Journal of Advanced Zoology

ISSN: 0253-7214

Volume 44 Issue-3 Year 2023 Page 1599:1606

COMPARATIVE PERFORMANCE ANALYSIS OF SOL–GEL METHOD AND HYDRO THERMAL METHOD FOR ZINC OXIDE NANOPARTICLE SYNTHESIS

[1]MADHAVI PRASANGI, [2]Dr.N.JAYA MADHURI

[1]Research Scholar, Department Of Physics, Jawaharlal Nehru Technological University, Ananthapuram, Andhra Pradesh, India.

[2]Assistant Professor, NBKR Institute Of Science And Technology, Vidyanagar, Nellore District, Andhra Pradesh, India

Article History

Received: 06 Sept 2022
Revised: 05 Oct 2023
Accepted:11Nov 2023

ABSTRACT: Nanomaterials have emerged as an amazing class of materials that consists of a broad spectrum of examples with at least one dimension in the range of 1 to 100 nm. Exceptionally high surface areas can be achieved through the rational design of nanomaterials. Nanomaterials can be produced with outstanding magnetic, electrical, optical, mechanical, and catalytic properties that are substantially different from their bulk counterparts. There has been considerable focus on the synthesis of metal nanoparticles with distinct and exceptional characteristics, making it a highly captivating area of research. Among these nanoparticles, Zinc Oxide NanoParticles (ZnO NPs) have gathered significant attention in the field of bioengineering. Various approaches have been explored for the preparation of ZnO nanoparticles. In this paper we are comparing two synthesis methods as Sol–Gel Method and Hydro Thermal Method for Zinc Oxide Nanoparticles. Sol-gel method based ZnO nanoparticles are compared with Hydrothermal based ZnO nanoparticles in terms of XRD, UV Visible Spectrum and SEM analysis. Sol-gel method based Al-doped Zinc Oxide Nanoparticle (AZnO) is compared with Hydrothermal based Al-doped Zinc Oxide Nanoparticle (AZnO) in terms of XRD, UV Visible Spectrum, SEM analysis and optical properties. At last Sol-gel method based Au-doped ZnO- Sm (NO3)3 is compared with Hydrothermal based Au-doped ZnO nanoparticles in terms of optical properties as transmittance and absorbance, structural properties as XRD analysis. From this paper we can conclude that, Sol-gel method based Au doped ZnO nano particles are efficient than Hydro thermal based ZnO nano particles.

KEYWORDS: Zinc Oxide Nanoparticle, Sol–Gel Method, Hydro Thermal Method, AZnO, UV Visible Spectrum, XRD and SEM analysis

.

I. INTRODUCTION

Nanomaterials have emerged as an exciting class of materials that are in high demand for a range of practical applications [1]. The length of a nanometer can be understood through the example of five silicon atoms or 10 hydrogen atoms lined up, which is one nanometer. Materials are defined as nanomaterials if their size or one of their dimensions is in the range of 1 to 100 nm.

The exact history of the utilization of nanosized objects by humans is difficult to clarify. However, the history of nanomaterial utilization is ancient, and human beings used these materials a long time ago for various applications, unknowingly. About 4500 years ago, humans exploited asbestos nanofibers to reinforce ceramic mixtures [2].
Nanotechnology is an excellent example of an emerging technology, offering engineered nanomaterials with the great potential for producing products with substantially improved performances [3]. Currently, nanomaterials find commercial roles in scratch-free paints, surface coatings, electronics, cosmetics, environmental remediation, sports equipment, sensors, and energystorage devices.14 This review attempts to provide information in a single platform about the basic concepts, advances, and trends relating to nanomaterials via covering the related information and discussing synthesis methods, properties, and possible opportunities relating to the broad and fascinating area of nanomaterials [4].

Chemical synthesis is one of the most important techniques which can be performed by using a range of precursors and different conditions like temperature, time, concentration of reactants, and so forth. Variation of these parameters leads to morphological differences in size and geometries of resulting nanoparticles [5].

