

Synthesis of Zinc Oxide Nanostructures using Ocimum Gratissimum Leaf Extract

M. V. Ramana¹, G. Anuradha², V. V. Janaki Rama Rao³, P. Anitha¹ and P. S. Lakshmi⁴

Department of Physics, SR & BGNR Govt.Arts & Science college, Khammam, Telangana, India.

mvr1508@gmail.com

Department of Chemistry, Acharya Nagarjuna University, Guntur, Andhra Pradesh, India.

anuradha.mythri@gmail.com

Department of Chemistry, SR & BGNR Govt.Arts & Science college, Khammam, Telangana, India.

vvjr Rao@gmail.com

Department of Computer Science, Swarna Bharathi Institute of Technology and Science, Khammam, Telangana, India.

lakshmicconnects@gmail.com

Abstract: ZnO nanostructures including nanorods with a hexagonal tip, flower-like structures, and star-like structures were synthesized by using the Ocimum gratissimum leaf extract, ZnCl₂ and NaOH solution using green synthesis method at room temperature. The solid ZnO products were washed, dried and characterized by using X-ray diffraction (XRD) and Scanning Electron microscopy (SEM). The proportion of raw materials influence the morphology of the ZnO nanostructures. It is observed that with variation of amount of water and amount of leaf extract to the Zinc chloride and sodium hydroxide, different nanostructures like nanorods, flower-like, star-like, regular hexagonal structures are formed. The structure and morphology In all the cases, ZnO nanostructures have Wurtzite structure, as evident from XRD patterns. This method of using the Ocimum gratissimum leaf extract for synthesis of ZnO nanostructures was found to be ease, low-cost and eco-friendly.

Keywords: Zinc nanostructures, Green synthesis, leaf extract, Scan Electron Microscopy (SEM).

1. Introduction

ZnO is a semiconductor material with potential applications in electronics, photoelectronics, sensors and catalysts due to its wide band gap and high exciton binding energy (Klingshrin, 2007; Xu et al., 2002, 2004). The change in the size and morphology of the ZnO crystals changes their optical and electronic properties. This provides the control over their physical properties. The control over the size and morphology of the nanometer or micrometer-sized ZnO crystals is a challenge to realize the design of new functional devices. Nanostructural transition metal oxides have been a constant interesting topic because of their interesting properties and applications in fields like catalysis, coatings, anode electrode, solid-state sensors, energy storage, optoelectronics, photothermal, and magnetism. Till now a large quantity of ZnO nano- and micro-structures with various exciting shapes have been successfully synthesized by using different techniques (Wie et al., 2005; Pan et al., 2001; Wang, 2004, 2007; Yang et al., 2006). The methods in which gas phase based processes are involved, are always involve vacuum environment, high temperature, and complicated controlling process, which may result in poor dispersion, impurity or the decomposition of the final product and are unfavorable for low-cost and large-scale production. In contrast, the solution-based synthesis approaches become a promising choice to solve the above problems due to their manipulation, low cost, and potential for scale-up by increasing the amount of the reactants and keeping the same molar concentration. As the temperature of the synthesis is low, the convenience and simplicity in fabrication process, hydrothermal treatments have been in the forefront for the nanostructure synthesis. This has been

extensively explored for synthesising the ZnO single crystals with a variety of morphologies, including rods, tubes, disks, flowers, or other nano- and micro-structures (Park et al., 2002; Koh and Loh, 2005; Muller et al., 2003; Yang and Chen, 2004). Most of the approaches for nanostructure synthesis are toxic and not environment friendly. The used organic solvents are problematic because of their toxic nature. Environmentally friendly chemical synthesis requires alternative solvents such as ionic liquids, liquid and water. Water is particularly attractive because it is inexpensive, environmentally benign and bestowed with many virtues especially under supercritical conditions (Lyu et al., 2002).

