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SYNTHESIS OF ORTHORHOMBIC SnS NANOFLOWER WITH THE WELL - CRYSTALLIZED MORPHOLOGIES

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ABSTRACT

In this study we synthesized orthorhombic SnS nanoflower with the well - crystallized morphologies as solid flower particles via solvothermal method. The synthesized samples were characterized with UV, XRD, FTIR. The SnS nanoflower structured particles have been solvothermal synthezised by using thiourea and $Sncl_2.2H_2O$ as raw materials and the solvent ethylene glycol with addition of capping agent acetic acid. The effect of capping agent on SnS nanoparticles change it morphology from sphere to flower with the size reduction of the particle.

Keywords: Synthesis, Orthorhombic, Nanoflower, UV, XRD, FTIR, Nanoparticles

INTRODUCTION

The study, manipulation, and engineering of materials, particles, and structures on the nanoscale scale is referred to as nanoscience (one millionth of a millimeter, the scale of atoms and molecules). The way molecules and atoms organise on the nanoscale into bigger structures determines important aspects of materials such as electrical, optical, thermal, and mechanical properties. Furthermore, because quantum mechanical effects become relevant in nanometer-sized structures, these features frequently diverge from those on the macroscale.

Nanoscience is study of phenomena & manipulation of materials at atomic, molecular & macromolecularscales. Their physical and chemical properties exhibited at atomic/molecular levels of dimensions ranging from few nanometers to less than 100nm; because at this scale properties differ significantly from those at a larger or bulk scale. Nanoparticles are fundamental units of Nanotechnology, They are used as raw materials for constructing Nanostructures, Nanomaterials, Nanomachines and Nanodevices. Nanoparticles may be defined as “A particle with at least one dimension of 100nm or smaller". Novel features that distinguish nanoparticles from bulk materials often emerge at a crucial length scale of less than 100 nm. [1]

The field of nanoscience and nanotechnology is truly interdisciplinary, and it is an emerging science that has the potential to impact nearly every aspect of human life, including communications, computing, textiles, cosmetics, sports, biomedical therapy, automobiles, environmental monitoring, fuel cells and energy devices, water purification, food and beverage industry, and so on. [2].

The abilityto tune objects in nanoscale forms is one of the exciting prospects in nanoscience research. It showsgreat potential for providingus in futurewith various breakthroughs thatwill revolutionize direction of technology in a wide range of applications. By improving material properties, investigators are able to find the applications for making different devices such as electronic devices, sensors, special polymer based membranes and magnetic devices etc in Fig 1.[3]

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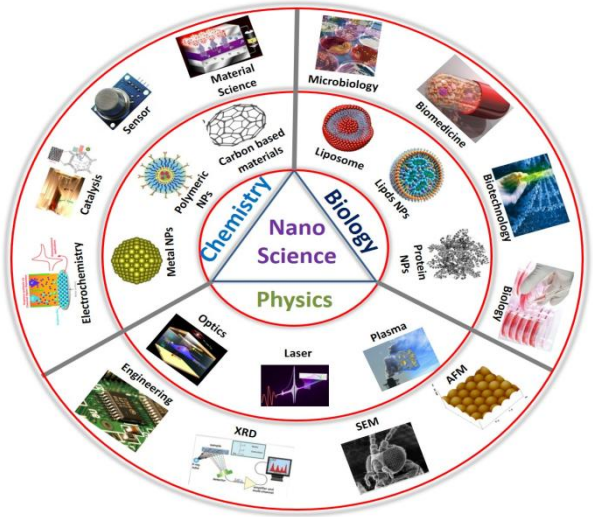

FIG 1 APPLICATIONS OF NANOSCIENCE AND NANOTECHNOLOGY

Band gap designed materials are quantum devices utilised in a wide range of electrical applications. Band gap engineering is a broad phrase that refers to the synthetic tailoring of band gaps in order to produce unexpected electrical transport and optical phenomena, as well as innovative technologies. [4]

Obviously, majority of semiconductor nanostructure-based quantum devices are band gap designed. The use of nanomaterials in the medical field is known as nanomedicine or nanobiomedical technology. This application uses nanometre scale materials & nano techniques to diagnose, treat, and prevent diseases such as cancer, musculoskeletal diseases, cardiovascular diseases, inflammatory conditions, neurodegenerative diseases, psychiatric diseases, diabetes, infectious diseases, bacterial & viral infections, & others.

