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Spectral, Structural and Characterization of NeodymiumIons Doped Zinc Oxide Nanomaterial

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Abstract: Zinc oxide with neodymium ions nanomaterials were synthesized by using the chemical synthesis method. The materials were characterized by XRD, FTIR, TEM, SEM and spectral analysis. From XRD, TEM and SEM images, the estimated average particle size are 20, 100 and 200nm respectively. Nearly hexagonal shapes for the dark spots in the TEM images indicate that the ZnO nanoparticles are almost hexagonal. SEM demonstrates clearly the formation of cluster type of ZnO nanoparticles and change of the morphology of the nanoparticles with the Nd^{3+} different ions concentration. UV- Visible absorption spectrum of the ZnO:Nd^{3+} nanomaterial was analyzed on the basis of Judd-Ofelt (J-O) theory. Nine absorption and four fluorescence bands have been observed at room temperature. Energy interaction and intensity parameters have been computed Fluorescence band have been assigned to transitions $^4F_{3/2} \rightarrow {}^4I_{9/2}$, $^4F_{3/2} \rightarrow {}^4I_{11/2}$, $^4F_{3/2} \rightarrow {}^4I_{13/2}$, and $^4F_{3/2} \rightarrow {}^4I_{15/2}$. The radiative properties were computed using the J-O intensity parameters and fluorescence data. The value of emission cross-section is an important parameter and signifies the rate of energy extraction from the optical material.

Keywords: Absorption and Fluorescence Spectrum, Judd-Ofelt (J-O) Parameters, Radiative Properties

1. Introduction

The rare earths are fairly abundant, but their availability is somewhat limited, primarily because their concentration levels in many ores are quite low (less than 5 percent by weight). An economically viable source should contain more than 5 percent rare earths, unless they are mined with another product [1]. RE doped ZnO nano material are those nano material in which two types of doped ions are used, one in which the outer orbital is exposed to the environment like the d-orbital in the transition metal ions and the other in which the outer orbital is shielded one, like the f-orbital in the tripositive rare earth ions. Nanosize particles of semiconductor materials have gained much more interest in recent years due to their desirable properties and applications in different areas such as catalysts [2], sensors [3], photoelectron devices [4], highly functional and effective devices [5]. These nanomaterials have novel electronic, structural, and thermal properties which are of high scientific interests in basic and applied fields [6].

The *Judd-Ofelt* theory [7, 8] is usually adopted to calculate transition probabilities from the data of absorption cross-sections of several *f-f* transitions. According to this theory, the strength of an *f-f* transition may be expressed by the sum of the products of three intensity parameters Ωi (i=2, 4, 6) times the squared matrix elements $U(i)$ between the initial J-states and the terminal J'-state. Once the phenomenological parameters Ωi have been calculated, it is possible to derive the strength of any absorption or emission transition, as well as the stimulated emission cross-section, the fluorescence branching ratio from level J to J', and the radiative lifetime of an excited level. For understanding the luminescent properties of rare earth ions, it is necessary to know their key energy levels. The energy level may be divided into three categories, those corresponding to $4f^n$ configuration, $4f^{n-1}5d$ configuration, and those corresponding to charge transfer involving the neighboring ions [9].

The optical studies confirm that Nd ion doping in ZnO leads to reduction in band gap and exhibits higher absorption

in the visible region. Nd doping in ZnO films was found to have enhanced photo catalytic activity and antibacterial activity. The influence of Nd doping on the photo catalytic activity of ZnO for the degradation of methylene blue dye was studied under visible light illumination. The decrease in grain size and light absorption over an extended visible region by Nd ion doping in ZnO nanofilms contributed equally to improve the photo catalytic activity [10].

In present work an effort has been made in the synthesis of Nd^{3+} doped ZnO nanomaterials by using simple techniques based on 50-70°C temperature chemical synthesis method to obtain various sizes of nanoparticles. Absorption and fluorescence spectra of Nd^{3+} doped ZnO have been recorded in the visible region at room temperature. The Nd: ZnO nanomaterials have been characterized by XRD, FTIR, TEM and SEM. Judd-Ofelt, Energy interaction and radiative propertieshave computed.

