Neodymium Ion doped ZnO Nanomaterial: Synthesis & Optical properties

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Abstract-*The present study involves the synthesis of Nd^{3+} doped ZnO nanomaterial by the zinc chloride and neodymium oxide chemical synthesis method. The synthesized nanomaterial was characterized with respect to their crystal structure, crystal morphology, particle size and photoluminescence (PL) properties using X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and PL-spectroscopy respectively. XRD results revealed that ZnO nanomaterial are single phase and spherical type structures. Further, PL spectra of ZnO: Nd^{3+} nanomaterial show approximate spherical shape to ZnO nanoparticles and the size of the particles around 100-20 nm. Their absorption spectra were measured in 300-1100 nm regions at room temperature. Various spectroscopic parameters such as Slater-Condon, Racah, spin-orbit interaction, Nephelauxetic ratio, bonding and Judd-Ofelt parameters have been computed from the observed band. The Fluorescence spectra have been recorded of Nd^{3+} doped ZnO nanomaterial using with intense absorption bands (583 nm) at room temperature in visible region. The formation of ZnO: Nd^{3+} nanomaterial was confirmed by Fourier transforms infrared (FTIR) and XRD spectra.*

Keywords: *Neodymium doped ZnO Nanomaterial, Absorption Spectra, Fluorescence Spectra and Radiative properties*

I. Introduction

Trivalent lanthanides doped semiconducting oxide nanostructures are one of the most promising nanomaterials for an active layer due to their stable intra-4f shell transitions in their ions. In this context, zinc oxide (ZnO) possesses a great potential as a host material for RE-doped semiconductor because it has a wide band gap of about 3.36 eV, a large exciton binding energy of 60 meV and native defects.

ZnO, with a wide energy band gap of about 3.37eV and large exciton binding energy of 60 meV finds wide range of application in light-emitting diodes[1], solar cells [2], transparent conductors [3], field emission displays [4], sensors [5,6] etc. The wide band gap of the ZnO increases the possibility of not only the excitation of the ions of the RE dopant but also in controlling its conductivity [7]. Various methods have been reported for the synthesis [8-12] $4fn-15d$ levels may be understood as formed by the electron in the $5d$ orbital interacting with $4fn-1$ core. As a consequence of this strong crystal field effect on the $5d$ electron, $4fn-15d$ configurations of RE ions in solids are very different from those of free ions. $4fn \rightarrow 4fn-15d$ absorption of most of the RE^{3+} and RE^{2+} ions exhibits two features. First, they consist of strong bands corresponding to the components of $5d$ orbital split in the crystal field. Consequently, their spectra are similar when ions are embedded in same type of host. Second, the structures of $5d$ bands can be fitted to energy differences in the ground multiplets of the $4f\,n-1$ configurations [13].

Neodymium is one of the most widely used elements for high power laser applications and recently these lasers have shown their usefulness in inertia confined fusion experiments. Furthermore, Nd^{3+} doping reduces the band gap energy and enhances the possibility of the photo degradation of dyes under visible light, also shown by us in the present study under UV light.

In the present study, Neodymium doped zinc oxide nanomaterial (ZnONM) was synthesized by the chemical synthesis method. The synthesized ZnONM was characterized by Fourier transform infrared (FT-IR) spectra, X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Moreover, the luminescent property was also studied using photoluminescence (PL) spectrophotometer.

II. Experimental Details

Nd^{3+} doped Nanomaterial have been prepared by chemical synthesis method and characterized by XRD, SEM, TEM, FTIR, Absorption and Fluorescence Spectra.

The nanomaterial with rare earth ions will be prepared by chemical synthesis method [14]. ZnO nanoparticles will be tried in alcoholic media like ethanol, methanol or propanol. In alcoholic media growth of

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oxide particles is slow and controllable [15]. Different solutions will be prepared by dissolving 0.2725 g of $ZnCl_2$ (10^{-1} M, 20 ml), 0.525 g NaOH (10^{-1} M, 100 ml) and *X* M glycerol in ethanol. Glycerol slowly added to NaOH solution while it will be continuously stirred. The resulting solution will be stirred for one hour before adding $ZnCl_2$ and Nd^{3+} solution to it. After three hours of constant stirring a milky white solution will be obtained. Size selective precipitation will be carried out using acetone as a non-solvent. The precipitate will be washed in methanol and ethanol will be allowed to evaporate at room temperature to obtain doped ZnO nanoparticles in white powder form.