These also include drug delivery systems & pharmaceuticals; new diagnostic tools; therapies; implants, tissueengineered constructs; imaging agents and methods. Nanomaterials offer various ways to reduce, prevent,sense and treat environmental pollution. Because nanotechnology enhances current technologies, the best ones can be developed. Nanomaterials can also be programmed to actively interact with a contaminant and degrade it into less dangerous forms. Any such breakthroughs will eventually rely on today's scientific research, which is setting the groundwork for tomorrow's nanoscience and nanotechnology on a daily basis.

COPPING AGENT

Metal chalcogenides were produced and reported in nano particle form utilizing a variety of methods. Tin chalcogenides SnS compounds, which belong to the periodic table's semiconductor IV-VI group, have been revealed to be promising candidates foroptoelectronic & solar cellapplications [5,6].

Tin atomsheets are sandwiched b/w tightly packed sulphuratom sheets to form SnS. Nanotechnology has evolved quickly and has been successfully employed in practical applications. Because of tiny bandgap properties of IV-VI semiconductors, optical activity in the infrared (IR) and near infrared (NIR) has received a lot of attention. It also has the benefit of having many constituent materials in nature and digesting any insignificant health & environmental issues. The current emphasis of researchers is on the fabrication of nanostructure materials with specific nano morphologies based on literature. SnS

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nanostructure particles were created by hydro thermal [7], co-precipitate [8], aqueous solution [9], sol gel processes, and other approaches.

Nanotechnology is manipulationof materials and systems at atomic & molecular levels, resulting in usage of nanomaterials in a wide range of applications. Because nanotechnology provides radically different properties than their macroscopic or bulk counterparts, new materials and new technologies have found applicationsin a widerange of areas.

SnS nano particles are generated in this study using a solvo thermal process with acetic acid as a capping agent [10]. Which modifies surface of nanoparticles and limits particle development to greater sizes [11]. For these samples, the influence of capping agent on optical absorption spectra has been examined utilizing XRD, FTIR, SEM, DLS, and TEM examinations. Due to the benign nature of SnS nano particles, bio-medical applications are being investigated.

OBJECTIVES OF THE STUDY

- To synthesize orthorhombic SnS nanoflower with the well - crystallized morphologies
- To synthesize SnS nanoparticles using capping agents by solvothermal method

LITERATURE REVIEW

Spray pyrolysis was found by N.K. Reddy and K.T.R.Reddy to create polycrystallineSnS films. Spray pyrolysis was utilised to form thin layers of tin monosulfide (SnS) onCorning 7059 glass substrateswith substratetemperatures varied from 300 to 350°C and other depositionparameters held constant. Thefilms' composition, structure, electricalresistivity, & optical energygap were all assessed. [12]

S. Schlecht and L. Kienle provide a straightforward solvothermalsynthesis of SnSnanoparticles from activated tinmetal & elemental sulphur. Uncoated Herzenbergite particles 20 40 nm in size were generated utilising diethyleneglycoldimethylether (diglyme) as a solvent at a reactiontemperature of 160° C. Powder X-ray diffraction (XRD), EDX analysis, & TEM investigation confirmed that SnS is single product.[13]

