Synthesis and Characterization of Zinc Oxide Nanorods

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***Abstract*—** Zinc Oxide nanorods have been fabricated by single step solid-state reaction using precipitation method at room temperature. It characterized by using X-ray diffraction, Scanning Electron Microscopy (SEM), UV visible spectroscopy and photoluminescence spectroscopy.

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I. INTRODUCTION

Zinc oxide is a versatile material due to properties such as: pyro electricity, semi conducting, piezoelectricity, luminescence, and catalytic activity. Its optical band gap, chemical and thermal stabilities are also very important characteristic. These properties are essential for electronic and photonic industry due to the wide range of technological applications in devices.

It had been fabricated by single step solid-state reaction using zinc acetate and sodium hydroxide, at room temperature. It characterized by using X-ray diffraction, Scanning Electron Microscopy (SEM),UV visible spectroscopy and photoluminescence spectroscopy. The size estimation by XRD and SEM confirmed that the ZnO nanorods are made of single crystals. The growth of zinc oxide crystals into rod shape was found to be closely related to its hexagonal nature. ZnO has attracted much interest in recent years due to its potential applications in optoelectronic devices. Some of the methods developed to produce ZnO nanorod arrays are chemical vapor deposition, physical vapor deposition metal-organic vapor phase epitaxy.

A. Synthesis of ZnO using precipitation method.

(a) ZnO with capping agent Glycine

- Plane ZnO

1) 7.375 gm. $(CH3COO)_2Zn(H_2O)$ dissolved in minimum amount of conductivity water. Adding 60ml Methanol it.
2) 4 gm KOH dissolved in minimum amount of conductivity water. Adding 40 ml methanol in it.
3) 3 gm glycine as capping agent, dissolved in minimum amount of conductivity water. Adding 20ml methanol in it.
4) Mixing the above solutions in round bottom flask. Continuous stirring of mixture & heating it for 70 °C for 3 days.
5) Sonication of the mixture for 20 min. daily.
6) On the 4th day centrifugation of the mixture at 10000 rpm for 5 min. & collection of the precipitates. Drying them in oven at 50-60 °C for 3 days.
7) Grinding the precipitates in powder form, which are the final ZnO.

- DOPING OF MN^{+2}

1) Repeating step 1 to 6.
2) On the 4th day now dividing the mixture in 2 parts.
3) 0.122 gm of $Mn(ac)_2$ (2.5%) dissolving in minimum amount of conductivity water and 60ml methanol in it. Also adding 0.085 gm KOH dissolving in 10 ml methanol, add it in 1 part.
4) Also 0.441 gm of $Mn(ac)_2$ (5%) dissolving in minimum amount of conductivity water and adding 60ml methanol in it. Also adding 0.085 gm KOH dissolving in 10 ml methanol, add it in 2nd part.
5) Now taking both mixtures in separate Rb. Stirring and heating these mixtures at 70 °C.
6) On 5th day centrifuging both the mixtures at 10000 rpm for 5 min & collection of the precipitates. Drying them in oven at 50-60 °C for 3 days.
7) Grinding the precipitates in powder form, which are the final ZnO doped with 2.5% & 5% concentration of Mn+2.

(b) ZnO with capping agent polyethylene glycol.

1) Repeat the same procedure (step 1 to 8) by using 2 ml Polyethylene glycol as capping agent instead of glycine.

B. Analysis

XRD

One of the best methods of determining a crystal's structure is by X-ray diffraction