2. Experimental Method

The $ZnO:Nd^{3+}$ nanomaterials prepared by chemical synthesis method [11]. $ZnO:Nd^{3+}$ nanoparticles have been tried in alcoholic media like ethanol, methanol or propanol. In alcoholic media growth of oxide particles is slow and convenient [12]. Different solutions have been prepared by dissolving 0.2725 g of $ZnCl_2$ (10^{-1} M, 20 ml), 0.545 g NaOH (10^{-1} M, 100 ml) and glycerol in ethanol. Glycerol slowly added to NaOH solution while it has been continuously stirred.

The resulting solution has been stirred for one hour before adding $ZnCl_2$ and (0.2mol %) Nd^{3+} ions solution to it. After two hours of constant stirring a milky white solution obtained. Size selective precipitation has been carried out using acetone as a non-solvent. The precipitate has been washed in methanol or ethanol has been allowed to evaporate at room temperature to obtain ZnO nanoparticles in white powder form. Similar type of method of ZnO with Pr^{3+} ion is also observed by Sharma et al for the nanopowder [13].

3. Characterization of $ZnO:Nd^{3+}$ Nanomaterials

3.1 Structural Characterization

ZnO nanomaterials with Nd^{3+} ionshave been characterized by XRD, SEM, TEM, FTIR to get exact information about the structure, surface morphology and particle size etc.

3.1.1. X-Ray Diffraction (XRD)

Sharp peaks in the XRD patterns indicate crystalline nature of the samples. XRD pattern of ZnO nano materials with Nd^{3+} ion have been shown in Figure 1. By using the Debye-Scherrer equation, $D=0.89\lambda/\beta\cos\theta$, where D is the average crystalline size [14]. The $ZnO:Nd^{3+}$ nanopowder's crystalline size is estimated to be about 100-20 nm. The peak position at 1434 and sharp diffraction bands at 2θ° (31.730) have been observed in $ZnO:Nd^{3+}$ nanoparticles (Figure 1). Similar type of behavior ofLa-doped ZnO ion is also observed by Mai M. A. Ahmed et al for the nano structured thin films [15].

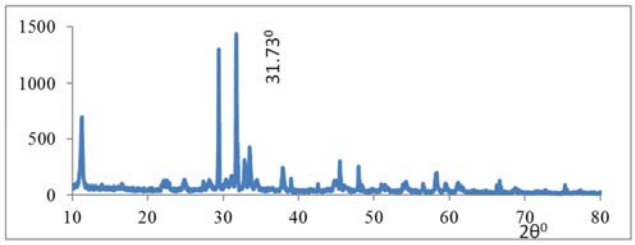

Figure 1. *XRD pattern of $ZnO:Nd^{3+}$ $ZnO:Nd^{3+}$ Nanomaterials with doping of 0.2 mol % Nd^{3+} ions.*

3.1.2. Scanning Electron Microscopy [SEM]

SEM images of ZnO nano materials with Nd^{3+} have been recorded and shown in Figure 2. The image shows cluster to ZnO nanoparticles and the size of the particles around 200-20 nm. It demonstrates clearly the formation of cluster type of ZnO nanoparticles, and change of the morphology of the nanoparticles with the Nd^{3+} different ions concentration. Similar type of behavior of ZnO: Dy^{3+} (3 wt%) ion is also observed by Prathibha Vasudevan et al for the nanophosphors [16].

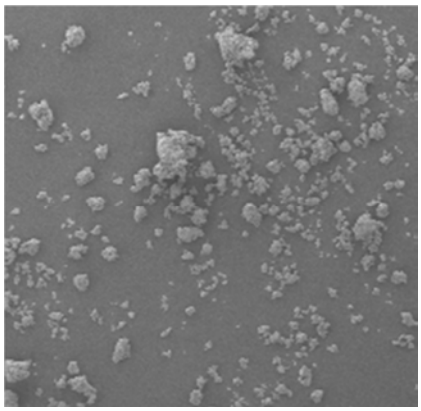

Figure 2. *SEM micrograph $Nd^{3+}ZnO:Nd^{3+}$ Nanomaterials with doping of 0.2 mol % Nd^{3+} ions.*

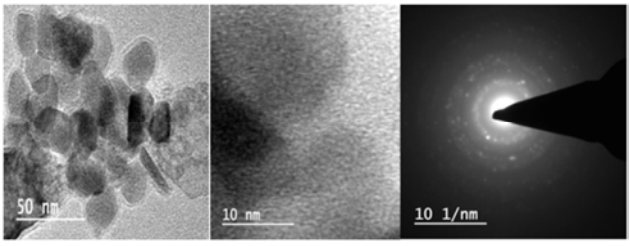

Figure 3. *TEM micrograph $Nd^{3+}ZnO:Nd^{3+}$ Nanomaterials with doping of 0.2 mol % Nd^{3+} ions.*

