Different synthetic approach and characteristic studies of ZnS nanomaterials: A review report

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Abstract:

This review study is aim to understand the structural properties of zinc sulphide nanoparticles with respect to different synthesis mechanism. The impact of capping agent on synthesis of pristine ZnS material has also been discussed here. The study also focused on ZnS-based polymer composite materials. Therefore this review report will be quiet helpful to the beginner researcher who opted to carry their research on this topic.

Keywords: Zinc Sulfide; Nanoparticles Semiconductor; Photoluminescence;

1.INTRODUCTION

The inorganic Zinc Sulfide (ZnS) compound have exist two types of forms called Sphalerite and Wurtzite. The first one possesses cubic structure it also called zinc blende where as wurtzite form has hexahedron structure. The optical band gap value of the bulk cubic and hexagonal form of ZnS samples are 3.68 eV and 3.77 eV respectively. Both belong to II – VI group semiconductors. Nanomaterials are found to have a relatively larger surface area compared to the same mass of material produced in a bulk form. The research and application of different form of nanomaterials based on the design such as spherical, thick and thin film, rod and tube like features have been drawn interest in different technological fields(1) These various shapes of ZnS nanomaterials can attribute to many important technological applications including waste water treatment (2), photovoltaic cells (3,4),flat panels (5),light emitting diodes(6,7),different optical sensors(8,9). Therefore depending upon the dimensions 0D to 3D various features of ZnS NPs can be evolved when bulk particles are transformed to nano or smaller scale.(10-13). Due to high brittleness and low stiffness property of ceramic structure, ZnS is not suitable for making flexible solar cells, foldable devices and movable sensors. To get rid of this problem nowadays ZnS NPs has been successfully synthesized in composite features in different conductive polymer matrix like poly (3,4-ethylenedioxythiophene), polyaniline, polythiophene as well as polysulphone. Now the embedded ZnS can exhibit more flexibility feature along with conducting polymers for the fabrication of portable electronic devices is concerned. The organic photo-voltaic cell has multipurpose electric production system and has various field of application when the processes involved light absorption and charge transportation to electricity (14–17).

In this review report, we have tried to present the gross review of the research works carried out regarding the ZnS nanomaterial. The different synthetic methods and their finds have been discussed. The overview was started from many

methods of synthetic route to an approach on ZnS based composite material. Discussions on variation in size and shape were present.

2. SYNTHESIS OF SULPHIDE NANOMATERIALS

There are two main approaches for nanomaterials synthesis;

a. Top-down: size reduction from bulk materials.

b. Bottom-up: material synthesis from atomic level.

Top-down approaches are physical methods which included in the typical solid –state processing of the materials. This approach is based on the bulk material and makes it smaller particles, thus breaking down the larger particles through physical processes such as crushing, milling or grinding. Bottom –up approach refers to chemical route that build-up of a material from the bottom: atom-by-atom, molecule-by-molecule or cluster-by-cluster. Chemical method includes like hydrothermal process (18), micro-emulsion method (19), sol-gel method (20), chemical coprecipitation method (21), sonochemical method (22), microwave irradiation (23) and solvothermal method (24) mechanochemical (25), etc and physical method based on ball-milling process (26). Although out of various synthesis process of ZnS NPs, Scientists are facing lots of challenges to get the better control over the particles size distribution, morphology, purity, quantity and quality while synthesizing nanomaterial through environment friendly economical processes. The choice of synthesis technique can be a important role in determining the effectiveness of the photovoltaic studies.