Two main approaches are used for the synthesis of nanomaterial: top-down approaches and bottom-up approaches. In top-down approaches, bulk materials are divided to produce nanostructured materials. Top-down methods include mechanical milling, laser ablation, etching, sputtering, and electro-explosion. Bottom-up approaches include Chemical vapor deposition (CVD) [6], Solvothermal and hydrothermal methods [7], Sol–gel method, Soft and hard templating methods and Reverse micelle methods.

The properties of matter at the nanoscale level are substantially distinct compared to bulk counterparts. Size-dependent effects become more prominent at the nanoscale. For example, Au solution appears yellow when in the bulk and it appears purple or red at the nanoscale level. The properties of nanomaterials can be tuned via tuning the nanomaterial size.92,93 At the nanoscale, the electronic properties are substantially changed compared to bulk materials [8]. For example, boron in bulk form is not considered a metal, whereas a two-dimensional network of boron (borophene) appears to be an excellent 2D metal.94 Compared to their bulk counterparts, the mechanical properties of nanomaterials are considerably improved due to increases in crystal perfection or reductions in crystallographic defects.

The optical properties of nanomaterials such as quantum dots strongly depend upon their shape and size.96 A photogenerated electron–hole pair has an exciton diameter on the scale of 1–10 nm. Thus, the absorption and emission of light by semiconductors could be controlled via tuning the nanoparticle size in this range. However, in the case of metals, the mean free path of electrons is B10–100 nm and, due to this, electronic and optical effects are expected to be observed in the range of B10–100 nm. The colors of aqueous solutions of metal nanoparticles can be changed via changing the aspect ratio.

Zinc oxide is the one of the most important n-type semiconductor materials with a 3.37 eV band gap at room temperature and 60 meV excitation binding energy that is in the

UV region and makes this nanoparticle as an efficient UV absorber [9]. Semiconductor nanomaterials have been received great attentions. Among these various semiconductor nanomaterials zinc oxide is a versatile material because of its physic-chemical properties such as mechanical, electrical, optical, magnetic and chemical sensing properties. Zinc oxide a chemical compound found naturally in the mineral called zincite has attracted much attention in recent times due to its low cost and because it can be obtained by simple techniques [10].

Among the nanoscale metal oxides, zinc oxide is a common host material that has been widely used due to its excellent chemical and thermal stability, low cost and environmental-friendliness. The high exciton binding energy of ZnO would allow for excitonic transitions even at room temperature, which could mean high radiative recombination efficiency for spontaneous emission as well as a lower threshold voltage for laser emission. The lack of a centre of symmetry in wurtzite, combined with a large electromechanical coupling, results in strong piezoelectric and pyroelectric properties and hence the use of ZnO in mechanical actuators and piezoelectric sensors.

ZnO nanostructures have a great advantage to apply to a catalytic reaction process due to their large surface area and high catalytic activity. Since zinc oxide shows different physical and chemical properties depending upon the morphology of nanostructures, not only various synthesis methods but also the physical and chemical properties of synthesized zinc oxide are to be investigated in terms its morphology. The demonstration of room temperature ultraviolet lasers, field effect transistors and field emission arrays based on ZnO nanorods have stimulated great interest in developing functional nanodevices. Moreover, the wide range of morphological diversity in the nano-regime has made this material a promising candidate in the field of nanotechnology and opened up new possibilities for the fabrication of high performance devices based on these nanostructures. The Sol–gel method is becoming one of the most important tools for advanced materials processing, particularly owing to its advantages in the processing of nanostructural materials for a wide variety of technological applications such as electronics, optoelectronics, catalysis, ceramics, magnetic data storage, biomedical, bio photonics, etc.

II. COMPARATIVE PERFORMANCE ANALYSIS

In this paper we are comparing two synthesis methods as Sol–Gel Method and Hydro Thermal Method for Zinc Oxide Nanoparticles.