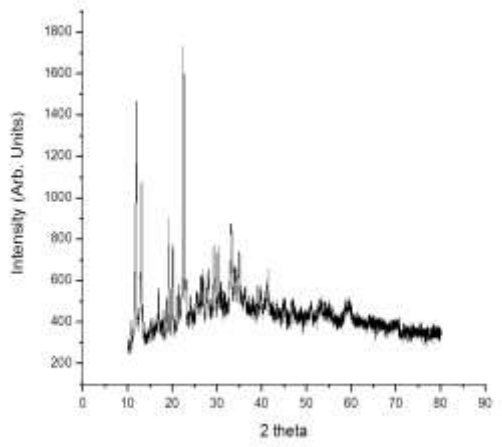

Fig.1. XRD image of ZnO with glycine as the capping agent

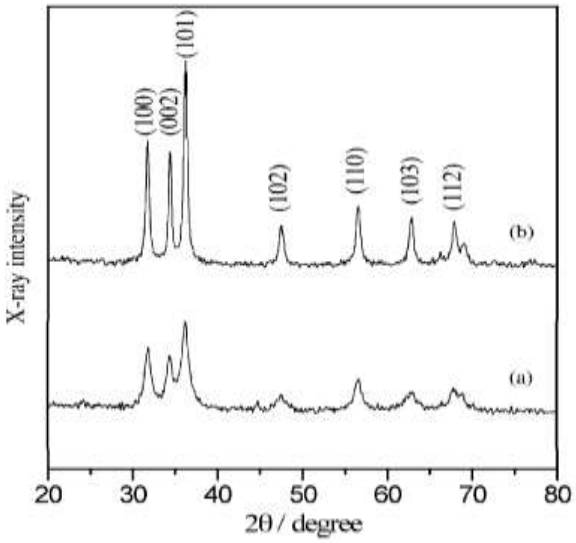

Fig.2. XRD of ZnO
(Courtesy: Journal of the Chinese Chemical Society, **2007**, *54*, 31-3)

XRD data

2θ	**θ**	**d=nλ/2sin θ (nm)**
22.37	11.18	3.96
11.75	5.87	7.52
13.00	6.5	6.80
19.26	9.63	4.60
33.14	16.57	2.69
20.6	10.3	4.30
30.2	15.1	2.95
29.16	14.58	3.05
34.9	17.45	2.56
41.31	20.65	2.18
33.98	16.99	2.63
37.94	18.97	2.37
36.10	10.05	4.41

Table. 1

(2θ values have been taken in the decreasing order of intensity of peaks)

JCPDS (36-1451) *Journal of the Chinese Chemical Society*, **2007**, *54*, 31-34		ZnO	
2θ	d	2θ	d
33.00	2.71	33.14	2.69
34.00	2.63	34.90	2.57
36.00	2.49	36.10	4.41

Table. 2

K=0.94 λ=1.54 A· Kλ=1.269x 10-^{9}

2θ	**θ**	Cos θ	FWHM (B)	L= kλ/ Bcosθ (nm)
22.3754	11.1877	0.9952	0.2094	60.89
11.7515	5.8757	0.9947	0.4432	28.70

Table. 3

Fig.1. Shows the XRD image of ZnO. It has several peaks at 2θ values of 22.3757, 11.7515, 13.00, 19.26, 33.14, and 34.9 and so on. Corresponding to these 2θ values the d values are shown in table no 1.

Some of the peaks in the synthesized sample are in agreement with the standard 2θ values and corresponding d values in literature as shown in table no 2.

From the available data of FWHM (full width at half maximum) of the synthesized ZnO the crystallite size has been calculated by using Scherer formula. The calculated values have been given in table 3.