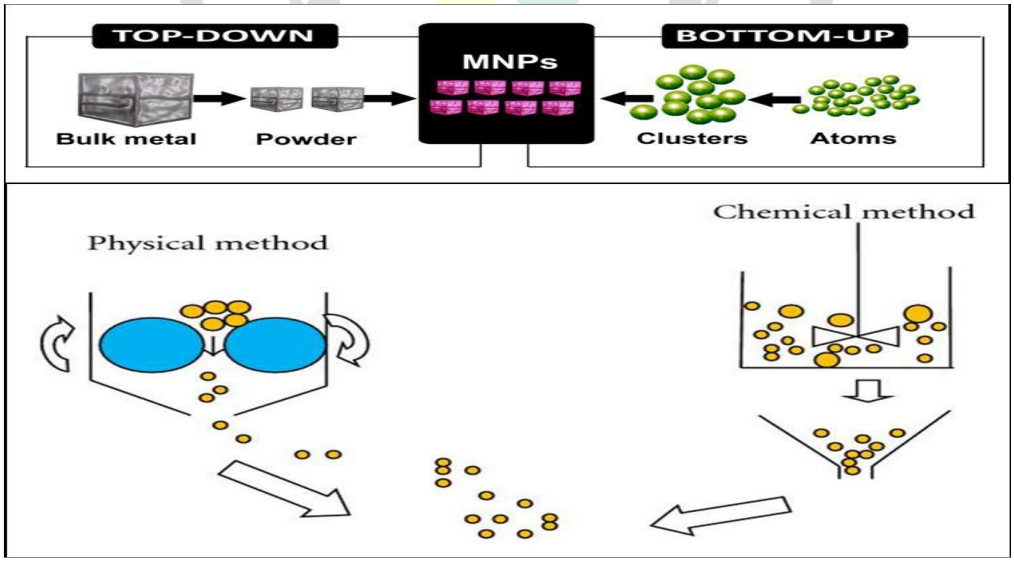

Figure1: Schematic representations of top down and bottom up approaches

Processing techniques:

2.1 Sol Gel Method: Sol-gel method (Fig. 2) is nothing but a Chemical Solution Deposition method. In this method under certain controlled condition molecules or particles having dimension of micros exist in the solution (sols) are tend to agglomerated to form a coherent matrix. The sol gel methods involve a set of process. Initially a homogeneous solution of precursor material is prepared in polar solvent and sol is formed by the hydrolysis and polymerization reactions with proper reagent that are normally inorganic metal salts or metal organic compounds such as metal sulphides or oxides (18).Then after hydrolysis and poly-condensation 'gel' formation is obtained (27). After gelation, the wet gel can be freely aged in its mother liquor, or in another solvent. Later it is repeatedly washed to get rid of different impurities. The 'aging' which is time between the formation of a gel and its drying is also an important parameter where the gel get shaped to diffent nano-formations such as nano-particles, nano-films, nano-rods, quantum dots etc which are sintered under desired temperature which is quiet low below the melting temperature of sulphides or oxides. That is why it is the process of low temperature synthesis Shahid M et al. (28). A flow chart in fig... Depicts the flow chart for sol-gel processing technique.

