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ABSTRACT. In this present investigation, pure and Fe doped Zinc oxide nanoparticles were successfully synthesized by sol gel method. The structural and optical properties were examined by using X-ray diffraction (XRD), Scanning electron microscope (SEM), Transmission electron microscope (TEM), Ultraviolet spectroscopy and Photoluminescence (PL) techniques. The structural characterization of XRD analysis confirmed the phase purity of the samples and crystallite size can be decreased with increasing doping concentrations. SEM image show that nanoparticles in spherical shape. The optical band gap calculated through UV-visible spectroscopy is found to be increasing from 3.48 to 3.57eV. TEM analysis depicted the crystallinity of nanoparticles prepared and chemical composition confirmed the EDAX analysis. The PL spectra reveal that, Fe doped ZnO exhibit a decrease in intensity of the band edge emission peak while the intensity of the deep level emission peak increases. The enhancement of low temperature ferromagnetism in ZnO: Fe was achieved.

Introduction. Recently, the diluted magnetic semiconductors (DMSs) have attracted much attention because new functions can be added and the functions can be tuned in these materials by transporting and controlling various types of spin states [1]. Zinc Oxide is one of the most important II-VI group elements with wide band gap (3.37eV) and large exciton binding energy (60 meV) at room temperature. It is a low cost and environmental friendly n-type semiconductor [2]. The undoped ZnO nanoparticles have only diamagnetic nature. Transition metal doping of ZnO has become an active research field ever since it was predicted to improve the optical and electronic properties of the oxide materials and particularly, leads to room temperature ferromagnetism [3]. By suitably adding transition metals such as Fe, Ag, Co, Cr and Al are an important class of semiconductor, one can tailor its physical, chemical and magnetic properties. The particles are transparent to visible light but they absorb UV light. ZnO has properties and versatile applications in transparent electronics, electrical and optical switching devices, chemicals gas sesors, laser diodes, solar cells, electrostatic dissipative coatings, varistors, luminescencesand spin based devices [4]. Fe doped ZnO nanoparticles have been prepared by the various method like, sol-gel method [5], co- precipitation method [6], solid state reaction method [7].

Experimental Method. The host precursor zinc acetate dihydrate (Zn (CH₃COO)₂.2H₂O) was dissolved in deionized water to obtain an aqueous solution, which was used as the starting solution (0.2 M). Ferricitrices (FeNO₃) were used dopant precursors for 1%, 3%, 5%, 7% respectively. The pH value of the starting solution was maintained at 9 by adding the required amount of NH₄OH solution. After, Tri-ethanolamine (C₆H₁₅NO₃) is added as surfactant to control size and morphology of nanoparticles. The resultant mixture was heated to 70°C and magnetically stirred for 2hrs. After completing the stirring process the precipitate was separated carefully by filtration and washed several times.

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times with a mixture of ethanol and water kept in the ratio of 1:3. The final product was irradiated with microwave oven for 30 min. Finally the powder calcinated at 500 °C for 2hrs.

**Result and Discussion**

**Structural Studies.** Fig.1 shows that doped ZnO nanoparticles have a polycrystalline structure with three orientations along (100), (002) and (101) diffraction planes. These patterns have been compared with standard JCPDS 89-0510.

![XRD patterns](image.png)

Fig. 1. XRD patterns of Fe doped ZnO nanoparticles.

It shows a decrease in crystallite size from 14nm to 11nm as the doping concentration increases. However, the crystallite size shows a decreasing trend, which consequently increased the dislocation density. The crystallite sizes of the synthesized powders are estimated from X-ray lines broadening using Scherer’s equation [8, 9],

\[
D = \frac{0.9\lambda}{\beta \cos\theta}
\]

where \( \beta \) is full width at half maximum (FWHM), \( \theta \) is diffraction angle and \( \lambda \) is wavelength of X-rays.

