Synthesis of Zinc Oxide Nanostructures using Ocimum Gratissiumum Leaf Extract

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Introduction

ZnO is a semiconductor material with potential applications in electronics, optoelectronics, sensors and catalysts due to its wide band gap and high exciton binding energy (Klingshirn, 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. Nanoostructural 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 high temperature and complicated controlling process, which may result in poor production. In contrast, the solution-based synthesis approach becomes 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., 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. Environmental 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.thrysiflorum (Anuradha et al, 2014), and Ocimum americanum L. leaf extracts (Anruadha et al 2014). Ocimum gratissimum leaf extract is also found to be a good candidate to be used in silver nanoparticle synthesis (Anuradha 2014).

Abstract: ZnO nanostructures including nanorods with a hexagonal tip, flower-like structures, and star-like structures were synthesized by using the Ocimum gratissiumum 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 sturcutre, as evident from XRD patterns. This method of using the Ocimum gratissiumum 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).
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 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 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 of 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 BSn 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.1. The supertant solution is characterized for the presence of nanostructures.

2.4. Nanostructure characterization

Synthesized Zinc nanostrucctures 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 supertant 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 is nanorods ranging from 348nm to 960nm, where as the diameterr is ranging from 54nm to 87nm.

![Figure 1: SEM image of Zinc nanoparticles](image1.png)

![Figure 2: SEM image of Zinc nanoflowers](image2.png)
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 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.

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.

### 4. Conclusions

It is concluded that the Ocimum gratissiumum 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 sturcutre, 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.

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References


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