1. Sol-gel method based ZnO nanoparticles versus Hydrothermal based ZnO nanoparticles:

Sol-gel method based ZnO nanoparticles are compared with Hydrothermal based ZnO nanoparticles in terms of XRD, UV Visible Spectrum and SEM analysis. These comparisons are represented in Table 1.

Table 1: COMPARATIVE PERFORMANCE ANALYSIS FOR ZnO NANOPARTICLES

Characterization Techniques	Sol-gel method based ZnO nanoparticles	Hydrothermal based ZnO nanoparticles
XRD	The nanoparticles average size is determined as14.36 nm. The size of the smallest crystallite is obtained by	Mostly spherical nanoparticles with average diameter of 14.5 nm was synthesized

	utilizing chloride as precursor at highest temperature of synthesis.	
SEM	The particles agglomeration is observed at higher resolution of SEM image.	Results indicate that agglomeration has taken place in the form of spherical.
UV Visible Spectrum	In the UV range radiations are absorbed by sample up to 362 nm and most of the visible spectrum radiations are transmitted through ZnO NPs	UV range radiations are absorbed by sample up to 368nm

X-Ray Diffraction (XRD) Spectrum

Sol-gel method:

The XRD spectrum of synthesized zinc oxide NPs and confirmed that, structure of wurtzite is hexagonal. The peaks of characteristics (100), (002), (101), (102), (110), (103), (200), (112), and (201) related to ZnO hexagonal structure (JCPDS Card no 01-075-1526) with the preferred orientation along (101) the plane.

The nanoparticles average size is determined as14.36 nm. The crystallite size calculation results using FWHM (Full Width at Half Maximum) technique from the spectrum of XRD exhibited that for (101) plane the size of the crystal is a function of precursor type and temperature of synthesis. The size of the smallest crystallite is obtained by utilizing chloride as precursor at highest temperature (90^{o}C) of synthesis.

Hydrothermal method:

XRD patterns of ZnO nanoparticles from hydrothermal method. The diffraction peaks are at 2θ values of 31.9456^0, 34.5903^0, 36.4341^0, 47.6971^0, 56.7894^0, 63.0411^0 and 68.1135^0. The peaks are identified to originate from (100), (002), (101), (102), (110), (103) and (112) planes. Based on the Scherrer equation the average crystallite size of the nanoparticles are observed as 14.5 nm.

Scanning Electron Microscopy (SEM)

Sol-gel method:

The SEM image of ZnO nanparticles are in spherical shape and have granular behavior. The particles agglomeration is observed at higher resolution of SEM image. Agglomeration is caused by aging.

Hydrothermal method:

The zinc oxide particles prepared are spherical shape using hydrothermal method. It also shows that a network formation of the zinc oxide nanoparticle has taken place which clearly indicates that agglomeration has taken place.

UV Visible Spectrum (UV-VIS)

Sol-gel method:

In the UV range radiations are absorbed by sample up to 362 nm and most of the visible spectrum radiations are transmitted through ZnO NPs. The energy of band gap is obtained through exploiting the curve is determined as 3.3 eV.

Hydrothermal method:

The UV-visible absorption spectrum of zinc oxide nanoparticle obtained from hydrothermal method. The absorption edge takes the value around 368 nm for zinc oxide nanoparticles prepared by hydrothermal method.

2. Sol-gel method based Al doped ZnO nanoparticles versus Hydrothermal based Al doped ZnO nanoparticles:

Sol-gel method based Al-doped Zinc Oxide Nanoparticle (AZnO) is compared with Hydrothermal based Al-doped Zinc Oxide

Nanoparticle (AZnO) in terms of XRD, UV Visible Spectrum, SEM analysis and Optical properties. These comparisons are represented in Table 2.

