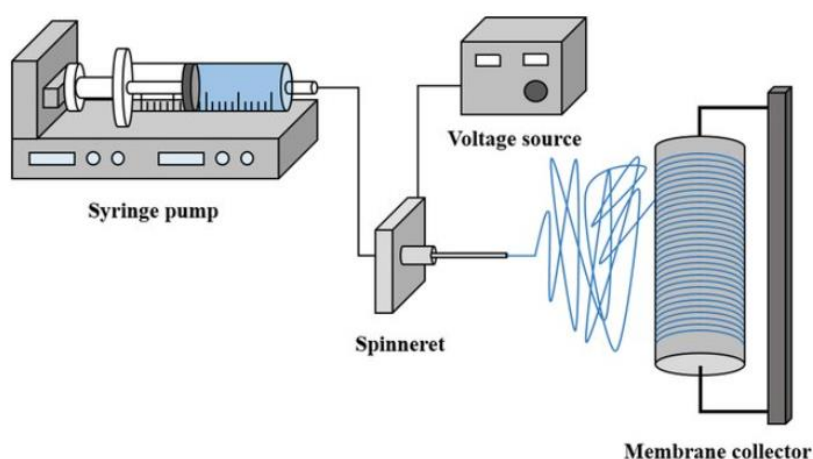

Figure-8 Stepwise process electrospinning setup for TMC's synthesis reproduced with permission from ref [157] copyright © 1996-2024 MDPI (Basel, Switzerland).

7.5. Chemical Vapor Deposition (CVD):

Chemical Vapour Deposition (CVD) is a widely used technique for the synthesis of transition metal chalcogenides, offering precise control over film thickness, uniformity, and crystal structure. The process involves the chemical reaction of volatile precursors in the vapour phase, leading to the deposition of chalcogenide materials onto a substrate illustrated in figure-9 [21]. In the context of transition metal chalcogenides, such as sulphides, selenides, and tellurides, CVD has proven to be a versatile and effective method for fabricating thin films and coatings with tailored properties.

Key Steps in CVD Process:

Precursor Selection:

Volatile precursors containing transition metal and chalcogen elements are carefully chosen to facilitate controlled deposition. These precursors can be in the form of metalorganic compounds, metal halides, or other volatile compounds.

Precursor Delivery:

The selected precursors are typically delivered into a reaction chamber as gases or vapors. Various methods, such as bubbling through a carrier gas or using liquid precursors in a vaporizing system, can be employed to introduce the precursors.

Reactant Activation:

The precursors undergo thermal or plasma activation to break chemical bonds and generate reactive species. This activation is critical for initiating the chemical reactions leading to the formation of chalcogenide compounds.

Chemical Reaction:

The reactive species generated in the activation step react with each other or with the substrate surface to form transition metal chalcogenides. The choice of reaction conditions, including temperature, pressure, and gas composition, influences the composition and structure of the deposited material.

Deposition onto Substrate:

The synthesized chalcogenide compounds are deposited onto a substrate placed within the reaction chamber. The substrate may be heated to promote adhesion and enhance the crystallinity of the deposited film.

Film Growth and Nucleation:

The chalcogenide material begins to nucleate and grow on the substrate surface as a thin film. The growth rate, morphology, and orientation of the film can be controlled by adjusting the deposition parameters.

Advantages of CVD for Transition Metal Chalcogenides:

Controlled Deposition:

CVD enables precise control over the deposition process, allowing for the production of uniform and well-defined thin films.

High Purity:

The use of high-purity precursors and controlled reaction conditions contributes to the formation of chalcogenide films with minimal impurities.

Scalability:

CVD is amenable to large-scale production, making it suitable for industrial applications in semiconductor device fabrication and other technology sectors.

