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SYNTHESIS AND CHARACTERISATION OF ZnO AND SIVER DOPED ZnO NANOPARTICLES

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Abstract: *ZnO Nano materials have attracted a wide attention of researchers due to their excellent properties and immense applications in many fields. In the present work pure ZnO and Ag doped ZnO nano particles with capping agent were synthesized by co-precipitation method. The prepared samples were characterized for their Structural, Morphological, Antibacterial and Photoluminescence properties. The average Grain size of ZnO, Ag doped ZnO was determined and was found to increase in the case of Ag doped ZnO samples when compared with undoped ZnO samples. The SEM image of Ag doped ZnO shows tube like structure. Ag doped ZnO shows inhibitory effect on the growth of E.Coli. PL spectra of the samples indicate the presence of narrow emission band in the uv region centered around 385 nm.*

Keywords: *ZnO nano particles, co-precipitation, antibacterial, photoluminescence*

1.INTRODUCTION

ZnO Nano materials have attracted the attention of many researchers due to their distinct properties and immense applications in many fields. ZnO has a direct wide band gap 3.37eV at room temperature[1] and also it has large exciton binding energy of 60meV. Pure ZnO nanostructures show weak optical features that results from point defects, therefore, it cannot be used directly in the industry. Doped ZnO nanostructures have attracted a great attention for their potential applications in various fields such as gas sensors, field emitters and photocatalytic applications. The distinct properties make ZnO and Ag doped ZnO, a great potential in the field of nanotechnology.

2.EXPERIMENTAL TECHNIQUES

Pure ZnO nano powders were synthesised by oxidation method and Silver doped ZnO by co-precipitation method.

2.1 Synthesis of Pure ZnO by oxidation method:

Zinc granules with purity of 99.9% were used for the synthesis of pure ZnO powders. Zinc granules were taken in the silica crucible and were kept in the muffle furnace. The furnace was first initialized for the temperature and timing segments and then Zinc granules were heated in the furnace at 600^{o}C and this temperature was set to attain in 3 hours and then maintained the same temperature for next 2 hours. Zinc was oxidized in the silica crucible in the normal atmosphere of air in the furnace and the oxygen present in the air facilitated the formation of Zinc oxide. The powders were then grinded in pestle and mortal and used for characterization.

2.2 Synthesis of pure ZnO by Co-precipitation method

The chemicals Zinc acetate and Silver nitrate were used for the synthesis of Ag doped ZnO nano powders. To 100 ml ethanol, 3.5g of Zinc acetate was added and was stirred in magnetic stirrer for one hour and then 0.25g Silver nitrate was added to the stirred solution. To the above clear solution, ammonia solution was added drop wise to reach PH 7. Finally few drops of Poly Vinyl Alcohol (PVA) / PVP was added and was stirred continuously for nearly 2 hours so that a glassy grey color gel was formed. The so formed gel was aged overnight and then filtered and washed 3 times with ethanol and double distilled water to remove impurities. The gel was allowed to dry at 100^{o} C in hot air oven and then annealed at 500^{o}C in the muffle furnace and the powders were collected and used for analysis.

3.Characterisation techniques

3.1 Structural analysis:

X-ray diffraction is a versatile, non-destructive technique that reveals detailed information about the chemical composition and crystallographic structure of materials. The structure of undoped ZnO nano particles and Silver doped ZnO

nanoparticles capped with PVA and PVP were characterized by X-Ray diffraction (XRD) technique using Cu-Kα radiation of wavelength 1.5418 A^0 with 2θ ranges from 20^0 - 90^0. Crystallite size was determined using Debye- Scherrer's formula.

3.2 Morphological Studies:

Morphological studies were performed using Scanning Electron Microscope(SEM). SEM micrographs of the undoped ZnO and that of Ag doped ZnO nanoparticles with PVA as capping agent were analysed.