Recently biosynthesis of nanoparticles using plant extracts has become popular because of its simple and economic feasibility (Ramana and Anuradha., 2014). Silver nanoparticles have been synthesized using biosynthesis routes, by this group recently, using Ocimum tenuiflorum L. Green and Purple (Anuradha et al, 2014), Ocimum basilicum L. Var.thyrsiflorum (Anuradha et al, 2014), and Ocimum americanum L. leaf extracts (Anuradha et al 2014). Ocimum gratissimum leaf extract is also found to be a good candidate to be used in silver nanoparticle synthesis (Anuradha 2014). With the positive results that were obtained using different plant and root extracts in the synthesis of silver nanoparticles, it is attempted to synthesis the ZnO nanoparticles with Ocimum gratissimum leaf extract. The present paper reports various nanostructures synthesized using Ocimum gratissimum leaf extract and their morphology. The synthesis route discussed these nanostructures is found to be simple, economic, fast and eco-friendly. Further it is found that this method is useful for the large scale synthesis.

UGC Sponsored National Conference on

Advanced Technology Oriented Materials (ATOM-2014), 8-9th Dec-2014

Department of Physics, Government College (A), Rajahmundry, Andhra Pradesh, India

2. Experimental

2.1 Preparation of leaf extract

Ocimum gratissimum leaves were collected from the botanical gardens, SR&BGNR Govt. Arts & Science College, Khammam and washed with flowing water for 15 minutes. The leaves were again washed with double distilled water and dried under sunlight for three days. The dried leaves were grounded into fine powder. 25 gms of *Ocimum gratissimum* leaf powder is taken in a 500ml glass beaker. An amount of 200 ml deionised water is added to this leaf powder and boiled for 5 minutes at 100°C. The thus boiled mixture is cooled to room temperature and filtered with Whatman No 1 filter paper. The filtrate was stored in a brown coloured glass container at 4°C. Herein this is called as solution LE.

2.2 Preparation of Zinc containing solution

The aqueous solutions of 10 M NaOH and 0.5 M ZnCl₂ in deionised water were prepared using analar grade chemicals. The two solutions are mixed together under constant magnetic agitation for the preparation of the homogenous solution 'A' with the desired molar ratio of Zn²⁺ : OH⁻ = 1 : 20.

2.3 Synthesis of nanostructures

The aqueous solutions of 10 M NaOH and 0.5 M ZnCl₂ in deionised water defined volume (mL) from solution 'A' is transferred into a 100ml glass beaker and is mixed with given amount of solution 'B', where solution B (coded as BS_n in table1) is a diluted solution of LE. The final mixture is filtered into another 100mL glass beaker and heated upto 90°C. A significant change in the colour of the solution is observed. The solution is then cooled to room temperature and filtered using Whatman filter paper. The supernatant solution is characterized for the presence of nanostructures.

2.4. Nanostructure characterization

Synthesized Zinc nanostructures were characterized by using Shimadzu UV-1800 spectrophotometer, in the range 300-700 nm.

Scanning electron microscopic (SEM) analysis was carried out using Zeiss, EV-18 model. A thin film of the sample was prepared on carbon coated copper grid by placing small amount of the sample on the grid. Then the film on the SEM grid was allowed to dry using mercury lamp for 5min.

Energy Dispersive X-ray analysis (EDX) was also carried out on the same sample prepared for that of SEM. XRD data was recorded using X-ray diffraction data were collected using an ENRAF NONNIUS- CAD 4 single crystal X-ray diffractometer

3. Results and Discussion

The composition of the leaf extract and the Zinc containing solution used in the present study is presented in table.1. Though other compositions were studied, the compositions

which gave significant results from the other compositions is mentioned in the present table.

Table 1: Composition of LE and Water of samples

Sample	LE	Deionised water
BS0	5mL	0mL
BS1	5mL	20mL
BS2	5mL	80mL
BS3	5mL	150mL
BS4	5mL	250mL
BS5	5mL	350mL

The earlier reported studies have indicated that biomolecules like protein, phenols, and anthroquinones play a vital role in reducing the ions to the nano size, but also play an important role in the capping of the nanoparticles.