SEM

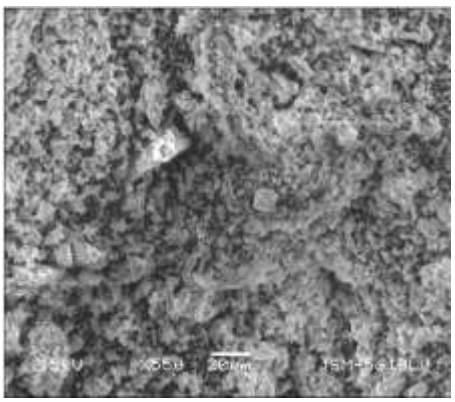

Fig.3. SEM image of ZnO

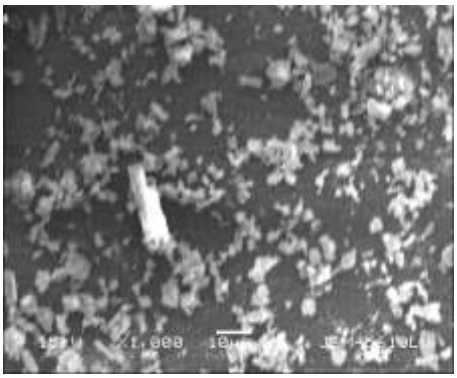

Fig.4. SEM image of ZnO

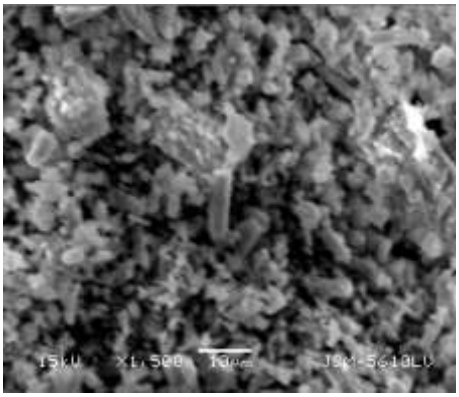

Fig.5. SEM image of ZnO

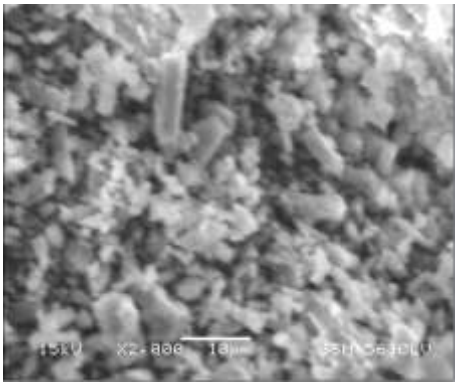

Fig.6. SEM image of ZnO

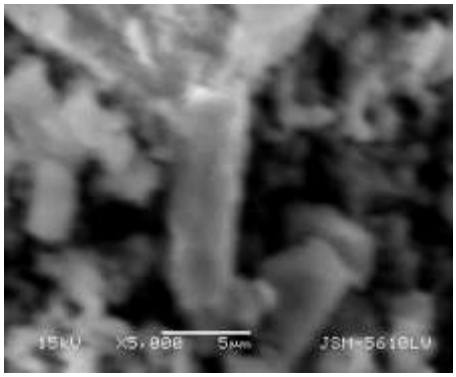

Fig.7. SEM image of ZnO

The samples were subjected to Scanning Electron Microscopy on a machine of JOEL make (M/s. Japan Electronics) model JSM-5610 LV. The scans were taken for different resolutions and scale as shown in images of Figure 3 to 7. The hazy images are due to accumulation of surface charges. Image in Fig.4 shows formation of nano rods, which become clear in the images of Figures 5 & 6. Image in Fig.7 has highest resolution. It provides an estimate of the rod size as the focus is on a single rod. Considering the scale of 5 μm in this image, the length of a typical rod is about 15 μm and diameter about 4 μm.

