

Concise Review of Nanomaterial Synthesis and Applications in Metal Sulphides

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ABSTRACT

Nanotechnology has garnered significant attention for its capacity to manipulate matter at atomic and molecular scales. This thorough review focuses on nanomaterials, with a specific emphasis on metal sulphides, elucidating their distinctive properties and applications. Nanoparticles, fundamental units in nanostructures, exhibit unique physical and chemical characteristics distinct from bulk materials, making them attractive for diverse industrial applications. The review provides a detailed categorization of nanomaterials based on dimensions, encompassing 1D (surface films), 2D (monolayer materials like graphene), and 3D (bulk powders, nanoparticle dispersions). Synthesis methods, categorized as top-down (breakdown) and bottom-up (build-up), are meticulously outlined, covering techniques such as dry/wet grinding, chemical vapor deposition, and liquid-phase methods. The synthesis methods and applications of metal sulphides, specifically Cadmium Sulphide (CdS), Nickel Sulphide (NiS), and Copper Sulphide (CuS), are explored in terms of crystal structures, quantum size effects, and their roles in solar cells, bioimaging, and photocatalysis. In conclusion, this review presents a comprehensive exploration of nanomaterials, synthesis methodologies, and the distinct applications of metal sulphides. The unique nanoscale properties of these materials hold promise for significant advancements across various fields, ranging from electronics to energy storage.

Keywords: Nanomaterials; Metal sulphides; Synthesis; Quantum size effects; Applications

1. Introduction

1.1 Nanotechnology

Nanoscience is the discipline dedicated to the examination of matter on a scale of one billionth of a meter (i.e., $10^{-9}\text{m} = 1\text{ nm}$). Similarly, nanotechnology involves the deliberate manipulation of matter at the atomic and molecular levels^{1,2}. Notably, a nanometre corresponds to one millionth of a millimetre, approximately 100,000 times smaller than the width of a human hair., as illustrated in (Figure 1).

At the core of crafting a nanostructure, a nanoparticle functions as the elemental building block. This minute entity resides on a scale markedly tinier than the everyday objects adhering to Newton's laws of motion, yet it surpasses the

dimensions of atoms or basic molecules governed by the principles of quantum mechanics^{3,4}.

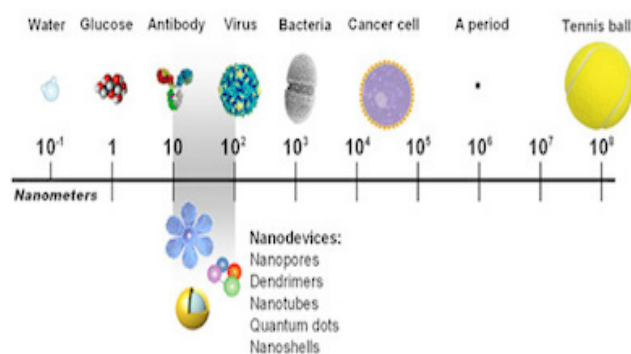


Figure 1: Scale Comparison Including Nanometres.

Typically ranging between 1 and 100 nm, nanoparticles exhibit distinct physical and chemical characteristics compared to bulk metals, such as lower melting points, higher specific surface areas, specific optical properties, mechanical strengths, and unique magnetization properties. These distinctive attributes make nanoparticles particularly appealing for diverse applications.

1.2 Nanomaterials

In the contemporary era, nanomaterials have become a point of interest due to their distinctive physical, chemical, and mechanical attributes. The ability to manipulate material properties, encompassing magnetic, optical, electrical traits, is achievable without altering the chemical composition. This mastery is attained by overseeing the structural configuration, and surface states of nanomaterials^{5,6}.

Nanomaterials attract attention due to the emergence of distinct optical, magnetic, electrical, and other properties at this scale. These unique characteristics possess considerable potential for advancements in electronics, medicine, and diverse fields.