3.1.3. Transmission Electron Microscope [TEM]

TEM images of $ZnO:Nd^{3+}$ Nanomaterial have been recorded and shown in Figure 3. From figure 3, nearly hexagonal shapes for the dark spots in the images indicate that the ZnO nanoparticles are almost hexagonal. The estimated average particle size corresponding to 0.2 mol% are 50, 100nm. These results are also consistent with other rare earth metal ions doped like Gd-doped ZnO Nanomaterial [17].

3.1.4. Fourier Transform Infrared (FT-IR) Spectra

FT-IR spectra of Nd^{3+} doped Zinc Oxide Nanomaterial have been recorded and shown in Figure 4. This spectral region encompasses several important stretch modes involving hydrogen bonded to oxygen and ZnO and collected in Table 1. FTIR spectra of Nd^{3+} doped ZnO were recorded, employing on PC using OMNIC software. The FTIR spectra of ZnO nanomaterial with doping 0.2mol% Nd^{3+} ions consists of several peaks which are broad and moderate in band width. Similar results are suggested by Odireleng et al [18] for Eu^{3+}, bonds observed from the 1.0 mol%Eu^{3+} doped ZnO spectrum around 714, 1050, 1080 and 3609 cm^{-1} are assigned to CO_3^2 vibration mode, C-H stretching mode and O-H group respectively [19-21].

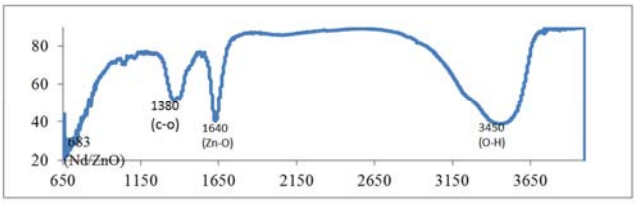

Figure 4. *FTIR pattern of ZnO:Nd^{3+} ZnO:Nd^{3+} Nanomaterials with doping of 0.2 mol % Nd^{3+} ions.*

Table 1. *FTIR-Observed band of ZnO:Nd^{3+} nanoparticles with doping 0.2mol% Nd^{3+} ions in Zinc Oxide.*

S. No	ZnO:Nd^{3+}	Assignment
	Wave Number (cm^{-1})	Bonds
1	677	Nd/ZnO
2	1380	C-O
3	1640	Zn-O
4	3450	O-H

3.2. Spectral Characterization

3.2.1. UV-Visible Absorption Spectrum

The absorption spectrum of Nd^{3+} doped ZnO Nanomaterial has been presented in Figure5 in terms of wave length (nm) vs relative absorption (I_o /I), where I and I_o are intensities of the radiation transmitted through specimens.

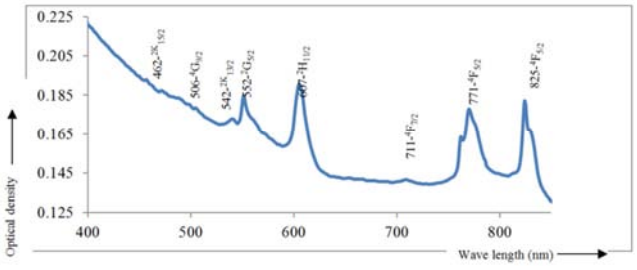

Figure 5. *Absorption spectrum of ZnO:Nd^{3+} ZnO:Nd^{3+} Nanomaterials with doping of 0.2 mol % Nd^{3+} ions.*

Nine absorption bands have been observed in ZnO:Nd^{3+} (0.2mol%) nanomaterial. 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 and collected in Table 2. From the absorption band, Energy interaction i.e. Slater – Condon (F_k), Racah (E^k) and Lande′ (ζ_{4f}) parameters have been computed and collected inTable 3.