Table 2: COMPARATIVE PERFORMANCE ANALYSIS for Al DOPED ZnO NANOPARTICLES

Parameters	Sol-gel method based Al doped ZnO nanoparticles	Hydrothermal based Al doped ZnO nanoparticles
XRD	The outcome demonstrates the excellent purity of the synthesized Al doped ZnO. Strong peaks in the pattern point and highly crystallized and oriented nature of the synthesized zinc oxide.	Based on the Scherrer equation the average crystallite size of the nanoparticles is observed as 18 nm. Moderate impurities were observed at peaks.
SEM	SEM analysis reveals a non-uniform surface of the films. As a result of the phenomenon, the Al-ZnO protecting is more resistant.	Agglomeration has taken place as spherical or flowers.
UV Visible Spectrum	The ZnO suspension's UV-visible absorption spectrum shows an absorption peak at 360nm	UV-visible absorption spectrum shows an absorption peak at 367 nm
Optical properties	A decrease in absorbance at higher doping level of Al may be attributed to the decrease in the size of nanoparticles and increase in porosity which ultimately widens the band gap. The optical transmittances of Al doped Zno are 88% and 87% respectively.	Size of nano particles are decreased slightly with increasing the doping levels in Hydrothermal method, which simultaneously decrease the absorbance. The optical transmittances of Al doped Zno are 84% and 86% respectively.

X-Ray Diffraction (XRD) Spectrum:

Sol-gel method:

XRD peaks match the zinc oxide structure seen in wurtzite, where zinc atoms are found in the tetrahedral positions of oxygen atoms that have been packed tightly into hexagonal shapes. The outcome demonstrates the excellent purity of the synthesized Al doped ZnO. Strong peaks in the pattern point and highly crystallized and oriented nature of the synthesized zinc oxide.

Hydrothermal method:

Based on the Scherrer equation the average crystallite size of the nanoparticles is observed as 18 nm. Moderate impurities were observed at peaks.

Scanning Electron Microscopy (SEM)

Sol-gel method:

The surface topography of the films with the fifth, tenth, fifteenth, and twentieth layers is displayed in the SEM images. SEM analysis reveals a non-uniform surface of the films. As a result of the phenomenon, the Al-ZnO protecting is more resistant.

Hydrothermal method:

Agglomeration has taken place as spherical or flowers.

UV Visible Spectrum (UV-VIS)

Sol-gel method:

The ZnO suspension's UV-visible absorption spectrum shows an absorption peak at 360 nm that is part of a different band for ZnO.

Although the ZnO nanoparticle's size of the UV-vis absorption band affects, it typically ranges from 330 to 380 nm. Here, the presence of a peak at 360 nm can serve as proof that ZnO NPs are present.

Hydrothermal method:

UV-visible absorption spectrum shows an absorption peak at 367 nm.

Optical Properties:

Sol-gel method:

The sharp increase in absorbance at $\lambda < 300$ nm is due to interband transitions at the fundamental edge. Band-gap energy increases with decreasing particle size due to quantum size effects. A decrease in absorbance at higher doping level of Al may be attributed to the decrease in the size of nanoparticles and increase in porosity which ultimately widens the band gap.

The optical transmittances of Al doped Zno are 88% and 87% respectively. Optical transmittances of undoped ZnO is 93%. It shows clearly that the transmittance decreases with the increment of Al content. This suggests that Al ions are causing the formation of defect centres in host lattice (zinc oxide) proportionate to Al content. The decrease in band gap with increasing Aluminum concentration was due to the contributions of hybridization of electronic states, as well as the Aluminum-induced intensification of p-d repulsion and tensile strain.

Hydrothermal method:

Size of nano particles are decreased slightly with increasing the doping levels in Hydrothermal method, which simultaneously decrease the absorbance. The optical transmittances of Al doped Zno are 84% and 86% respectively.

3. Sol-gel method based Au doped ZnO nanoparticles versus Hydrothermal based Au doped ZnO nanoparticles:

Sol-gel method based Au-doped ZnO- Sm (NO3)3 is compared with Hydrothermal based Au-doped ZnO nanoparticles in terms of optical properties as transmittance and absorbance, structural properties as XRD analysis. These comparisons are represented in Table 3.