As the particle radius approaches the asymptotic Bohr radii, quantum confinement effects come into play, especially observable in nanostructured semiconductors showcasing captivating electro-optical properties and catalytic behaviour. This emphasizes the significant influence of surface properties on the structural and optical traits of nanomaterials. Furthermore, the alteration of nanomaterial surfaces through the introduction of diverse inorganic species serves to eradicate surface defects and shape their optical properties.

The versatile applications of nanomaterials encompass fields such as light-emitting diodes, gas sensors, nanothermometers, solar cells, fuel cells, piezoelectric nanogenerators, and lithium-ion batteries^{7,8}.

1.3 Types of nanomaterials

Nanomaterials are characterized by their exceedingly small size, with at least one dimension measuring 100 nm or less. These materials can manifest in nanoscale dimensions along one (e.g., surface films), two (e.g., strands or fibers), or three dimensions (e.g., particles). They may exist in several forms, including single, fused, aggregated, or agglomerated structures, exhibiting spherical, tubular, or irregular shapes, as illustrated in (Figure 2). Prominent examples of nanomaterials encompass nanotubes, dendrimers, quantum dots, and fullerenes⁹⁻¹³.

- **One-dimensional nanomaterials (1D):** Systems with a single dimension, such as thin films or manufactured surfaces, have been employed for decades. Thin films with sizes ranging from 1 to 100 nm, or monolayers, have become integral components in diverse fields such as solar cells, sensing technologies, information storage systems, magneto-optics, optical devices, and fiber-optic systems.
- **Two-dimensional nanomaterials (2D):** Beyond the nanoscale, these materials have a single dimension comprising only a single or a few atomic layers. This category includes plate-like shapes, such as graphene and other monolayer materials like MXenes, black phosphorous (phosphorene), diatomic hexagonal boron nitride, and carbon nanotubes.
- **Three-dimensional nanomaterials (3D):** Materials in this category are not confined to the nanoscale in any

dimension. They encompass bulk powders, dispersions of nanoparticles, bundles of nanowires, and multi-nanolayers, as exemplified by dendrimers, quantum dots, and fullerenes (e.g., Carbon 60).

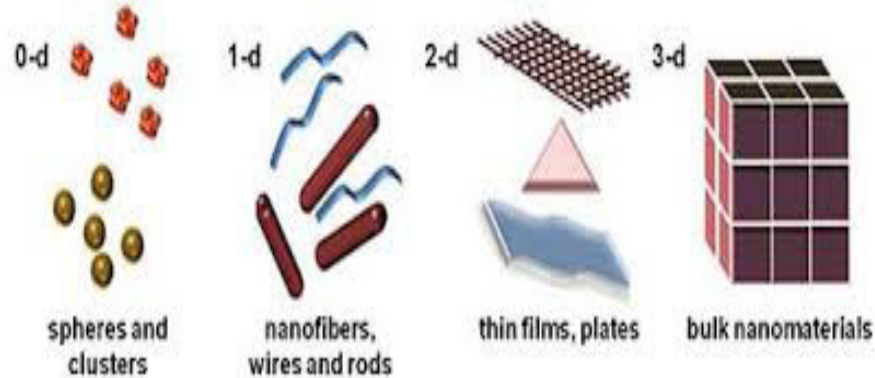


Figure 2: Categorization of Nanomaterials.

1.4 Nanomaterials Synthesis Methods

Two distinct approaches have historically been employed in the production of ultrafine particles. The first method involves the breakdown (top-down) approach, where an external force is applied to a solid, causing it to disintegrate into smaller particles. Another method, known as the build-up (bottom-up) approach, generates nanoparticles by commencing with gas or liquid atoms, relying on atomic transformations or molecular condensations, as illustrated in (Figure 3)¹⁴⁻¹⁷.