H. Su et al. devised a simple ethanol thermal method for manufacturing and controlling the morphology of rod-like nanocrystalline tin sulphides. At a low temperature (100°C for 10 hours), simple ethanol thermal reactions with tin chlorides & thioacetamide produced rod-like nanocrystalline tin sulphides of various compositions & phases. Solvents, reaction temperatures, & trace water in the system were shown to be important determinants in the formation of one-dimensional nanocrystalline tin sulphides. The mechanism of the reaction has also been examined. [14]

Shen G. et al. developed a new polyol method for flaky nanoscale tin sulphides. A unique mild solution approach, the polyol pathway, was developed to synthesise nanoscale tin sulphides (SnS & SnS2) flaky crystallines. The XRD technique shows that goods are beautifully crystalline. The commodities exhibit a constant flaky structure with unequal drapes, according to TEM examination. Furthermore, the most likely mode of manufacturing is investigated.[15]

Using thiourea hydrolysis, Rao M.M. et al. examine the time-selective hydrothermal synthesis of SnS nanorods & nanoparticles. In an autoclave at 180 °C for 2 and 8 hours, respectively, thiourea hydrolysis was utilised to make SnS nanorods and nanoparticles. The synthesis of nanorods in absence of a structure-directing agent

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was attributed to the templating property of ammonium ion generated in situ during hydrolysis.[16]

Khel et al. investigate formation of SnS thin films on an aluminium plate using normal electrochemical deposition. ECD was utilised to produce SnS thin films onto aluminium sheets from aqueous solutions comprising $SnSO_4$ and Na $2S_2O_3$. The deposited SnS was polycrystalline & orthorhombic in structure, with an S-rich composition in acidicpH and Sn-rich composition in higher pHvalues. The relationship b/w film properties and deposition parameters was investigated in order to optimise deposition condition. [17]

A seedless solution approach was utilised by Greyson et al. to manufacture tin sulphide (SnS) nano- and microcrystals. The ZB structure produced three-dimensional tetrahedral particles, whereas the orthorhombic phase produced two-dimensional platelike particles. Although the optical characteristics of the orthorhombic plates were equivalent to those of bulk SnS, the absorption edge of the ZB tetrahedra was roughly 300 nm bluer. [18]

C. Zhai et al. look into the large-scale manufacturing of ultrathin hexagonal tin disulfide nanosheets with extremely reversible lithium storage. A simple hydrothermal process is used to create ultrathin hexagonal SnS_2 nanosheets. The nanosheets were used as an anode in a lithium-ion battery and shown great reversibility and cycling stability, with 96% capacity retention after 50 cycles. [19]

Li Ren et al. used a triethanolamine-assisted diethylene glycol solution synthesis to produce and characterise SnS nanocrystals. Stoichiometric, phase-controllable SnS nanocrystals (NCs) were synthesised at injection temperatures ranging from 180 to 220°C using a triethanolamine-assisted diethylene glycol solvent, tin(II) chloride, & thioacetamide as precursors. The effects of triethanolamine additive amounts, injection temperature, and refluxing time on SnS NC crystal phase, growth morphology, and optical characteristics were investigated. The findings showed that by adjusting the amount of triethanolamine, orthorhombic (OR) & zinc-blende (ZB) phases of SnS NCs may be generated.[20]

The development of catalytic olefin oxidation is described by Zhang Yongmei and You Hongjun. The catalytic oxidation of olefins synthesis method is also discussed in detail, including catalytic gas-phase oxidation of olefins, catalytic liquid-phase oxidation of olefins, catalytic oxidation of enzyme, catalytic oxidation of olefins using sodium hypochlorite and hydrogen peroxide as the catalyst, and directly catalytic oxidation of olefins. When hydrogen peroxide is employed as the catalyst, there is no pollution and no danger to the public. [21]