Tailored Properties:

By adjusting the deposition parameters, researchers can tailor the properties of the synthesized chalcogenide films, including thickness, composition, and crystal structure.
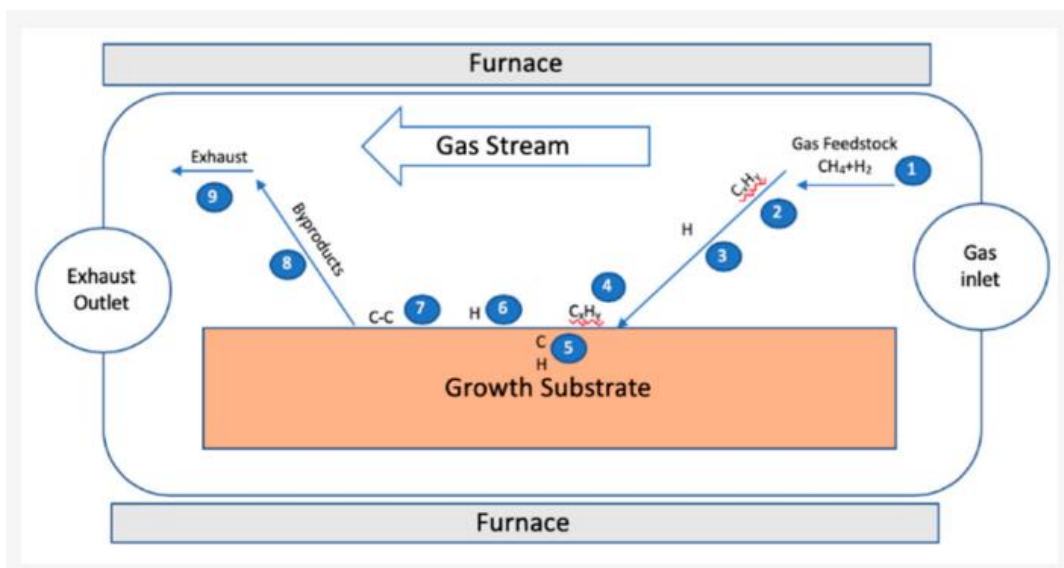

Figure-9 Chemical vapour deposition setup for TMC's synthesis reproduced with permission from ref [158] copyright © 1996-2024 MDPI (Basel, Switzerland).

7.6. Sol-Gel Method:

The sol-gel method is a versatile and widely used technique for the synthesis of transition metal chalcogenides, offering control over the composition, structure, and properties of the resulting materials. The method involves the transformation of a precursor sol or gel into the desired chalcogenide material through a series of chemical reactions and subsequent thermal treatment [159]–[161]. Here is a step-by-step description of the sol-gel method for synthesizing transition metal chalcogenides:

Precursor Preparation:

Metal and chalcogenide precursors, typically in the form of metal salts and chalcogenide sources such as alkoxides, are dissolved in a suitable solvent to form a precursor solution. This solution is often referred to as the sol.

Gel Formation:

The precursor sol undergoes a gelation process to form a three-dimensional network, known as the gel. Gelation can occur through various mechanisms, such as hydrolysis and condensation reactions. The gel structure is crucial as it determines the final morphology and porosity of the chalcogenide material.

Aging:

The gel is allowed to age for a specific duration. During this period, the precursor molecules continue to react, leading to the formation of a more stable and well-defined gel structure. Aging is critical for achieving homogeneity and controlling the size of the nanoparticles formed during subsequent thermal treatment.

Drying:

The gel is subjected to a drying process to remove the solvent and water content, leaving behind a porous matrix. Drying can be achieved through various methods, such as air-drying, freeze-drying, or supercritical fluid drying.

Thermal Treatment (Calcination):

The dried gel undergoes thermal treatment, typically referred to as calcination, in a controlled atmosphere. During calcination, the organic components of the gel are

decomposed and removed, and the remaining inorganic components undergo further reactions to form the desired transition metal chalcogenide phase.

The sol-gel method offers several advantages, including the ability to produce homogeneous materials with controlled porosity and particle size. It is applicable to various transition metals and chalcogenides, allowing for the synthesis of a wide range of materials with tailored properties. Additionally, the method is versatile and scalable, making it suitable for both laboratory-scale research and industrial applications in fields such as catalysis, sensing, and energy storage.