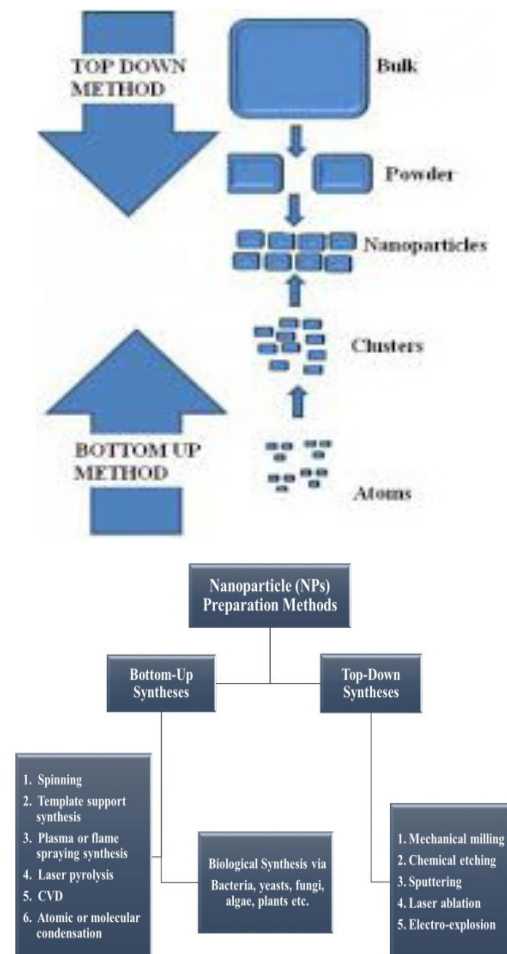


Figure 3: Nanoparticle synthesis methods.

1.4.1 Top-down method

The top-down approach entails the disintegration of a solid material into extremely fine particles, and it can be classified as dry and wet grinding. In the realm of dry grinding, the solid

material experiences pulverization through the application of shock, compression, or friction. This process is executed through various methods, including jet mills, hammer mills, shearing mills, roller mills, shock shearing mills, ball mills, and tumbling mills. Achieving particle sizes below 3 μm through grain refinement is challenging due to simultaneous particle condensation during pulverization.

In the wet grinding approach, a solid substrate is processed utilizing specialized equipment like tumbling ball mills, vibratory ball mills, planetary ball mills, centrifugal fluid mills, agitating beads mills, flow conduit bead mills, annular gap beads mills, or wet jet mills. Unlike the dry method, wet processing proves advantageous in averting the agglomeration of generated nanoparticles, promoting the creation of finely dispersed nanoparticles. Moreover, within the top-down methodologies, the mechanochemical method and mechanical alloying method are incorporated¹.

1.4.2 Bottom-up method

The bottom-up approach can be broadly categorized into gaseous phase methods and liquid phase methods. In gaseous phase methods, the chemical vapor deposition method (CVD) involves a chemical reaction, while the physical vapor deposition method (PVD) relies on the cooling of evaporated material. Although gaseous phase methods minimize the presence of organic impurities compared to liquid phase methods, they require complex vacuum equipment, leading to high costs and low productivity. The CVD procedure can generate ultrafine particles of less than 1 μm through chemical reactions in the gaseous phase, and careful control of the reaction allows the production of nanoparticles ranging from 10 to 100 nm. Elevated temperature chemical reactions in the CVD approach require heat sources like a chemical flame, plasma process, laser, or electric furnace. In contrast, the PVD technique involves the evaporation of solid or liquid material, followed by swift cooling of the resulting vapor to generate the desired nanoparticles. The evaporation of materials can be achieved using an arc discharge method. The straightforward thermal decomposition method has demonstrated notable efficacy in generating metal oxide and various particle varieties, establishing itself as a widely embraced synthetic approach in the industrial domain. Liquid phase methods, especially liquid/liquid methods and sedimentation methods, have traditionally served as the predominant techniques for nanoparticle preparation over many years^{18,19}.

The chemical reduction of metal ions exemplifies a liquid/liquid method, offering the primary advantage of easily fabricating particles in diverse shapes, including nanorods, nanowires, nano-prisms, nanoplates, and hollow nanoparticles. Through the chemical reduction method, precise control over the form (shape) and size of nanoparticles can be achieved by adjusting factors such as the dispersing agent, reducing agent, reaction time, and temperature.