3.3 Antibacterial Studies:

Antibacterial testing was performed against E Coli (Escherichia coli) and for the bacterial growth Mueller Hinton Agar (MHA) medium was used. The sample Ag doped ZnO with PVA as capping agent of volume 40 µl, 60 µl 80 µl and 100 µl with different concentrations 300 µg/ml, 400 µg/ml and 500 µg/ml were used for the study of the antibacterial activity.

3.4 Photoluminescence studies

To investigate the effect of Ag on the optical properties of ZnO nanoparticles, Photoluminescence (PL) studies were taken.

4. RESULTS AND DISSCUSSION

The XRD pattern of the prepared samples were shown in the Fig 1. The diffraction peaks of undoped ZnO sample represents the formation of pure wurtzite structure of ZnO and was quite matching with the hexagonal wurtzite structure of ZnO data [JCPDS.No: 89-0510] [2]

Fig 2 represents the diffraction peaks of Ag doped ZnO with PVA as capping agent and fig.3 represents the diffraction peaks of Ag doped ZnO with PVP as capping agent. The diffraction peaks were quite matching with the undoped ZnO hexagonal wurtzite structure with some additional diffraction peaks which were shown with mark * **[3].** The additional peaks were observed at 38.17°, 44.31°, 64°, 77° (2Ɵ) which corresponds (1 1 1), (2 0 0), (2 2 0) and (3 1 1) planes of Silver[4],[5] and was in good agreement with (JCPDS 4-0783) values[6]. The coexistence of Ag peaks with ZnO peaks indicate the presence of both Ag^+ ions into the ZnO lattice. From diffraction pattern it was noted that the intensity of peaks corresponding to Ag was slightly greater for PVP capping sample than for PVA capping sample.

Average Grain size and average strain produced in the samples were calculated and the average grain size for pure ZnO, Ag doped ZnO with PVA and Ag doped ZnO with PVP was found to be 8.62996 nm, 11.8433 nm and 16.964 nm respectively. The average Grain size was found to increase in the case of Ag doped ZnO samples when compared with undoped ZnO samples. The average strain introduced in the sample was found to be less for Silver doped samples than pure ZnO particles (Table 1). The average strain is least for the sample with PVP as capping agent. The observed variation in the crystallite size and lattice strain may be due to the incorporation of silver in ZnO [7]. The lattice parameters a and c of the samples are in good agreement with the reference values.

The SEM images of pure ZnO shows clear tetra pod structures (fig.4). The micrograph images of Ag doped ZnO particles shows tube / rod like structure (fig 5 & fig 6) and a hexagonal cross section was also seen in the tube / rod [8]. From SEM images it was noted that the particle size varies from 37µm to 250 µm. The length of the rod like structure varies from 80 µm to 260 µm was noted from the SEM images. The SEM images of pure ZnO synthesized by oxidation method was found to be clear when compared to the images of Ag- doped ZnO synthesized by Co-precipitation method. This may be due to the presence of impuities in the sample Ag doped ZnO.

Ag doped ZnO shows inhibitory effect on the growth of E.Coli[9]. The table 2 shows that with the increase of the concentration, the growth inhibition of E Coli was found to increase in the case of Ag doped ZnO. The zone of inhibition diameter was found to increase with the increase of sample volume. The fig 7 and fig 8 shows the antibacterial efficiencies of the prepared ZnO and Ag–ZnO do not differ significantly.

Fig 9 shows Photoluminescence (PL) spectra of pure ZnO and Fig.10 shows that of Ag doped ZnO samples with 300 nm excitation wavelength at room temperature. PL spectra of both the samples indicate the presence of narrow emission band in the uv region centered around 385 nm. This may be due to the free excitonic (electron – hole) recombination in ZnO. Due to Ag doping no appreciable change was observed. In addition to this UV band, there was another wider emission band in the visible spectral range 490 to 520 nm (cyan) of ZnO and wider emission band in the visible spectral range 580 to 610 nm (yellow - orange) of Ag doped ZnO nano structures. This multi component emission may be due to the defects in the samples. The intensity variation observed may be due to the incorporation of Ag^+ ions into the ZnO nano structures[10] and this Ag concentration in the nano structures incorporates larger structural defects.