**Morphological Studies.** Fig. 2 shows the SEM micrograph exhibiting the morphology of assynthesized by ZnO nanoparticles. The surface contains spherical structure without any isolated grains or larger agglomerates with nano crystallites revealing the polycrystalline nature as observed from the XRD result [10].

The typical EDAXspectrum of Fe doped ZnO with 1%, 3%, 5%, 7% Fe elemental compositional and calcined at 500°C is shown in Fig. 2.

**TEM studies**
Table 1. Values of crystallite size and dislocation density from XRD of ZnO samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Crystallite size (nm)</th>
<th>Dislocation density ($\delta \times 10^{-3}$) (nm$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure ZnO</td>
<td>14.47</td>
<td>4.77</td>
</tr>
<tr>
<td>1%</td>
<td>13.54</td>
<td>5.45</td>
</tr>
<tr>
<td>3%</td>
<td>12.06</td>
<td>6.87</td>
</tr>
<tr>
<td>5%</td>
<td>11.44</td>
<td>7.64</td>
</tr>
<tr>
<td>7%</td>
<td>11.04</td>
<td>8.20</td>
</tr>
</tbody>
</table>

Fig. 2. SEM and EDAX image of Fe-doped ZnO nanoparticles.

Fig. 3. TEM and SAED image of Pure and Fe doped ZnO nanoparticles.
Fig. 3 shows the plane-view TEM images of ZnO nanoparticles. There are no aggregates or secondary phases at the inter grain boundaries. The grain size reduction observed is in good agreement with the morphological and structural results revealed by the SEM and XRD studies and the selected area electron diffraction (SAED) patterns for the ZnO nanoparticles. The other rings assigned to (002), (100) are also confirming the formation of monophase ZnO nanoparticles with hexagonal structure.

**UV-Studies.** Fig. 4 shows the optical absorption spectra of as prepared Fe doped ZnO nanoparticles in the visible range. It can be seen that when the doping concentration is increased and band gap can be increased.

![UV-vis spectrum and Tauo plot of Fe doped ZnO nanoparticles](image)

Fig. 4. UV-vis spectrum and Tauo plot of Fe doped ZnO nanoparticles

Fig. 5 shows the optical band gap of the Fe doped ZnO nanoparticles estimated by extrapolation of the linear portion of (Tauc’s plot) using the relation \( a h v = A (h v-E_g)^n \), where \( a \) is the absorption coefficient, \( h v \) the photon energy and \( E_g \) is the optical band gap.

The optical bandgap values of as prepared by nanoparticles have been given in Table 2.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Band gap (eV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure ZnO</td>
<td>3.48</td>
</tr>
<tr>
<td>1%</td>
<td>3.51</td>
</tr>
<tr>
<td>3%</td>
<td>3.52</td>
</tr>
<tr>
<td>5%</td>
<td>3.55</td>
</tr>
<tr>
<td>7%</td>
<td>3.57</td>
</tr>
</tbody>
</table>

**PL Studies.** Fig. 5 shows the room temperature photoluminescence (PL) spectra of ZnO nanoparticles. In the room temperature PL spectra of Fe doped ZnO, a dominant peak at about 389 and 390 nm has been observed. The peak in the UV region corresponds to the near band edge emission (NBE), because this peak is located close to the band gap energy (~3.3 eV), of ZnO material/crystals at room temperature.
The origin of the peak around 415 nm could be ascribed due to the transition occurring from Zn interstitials to the valance band [11].

**Summary.** Fe doped ZnO nanoparticles were prepared by sol-gel techniques. Structural analysis indicates that the Fe doped ZnO nanoparticles crystallized in hexagonal wurtzite structure and the crystallite size decreases as the doping concentration is increased. From the optical band gap of the ZnO:Fe shows an increase with the doping level increases. The transmittance range of Fe doped Zinc oxide nanoparticles in the visible range is about 85%. PL spectra shows that the emission range in 389-391 nm. SEM and TEM images indicate that the nanoparticles have spherical shapes of ZnO nanostructures.

**References**


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