7.7. Microwave-Assisted Synthesis:

Microwave-assisted synthesis is a specialized method employed in the preparation of transition metal chalcogenides, offering advantages such as rapid reaction rates, enhanced purity, and controlled morphology. This technique utilizes microwave radiation to heat reaction mixtures, promoting faster and more efficient chemical reactions compared to traditional heating methods represented in figure-10 [146], [162]–[164]. The microwave-assisted synthesis of transition metal chalcogenides typically involves the following key steps:

Precursor Preparation:

Transition metal salts and chalcogenide sources, such as metal precursors and sulfur or selenium-containing compounds, are selected as starting materials. These precursors are dissolved or dispersed in a suitable solvent to form a reaction mixture.

Reaction Vessel Setup:

The reaction mixture is placed in a microwave-transparent reaction vessel. The vessel material should be compatible with microwave radiation to allow efficient energy transfer. The reaction vessel may also contain additional components such as surfactants or capping agents to control the size and morphology of the resulting chalcogenide nanoparticles.

Microwave Irradiation:

The reaction vessel is exposed to microwave radiation. Microwaves penetrate the reaction mixture, leading to efficient and selective heating of the reaction components. The microwave energy is absorbed by polar molecules in the mixture, generating heat rapidly and uniformly throughout the reaction volume.

Reaction Progress Monitoring:

The progress of the reaction is monitored using in-situ techniques, if applicable, to observe the formation of transition metal chalcogenides. Real-time monitoring allows researchers to optimize reaction conditions and control the size, shape, and phase purity of the synthesized materials.

Quenching and Purification:

After the desired reaction time, the reaction is quenched to halt further chemical transformations. The resulting product is then typically subjected to purification steps, such as solvent washing or centrifugation, to remove unreacted precursors, by-products, or impurities.

Advantages of Microwave-Assisted Synthesis:

Rapid Heating: Microwave radiation enables fast and uniform heating, reducing reaction times.

Enhanced Purity: The rapid and localized heating minimizes side reactions, leading to higher purity products.

Controlled Morphology: Microwave synthesis often allows better control over the size and morphology of the resulting chalcogenide nanoparticles.

Green Chemistry: Reduced reaction times and energy consumption contribute to the environmentally friendly aspects of the method.
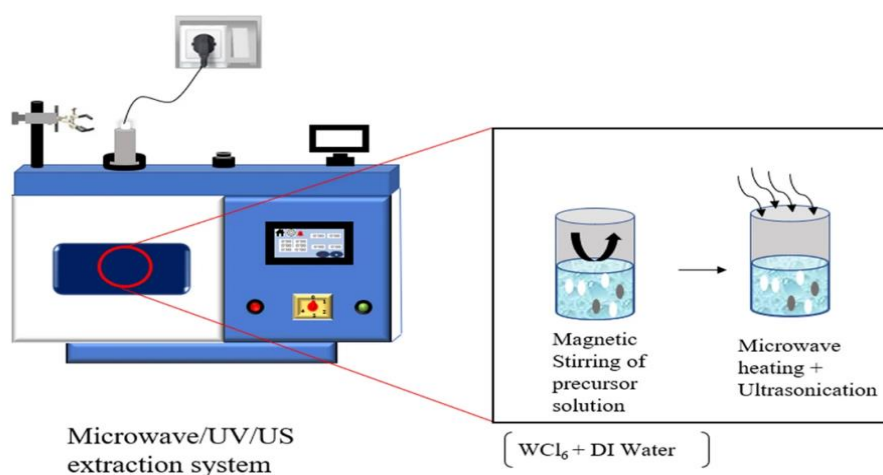

Figure-10 Stepwise reproduced with permission from ref [146] copyright © 2023 Springer Nature.