The chemical reduction method involves reducing metal ions to their 0 oxidation states ($M^{+n} \rightarrow M^0$) through a chemical process. This method employs simple equipment and proves cost-effective, enabling the production of substantial quantities of nanoparticles in a short duration. Notably, microwave radiation serves as an efficient heat source, contributing to the swift generation of high-quality nanoparticles in this process. In addition to the chemical reduction method, which involves the introduction of a reducing agent (known as

the direct reduction method), various alternative reduction techniques are recognized. These include photoreduction using ultrasonic waves, gamma rays, and liquid plasma, all viable for nanoparticle synthesis. A distinctive feature of these methods, characterized by the absence of chemical reducing substances, is their ability to introduce no additional impurities introduced to the nanoparticles. Additionally, other well-known methods in this domain encompass spray drying, solvothermal synthesis, spray pyrolysis, and the supercritical method¹⁸⁻²².

The sedimentation method commonly relies on a sol-gel process, widely applied for manufacturing metal oxide nanoparticles. This process begins by converting a metal alkoxide solution into a sol through hydrolysis, followed by polycondensation resulting in gel formation. Unlike the dry method, the wet process, operating in the liquid phase, ensures a superior dispersion of nanoparticles. However, when the resultant nanoparticles undergo drying, particle aggregation promptly occurs. In such instances, re-dispersion can be executed using the procedures employed in the solid phase method^{1,18-22}.

1.5 Metal sulphides

Metal sulphides nanomaterials have garnered considerable attention owing to their exceptional properties and promising applications in electronic, optical, and optoelectronic devices. The extensive study of nanostructured metal sulphides is driven by their crucial role in elucidating quantum size effects and their applications across various devices, including solar cells, light-emitting diodes, sensors, thermoelectric devices, lithium-ion batteries, fuel cells, and nonvolatile memory devices²³⁻²⁸. Representing a major group of minerals, metal sulphides offer a rich field for crystal chemists due to their diverse structural types. Abundant and cost-effective, these sulphides are commonly found in nature as minerals such as heazlewoodite (Ni_3S_2), chalcocite (Cu_2S), pyrite (FeS_2), CdS, and others.

This work will focus on a significant subgroup of nanoparticles which are metal sulphides. Subsequent sections will delve into the exploration of this nanoparticle group.

1.5.1 Cadmium Sulphide (CdS)

Cadmium sulphide (CdS), classified as an II-VI semiconductor, demonstrates insolubility in water but solubility in dilute mineral acids. Its intrinsic n-type conductivity is attributed to sulfur vacancies resulting from excess cadmium atoms. In bulk, CdS possesses a band gap energy of 2.42 eV at 300K, with absorption maxima at 515nm²⁹⁻³¹. CdS can adopt three crystal structures—wurtzite, zinc blend, and high-pressure rock-salt phases. Among these, the wurtzite phase, known for its stability, can be readily synthesized. While both bulk and nanocrystalline CdS exhibit the wurtzite phase, cubic and rock-salt phases are exclusive to nanocrystalline CdS^{32,33}.

Nanoparticles of CdS exhibit distinct physical, chemical, and structural properties compared to their bulk counterparts. The size of CdS nanoparticles influences various properties, including melting point, electronic absorption spectra, band gap energy, and crystal structure^{34,35}. The quantum size effect in CdS nanoparticles is evident in the direct relationship between the particle size and absorption wavelength. The electronic properties of nanocrystalline CdS can vary based on size reduction and reaction conditions, leading to different crystalline structures.

Cadmium sulphide finds applications across diverse fields such as solar cells, bioimaging^{36,37}, photoconductive devices³⁸, chemiluminescence³⁹, and sensing applications⁴⁰. It is extensively

utilized as a visible-light-driven photocatalyst, despite challenges like photocorrosion, facile recombination of electron-hole pairs, and lower efficiency in surface reactions. The development of stable CdS-based photocatalysts, ensuring efficient charge separation and high photocatalytic activity, is imperative for enhancing their practical utility. Various hierarchical nanostructures of CdS have been prepared using methods like microwave-assisted synthesis⁴¹, chemical vapor deposition (CVD)⁴², hydrothermal routes⁴³, chemical bath deposition (CBD)⁴⁴, UV irradiation technology⁴⁵, and electrochemical synthesis⁴⁶. Some of these nanostructures obtained through these methods have found applications in catalysis.