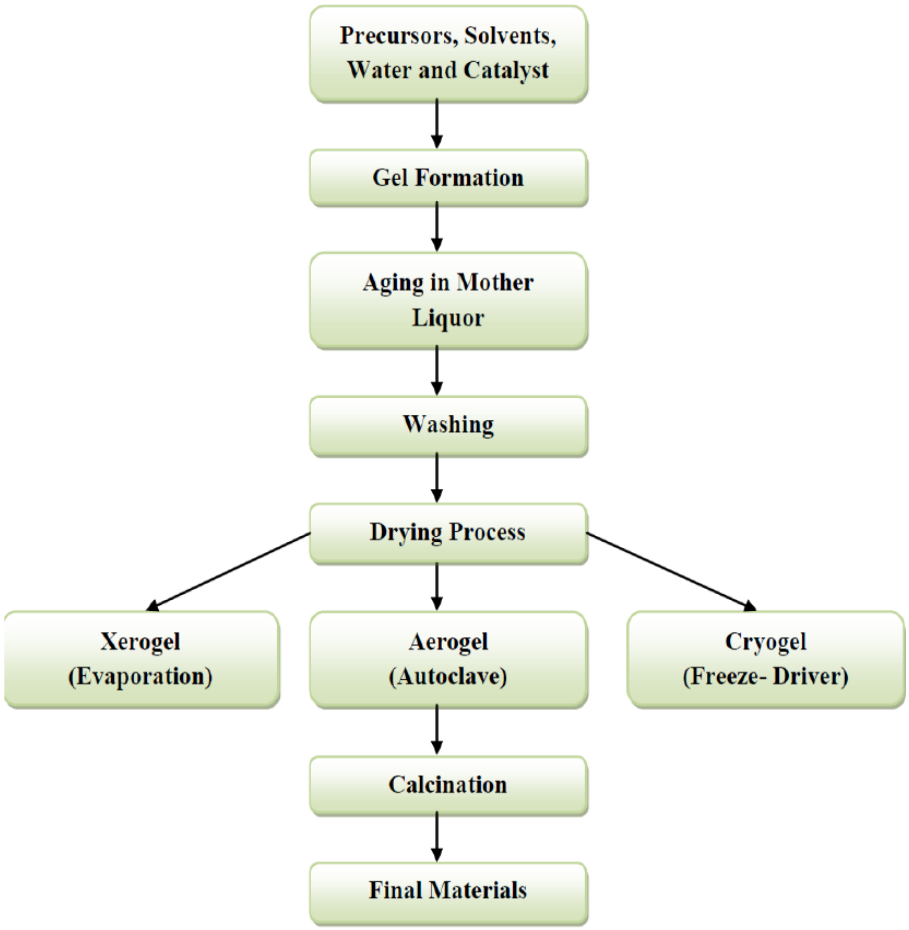

Figure 2 : Sol-Gel and Drying Flowchart

Table. 1 Importance parameter in the various steps of a sol-gel process

Step	**Purpose**	**Important parameters**
Solution Chemistry	To form gel	Nature and concentration of Precursor, Solvent type, Water Content, Temperature, pH of the solution
Aging	To allow a gel undergo changes in parameters	Aging temperature, environment and composition of the pored liquid,
Drying	To remove solvent from gel	Drying method (evaporative super critical & freeze drying),Temperature and heating rate, Pressure and pressurization rate, Time
Calcination	To change the physical /chemical properties of the solid, often resulting in crystallization and densification	Temperature and heating rate, Time, Gaseous environment (inert, reactive gases)

AL-Zahra et al. (29) have prepared ZnS nanoparticles by using sol-gel technique which were calcined with four different temperatures. X-ray diffraction revealed ZnS's cubic crystal structure with average particle size 15-24 nm. In spectroscopy, the zinc-sulfur band can be seen at 420 cm^{-1}. ZnS NP's visible and ultraviolet absorption spectra revealed a blue shift at 315 nm. Bhattacharjee et al.(30) have synthesized and characterized undoped and Mn^{2+} doped ZnS nanocrystallites with radius 2-3 nm embedded in a partially densified silica gel matrix utilising sol-gel method. Measurements of optical transmittance, photoluminescence (PL), ellipsometry, and electron spin resonance revealed evidence of the quantum size effect. At room temperature, PL spectra revealed a broad blue emission signal centred at 420 nm and a Mn^{2+} related yellow-orange band centred at 590 nm, whereas ESR revealed that Mn in ZnS was present as a dispersed impurity rather than a Mn cluster.

2.2. Hydrothermal method:

The hydrothermal technique was one of the most effective methods for preparing ZnS particles. Fundamentally, it was important to note that hydrothermal technique was involved in the crystallisation of substances from high temperature aqueous solutions at high vapour pressures (31, 32). It was theoretically defined as a method of synthesis of single crystals based on the solubility of minerals in water under high pressure. In general, crystal growth occurs in an apparatus consisting of a steel pressure vessel known as an autoclave, into which a sample is supplied along with water. A temperature gradient is maintained between the opposite ends of the growth chamber. The ability to create crystalline phases that are not stable at the melting point was considered an advantage of hydrothermal technique. Furthermore, it can effectively provide the controllable properties and composition during synthesis. The hydrothermal synthesis successfully synthesized controllable properties as well as composition of ZnS particles. Chemical reagent sources of zinc such as zinc nitrate, zinc acetate, and zinc chloride were used, as well as polymeric-based materials such as polyvinylpyrrolidone and polyethylene glycol for stability (33, 34).