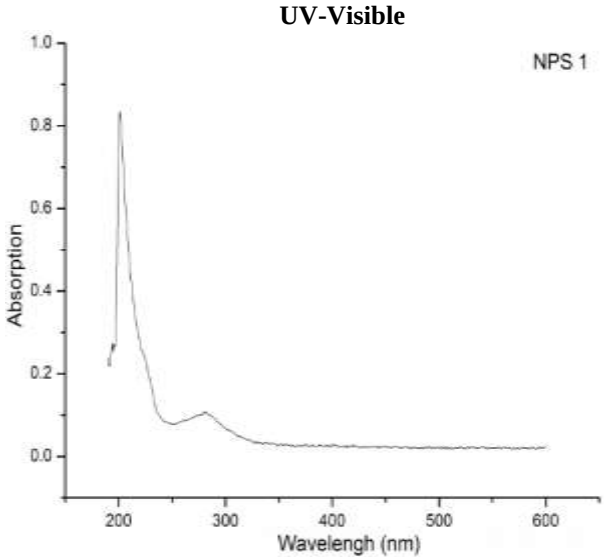

Fig.8. UV – Visible characteristics of ZnO with glycine as capping agent

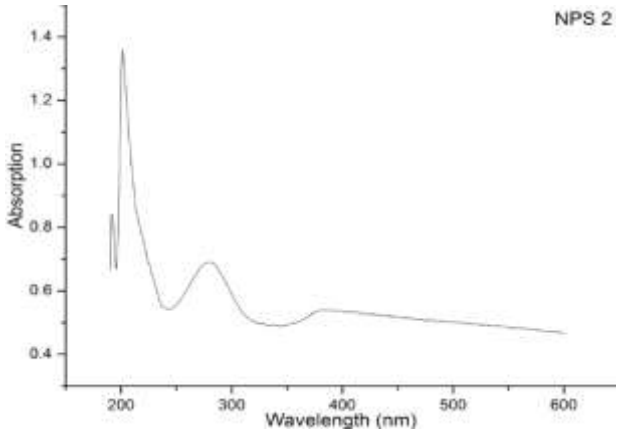

Fig.9. UV – Visible characteristics of ZnO with polyethylene glycol as capping agent

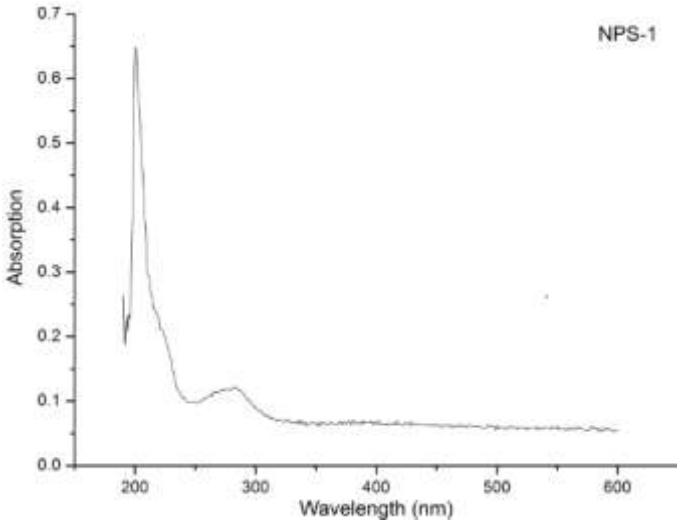

Fig.10. UV – Visible characteristics of ZnO with glycine as capping agent and doped with 2.5% Mn^{2+}

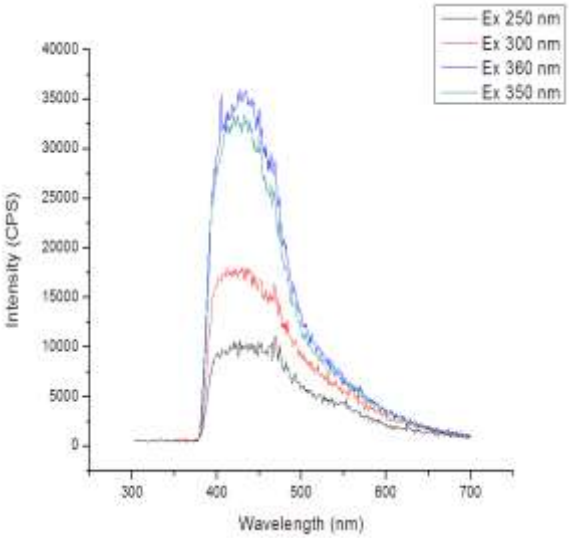

Fig.12. Emission spectra of ZnO with glycine as capping agent for excitation at 250, 300, 350 and 360 nm

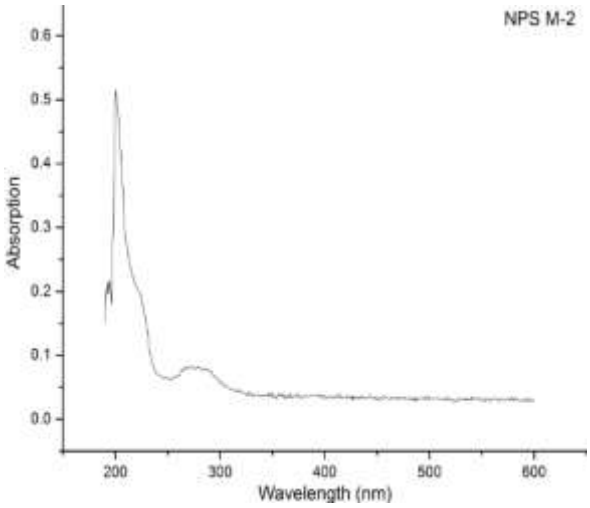

Fig.11. UV – Visible characteristics of ZnO with glycine as capping agent and doped with 5% Mn^{2+}