1.5.2 Nickel Sulphide (NiS)

Nickel sulphide (NiS), a significant member within the extensive family of transition metal (TM) sulphides, serves various purposes such as a potential cathode material for rechargeable lithium batteries, a catalyst in the degradation of organic dyes, and in magnetic devices and certain non-linear optical devices⁴⁷. Catalysts, including NiS, play a crucial role in the oil industry for separating elements with hydrocompounds from insulators⁴⁸. Given its diverse applications, numerous synthesis methods have been employed to prepare NiS nanoparticles, including the sol-gel method, laser ablation, solvothermal processes, UV irradiation, and the colloidal microemulsion method⁴⁹. Among these, the chemical precipitation method stands out as a significant approach for nanoparticle synthesis.

Nickel sulphide showcases complex compositional, structural, optical, electrical, and magnetic phase responses. Different binary nickel sulphides, such as Ni_3S_2 , $\text{Ni}_{3+x}\text{S}_2$, $\text{Ni}_4\text{S}_{3+x}$, Ni_6S_5 , Ni_7S_6 , Ni_9S_8 , Ni_3S_4 , and NiS, have been documented, depending on the chosen synthesis method⁵⁰.

1.5.3 Copper Sulphide (CuS)

Copper sulphide (CuS) stands as a significant p-type semiconductor with considerable potential applications, including its use as cathode materials for lithium-ion batteries, solar radiation absorbers, and nonlinear optical materials. Its appeal lies in being an exceptionally thin absorber layer for solar cells, owing to its nearly ideal band gap of 1.2 eV and cost-effectiveness. Furthermore, CuS holds promise for nanoscale switches due to its nature as a mixed Cu ionic/electronic conductor^{51,52}.

Monocrystalline copper sulphide serves as an n-type semiconductor, demonstrating at least five stable phases at room temperature with varying Cu:S molar ratios (Cu_xS). These phases include covellite (CuS), anilide ($\text{Cu}_{1.75}\text{S}$), digenite ($\text{Cu}_{1.8}\text{S}$), djurleite ($\text{Cu}_{1.95}\text{S}$), and chalcocite (Cu_2S), each possessing a crystal structure ranging from orthogonal to hexagonal⁵³.

1.6 Sulphur sources

Many sulphur sources were used in the preparation of metal sulphide nanoparticles such as, thiourea, thioglycolic acid, dithiocarbamate.... etc.

In the present work, we will use three organic sulphur sources to obtain metal sulphide nanoparticles materials. These organic sulphur sources are thiocarbohydrazide TCH, thiocarbonic acid dipotassium salt and thiocarbonyl-bis-thioglycolic acid. In the following we will summarize the previous works deal with this organic sulphur source.

1.6.1 Thiocarbohydrazide (TCH)

Thiocarbohydrazide, a straightforward hydrazine derivative of thiocarbonic acid, finds application in organic synthesis,

as well as in the industrial manufacturing of insecticides, fungicides, and various agricultural chemicals. Moreover, it serves as a chemical reagent in laboratory settings^{54,55}. TCH presents as a colorless crystalline solid, exhibiting decomposition at approximately 171 °C and facilitating recrystallization from water^{56,57}. Multiple synthesis methods for TCH exist, including the Taguchi method⁵⁶⁻⁵⁸, Solomon Omwoma Lugasil⁵⁹, Audrieth and colleagues⁶⁰, and Mohamed A. Metwally et al.⁶¹⁻⁶⁷. Recent comprehensive reviews delve into the chemistry and applications of thiocarbohydrazide in the realms of synthetic organic chemistry and biological sciences⁶⁸. Beyond its role in organic synthesis, thiocarbohydrazide finds application in diverse areas, serving as fogging agents recognized for safety, storability, and cool-burning characteristics in pyrotechnic compounds used for smoke dissemination and chemical warfare agent dispersal. Moreover, it functions as a therapeutic agent, showcasing its efficacy as a highly selective adsorbent for heavy metal ions and as a complexing agent in various solvent extraction and separation methodologies⁶⁸.