Ying Xu et al. look into the Synthesis & Size Dependent Reflectance of Water Soluble SnS Nanoparticles. A facile, one-step solution-based method based on ethanolamine ligands yields near-monodispersed water soluble SnS nanoparticles with sizes ranging from 3-6 nm. The optimal dosage of triethanolamine is being studied. It is discussed how further heat treatment influences size of these SnS nanoparticles. Diffuse reflectance study of SnS nanoparticles verifies the predictions of the quantum confinement paradigm.[22]

Hongrui Peng and coworkers, The researchers created morphologically controlled tin sulphide nanostructures. The effect of synthetic factors like reaction temperature and sulphur sources on tin sulphide morphologies has been studied. Using an effective solvothermal approach, researchers developed a variety of tin sulphide nanostructures such as nanobelts, nanorightangles, nanorods, and nanosheets. SnS nanobelts have a

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thickness of less than 30 nm and a breadth of 50-300 nm. Two nanobelts combine to form a nanorightangle. [23]

X-raydiffraction (XRD), secondary electronmicroscopy (SEM), diffuse reflectance spectroscopy, & FTIR were utilised by W. A. A. Syed et al. to examine structural, morphological, and optical aspects, respectively. The co-precipitation approach was used to create nanoparticles of tin mono sulphide (SnS), a critical and ecologically acceptable material for use in optoelectronic devices. XRD and SEM measurements of phase and particle size reveal homogeneous spherical shaped nanoparticles with sizes ranging from 44 to 57 nm. The computed direct and indirect energy band gaps are 3.2 and 2.7eV, respectively. The molecule's functional groups were explored further using FTIR, and the results are consistent with previous research.[24]

Mukesh Upadhyay et al. explored how tin sulphide nanoparticles were successfully synthesised using a wet chemical process, as well as their structural properties. The nanoparticles were examined using X-raydiffraction (XRD) and Raman spectroscopy. According to XRD data, Tin Sulfide nanoparticles have an orthorhombic structure and a particle size of around 12.72nm. The Raman spectrum reveals frequency of phonon in these nanoparticles, & it was revealed that the Raman modes of Tin Sulfide nanoparticles travel towards the lower wave number side.[25]

Hudson, Benjamin Baby, and colleagues present the single phase synthesis of orthorhombic SnS nanorods in a solvothermal process with reduced reaction time and temperature using CTAB as a surfactant. The microstructure study investigates how CTAB concentration, initial mixing, & reaction time impact orthorhombic SnS phase stability. Morphological, optical, & electrical studies confirm the controlled growth of SnS nanorods with an energybandgap of 1.6 eV, p-type conductivity, resistivity of 106 Ω cm, & carrierconcentration of 1012 cm-3. Because of the prolonged reaction time, crystalline Sn2S3 and SnS with a red shift in direct energy band gap were produced. [26]

In this study, Hamit ztürk and Ferhat Aslan proved that exceptional grade SnS powder can be created using a powder preparation technique at extremely low temperatures. Thermal evaporation was used to deposit SnS layers from the resultant powder. After that, SnS layers were formed on molybdenum-coated and untreated glass substrates. It was discovered that the crystalline structures of films produced on molybdenum coated substrates were improved. SnS thin films exhibit p-type conductivity and may be used in heterojunction solar cells and self-powered photodetectors with a forbidden band gap of 1.5 eV, according to optical and electrical studies. [27]

MATERIALS

Sigma Aldrich provided the first ingredients, which included stannouschloride dehydrate ($Sncl_2.2H_2O$, 98%), thiourea ($CS(NH_2)_2$) 99%, ethylene glycol, acetic acid, and 100% ethanol. As a solvent, ethylene glycol was employed, while acetic acid was used as a capping agent. All of chemicals employed were of high purity analytical grade & did not require additional purification.