3.1 UV-Visible spectral analysis

The nanoparticles were preliminarily characterized by UV-Visible Spectroscopy, which is proved to be a very useful technique for the analysis of nanoparticles. As the leaf extract samples from BS0 to BS5 were mixed with the solution A, the colour of the solution change from pale greenish yellow to brownish gold colour due to excitation of the surface plasma vibrations indicate the formation of the Zinc nanoparticles. The UV spectrum absorption is recorded for all the samples. The spectrum is almost identical to the reported spectra in the literature for ZnO nanostructures. In all the samples the surface plasma vibrations were observed with a corresponding peak around 309 nm.

3.2 SEM analysis

The SEM images of the samples prepared for SEM using the supernatant solutions described earlier for all the samples were presented in Figure 2. The SEM images show different morphologies. The resultant nanostructural morphology is presented in table.2. In the present study, nano particles, nanorods and nanoflowers were observed. All these nano structures were clearly distinguishable. The length of the nanorods ranging from 348nm to 960nm, where as the diameter is ranging from 54nm to 87nm.

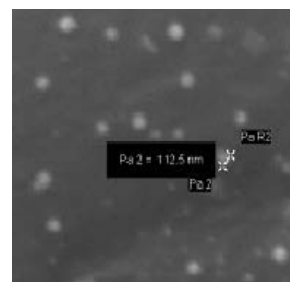


Figure 1: SEM image of Zinc nanoparticles

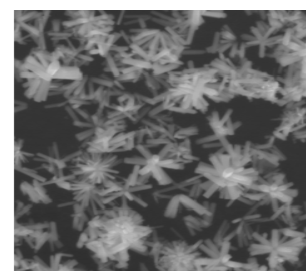


Figure 2: SEM image of Zinc nanoflowers

UGC Sponsored National Conference on

Advanced Technology Oriented Materials (ATOM-2014), 8-9th Dec-2014

Department of Physics, Government College (A), Rajahmundry, Andhra Pradesh, India

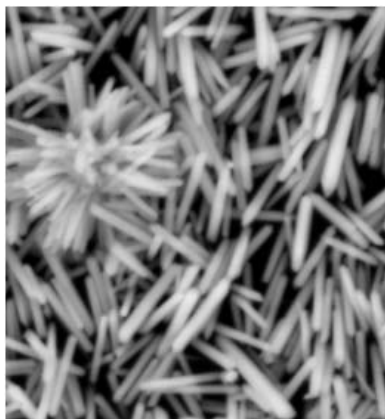


Figure 3: SEM image of Zinc nanorods

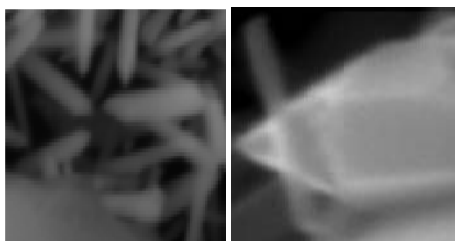


Figure 1: SEM image of Zinc prismatic nanostructures

Table 2: Samples and observed nanostructures

Sample	Nanostructures observed
BS0	Prismatic, pyramid and nanorods
BS1	Nanorods, prisms
BS2	Nanorods
BS3	Nanoflowers, nanorods
BS4	Nanoflowers, nanoparticles
BS5	Nanoparticles

The observed nanostructures and the samples in which they were observed in the present study were table.2. In few samples more than one type of nanostructures were observed. Only dominant nanostructures were mentioned in the table. Almost every nanostructure is observed in all the samples. The majority of the present nanostructures is made a mention in the table.

3.3 EDX analysis

The EDX spectra show the purity of the material and the complete chemical composition of synthesized Zinc nanoparticles. In the present synthesis EDX analysis shows 91% to 94% purity of the Zinc in the zinc nanostructures developed in this study.