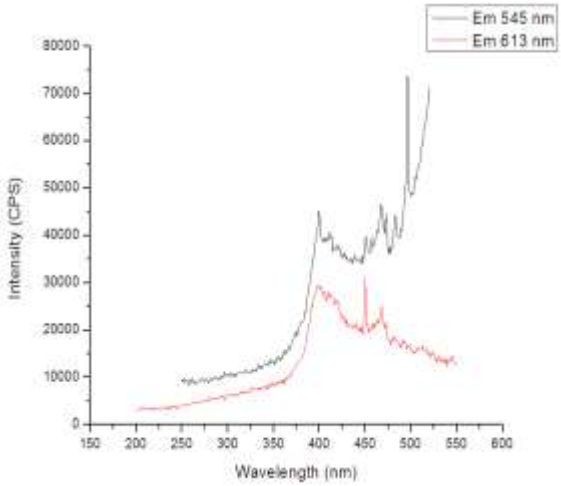

Fig.13. Excitation spectra of ZnO with polyethylene glycol as capping agent for the emission wavelengths 545 and 613 nm

Figures 8 to 11 show the UV – Visible characteristics of the synthesized ZnO samples. The peaks for all the samples are found to be around 280 nm with varying amount of absorbance. The highest absorbance is observed in the sample with polyethylene glycol as capping agent (Figure 9). In this sample, another peak is also observed around 390 nm. Thus there is good absorption in the UV region. Beyond UV, in the visible region the absorption remains almost constant with wavelength up to 600 nm

Photoluminescence

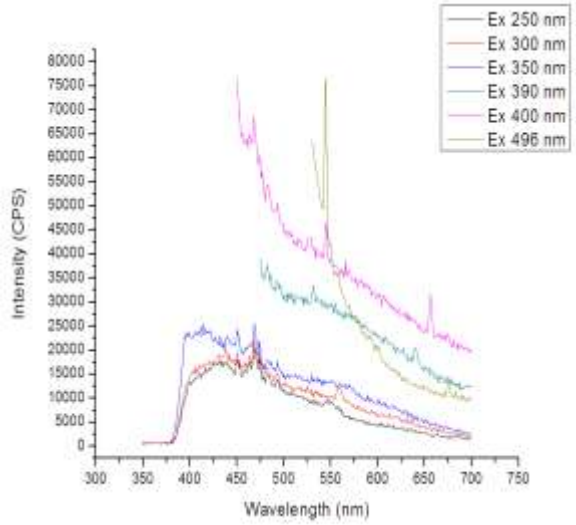

Fig.14. Emission spectra of ZnO with polyethylene glycol as capping agent excitation at 250, 300, 350, 390, 400 and 496 nm

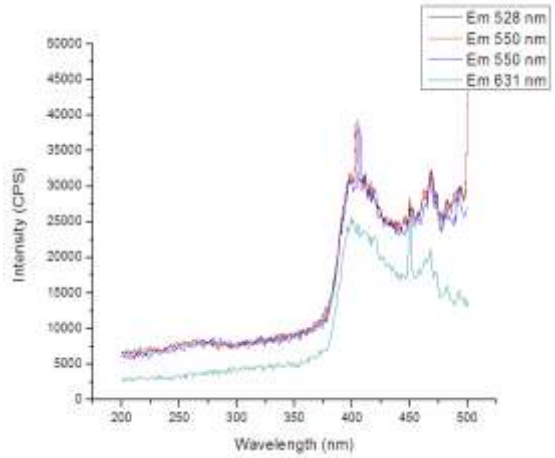

Fig.15. Excitation spectra of ZnO with glycine as capping agent and doped with 2.5% Mn^{2+} for the emission wavelengths 528, 550 and 631 nm