III. Results And Discussion

X-Ray Diffraction (XRD)

The XRD patterns indicated that RE ions were successfully incorporated into the crystal lattice of ZnO matrix. The XRD results also confirmed the proper hexagonal phase formation and improved crystallinity with increasing dopant concentration. The X-ray diffraction pattern of typical ZnO: Nd^{3+} nanoparticles is given in Fig. 1.It can be noted that all of the diffraction peaks could be well indexed to the Hexagonal Wurtzite crystal structure. In this Nd^{3+} doped sample, no additional phase was observed, indicating that manganese was successfully doped in the ZnO crystalline lattice. The width of the diffraction lines is broadened because of the small size of the crystallites. The XRD confirmed the proper hexagonal phase formation and improved crystalline with increasing dopant concentration [16]. Almost similar values have also been reported by Yadav [17].

SCANNING ELECTRON MICROSCOPY [SEM]

The SEM image in Fig.2 of Nd^{3+} doped ZnO nanoparticles prepared by chemical synthesis method at room temperature. The image shows approximate Hexagonal Wurtzite crystal structure of ZnO nanoparticles and the size of the particles around 100-20 nm. Similar type of behavior of ZnO with Pr^{3+} ion is also observed by Sharma et al for the nanopowder [18].

TRANSMISSION ELECTRON MICROSCOPE (TEM)

A TEM image of ZnO: Nd^{3+} is presented in Fig.2**.** It can be seen from the figures that the ZnO: Nd^{3+} sample occurs through the aggregation of the nanocrystals exhibiting sizes of about 20 nm. Nearly hexagonal shapes for the dark spots in the images indicate that the ZnO nanoparticles are almost hexagonal. These results are also consistent with other rare earth metal ions doped like Gd-doped ZnO Nanomaterial [19]. The particle sizes are in the range of 100-20nm. The HR-TEM patterns consist of dots, which are characteristic of randomly oriented single crystalline nanoparticles. Based on the TEM studies it is inferred that as prepared Nd^{3+} containing sample consists of crystalline ZnONM of average size of 20 nm. This suggests that Nd^{3+} doping has no effect on the particle size of ZnO nanoparticles. Even though the particle size and crystallinity of the ZnONM containing different amounts of Nd^{3+} ions are same, it is quite possible that the Fluorescence properties of Nd^{3+} ions can change significantly depending on the interaction of Nd^{3+} ions with the ZnONM.

FOURIER TRANSFORM INFRA RED (FTIR)

FTIR image of ZnO: Nd^{3+} is presented in Fig.3 and table 2**.** The FTIR spectra of Nd^{3+} doped ZnONM consists of several peaks which are broad and moderate in band width. The peaks in the range 685 cm^{-1} are due to metal–oxygen bonds Nd/ZNO. The peak around 1650 cm^{-1} is assigned to the asymmetric stretching vibrations of Zn–O bonds from metal-oxygen group. The broad band in all the ZnONM matrices around 3350 cm^{-1} is corresponding to the fundamental stretching vibrations of O–H indicating the presence of hydrogen groups. The band positions and their assignments for observed FTIR bands are reported in Table 2 for Nd^{3+} doped ZnONM. [20-23].

UV-VISIBLE STUDY

The UV-visible study of Neodymium doped Zinc Oxide nanomaterial has been done by recording optical absorption using Spectrophotometer (2375 Double Beam Spectrophotometer and Varian Carry). The absorption spectra of Neodymium doped Zinc Oxide nanomaterial is shown in Fig. 4. It has been observed that the absorption peaks appears at wavelengths 457, 462 506, 542, 551, 604, 641,770 and 824nm as the concentration of dopant as 0.3 mol% in Neodymium doped Zinc Oxide nanomaterial matrix. The shifting occurred in the spectra may be due to the polarity of solvent used in the synthesis or may be due to the dispersion of rare earth oxide particles in the Neodymium doped Zinc Oxide nanomaterial matrix.