3.4 XRD analysis

The XRD patterns reveal that the samples which is in the form of flowerlike structures has a variety of orientation and a low crystallinity is observed. The Miller indices are calculated from peak angles and corresponded to hexagonal structure. The lattice distance d_{hkl} (Å) is estimated from Bragg's law. The lattice constants are estimated as following: For the high crystalline materials: $a=b=3.287$ Å, $c=5.13$ Å and $a=b=3.31$ Å, $c=5.28$ Å for others. This is in consistency with the reported values indicating the variation

for $a=b$ from 3.27 Å up to 3.41 Å and c from 5.01 Å up to 5.37 Å.

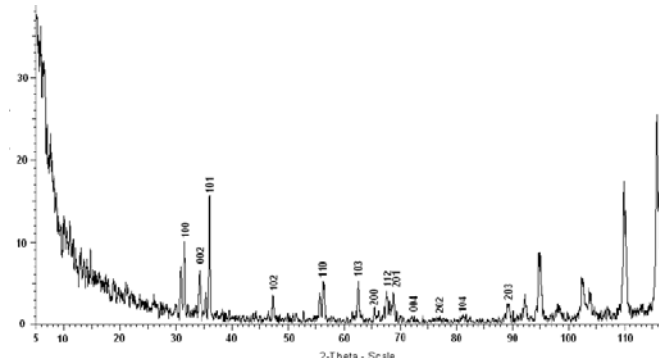


Figure 5: XRD pattern of sample BS3.

The morphologies of straight ZnO nanorods either ending with regular hexagonal prism or with hexagonal pyramid, as observed from SEM images, are in accordance with XRD major reflexes. According to literature [4], the crystallographic faces (100), (101), (001) and (001) correspond to m, +p, -c and +c, accordingly. The schematic representation of regular hexagonal pyramid structure (p), regular hexagonal prism structure (c) and side structure (m) is shown in following figure.

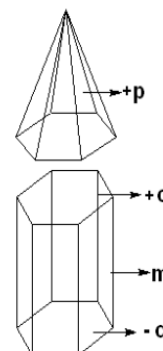


Figure 6: Crystallographic structures. +p: regular hexagonal pyramid, +c, -c: regular hexagonal prism, m: side structure : Courtesy: P.Georgiou *et al.*

4. Conclusions

It is concluded that the *Ocimum gratissimum* leaf extract can be used for synthesis of ZnO nanostructures. This method was found to be ease, low-cost and eco-friendly.

The change in the leaf concentration as evident from the composition of the samples under study, different nanostructures like nanorods, flower-like, star-like, regular hexagonal structures are formed. This strongly suggests that the morphology of these nanostructures was influenced by the content of leaf extract in the sample. The proportion of raw materials influence the morphology of the ZnO nanostructures. It is observed that with variation of amount of water and amount of leaf extract to the Zinc chloride and sodium hydroxide influences the morphology. The structure and morphology In all the cases, ZnO nanostructures have Wurtzite structure, as evident from XRD patterns.

The growth of the nanocrystalline materials along the c-axis direction may be under the influence of the leaf extract content in the sample. A further systematic study is under

progress to identify the composition of the solution which yields only the desired nanostructure.

5. Acknowledgements

The authors wish to acknowledge the Department of Physics Osmania University for SEM, EDX spectral analysis. One of the authors (MVR) would like to thank University Grants Commission, New Delhi for the financial support provided through research grant No.39-464/210(SR).