5.Conclusion

Pure ZnO nano particles and Ag doped ZnO nanoparticles were synthesized by oxidation and chemical co-precipitation method respectively. The characterization studies such as XRD, SEM, Antibacterial and PL has been carried out for the prepared samples.

The structural details of the samples show hexagonal wurtzite phase and the increase of average grain size was noted in the case of Ag doped ZnO samples capped with PVA when compared with undoped ZnO samples. The average strain introduced in the sample is found to decrease for Silver doped ZnO than pure ZnO particles. The grain size of Ag-doped ZnO capped with PVA is slightly greater than Ag-doped ZnO capped with PVP but strain is lesser. SEM micrographs of the undoped ZnO shows clear tetra pod structure and Ag doped ZnO nasnoparticles with PVA as capping agent shows rod / tube like structure. The particle size of silver doped ZnO nano structures is greater than that of pure ZnO Nano particles. Antibacterial testing performed for Ag doped ZnO structures shows inhibitory effect on the growth of E. coli. PL spectra of pure ZnO shows emission band in the UV region and that of Ag doped ZnO shows emission band in the cyan region.

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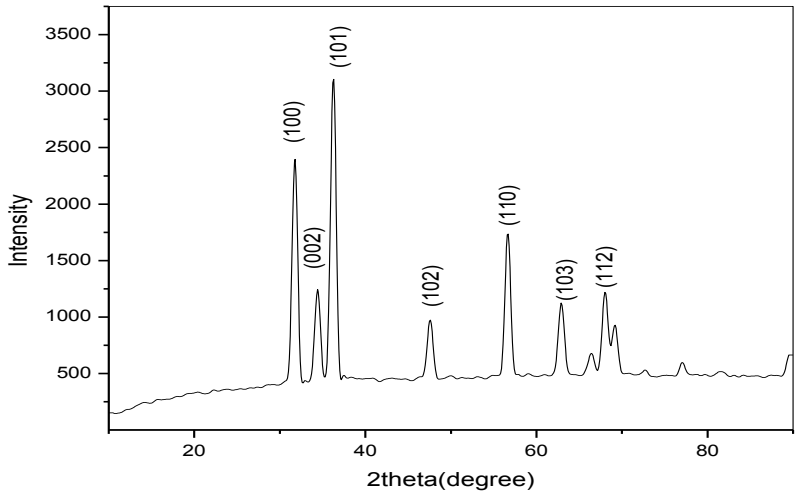

Fig. 1 XRD pattern of undoped ZnO nano particles

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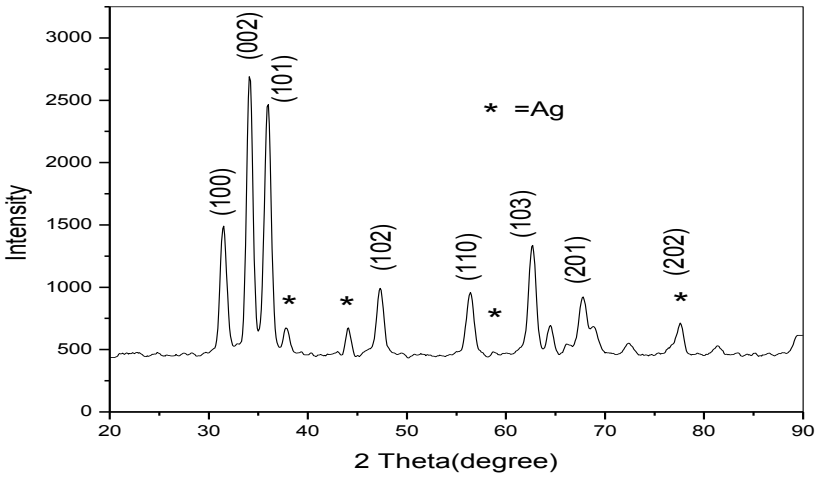