SYNTHESIS OF SnS NANOFLOWER

Via acetic acid as capping agents, SnS nano particles were produced using a solvo thermal approach. The concept at work in this approach is precipitation of metal ions insolution with sulfide ions. In presence of ethylene glycol as asolvent and cappingagent of acetic acid 2ml, 5mmol aqueous solution of stannous chloride dihydrate and 7mmol of thiourea were combined together and agitated for 1 hour on a magnetic stirrer to get a

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homogenoussolution. The solutionwas put into a100 cc stainless steelautoclave reaction chamber coated with Teflon. The autoclave champer was sealed and placed in an electrical furnace for six hours at a temperature of 1800 C. A black precipitate was formed after centrifugation and multiple washes with double distilled water & ethanol solutions. To get a powder sample, the precipitate was dried in anoven at 70°C for 2 hours.

RESULT AND DISCUSSION

XRD ANALYSIS

X-Ray Powder Diffraction(XRD) apparatus isan essential analytical technique forphase identification & information on crystalline material unit cell dimension. The Bragg's Law condition, constructive interference, and adiffracted ray created incident rays interact with the sample. The X-Ray diffraction (XRD) pattern was used to assess the phase & purity of as-obtained tin sulphide material. XRD spectrum of flower-shaped SnS nanoparticles produced by solvothermal technique with acetic acid as capping agent. The measured diffraction peaks for the produced SnS nanoparticles of flower can be properly indexed to orthorhombicstructure of SnS with lattice a= 4.32, b=11.18, and c= 3.98. The presenceof strong peaks in XRD pattern indicates that the solvothermal product of SnS nanoflower was successfully crystallized with the pure SnS phase. Which contain diffraction planes matching to (1 2 0), (1 1 1), (0 4 0), (1 3 1), (2 2 0), and (151) and are in good agreement with JCPDS Card No: 39-0354 [28].

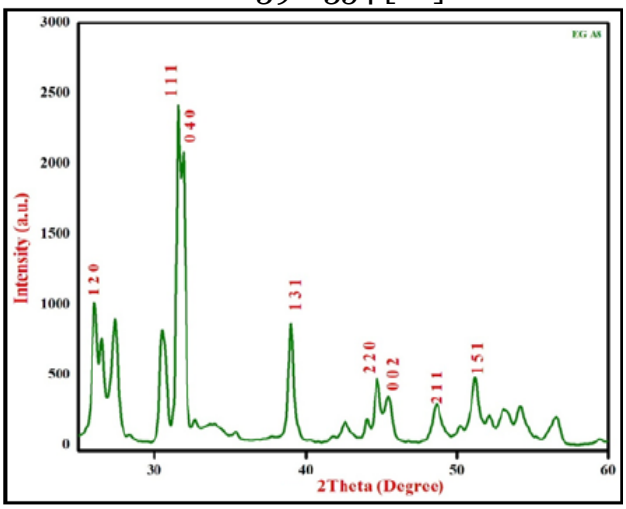

FIG 1 X-RAY DIFFRACTION SPECTRUM OF SNS NANOFLOWER PARTICLES

The planes (1 1 1) and (0 4 0) in Fig. 6.1 confirm the coexistence of SnS crystal planes with layeredstructure and layer planeperpendicular to crystallographic from literature review suggests the dominating planes peaks indexed for orthorhombic crystal structure. The absence of further impurity peaks confirms formation of a single pure phase tin monosulphide [29].

UV-ANALYSIS

When nanoparticles interact with infrared light,molecules undergovibrational transitions; nevertheless, shorter wavelength, higherenergy radiation in visible (400-700 nm) and infrared (200-400 nm) portions of electromagnetic spectrumcauses many molecules to undergoelectronic alterations. When a molecule absorbs UV or visible light energy, one of its electrons shifts from a lower energy molecular orbital to a higherenergy molecularorbital. According to the optical absorption investigation at SnS nano flower

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particles, the quantity of capping agent used during the synthesis regulates the optical electronic state transition in the SnS nano flower particles.