The hydrothermal technique was used to prepare ZnS to nano-scale particles of heterostructure of ZnS-based composite materials. To create a ZnS-based composite, two different chemical reagent sources of ZnS and other materials, such as carbon tubes and graphene, were chemically synthesised in situ using the in situ polymerization technique (35–37). To date, preparing ZnS particles on a smaller scale to be quantum dots has resulted in a wide range of applications.

Among the methods offered for the synthesis of metal sulphide nanoparticles, continuous hydrothermal synthesis in supercritical water (SCW) offers the best advantages and potential [38–39]. The size and characteristics of the particles can be controlled using this straightforward implementation and scaling mechanism. However the method's main flaw is its poor ability to manage the aggregation of nanoparticles. Yue et al.(40) reported Wurtzite ZnS nanowires can be synthesised hydrothermally at a low temperature (180 °C) in the presence of ethylenediamine (en). The samples' structure

and morphology are investigated, and the growth mechanism is discussed. The photoluminescence spectra shows a peak around 376 nm, which could be attributed to hole traps originating from the surface S atoms' unsaturated sp3 orbital. Peaks at 464 and 505 nm are associated with ZnS host defect-related emission. The TGA curve demonstrates that only a small amount of organic molecules remained bound to the washed ZnS nanowires.

2.3. Solid state reaction technique:

On solid state reaction, one of the effective routes for ZnS preparation was extensively considered. Fundamentally, solid state reaction was used to create polycrystalline solids from a mixture of solid starting materials (41, 42). Solids do not react together over normal time scales at room temperature, but the reaction can occur if the temperature is raised. In general, the temperature range for ZnS was estimated to be 1300 °C and higher. Reaction conditions, structural properties of reactants, surface area of solid and reactivity, and thermodynamic free energy of reactant are all factors that influence the feasibility and rate of a solid state reaction (43,44).The fundamental disadvantage was related to uncontrollable particle size and shape, as well as the process's high temperature. It is unsuitable for high production in industrial commercialization.Wang et al. (45) used novel solid-state method to synthesize zinc sulphide nanoparticles with various sizes through solid-state reaction of zinc acetate and thioacetamide at low temperatures. The preparation temperature ranged from room temperature to 300°C. X-ray diffraction (XRD), transmission electron microscopy (TEM), differential thermal analysis (DTA), and a photoluminescence spectrum were used to characterise the particles. The particles had a pure zinc-blende crystal structure, and the particle size increased with increasing temperature, according to X-ray diffraction patterns. The TEM micrograph revealed that the sample heated to 100°C had a mean particle size of about 40 nm. The photoluminescence emission spectrum showed a blue shift. Rita John et al. (46) synthesised ZnS nanoparticles from Zn $(CH3COO)_2$ H2O and Na_2S using a solid-state reaction method. The resulting particles are 11nm in size. The phase that was created has a cubic structure.

2.4. Wet chemical method: Modification of ZnS as composite based material

One of the strategic approaches considered in order to gain more and more feasibility on the use of ZnS was the design of ZnS-based polymer composite. To insert the other part of materials to ZnS can be provided the other layer of energy from the standpoint of optical-based materials. Many different types of subshell energy can be used to activate light. Modifications to ZnS quality may involve the design of ZnS and CdS compounds (47, 48). On the other hand, in order to broaden the use of ZnS for a wide range of applications, it would be preferable to focus on mechanical properties. ZnS was created through an in-situ polymerization process to increase flexibility. It was critical that the in-situ polymerization process of ZnS was regarded as a polymer as a matrix and ZnS was chemically synthesised as a particle. As a composite, it was well dispersed in the polymer matrix. This composite feature can currently be scaled down to the nano-range. The

most important aspect of ZnS-based composite was the significant improvement in properties and application. From a fundamental standpoint, ZnS was regarded as a ceramic-based material. It resulted in some specific applications requiring small displacement. To improve the ability to use ZnS, a small amount of this particle was embedded in polymer matrix. Many polymers have been used as matrix materials up to the present day. Maity et al.(18) used wet-chemical route to grow ZnS nanocrystals into the nanopores of a polyvinyl pyrrolidone (PVP) matrix on glass and Si substrates. The formation of cubic phase of ZnS nanocrystals into the polymer matrix was confirmed by X-ray diffraction (XRD) analysis. Changing the polymer mole fraction in the starting solution caused the particle size to vary. Transmission electron microscopy images and XRD studies both confirmed the formation of nanometer-sized particles within the polymer matrix. The average particle size was discovered to be between 4 and 7 nm. UV-Vis-NIR optical spectra demonstrated 90-95% transmittance in the visible and near infrared regions. The direct optical bandgap of the polymer-capped nano- ZnS film ranged from 3.87 to 3.98 eV for various PVP.