Fig. 2 XRD pattern of Ag doped ZnO (capping agent PVA)

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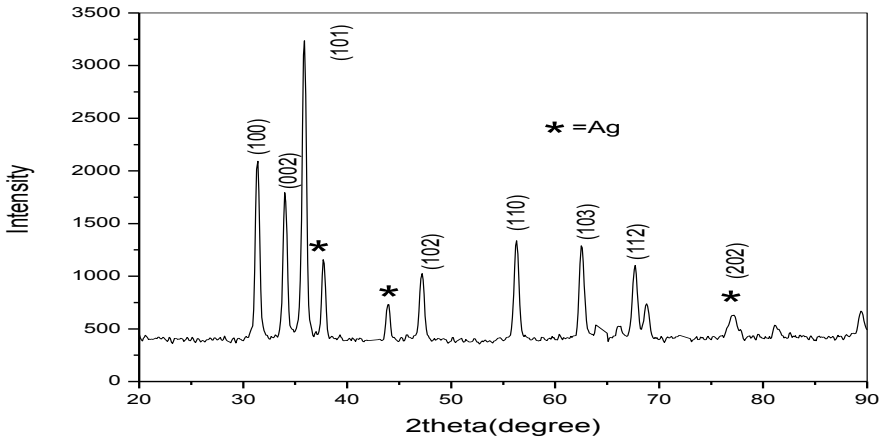

Fig. 3 XRD pattern of Ag doped ZnO (capping agent PVP)

Sample	Average Grain size (nm)	Average Strain
Undoped ZnO	8.62996	0.0111
Ag-doped ZnO with PVA	11.8433	0.00742
Ag-doped ZnO with PVP	16.964	0.00485

Table 1

SYNTHESIS AND CHARACTERISATION OF ZnO AND SIVER DOPED ZnO NANOPARTICLES

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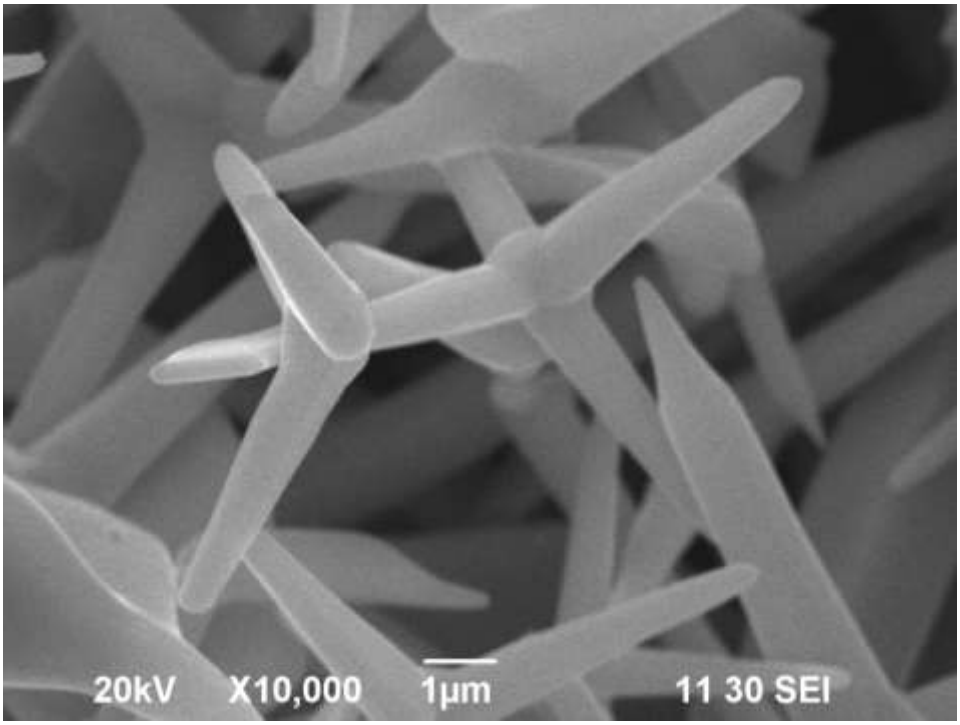