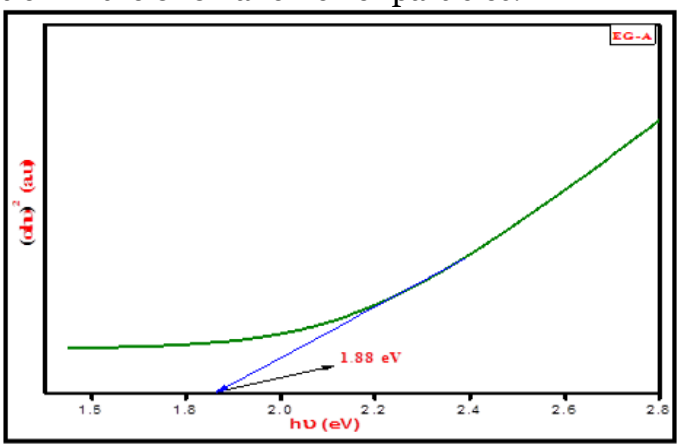

FIG 2 TAUCPLOT OF SNS NANOFLOWER PARTICLES

A 2% acetic acid capping agent was used in the manufacture of SnS nano flower particles, resulting in an energy gap of 1.8eV. It suggests that band gap energy is inverselyproportional to size of nano particles and has an increase dependent property of 1.8 eV [30].

As a result, increase in band gap with addition of a capping agent implies that the size of nano particles may be controlled. As particle size reduces, the material's band gap energy increases, confirming the quantum confinement of quantum effect [31].

FTIR ANALYSIS

FTIR Spectroscopy provides a wide range of analytical options in the laboratory. When IR radiationis delivered through asample, part of it is absorbedby sample & some goes through the detector, resulting in a spectrum at the detector. The typical FTIR absorption bands of SnS nano particles are depicted in Fig 3 [32].

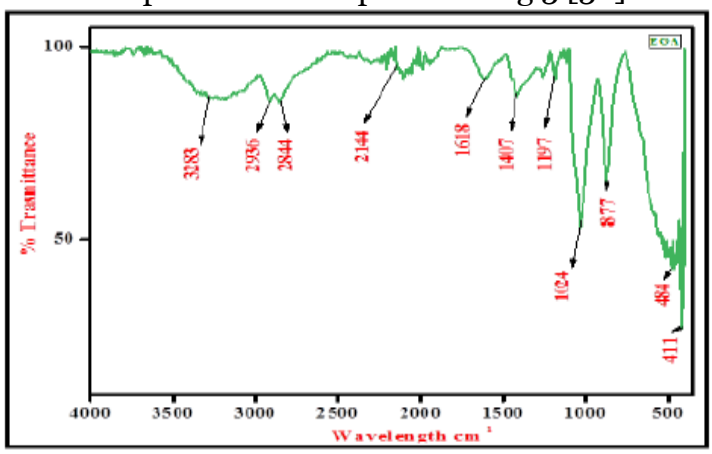

FIG 3 FOURIER TRANSFORM INFRARED SPECTRUM OF SNS NANOFLOWER PARTICLES

Peaks linked to O-H bending modes were found at 1618 cm-1, which might have been caused by surface hydroxyls. The line at 1407 cm-1 is attributed to C-OH vibrations, whereas bands at 1024 cm-1 & 1197 cm-1 indicate presence of inorganic compounds. Theband at 2936 cm-1 - 2844 cm-1 confirms the presence of carboxylic acid, revealing the involvement of morphological change in the SnS nano flower synthesis [33].

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CONCLUSIONS

In summary, we have successfully prepared SnS nanoflower particles by using a solvothermal method. The nanoparticles were characterized with XRD, UV and FTIR. The results of the XRD analyses show that the nanoparticles have an average size of 4.5 nm, X-Ray Diffraction as well as the corresponding structures are measured lattice parameters are a = 4.3283Å, b = 11.2015Å, and c = 3.9925 Å for orthorhombic SnS. The optical behavior and FTIR results agree well with the presence of function group of Sn-S.

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