References

- [1] Z. Fan and J. G. Lu, Journal of Nanosci. Nanotechnol., Vol. 5, No. 10, p. 1561, 2005
- [2] X. Liu, X. Wu, H. Cao and R.P.H. Chang: J. Appl. Phys., Vol. 95, No.6, p. 3141., 2004
- [3] T. Gao, Q. Li and T. Wang: Chem. Mater., Vol. 17, No. 4, p. 887, 2005
- [4] W. Zheng, F. Gao, and Y. Qian: Adv. Funct. Mater., Vol. 15, No. 2, p. 331, 2005
- [5] H.S. Qian, S.H. Yu, J.Y. Gong, L.B. Luo and L.L. Wen: Crystal Growth & Design, Vol. 5, No.3, p. 935, 2005
- [6] X. Wu, H. Bai, C. Li, G. Lu and G. Shi: Chem. Commun., p. 1655, 2006
- [7] B. Baruwati, D.K. Kumar, S.V. Manorama: Sensors and Actuators B, Vol. 119, p. 676,2006
- [8] A.N. Baranov, C.H. Chang, O.A. Shlyakhtin, G. N. Panin: Nanotechnology, Vol. 15, p.1613, 2004.
- [9] W.J. Li, E.W. Shi, Y.Q. Zheng, Z.W. Yin: Journal of Materials Science Letters, Vol. 20, p. 1381, 2001.
- [10] M. Guo, P. Diao and S. Cai: Journal of Solid State Chemistry, Vol. 178, p. 1864, 2005
- [11] D. Andeen, L. Loeffler, N. Padture and F.F. Lange: Journal of Crystal Growth, Vol. 259, p. 103, 2003
- [12] K. Sue, K. Kimura, M. Yamanoto, K. Arai: Materials Letters, Vol. 58, (2004), p. 3350, 2004.
- [13] D. Tao, W. Qian, Y. Huang and F. Wie: Journal of Crystal Growth, Vol. 271, p. 353, 2004.
- [14] X. Zhang, H. Zhao, X. Tao, Y. Zhao and Z. Zhang, Materials Letters, Vol. 59, p. 1745, 2005.
- [15] J. Yu and X. Yu: Environ. Sci. Technol., Vol. 42, p.490, 2008..
- [16] P. Si, X. Bian, H. Li and Y. Liu: Materials Letters, Vol. 57, p. 4079,2003.
- [17] Anuradha. G, B. Syama Sundar and M. V. Ramana, Scholars Research Library, Archives of Applied Science Research, Vol.6 (3), p. 59, 2014.
- [18] Anuradha. G, B. Syama Sundar, M. V. Ramana, J. Sreekanth kumar and T. Sujatha, IOSR Journal of Applied Chemistry, vol.7, p. 123, 2014.
- [19] M.V.Ramana and G.Anuradha, A Review on Biosynthesis of noble metal nanoparticles, e-book, Lulu Publications, 2014.
- [20] G.Anuradha, Ph.D Thesis, Acharya Nagarjuna University, Nagarjunanagar, 2014.

Author Profile



M.V.Ramana has received his B.Sc, M.Sc and Ph.D from Osmania University in 1981,1985 and 1990. Presently he is working in SR & BGNR Govt. Arts & Science College, Khammam, Telangana, India. He has number of International and National research publications. He is author of 19 books and member of number of academic bodies. He is Fellow of International Science Congress Association. His fields of interest include spectroscopy of rare-earth ions, transition metal ions in complexes and solids, nano materials and non-linear dynamics.



Anuradha.G has received B.Sc., degree in Biochemistry from Kakatiya University in 1999 and M.Sc., degree in Medicinal chemistry from Osmania University in 2001. She received M.Phil degree from Acharya Nagarjuna University, A.P, India, in 2006 and presently in pursuit of Ph.D., from the same university.



V.V.Janaki Rama Rao has received B.Sc from Osmania University and M.Sc from Sri Krishnadevaraya University in 1983 and 1987. He has registered for his Ph.D with Krishna University. Nanochemistry is the area of his interest. Presently he is working in SR & BGNR Govt. Arts & Science College, Khammam, Telangana, India.



P.Anitha has received B.Sc and M.Sc from Kakatiya University in 1995 and 1999. She has been working in the area of biosynthesis of nanoparticles. Presently she is working in SR & BGNR Govt. Arts & Science College, Khammam, Telangana, India.



Lakshmi.P.S has received B.Sc and M.Sc from Andhra University in 1986 and 1988. M.Tech from Jawaharlal Nehru Technological University in 2012. She has Nine International publications to her credit. Computer simulation. Computer networks, spectroscopy and Nanomaterials are the topics of her research interest.