2.5. Microwave assisted synthesis:

The precipitation of ZnS nanoparticles was performed from homogeneous solutions of 0.05M zinc acetate ($Zn(CH3COO)_2 \cdot 2H2O$) and 0.05M sodium sulphide. Simultaneously, the mixture was slowly stirred and ultrasonicated in a round-bottom flask. The flask was then placed in a refitted domestic microwave oven, and a water-cooled condenser outside the microwave oven cavity was connected by a glass joint to the round-bottomed flask stably set inside, forming the so-called refluxing system. An aluminium lamella was placed over the glass joint to prevent microwave leakage. For 10 minutes, the reaction was microwave-irradiated (2450 MHz). After cooling to room temperature, the precipitate was centrifuged at 4000 rpm, washed several times with deionised water, and dried for about 10 hours at 100 degrees Celsius. Using this method Yang et al. (49) obtained 6.5 nm diameter crystallite size of pristine ZnS NPs where the size of the crystallites is independent of microwave irradiation time. It was also reported with increasing microwave irradiation time, the intensity of photoluminescence (PL) emission reaches a maximum and then decreases. Kashinath et al. (50) synthesized the assembly of nanosized ZnS particles grown on the surface of a 2D platform of reduced graphene oxide (RGO) sheets using the sol-gel method and treatment with a microwave irradiation technique was reported in this work. During this time, the RGO Sheets were formed simultaneously to act as a substrate, matrix, and fine template for the growth/decoration of isometric ZnS nanoparticles and graphene sheets.The size of the crystallites were varying between 1-4nm. Under UV light radiation, the nanocomposites were used as photocatalyst materials for the degradation of Brilliant Blue and Brilliant Yellow dyes. The findings indicate that the nanocomposites have higher photocatalytic activity than pristine ZnS and RGO due to their high specific surface area and the reduction of photo induced electron-hole pair recombination in ZnS. This is due to the inclusion of RGO and its synergistic effects. Reduced graphene oxide is an

excellent electron transporting material that effectively suppresses charge recombination in ZnS-RGO nanocomposites with significantly higher photoresponse activity.

2.6.Chemical precipitation method.

Chemical precipitation is the separation of a solid substance from a solution by either changing the form of the substance into a less soluble or insoluble one or decreasing the solubility of the substance by changing the solvent composition. Precipitation is regularly used to remove metal ions from solvents. Gayatri et al. employed Coprecipitation method to create ZnS nanoparticles using various capping agents such as PVA (polyvinyl alcohol) and PEG-4000 (polyethylene glycol). UV-Visible absorption spectra were used to calculate the optical band gap, which was found to be in the 3.0-3.4eV range. The particle size of nanoparticles calculated from an XRD pattern was in the 10-12 nm range. It has also been discovered that the nature of the capping agent influences the particle size of nanoparticles(51). Ashutosh K. Shahi et al. (52) used chemical precipitation to create small-sized ZnS particles by varying the concentration of the cationic surfactant N-cetyl-N, N, trimethyl ammonium bromide. This has resulted in small Zinc Sulphide nanoparticles (2-5nm).

Tamrakar et al.(53) utilising chemical precipitation technique have prepared zinc sulphide (ZnS) nanoparticles with varying concentrations of capping agent. The range of obtained particle size from XRD analysis is 2-3 nm. The particle size decreases as the concentration of capping agent increases. Optical absorption studies show that as the capping agent concentration increases, the absorption edge shifts towards the blue region, indicating that the effective band gap energy increases as particle size decreases. Photoluminescence (PL) experiments show that ZnS samples produce a single peak with a stoke shift. The PL emission peak is measured at 460 nm for uncapped nanoparticle. The PL spectra of ZnS nanoparticles with varying capping agent concentrations show that as particle size is reduced, the emission becomes more intense and shifts towards blue. Iranmanesh et al. (54) have also used the same method to obtain ZnS NPs with average diameter of the particle 5.5 nm. The PL spectrum consists of 428-448nm blue emission band.