Table 3: COMPARATIVE PERFORMANCE ANALYSIS for Au doped ZnO NANOPARTICLES

Parameters	Sol-gel method based Au doped ZnO nanoparticles	Hydrothermal based Au doped ZnO nanoparticles
XRD	Zero impurity peaks were seen in the manufactured samples according to XRD spectra, but there was only a small shift in the peak location, indicating that this doping is producing internal tensions.	The average crystallite size of the nanoparticles is observed as 14 nm. Little amount of impurities of peaks were observed.
Optical properties	In the 300–800 nm wavelength region, the optical transmittance ranged from 25–70%. Transmittance decreased further of 1.5 percent Au-doped ZnO-Sm nanoparticle films to 25%, as Au-doping increased from 0 to 1.5 percent. Au doped Zinc oxide absorption spectrum matched the optical band gap of Au doped ZnO,	In the 350–800 nm wavelength region, the optical transmittance ranged from 20-70%. Transmittance decreased further of 1 percent Au-doped ZnO-Sm nanoparticle films using hydro thermal method to 20%, as Au- doping increased from 0 to 2 percent. Au doped Zinc oxide nanoparticles' absorption spectrum matched the optical band gap of Au

	which is established employing Tauc's equation and determined to be ~ 3.41 nm, which showed a peak positioned at 363 nm.	doped ZnO. Showed a absorption peak positioned at 360 nm.

X-Ray Diffraction (XRD) Spectrum:

Sol-gel method:

The (XRD) **X-ray diffraction** pattern of Au-doped ZnO-Sm (NO3)3 nanoparticle films confirmed that the wurtzite hexagonal single phase of ZnO. Zero impurity peaks were seen in the manufactured samples according to XRD spectra, but there was only a small shift in the peak location, indicating that this doping is producing internal tensions.

Hydrothermal method:

The average crystallite size of the nanoparticles is observed as 14 nm. Little amount of impurities of peaks were observed.

Optical Properties:

Sol-gel method:

In the 300–800 nm wavelength region, the optical transmittance ranged from 25–70%. Transmittance decreased further of 1.5 percent Au-doped ZnO-Sm nanoparticle films to 25%, as Au- doping increased from 0 to 1.5 percent.

The doping of Sm and Au enhanced absorption due to the visible light range doping form of tiny and large dimension nanoparticles. Au doped Zinc oxide nanoparticles' absorption spectrum matched the optical band gap of Au doped ZnO, which is established employing Tauc's equation and determined to be ~ 3.41 nm, which showed a peak positioned at 363 nm.

Hydrothermal method:

In the 350–800 nm wavelength region, the optical transmittance ranged from 20-70%. Transmittance decreased further of 1 percent Au-doped ZnO-Sm nanoparticle films using hydro thermal method to 20%, as Au- doping increased from 0 to 2 percent.

Au doped Zinc oxide nanoparticles' absorption spectrum matched the optical band gap of Au doped ZnO. Showed a absorption peak positioned at 360 nm.

III. CONCLUSION

This paper presents, Comparative Performance Analysis of Sol–Gel Method and Hydro Thermal Method for Zinc Oxide Nanoparticles Synthesis. ZnO nanostructures have a great advantage to apply to a catalytic reaction process due to their large surface area and high catalytic activity. Sol-gel method based ZnO nanoparticles are compared with Hydrothermal based ZnO nanoparticles in terms of XRD, UV Visible Spectrum and SEM analysis. Sol-gel method based Al-doped Zinc Oxide Nanoparticle (AZnO) is compared with Hydrothermal based Al-doped Zinc Oxide Nanoparticle (AZnO) in terms of XRD, UV Visible Spectrum, SEM analysis and optical properties. At last Sol-gel method based Au-doped ZnO- Sm (NO3)3 is compared with Hydrothermal based Au-doped ZnO nanoparticles in terms of optical properties as transmittance and absorbance, structural properties as XRD analysis.