Table 2. *Experimental energy (E_{exp}) and Calculated energy (E_{cal}) with their Differences (ΔE) for various absorption bands of ZnO:Nd^{3+} (0.2mol%) nanomaterials.*

Absorption levels	E_{exp} (cm^{-1})	E_{cal} (cm^{-1})	ΔE
$^4F_{5/2}$	12953	12645.05	307.94
$^4F_{7/2}$	14064	13649.72	414.283
$^4F_{9/2}$	14727	15436.54	-709.539
$^2H_{11/2}$	16447	16412.01	34.9941
$^4G_{5/2}$	18083	16837.82	1245.178
$^2K_{13/2}$	18450	18879.05	-429.044
$^4G_{9/2}$	19762	19836.7	-74.464
$^2K_{15/2}$	21786	21416	370.003
$^4D_{3/2}$	26385	27877	-1492.299

Table 3. *Computed values of Slater - Condon, Lande′, Racah, Nephelauxetic ratio, Bonding and Judd-Ofelt Parameters for ZnO:Nd^{3+} (0.2mol%) nanomaterials.*

PARAMETERS	FREE IONS	ZnO:Nd^{3+} (0.2mol %)
F_2 (cm^{-1})	331.09	340.86
F_4 (cm^{-1})	50.72	43.22
F_6 (cm^{-1})	5.15	5.72
ζ_{4F} (cm^{-1})	884.00	895.595
E^1 (cm^{-1})	5024.00	5033.51
E^2 (cm^{-1})	23.90	24.91
E^3 (cm^{-1})	497.00	480.85
F_4 / F_2	0.15	0.12
F_6 / F_2	0.02	0.01
E^1/E^3	10.11	10.46
E^2/E^3	0.05	0.05
β’	----	0.9291
$b_{1/2}$	-----	0.02
Ω_2 (10^{-20})	-	3.40
Ω_4 (10^{-20})	-	5.67
Ω_6 (10^{-20})	-	4.04

3.2.2. Judd-Ofelt Intensity Parameters

The phenomenological JO parameters have been calculated from observed line strength and collected in Table 3. These parameters show the general tendency $\Omega_2 < \Omega_6 < \Omega_4$. 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. It is necessary to study the various transitions giving rise to fluorescence and compute their radiative properties utilizing the Judd-Ofelt parameters and fluorescence data. [22].

3.2.3. Fluorescence Spectrum

The fluorescence spectra of Nd^{3+} doped ZnO Nanomaterial have been recorded and shown in Figures 6&7 in terms of wavelength (nm) vs relative fluorescence in arbitrary units (a.u.). The wave length of exciting radiation is written on each spectrum. Fluorescence peak values (λ_p), and effective line width ($\Delta\lambda_{eff}$) for Nd^{3+} doped ZnO nanomaterials at exciting wavelength (λ) have been given in Table 4. Four fluorescence

bands have been observed in ZnO:Nd^{3+} (0.2mol %) nanomaterials. They have been assigned to transitions $^4F_{3/2} \rightarrow {}^4I_{9/2}$, $^4F_{3/2} \rightarrow {}^4I_{11/2}$, $^4F_{3/2} \rightarrow {}^4I_{13/2}$ and $^4F_{3/2} \rightarrow {}^4I_{15/2}$. The values of 'β' for ZnO:Nd^{3+} has been calculated for different transitions. The results are given in Table 5. The values of 'β' are maximum for $^4F_{3/2} \rightarrow {}^4I_{13/2}$ transition and is closely followed by $^4F_{3/2} \rightarrow {}^4I_{9/2}$ for the neodymium doped ZnOspecimens, suggesting that, these transitions are to be used as a laser transition. The radiative life time 'τ' for a transition is reciprocal of spontaneous emission probability A. The values of 'τ' are given in Table 5 for ZnO:Nd^{3+} specimens. The minimum 'τ' values have been obtained for $^4F_{3/2} \rightarrow {}^4I_{13/2}$ and $^4F_{3/2} \rightarrow {}^4I_{11/2}$ transitions in comparison to two others transitions [23, 24].

The stimulated emission cross-section, σ_p is calculated using the observed peak values of the fluorescence λ_p, their effective line width. $\Delta\lambda_{eff}$ (Table 4) and the A values (Table 5). The σ_p parameter is the most important parameter. Its value (Table 5) signifies the rate of energy extraction from the laser material and is generally used to predict laser action in RE doped material prepared on laboratory scale. A large stimulated emission cross-section is of benefit for a low threshold and a high gain in laser operation.