2.7. Ball milling or Mechanochemical process:

The material is crushed by a strong mechanical force in the mechanochemical reaction method (Fig. 3), resulting in the formation of a different structure. Currently, a typical planetary ball mill machine employing a mechanochemical process for material crushing is in use. Grinding is accomplished by continuously rotating the large surface and, as a result, the containers. The plate's centrifugal speed and the container's planetary (rotation) speed can be adjusted independently. The collision of balls is important in the transfer of raw materials during this grinding process. When the crystallites receive energy, they rupture. This resulted in a reduction in particle size and an increase in surface area and surface energy. Such a collision effect can cause detectable structural changes and even chemical reactions in materials, known as mechanochemical reactions (55).The mechanochemical method can be used to produce large quantities of materials in a

short period of time, particularly for industrial purposes. T. Tsuzuki et al. (56) reported the size of obtained nanoparticles via this method is between 4-20nm although particle sizes are highly depend upon the milling condition. Pathak et al. (57) used the ball milling method to create ZnS nanoparticles from Zn $(CH3COO)_2$ and Na_2S as initial starting components. ZnS nanoparticles have a size of about 2 nm and an optical band gap of about 4.71-5.17eV. Their XRD report demonstrated that the ZnS material has a cubic structure. Pathak et al. (58) used a mechanochemical route to create ZnS nanoparticles from zinc acetate and sodium sulphide as precursors. The resulting ZnS nanoparticles have a size range of 4-7 nm and an optical band gap of 4.04-4.6 eV. EDAX spectrum analysis confirmed the composition of ZnS nanoparticles. The cubic phase structure of the synthesised ZnS nanoparticles was identical to that of standard cubic ZnS. Jianfeng Chen et al. (59)**.**used a rotating packed bed reactor to prepare ZnS nanoparticles using a mechanochemical method (RPBR). As precursors, they used Zn $(NO_3)_2$ solutions and H2S gas. As a result, the obtained nanosized ZnS has significantly improved absorption capacity for light with wavelengths ranging from 200 to 330 nm. They also discovered ZnS NPs with cubic phase Hamaguchi et al.(60) reported ball-milling method can be employed to create ZnS:Mn^{2+} nanoparticles and other sulpher-based ternary compound nanoparticles. Comminution of materials in a solvent resulted in several-nanometer-sized ZnS:Mn^{2+} nanoparticles. X-ray diffraction (XRD) and transmission electron microscopy (TEM) both indicated that the particles were milled down to nanometer size. The ball-milling process had no effect on the stoichiometry of the starting powder, according to TEM-energy-dispersive X-ray spectroscopy (TEM-EDX). The orange-colored luminescence emitted by Mn^{2+} was enhanced by reducing particle size to a few nanometers. This technique was also used to investigate red, green, and blue (RGB) coloured phosphors. Without affecting the crystal structure or stoichiometry.

3. Conclusion:

Various efforts have been made to date to conduct extensive research on ZnS materials. Many synthetic routes and modifications on physical and chemical properties have been extensively researched. To prepare ZnS-based materials, sol gel formation, hydrothermal technique, and solid state reaction, microwave synthesis, chemical precipitation, Mechanochemical etc were successfully used.. This review report provides an in-depth look at ZnS nanomaterials, its unique properties, and its main applications. More emphasis is placed on luminescence properties, as well as physical and chemical properties prepared using a variety of techniques. The enhancement of Several doping impacts the luminescent properties greatly. The variation of capping agent tailored the size of the particle which significantly blue shifted and enhances the luminescence intensity as size of nanocrystallites reduced. The ZnS-based polymer composite gives the more feasibility and flexibility to its structural morphology and characteristic as well. In electronic applications,

structural morphology and sizes play an important role. Although this field has made many advances, several key issues have emerged that must be addressed before practical applications can be implemented.

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