1.6.2 Synthesis of thiocarbonic acid dipotassium salt:

Thiocarbonic acid dipotassium salt was obtained by the reaction between K_2S and carbon disulphide in 35 ml distilled water by Holmberg synthesis method⁶⁹.

1.6.3 Synthesis of the thiocarbonyl-bis-thioglycolic acid

Thiocarbonyl-bis-thioglycolic acid can be prepared by Holmberg synthesis method⁶⁹.

1.7 Literature Survey and previous work

1.7.1 CdS nanoparticles

Various methodologies and precursor combinations were employed to synthesize CdS nanoparticles. Ristic et al⁷⁰ achieved the synthesis of cubic CdS nanoparticles with a size range of 2-3 nm by reacting H_2S gas with a 10% aqueous solution of cadmium acetate at room temperature. Beggasa et al⁷¹ utilized the chemical bath deposition technique to produce hexagonal CdS nanoparticles, demonstrating an average size spanning 14.3 to 30.4 nm. In their approach, a bath solution consisting of Cadmium carbonate and thiourea was employed, with ammonia serving as a complexing agent. The transmittance of CdS exceeded 70% in the invisible region, and the band gap energy ranged from 2.46 to 2.42 eV. Yang et al⁷² opted for an organic synthesis method to prepare CdS, resulting in the development of Se-doped CdS semiconductor nanocrystals (NCs). Billakant et al⁷³ synthesized CdS nanoparticles using a solution-phase hexamethyldisilazane (HMDS)-assisted chemical synthetic method, with CdCl_2 and thiourea as precursors. Lahewil et al⁷⁴ engineered CdS thin films with a nanostructure, depositing them on glass substrates with Cd:S ratios ranging from 1.2 to 0.05 mol/L. The obtained films underwent annealing at 400 °C, with different spin coating speeds (1000 and 5000 rpm) influencing the average grain size, varying from 1.35 to 2.66 nm for films prepared at 1000 and 5000 rpm, respectively. Amorphous CdS nanoparticles, capped with cetyltrimethyl ammonium bromide (CTAB), were synthesized under diverse conditions using a co-precipitation method, resulting in a blue shift in the band gap and an approximate CdS size of 8 nm⁷⁵.

Mahdi et al⁷⁶ employed the microwave-assisted chemical bath deposition method to fabricate CdS thin films onto glass substrates at 80 °C, achieving films with robust adhesion and an absence of pinholes. Aqueous solutions of cadmium chloride or cadmium acetate, along with thiourea, served as the sources

for Cd²⁺ and S²⁻ ions, respectively. Moualkia et al⁷⁷ utilized the chemical bath deposition (CBD) technique to produce cubic CdS thin films exhibiting a preferential orientation along the (111) plane. The process involved NH₄OH, CdSO₄, and CS (NH₂)₂. Alonso et al⁷⁸ synthesized hexagonal CdS, characterized as an n-type semiconductor, through microwave heating, utilizing thioacetamide as the sulfur source. Abo-Bakr et al⁷⁹ synthesized two CdS nanocrystals using a novel organic salt named Potassium N'-[4-(N'-dithiocarboxy-hydrazino)-4-oxo-butyryl]-hydrazinecarbo-dithionate, which was dissolved in different solvents (200 ml water and 100 ml water with 100 ml ethanol). Duchaniya⁸⁰ employed the sol-gel technique to synthesize cubic CdS with a crystallite size ~ 10 nm. Al-Douri et al⁸¹ fabricated CdS nanostructures on glass substrates using the spin coating technique with varying spin coating speeds (1000, 3000, and 5000 rpm). The resulting films underwent annealing at 400 °C, yielding particle sizes for CdS nanostructures of 1.40, 1.78, and 2.31 nm at 1000, 3000, and 5000 rpm, respectively.