7.8. Template-Assisted Synthesis:

Template-assisted synthesis is a versatile approach used to control the morphology and structure of materials, including transition metal chalcogenides. This method involves the use of templates or scaffolds, which can be sacrificial or nanoporous materials, to guide the growth of chalcogenide

materials into specific shapes or structures. The template serves as a mold or framework, influencing the arrangement and organization of atoms during the synthesis process [165], [166]. Here is an overview of the template-assisted synthesis for transition metal chalcogenides:

Choice of Template:
The first step in template-assisted synthesis is the selection of an appropriate template. Templates can be divided into two main types:

> **Sacrificial Templates:** These templates are later removed or etched away, leaving behind the desired chalcogenide structure. Examples include polymers, organic molecules, or inorganic salts.
>
> **Nanoporous Templates:** These templates have a porous structure that allows the controlled growth of chalcogenides within the pores. Common nanoporous templates include porous alumina, zeolites, and other porous materials.

Deposition of Precursor Materials:
> The template is coated or impregnated with precursor materials for the transition metal and chalcogen. These precursors may be in the form of salts, metal-organic complexes, or other compounds that can be converted into the desired chalcogenide.

Template-Assisted Growth:
> The template, with the deposited precursors, is subjected to specific conditions such as elevated temperature or chemical reactions. Under these conditions, the precursors undergo chemical transformations, and the resulting transition metal chalcogenide begins to grow within or on the template.

Removal of Template:
> For sacrificial templates, once the chalcogenide material has formed and solidified, the template is removed. This removal process can involve chemical etching, thermal decomposition, or other methods that selectively dissolve or eliminate the template while leaving the chalcogenide intact.

Structure and Morphology Control:
> The template provides a framework that dictates the structure and morphology of the resulting transition metal chalcogenide. By modifying the template properties, such as pore size or surface chemistry, researchers can precisely control the size, shape, and arrangement of the synthesized chalcogenide.

7.9. Chemical Precipitation:

Chemical precipitation is a common and straightforward method for the synthesis of transition metal chalcogenides. This method involves the precipitation of solid particles from a solution containing metal and chalcogenide precursors. The precipitation occurs through the addition of a suitable precipitating agent, leading to the formation of insoluble compounds that can be collected, washed, and further

processed to obtain the desired transition metal chalcogenide material [83], [93], [167].
Here's a step-by-step description of the chemical precipitation process for synthesizing transition metal chalcogenides:

Precursor Preparation:
> Metal salts (e.g., metal chlorides, nitrates) and chalcogenide sources (e.g., sulfides, selenides) are selected as precursor compounds. These precursors are dissolved in a solvent to form a homogeneous solution.

Formation of Precipitating Agent:
> A precipitating agent is introduced to the precursor solution. Common precipitating agents include sodium sulfide (Na_2S) for sulfides, sodium selenide (Na_2Se) for selenides, and sodium telluride (Na_2Te) for tellurides. The choice of the precipitating agent depends on the specific chalcogenide being synthesized.

Precipitation Reaction:
> The addition of the precipitating agent results in a chemical reaction between the metal ions and chalcogenide ions, leading to the formation of insoluble metal chalcogenide compounds. The reaction can be represented as follows for the generic case of a metal M and chalcogenide X:
> $$M^{n+} + X^{2-} \rightarrow MX\downarrow$$
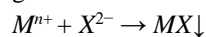
Formation of Precipitate:
> The newly formed metal chalcogenide compound precipitates as solid particles in the solution. The size and morphology of the particles can be influenced by reaction conditions such as temperature, pH, and the rate of addition of the precipitating agent.

Filtration and Washing:
> The precipitate is separated from the liquid phase by filtration. The collected solid is then washed several times with a suitable solvent to remove impurities, unreacted precursors, and the precipitating agent.

Drying and Annealing:
> The washed precipitate is dried to remove any residual solvent. Subsequent annealing (heat treatment) is often performed to enhance the crystallinity and optimize the properties of the transition metal chalcogenide. The annealing conditions can be adjusted to achieve the desired phase and structure.

Chemical precipitation is advantageous for its simplicity, cost-effectiveness, and potential for large-scale production. However, controlling particle size, uniformity, and purity can be challenging. Optimization of reaction parameters is crucial to achieving the desired properties of the transition metal chalcogenide material for specific applications, including those in energy storage, catalysis, and optoelectronics.