Fig. 4 SEM image of undoped ZnO

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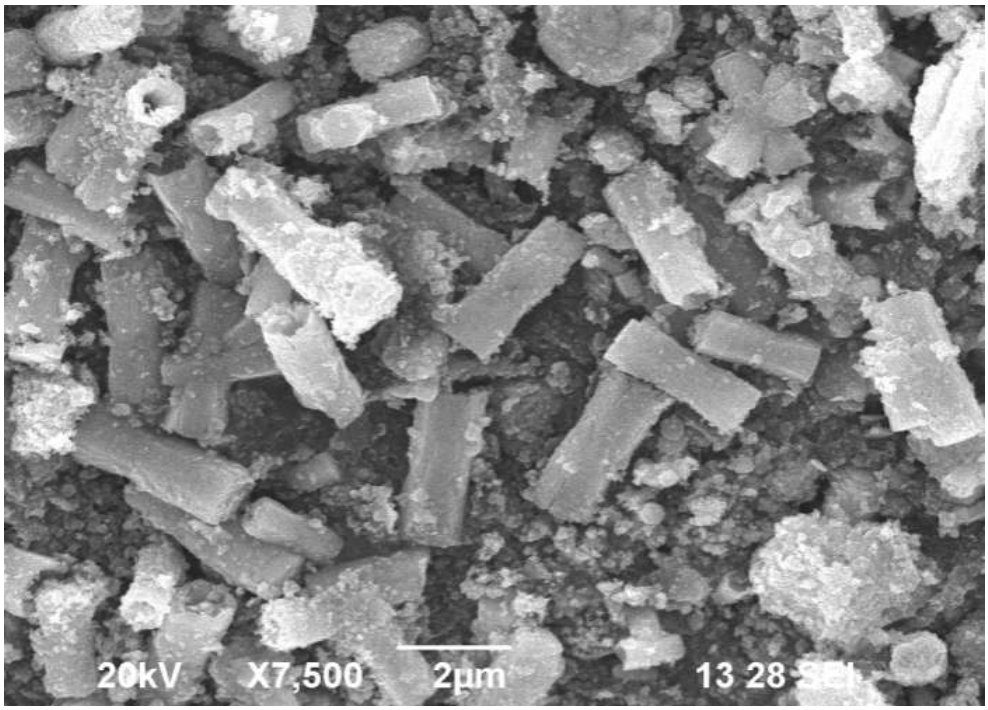

Fig.5 SEM image of Ag doped ZnO

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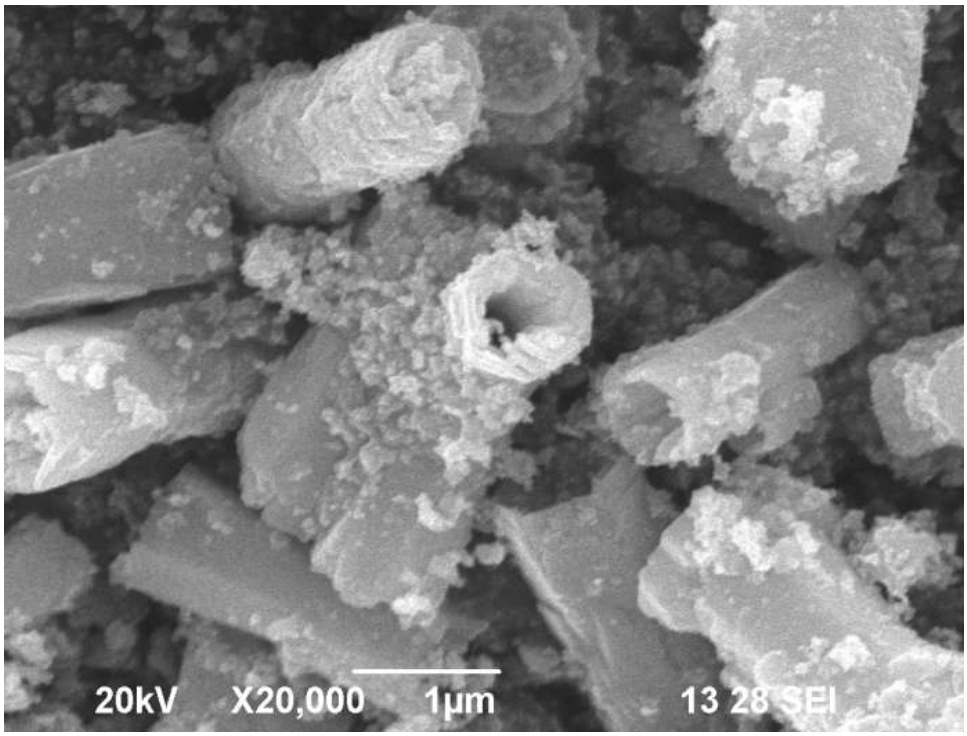