From this paper we can conclude that, Sol-gel method based Au doped ZnO nano particles are efficient than Hydro thermal based ZnO nano particles. The calculated band gap of Au doped Zinc oxide nanoparticles approximately 3.41 eV indicates the semiconductor nature of the material, which is highly advantageous for solar cell production.

IV. REFERENCES

[1] R. Ramamurti *et al.*, "Atmospheric Pressure Plasma Printing of Nanomaterials for IoT Applications," in *IEEE Open Journal of Nanotechnology*, vol. 1, pp. 47-56, 2020, doi: 10.1109/OJNANO.2020.3009882.

[2] N. Kawamoto *et al.*, "Transmission electron microscope as an ultimate tool for nanomaterial property studies," in *Microscopy*, vol. 62, no. 1, pp. 157-175, Feb. 2013, doi: 10.1093/jmicro/dfs078.

[3] D. M. Newberry, "A modularized approach to nanotechnology education: Opportunities, challenges and requirements," *2012 12th IEEE International Conference on Nanotechnology (IEEE-NANO)*, Birmingham, UK, 2012, pp. 1-2, doi: 10.1109/NANO.2012.6322144.

[4] A. Meghea, "Templating of nanostructures by soft nanotechnology techniques," *2007 ICTON Mediterranean Winter Conference*, Sousse, Tunisia, 2007, pp. 1-1, doi: 10.1109/ICTONMW.2007.4446908.

[5] Hongyu Zhang, Yanyan Wang, Guoqiang Tao, Yongming Chai and Guohe Que, "Chemical synthesis of Fe nanocrystals via hydrogenation of ferric acetylacetonate," *2011 International Conference on Materials for Renewable Energy & Environment*, Shanghai, 2011, pp. 2066-2070, doi: 10.1109/ICMREE.2011.5930744.

[6] Y. Jin, K. Kasuya, K. Nagato, H. Morii, T. Ooi and M. Nakao, "Local Synthesis of Single-walled Carbon Nanotubes on Zeolite-covered Silicon Substrate by Laser-heating Chemical Vapor Depositon," *2007 2nd IEEE International Conference on Nano/Micro Engineered and Molecular Systems*, Bangkok, Thailand, 2007, pp. 462-466, doi: 10.1109/NEMS.2007.352059.

[7] M. S. M. Yusoff, W. Paulus and M. Muslim, "Synthesis of nano-sized alpha alumina using solvothermal and hydrothermal methods: A comparative study," *2012 International Conference on Enabling Science and Nanotechnology*, Johor Bahru, Malaysia, 2012, pp. 1-2, doi: 10.1109/ESciNano.2012.6149664.

[8] L. Oleksenko, I. Matushko, N. Maksymovych, G. Fedorenko, L. Lutsenko and H. Arinarkhova, "Morphology, Gas Sensitive and Catalytic Properties of Ce-containing Nanomaterials Based on Tin Dioxide Doped with Sb," *2019 IEEE 9th International Conference Nanomaterials: Applications & Properties (NAP)*, Odessa, UKraine, 2019, pp. 02NEE05-1-02NEE05-5, doi: 10.1109/NAP47236.2019.216988.

[9] V. Postica *et al.*, "Properties of ZnO:Fe nanostructured films grown by successive chemical synthesis," *2016 International Conference on Nanomaterials: Application & Properties (NAP)*, Lviv, Ukraine, 2016, pp. 01NTF02-1-01NTF02-4, doi: 10.1109/NAP.2016.7757233.

[10] A. I. Oliva and J. M. Lugo, "The physical properties of nanomaterials: A challenge in materials science," *2015 12th International Conference on Electrical Engineering, Computing Science and Automatic Control (CCE)*, Mexico City, Mexico, 2015, pp. 1-6, doi: 10.1109/ICEEE.2015.7357902.