7.10. Mechanical Alloying:

Mechanical alloying is a solid-state powder processing technique used for the synthesis of transition metal chalcogenides. It involves the repeated cold welding, fracturing, and rewelding of powder particles in a high-energy ball mill. This process is particularly effective for producing finely divided and homogeneously mixed powders, leading to the formation of transition metal chalcogenides with enhanced properties [168].

Here's a step-by-step description of the mechanical alloying process for synthesizing transition metal chalcogenides:

Selection of Precursors:

Elemental powders of transition metals (e.g., nickel, cobalt, iron) and chalcogenides (e.g., sulfur, selenium, tellurium) are selected as precursor materials. The choice of elements depends on the desired composition of the final chalcogenide compound.

Loading the Ball Mill:

The selected elemental powders are loaded into a high-energy ball mill along with grinding media (usually metallic balls) that serve as impactors. The ratio of balls to powder and the milling atmosphere (e.g., under inert gas to prevent oxidation) are carefully controlled.

Milling Process:

The ball mill is set into motion, causing the grinding media to impact the powder mixture. The repeated collisions and mechanical forces generated during milling lead to the cold welding of powder particles. This process results in the formation of alloyed powders as the elemental components mix at the atomic level.

Fracturing and Rewelding:

As the milling continues, the powders undergo fracturing due to the impact of the grinding media. The newly exposed surfaces then undergo further cold welding, promoting the homogenization of the powder mixture. This cycle of fracturing and rewelding continues until the desired level of alloying is achieved.

Formation of Transition Metal Chalcogenides:

The mechanical alloying process induces chemical reactions between the transition metal and chalcogen elements, leading to the formation of transition metal chalcogenides. The high-energy milling facilitates the reduction of particle size and enhances the reactivity between the elements.

Heat Treatment:

In some cases, a subsequent heat treatment or annealing step may be employed to optimize the crystal structure and properties of the synthesized transition metal chalcogenides. This step is particularly useful for promoting crystallinity and removing any defects introduced during mechanical alloying.

Mechanical alloying is advantageous for its simplicity, scalability, and the ability to produce fine and uniform powders. It is widely used in the synthesis of transition metal chalcogenides for applications such as catalysis, energy storage, and electronic devices. The controlled milling conditions play a crucial role in determining the properties of the final chalcogenide material.

8. IDENTIFICATION OF PROBLEMS AND ISSUES

While the research on chalcogenide nanocomposites for energy applications has made significant progress, there are still notable gaps and challenges in the existing literature that warrant attention. Some key gaps and challenges include:

8.1.1. Standardization of Synthesis Methods:

Existing literature lacks standardization in the synthesis methods of chalcogenide nanocomposites. Different studies employ varied approaches, making it challenging to compare results and establish a universal set of optimized conditions. The present work however only focuses on microwave assisted and hydrothermal assisted route making the work more precise and clear.

8.1.2. Long-Term Stability and Durability:

Many studies focus on the initial performance of chalcogenide nanocomposites, but there is a lack of comprehensive investigation into their long-term stability and durability. All the energy materials such as cinnabar, Cd especially for core shells, lack electrochemical investigation perfumed in the thesis.

8.1.3. Environmental Impact and Sustainability:

The environmental impact of chalcogenide nanocomposites, especially those containing elements like cadmium (Cd) or mercury (Hg), needs thorough consideration. The potential toxicity and environmental implications associated with the production, use, and disposal of these materials are essential aspects that require more attention in the literature.

8.1.4. Integration into Energy Device Architectures:

While the literature explores the synthesis and characterization of chalcogenide nanocomposites, there is a gap in understanding their integration into practical energy device architectures. Studies often lack insights into the scalability and feasibility of incorporating these materials into real-world devices, such as batteries, supercapacitors, or solar cells. The present work explores the use of these material as an electrode either for battery, capacitor or solar cell.

8.1.5. Optimization of Electrochemical Performance:

Achieving optimal electrochemical performance, including high energy density, power density, and cyclic stability, remains a challenge. The literature needs more systematic investigations into the factors influencing electrochemical performance, such as electrode design, electrolyte composition, and charge storage mechanisms.