Fig 6: SEM image of Ag doped ZnO

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Conc. of Ag doped ZnO	Zone of inhibition for 40 µl		Zone of inhibition for 60 µl		Zone of inhibition for 80 µl		Zone of inhibition for 100 µl	
	Trial 1	Trial 1I	Trial 1	Trial 1I	Trial 1	Trial 1I	Trial 1	Trial 1I
300 µg/ml	11	10.5	11	11.5	12	12	12	12
400 µg/ml	11.5	11	12	12	13	13	13.5	12.5
500 µg/ml	13	11	13.5	13.5	11.5	12.5	14	13.5

Table 2 Antibacterial activity of Ag doped ZnO

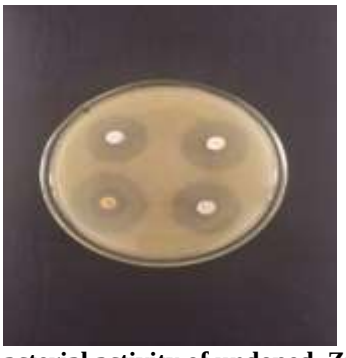

Fig. 7 Antibacterial activity of undoped ZnO for E.coli

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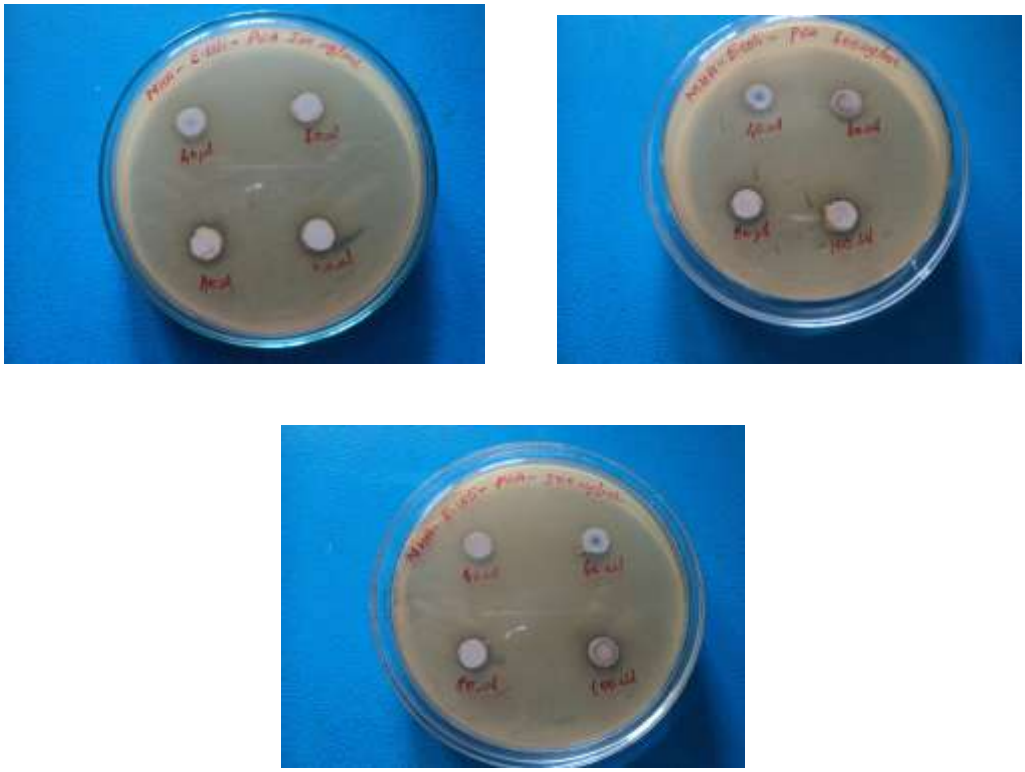