Nine absorption bands have been observed for 0.3mol% dopent concentration of Nd^{3+} ion ranging in ZnO Nanomaterial Figures. 4. The assignment of these bands from ground state $^4I_{9/2}$ to the excited states $^4F_{5/2}$, $^4F_{7/2}$, $^4F_{9/2}$, $^2H_{11/2}$, $^4G_{5/2}$, $^2K_{13/2}$, $^4G_{9/2}$, $^2K_{15/2}$ and $^4D_{3/2}$ energy levels are observed in ZnO nanomaterial. The values

(table 3) of Slater – Condon (F_k), Racah (E^k) and Landé′(ζ_{4f}) parameters have been computed by using the observed energies of the bands, E_{0j} and partial derivatives by the help of partial regression method [24].

JUDD OFELT INTENSITY PARAMETERS:
   A model to calculate radiative transition probabilities between electronic energy levels of trivalent rare-earth ions in solids has been developed by Judd [25] and by Ofelt [26]. The J-O model has been applied to several rare-earth-doped crystalline solids [27-29] and such studies have recently led to the prediction [30, 31] and observation of new crystalline rare-earth lasers.

   These parameters show in table 4 the general tendency $\Omega_2 < \Omega_4 < \Omega_6$. The same tendency is observed for Nd^{3+} ion in ZnONM. The Ω_λ parameters are very important since they are used in the calculation of laser parameters. Ω_2 parameter involves the long range terms in the crystal field potential and is most sensitive to the local structural changes. The intensity of $^4I_{9/2} \rightarrow {}^4G_{5/2}$, $^2G_{7/2}$ transition is the principal determiners of the Ω_2. This transition satisfies the selection rule $|\Delta J| \leq 2$ and is known as hypersensitive transition. A good agreement is found between the calculated and experimental line strengths.

FLUORESCENCE STUDY
   Fluorescence spectra of Neodymium doped Zinc Oxide nanomaterial samples are shown in Figure 5 and tables 5-7. Fluorescence study of Neodymium doped Zinc Oxide nanomaterial in which the concentration of dopant as 0.3 mol% shows emission peaks at wavelength 882.2, 1046 1380 and 1403nm. It has been observed from fluorescence spectra that the emission peak has maximum intensity at concentration 0.3mol% corresponding to wavelength 1048nm. A slight shifting has been observed in the spectra as concentration of dopants changes in the host matrix. The spectra show narrow emission peaks resulting from 4f-4f transitions within Nd^{3+} ions, with the most intense peak at 1046 nm corresponding to the $^4F_{3/2} \rightarrow {}^4I_{11/2}$ transition. Three other peaks are observed at 882.6 nm $^4F_{3/2} \rightarrow {}^4I_{9/2}$, 1383 nm $^4F_{3/2} \rightarrow {}^4I_{13/2}$ and 1408 nm $^4F_{3/2} \rightarrow {}^4I_{15/2}$ respectively.

RADIATIVE PROPERTIES
   The fluorescence spectra of Nd^{3+} doped ZnONM have been recorded and presented in Figs.5 The spontaneous emission probability (A) values has been calculated for the fluorescence bands at 882.6nm. Fluorescence branching ratio (β) ZnONMNd^{3+} have been calculated for different transitions The radiative life time 'τ' for a transition is reciprocal of spontaneous emission probability A. The stimulated emission cross-section, σ_p is calculated using the observed peak values of the fluorescence λ_p, their effective line width. Similar type results are also consistent with other rare earth metal ions doped like Sm-doped ZnO Nanomaterial [32].