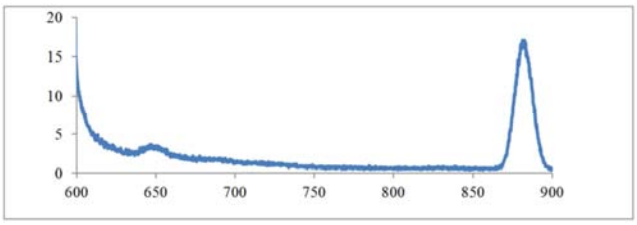

Figure 6. *Fluorescence spectrum of ZnO:Nd^{3+} Nanomaterials with doping of 0.2 mol % Nd^{3+} ions.*

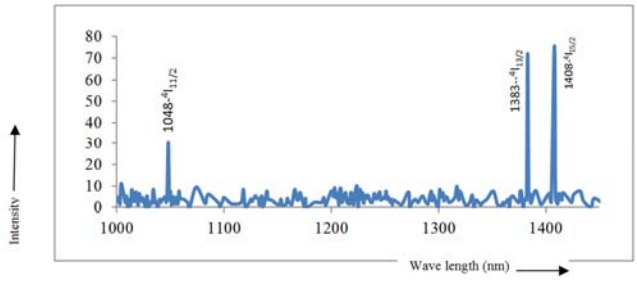

Figure 7. *Fluorescence spectrum of ZnO:Nd^{3+} ZnO:Nd^{3+} Nanomaterials with doping of 0.2 mol % Nd^{3+} ions.*

Table 4. *Matrix elements (U^{λ}) [25-27] and Observed values of wavelength (λ) and half band width ($\Delta\lambda_{eff}$) of various fluorescence peaks for ZnO:Nd^{3+} (0.2mol%) with Excitation wavelength.*

Excitation Wavelength λ(nm)	Assignment	$[U^{(2)}]^2$	$[U^{(4)}]^2$	$[U^{(6)}]^2$	Λ (nm)	$\Delta\lambda_{eff}$ (nm)
583	$^4F_{3/2} \rightarrow {}^4I_{9/2}$	0.0000	0.2290	0.0550	882.6	13.0
	$^4F_{3/2} \rightarrow {}^4I_{11/2}$	0.0000	0.1330	0.3630	1047.0	100
	$^4F_{3/2} \rightarrow {}^4I_{13/2}$	0.0000	0.0000	0.2310	1380.0	100
	$^4F_{3/2} \rightarrow {}^4I_{15/2}$	0.0000	0.0000	0.0160	1405.0	300

Table 5. *Spontaneous emission probability (A), Fluorescence branching ratio (β) and Radiative time (τ) and stimulated emission cross section (σ_p) for various levels of ZnO:Nd^{3+} (0.2mol%) nanomaterials.*

Assignment	λ(nm)	A(Sec^{-1}) (10^{+3})	β	τ (μ sec)	σ_p (10^{-20})
$^4F_{3/2} \rightarrow {}^4I_{9/2}$	882.6	1.23	5.15	813.00	1.73
$^4F_{3/2} \rightarrow {}^4I_{11/2}$	1047.0	1.08	0.42	929.00	2.32
$^4F_{3/2} \rightarrow {}^4I_{13/2}$	1380.0	0.19	0.09	509.00	1.28
$^4F_{3/2} \rightarrow {}^4I_{15/2}$	1405.0	0.001	0.06	771.00	2.15

4. Conclusions

Nd^{3+} doped ZnO nanomaterial have been prepared by chemical synthesis method. Structural, spectral, and morphological composition were studied after a successfully synthesis and characterized by using different techniques such XRD, SEM, TEM, FTIR, UV-VIS absorption and fluorescence spectrum. The XRD study revealed, ZnO:Nd^{3+} nanopowder's crystalline size is estimated to be about 100-20 nm. From TEM image, nearly hexagonal shapes for the dark spots in the images indicate that the ZnO nanoparticles are almost hexagonal. The estimated average particle size are 50, 100nm. SEM demonstrates clearly the formation of cluster type of ZnO nanoparticles and change of the morphology of the nanoparticles with the Nd^{3+} different ions concentration. Nine absorption and four fluorescence bands have been observed in ZnO:Nd^{3+} nanomaterials at room temperature. The energy interaction and Judd-Ofelt parameters have been computed using absorption spectrum data. The radiative properties, such as radiative transition probability, branching ratio, stimulated emission cross section were investigated using the J-O intensity parameters and fluorescence data.

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