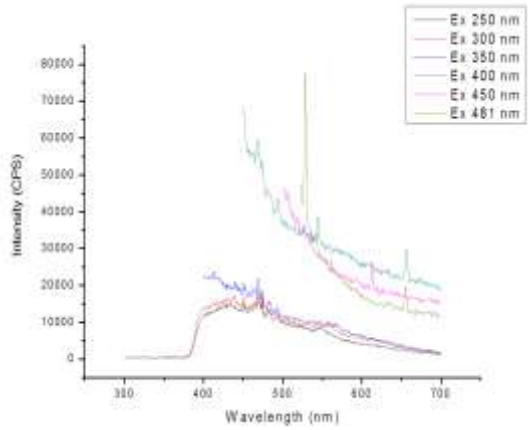

Fig.16. Emission spectra of ZnO with glycine as capping agent and doped with 2.5% Mn^{2+} for excitation at 250, 300, 350, 400, 450 and 481 nm

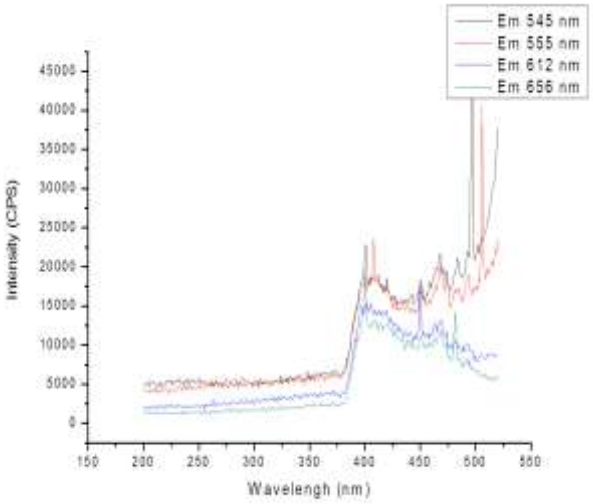

Fig.17. Excitation spectra of ZnO with glycine as capping agent and doped with 5% Mn^{2+} for emission wavelengths 545, 555, 612 and 656 nm

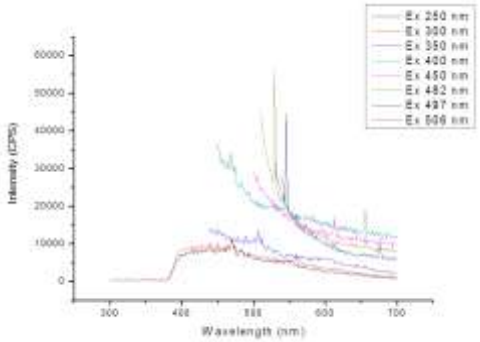

Fig.18. Emission spectra of ZnO with glycine as capping agent and doped with 5% Mn^{2+} for excitation at 250, 300, 350, 400, 450, 482, 497 and 506 nm

The emission characteristics of the samples exhibit a common feature in the form of a broad band with peak at around 430 nm. The emission characteristics were taken for a number of excitation wavelengths from 250 nm to 500 nm. For excitation beyond 400 nm, some sharp features are observed at wavelengths above 500 nm, the most prominent being around 550 nm. The intensity counts in the emission spectra was found to be highest for the sample ZnO with glycine as capping agent. The broad band feature is generally a characteristic of Zinc Oxide.

The excitation characteristics for the samples also show similarities except for the sample ZnO with glycine as capping agent. The samples show very low excitation counts in the visible region. The intensity increases beyond 400 nm in the visible region showing that the samples absorb reasonably well in the visible region also. For ZnO with glycine as capping agent, there is good absorption in the UV region. There are sharp features at 400 nm and beyond. The doping of Manganese does not seem to be enhancing the either the absorption or emission characteristics. This can be due to an increase in the non-radiative transitions on account of the Manganese ions or lack of incorporation of Manganese ions in the ZnO lattice.

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