Marathe et al⁸² employed both the sol-gel method and spray pyrolysis to fabricate CdS nanocrystalline thin films on glass substrates. The films obtained through the sol-gel method exhibited a band gap of approximately 3.25 eV, while those produced by the spray pyrolysis method had a band gap of 2.87 eV. Limin Qi et al⁸³ synthesized CdS nanoparticles with particle sizes ranging between 2 and 4 nm. They utilized double hydrophilic block copolymers, consisting of a solvating poly(ethylene glycol) (PEG) and a poly(ethylene imine) (PEI), as effective stabilizers for CdS nanoparticle solutions in water and methanol. Jinxin et al⁸⁴ employed the hydrothermal method in a microemulsion composed of polyoxyethylene lauryl ether, water, cyclohexane, and butanol to synthesize hexagonal CdS nanoparticles with a minimum diameter of approximately 10 nm. The study revealed a decrease in the diameter of CdS nanoparticles with an increase in the molar ratio of water to surfactant. Zang et al⁸⁵ utilized a solvothermal method with oxalic acid as an auxiliary agent to prepare hexagonal-phase CdS. The resulting hollow microspheres of CdS had a diameter of about 5 µm, with a center hole measuring approximately 500 nm. The optical energy band gap was determined to be 2.31 eV. Weiguang et al⁸⁶ prepared CdS Q-nanoparticles with a narrow size distribution, featuring mean diameters ranging from 2 to 5 nm. This was achieved through size-selective precipitation techniques. Raevskaya et al⁸⁷ synthesized CdS nanoparticles with a diameter of about 2 nm using polyethylenimine, exhibiting a narrow size (~10%). These nanoparticles demonstrated luminescence in the range of 400-600 nm, with a quantum yield of about 10%. Niasari et al⁸⁸ employed a cyclic microwave route and [Cd (C₂O₄)·3H₂O] powder as a precursor to produce CdS nanoparticles with an average size of approximately 15 nm. Lingdong et al⁸⁹ prepared CdS nanoparticles with an average particle size of 2.5 nm, utilizing a carboxylic-containing copolymer, polystyrene-maleic anhydride (PSM), as a template.

1.7.2 CuS nanoparticles

Various techniques and source materials were employed in the production of copper sulphide (CuS) nanoparticles. Wang et al⁹⁰ utilized a sonochemical route, involving an aqueous solution containing metal monosulphide and thioacetamide, with triethanolamine acetate as a complexing agent under ambient air conditions to achieve CuS nanoparticle synthesis. Sandhya et al⁹¹ employed a simple chemical co-precipitation method using copper acetate and sodium thiosulfate as precursors. The pH of the solution varied from 5.5 to 9.5, resulting in the preparation

of CuS nanoparticles with an optical band gap ranging from 3.27 to 3.66 eV. Li et al⁹² conducted a solvothermal synthesis using copper nitrate trihydrate and thiourea to produce CuS nanomaterials. The UV-visible spectrum exhibited broad absorption in the visible range, and the photoluminescence spectrum revealed a strong green emission. Ramamoorth et al⁹³ synthesized hexagonal CuS nanoparticles by employing copper acetate and thiourea in the presence of water-butanol and water-cyclohexanol as a mixed medium. The transmittance of the resulting CuS nanoparticles varied from 35% to 70% up to 450 nm in the electromagnetic spectra, with the band gap ranging from 2.31 to 2.51 eV.