IV. Conclusion
   ZnO: Nd^{3+} nanomaterial were successfully prepared by Chemical synthesis method. The crystal structure and surface of nanomaterials were analyzed by X-ray diffraction, TEM, FTIR, Absorption and Fluorescence characters were studied by high-resolution Fluorescence with 583nm excited wavelength. In the Nd^{3+} doped ZnO Nanomaterials, the diffraction peaks are almost similar. It is possible for Nd^{3+} ions cooperate with the matrix of ZnO particles to form Nd-Zn-O solid solutions. The X-ray diffraction and TEM results show that our samples are nanomaterials with particle size about 100–10 nm. SEM image shows approximate Hexagonal shape to ZnO nanoparticles and the size of the particles around 100-20nm. It demonstrates clearly the formation of Hexagonal ZnO nanoparticles, and change of the morphology of the nanoparticles with the Nd^{3+} different ions concentration The FTIR spectra of Nd^{3+} doped ZnONM consists of several peaks which are broad and moderate in band width..

   The spectroscopic properties of RE ions doped ZnONM have been analyzed on the basis of Judd-Ofelt theory. Judd - Ofelt intensity parameters Ω_2, Ω_4 and Ω_6 have been computed Nd^{3+} doped Zinc Oxide Nanomaterial specimens. Fluorescence spectra were studied in detail. They have strong PL intensity and their color can be modified by concentration of Nd^{3+}. The promising material for imaging technology, excited by He-Cd laser, ZnO: Nd^{3+} also be excited by a 583nm diode. The optimal concentration of Nd^{3+} is 0.3mol%. ZnO: Nd^{3+} Nanomaterial have been the subject of much research due to their wide application in semiconductor-electronic technologies. The influence of concentration has been discussed. The neodymium doped ZnONMNd specimen, suggesting that, these transitions are to be used as a laser transition. We used photocatalytic and sonocatalytic techniques independently for degradation of organic dyes. These both techniques can be used collectively for fast degradation of organic dyes.
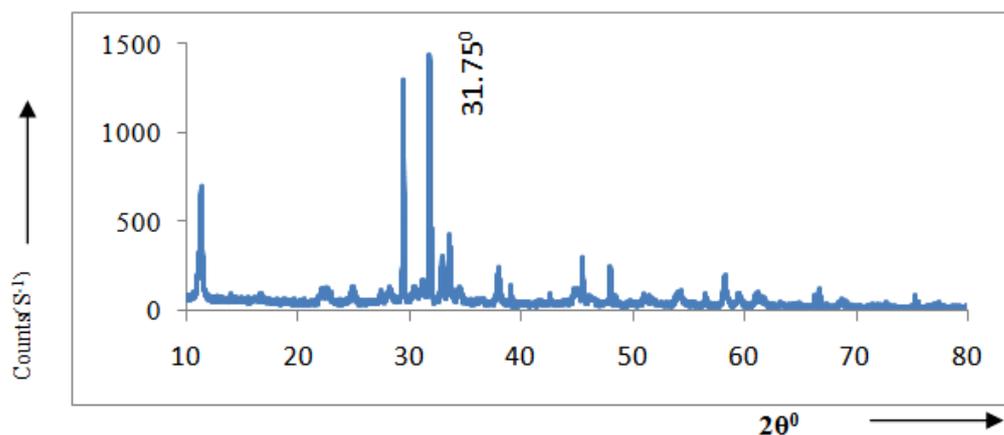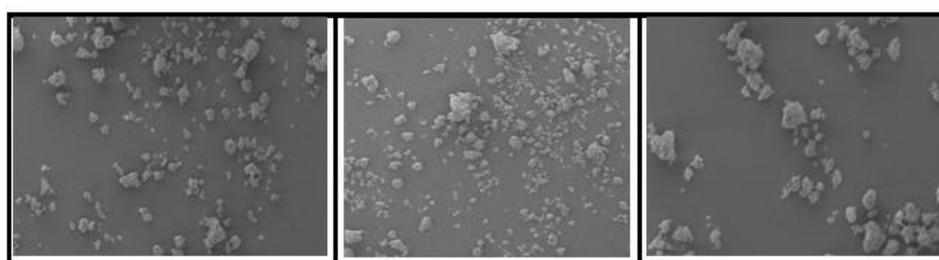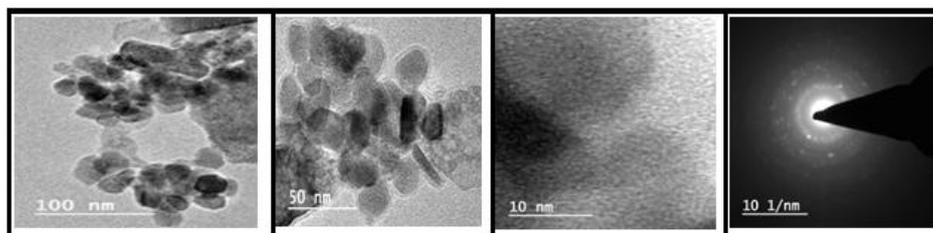
Fig .1-XRD pattern of ZnO Nanomaterial with 0.3mol% of Nd^{3+} ion