8.1.6. Understanding Interface Interactions:

The interactions at the interfaces between different components in chalcogenide nanocomposites are complex and play a crucial role in determining overall performance. Existing literature often lacks in-depth analyses of these interface interactions, including charge transfer kinetics, ion diffusion dynamics, and electronic conductivity across interfaces. Chapter-3, 4 and 5 thoroughly discuss the charge transfer kinetics, ion diffusion dynamics, and electronic conductivity across interfaces

8.1.7. Multifunctional Nanocomposites:

Many energy applications demand multifunctional nanocomposites with tailored properties for specific functions. Current literature often focuses on individual aspects, such as energy storage or conversion, without exploring the full potential of chalcogenide nanocomposites to serve multiple functions within the same device or system.

8.1.8. Performance Limitations:

Issues related to the performance of chalcogenide nanocomposites in energy storage and conversion applications, such as limited charge/discharge cycles and suboptimal efficiency, pose significant challenges that need careful consideration and targeted solutions. Addressing these issues requires a multifaceted approach involving materials design, engineering, and comprehensive understanding of the underlying mechanisms. Some of the performance limitations are: (a) Cycle Life Improvement, (b) Materials Selection, (c) Enhancing Conductivity, (d) Optimizing Electrode Design, (e) Understanding Degradation Mechanisms, (f) Optimizing Synthesis Conditions

8.1.9. Environmental Impact:

The synthesis and use of chalcogenide nanocomposites for energy materials raise environmental concerns primarily related to the presence of toxic elements, such as cadmium (Cd) and mercury (Hg), in some chalcogenide compounds. CdS and HgS are examples commonly used in nanocomposites, and their toxicity poses risks to both human health and the environment. Cd is a known carcinogen and can accumulate in ecosystems, potentially causing adverse effects on aquatic life. Additionally, the disposal of materials containing Cd and Hg at the end of their lifecycle may contribute to environmental pollution. Furthermore, the extraction and processing of raw materials for chalcogenide nanocomposites can have environmental impacts, including habitat disruption and energy consumption. Addressing these concerns necessitates a focus on developing environmentally friendly synthesis methods, substituting toxic elements with safer alternatives, and implementing effective recycling strategies to minimize the environmental footprint of chalcogenide nanocomposites in energy materials. Sustainable approaches that prioritize both performance and environmental responsibility are crucial for the responsible development and deployment of chalcogenide nanocomposites in energy technologies.

9. CONCLUSION

In conclusion, our discussion has shed light on the multifaceted landscape of chalcogenide nanocomposites for energy applications. We explored diverse synthesis techniques, from hydrothermal to UV-assisted methods, highlighting their roles in achieving sustainable and efficient material fabrication. Despite the promising advancements, challenges such as limited cycle life and suboptimal efficiency persist, underscoring the need for continued research and innovation. Addressing these challenges requires a holistic approach, encompassing materials design, characterization, and performance optimization. By enhancing conductivity, optimizing electrode design, and understanding degradation mechanisms, we can unlock the full potential of chalcogenide nanocomposites in energy storage and conversion.

Moreover, our dialogue emphasized the importance of sustainability in materials synthesis, advocating for greener synthesis approaches and environmental responsibility. Through collaborative efforts and interdisciplinary research, we can propel chalcogenide nanocomposites toward a brighter energy future—one that is both efficient and environmentally conscious. As we move forward, let us remain committed to harnessing the transformative power of these materials for a more sustainable world.

DECLARATION OF COMPETING INTEREST

It is declared that this article is original and written by the stated authors. There is no conflict of interest between the authors.

CONFLICTS OF INTEREST OR COMPETING INTERESTS

No conflicts of interest exist among the authors.

DATA AND CODE AVAILABILITY

All the data is presented in the manuscript. No supplementary data is needed.

SUPPLEMENTARY INFORMATION

No supplementary data is required as no additional material omitted from the main manuscript.

ETHICAL APPROVAL

All the authors have worked in accordance with ethical standard and approved by Netaji Subhas University of Technology.

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