Ajibade⁹⁴ conducted the synthesis of hexagonal copper sulphide nanocrystals using copper (II) dithiocarbamate single molecule precursors. The estimated crystallite sizes, as determined by XRD, ranged from 17.3 to 18.6 nm. TEM images further revealed particles with average crystallite sizes within the range of 3 to 9.8 nm. Castillón et al⁹⁵ employed the polyol method to prepare copper sulphide nanoparticles by utilizing copper nitrate and sodium sulphide as raw materials in the presence of ethylene glycol at various temperatures. The resulting particles exhibited a size of 10 nm, and the band gap energy value for the nanoparticles was estimated to be 2.15 eV. Pal et al⁹⁶ utilized a wet chemical method to produce hexagonal phase CuS nanoparticles with sizes in the nanometer range. The estimated band gap energy for these nanoparticles was found to be 2.05 eV. Nemade et al⁹⁷ employed the spray pyrolysis technique to synthesize CuS nanoparticles at different substrate temperatures. Riyaz et al⁹⁸ prepared CuS nanoparticles using the sol-gel route in the presence of distilled water at 100 °C for 3 hours. The crystallite size, determined by the Debye-Scherrer formula, was found to be 17.73 nm, and the band gap was calculated using the Tauc relation, resulting in a value of 2.89 eV.

1.7.3 NiS nanoparticles

Wang et al⁹⁰ employed a sonochemical route to prepare NiS from an aqueous solution containing metal monosulphide and thioacetamide, with triethanolamine acetate serving as a complexing agent under ambient air. Kristl et al⁹⁹ sonochemically synthesized NiS and Ni₃S₄ from nickel acetate and sulfur using a direct immersion ultrasonic probe. The resulting nanoparticles exhibited an average crystallite size ranging from 7 to 30 nm, with optical band gap energy in the range of 3.3 eV to 3.8 eV. Shajudheen et al¹⁰⁰ utilized the chemical precipitation method to prepare orthorhombic NiS nanoparticles, employing triethanolamine as a capping agent. Lili et al¹⁰¹ obtained the hexagonal NiS phase through a hydrothermal method starting from nickel acetate and sodium thiosulfate at 200 °C for 12 hours. Additionally, Ni₂ microsphere cubic phase was prepared by incorporating ethylenediaminetetraacetic acid (EDTA). Yang et al¹⁰² synthesized NiS nanorods through a solvothermal synthetic route using sulfur and nickel powders as reagents in ethylenediamine as the solvent at 200 °C. Rozue et al¹⁰³ utilized nickel acetate, sodium sulphide, and sodium hydroxide to obtain β-NiS nanoparticles. The samples were calcinated at 500 °C and 1000 °C for 1 hour, resulting in band gap values of 4.8 eV and 2.8 eV, respectively. Abd El-Raady et al¹⁰⁴ prepared nickel monosulphide using (Potassium N'-[4-(N'-dithiocarboxy-hydrazino)-4-oxo-butyryl]-hydrazinecarbodithionate (I) and thiourea (II)) through a simple chemical method in an aqueous ethanolic solution¹⁰⁵⁻¹⁰⁷.

2. Conclusion

In summary, this review highlights the rapidly advancing field of nanotechnology, with a specific emphasis on nanomaterials, synthesis techniques, and the practical applications of metal sulphides. The capacity to control matter at the atomic and molecular levels has given rise to a range of unique materials with varied properties, presenting numerous opportunities across different industrial sectors. Both top-down and bottom-up synthesis methods provide a comprehensive toolkit for crafting nanomaterials tailored to specific requirements. The article explores the intricacies of these methods, from dry/wet grinding to chemical vapor deposition, shedding light on the advantages and challenges associated with each approach.

The focus on metal sulphides, such as Cadmium Sulphide (CdS), Nickel Sulphide (NiS), and Copper Sulphide (CuS), unveils their distinctive properties and versatile applications. From their quantum size effects to their roles in solar cells, batteries, and catalysis, metal sulphides exemplify the transformative potential of nanomaterials. The examination of three organic sulphur sources for synthesizing metal sulphide nanoparticles introduces a layer of specificity to the discourse, demonstrating the diverse strategies employed by researchers to customize materials for intended applications. In essence, this review serves as a guide for researchers, providing valuable insights into the current state of nanotechnology, nanomaterials, and their applications. Simultaneously, it directs attention towards exciting avenues for future exploration and innovation in the field.

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