SEM-ZnONd

TEM-ZnONd

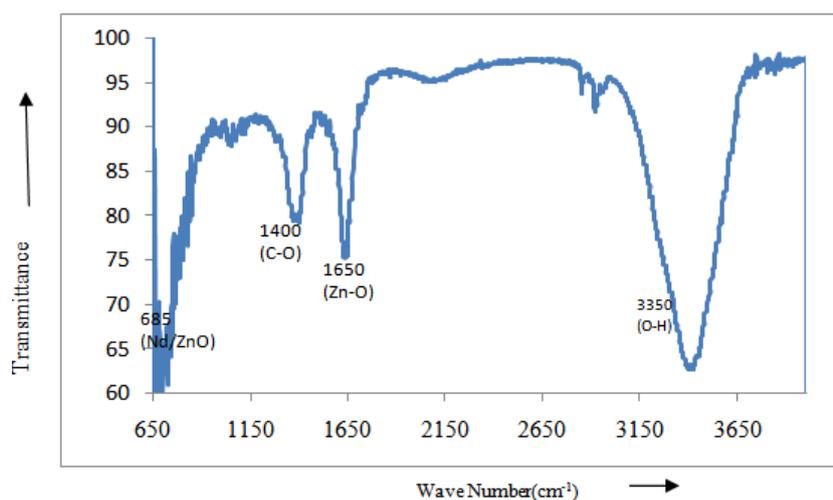**Fig. 2:-** SEM and TEM micrograph ZnO Nanomaterial with 0.3mol% Nd^{+3} ion.

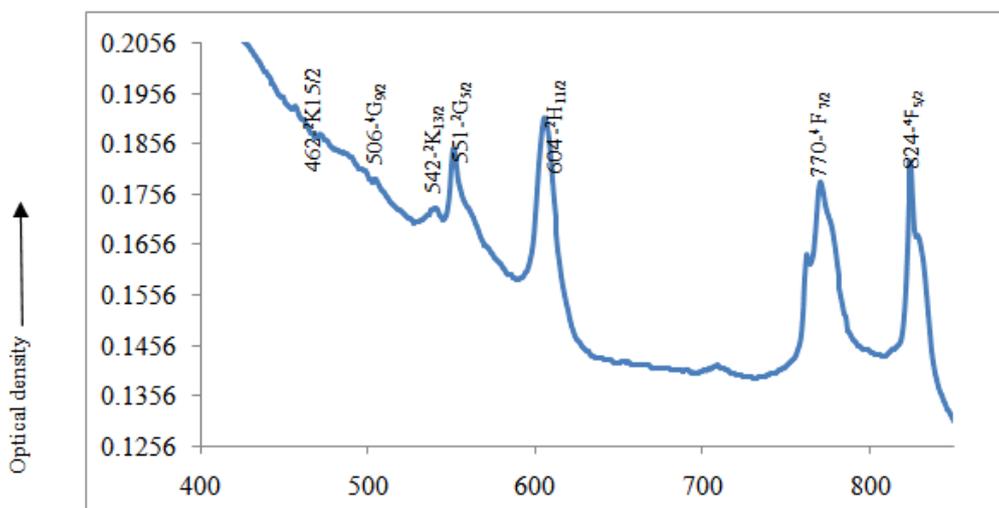**Fig 3:-** FTIR pattern of ZnO Nanomaterial with 0.3 mol% of Nd^{3+}

Fig. 4:- Absorption spectrum of ZnO Nanomaterial with 0.3 mol % Nd^{3+} ion

Fig.5:- Fluorescence spectrum of ZnO Nanomaterial with 0.3 mol % Nd3+ ion.