Fig. 8 Antibacterial activity of Ag doped ZnO for E. coli
300 µg/ml, 400 µg/ml, 500 µg/ml.

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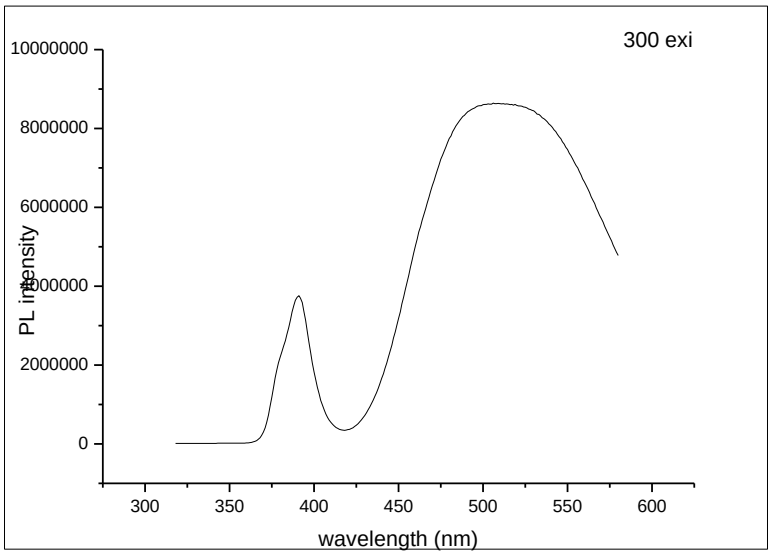

Fig. 9 PL spectra of ZnO nano particles

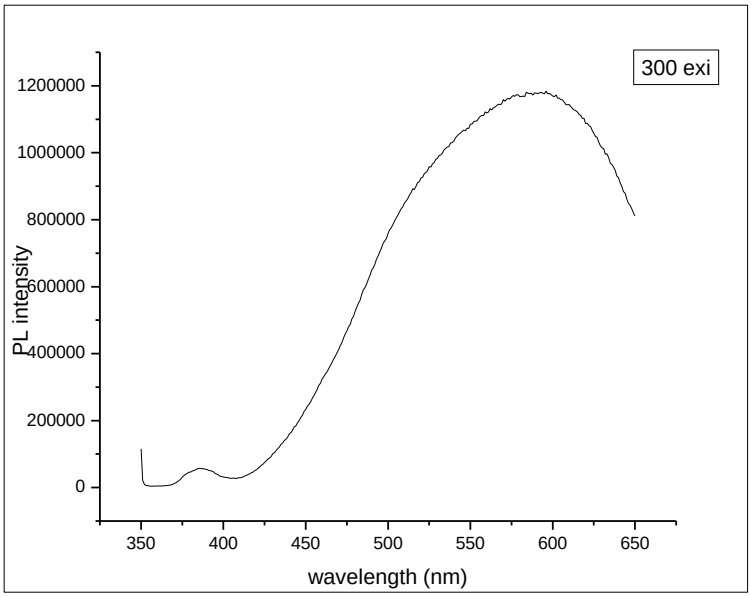

Fig. 10 PL spectra of Ag doped ZnO nano structures