TABLE 1:- Observed peak height and sharp diffraction of Nd^{3+} doped ZnO Nanomaterial with 0.3mol% concentration

ZnONMNd	0.3 mol %	1434	31.75^0

TABLE 2:-Observed band of Nd^{3+} doped ZnO Nanomaterial with 0.3mol% Concentrations

ZnONMNd	Assignment
Wave Number (cm^{-1})	BONDS
685	Nd/ZnO
1400	C-O
1650	Zn-O
3350	O-H

TABLE 3:- Computed values of Slater - Condon, Lande′, Racah, Nephelauxetic ratio and Bonding parameters for 0.3mol% Nd^{3+} doped ZnO Nanomaterial

PARAMETERS	FREE IONS	ZnONMNd
$F_2(cm^{-1})$	331.09	335.63
$F_4(cm^{-1})$	50.72	51.99
$F_6(cm^{-1})$	5.15	5.04
$\zeta_{4F}(cm^{-1})$	884.00	882.24
$E^1(cm^{-1})$	5024.00	5004.12
$E^2(cm^{-1})$	23.90	24.99
$E^3(cm^{-1})$	497.00	483.90
F_4 / F_2	0.15	0.17
F_6 / F_2	0.02	0.013
E^1/E^3	10.11	9.674724
E^2/E^3	0.05	0.05
β'	----	0.8746
$b_{1/2}$	-----	0.025

TABLE 4:- Judd Ofelt Intensity Parameters for Nd^{3+} doped ZnO Nanomaterial with 0.3mol% doping concentration

Omega parameters	ZnONMNd
	0.3mol%
$\Omega_2(10^{-20})$	1.25
$\Omega_4(10^{-20})$	8.32
$\Omega_6(10^{-20})$	8.34
Ω_4/Ω_6	0.99

TABLE 5:- Observed values of wavelength (λ), energy (ν) and half band width (Δ λ_{eff}) of various fluorescence peaks for Nd^{3+} doped ZnO Nanomaterial with 0.3mol% doping concentration

Dopant concentration	Excitation λ(nm)	Assignment	λ(nm)	Δλ_{eff} (nm)
0.3 mol%	583	$^4F_{3/2} \rightarrow {}^4I_{9/2}$	882.6	12.6
		$^4F_{3/2} \rightarrow {}^4I_{11/2}$	1048.0	100
		$^4F_{3/2} \rightarrow {}^4I_{13/2}$	1383.0	100
		$^4F_{3/2} \rightarrow {}^4I_{15/2}$	1408.0	100

TABLE 6:- Spontaneous emission probability (A), Fluorescence Branching ratio (β) and Radiative time (τ) for various levels of Nd^{3+} doped Zinc Oxide Nanomaterial

Dopant concentration	Excitation λ(nm)	Assignment	λ(nm)	A(Sec^{-1}) (10^{+3})	Δλ_{eff} (nm)	β	τ (μ sec)
0.3mol%	583	$^4F_{3/2} \rightarrow {}^4I_{9/2}$	882.6	1.91	12.60	4.40	376.00
		$^4F_{3/2} \rightarrow {}^4I_{11/2}$	1048.0	2.00	100.00	0.46	501.00
		$^4F_{3/2} \rightarrow {}^4I_{13/2}$	1383.0	0.40	100.00	0.09	247.00
		$^4F_{3/2} \rightarrow {}^4I_{15/2}$	1408.0	0.002	100.00	0.06	376.00

TABLE 7:- Emission cross section (σ_p) for various levels of Nd^{3+} doped Zinc Oxide Nanomaterial

Dopant concentration	Excitation λ(nm)	Assignment	λ(nm)	$\sigma_p(10^{-20})$
0.3mol%	583	$^4F_{3/2} \rightarrow {}^4I_{9/2}$	882.6	2.80
		$^4F_{3/2} \rightarrow {}^4I_{11/2}$	1048.0	6.47
		$^4F_{3/2} \rightarrow {}^4I_{13/2}$	1383.0	3.97
		$^4F_{3/2} \rightarrow {}^4I_{15/2}$	1408.0	2.80

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