Synthesis, characterization and photoluminescence study of Nd^{3+}/Ho^{3+} doped $GdPO_4$ nanomaterials

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Abstract

The present study reports the preparation of nanomaterials by successful method of solvothermal synthesis of $GdPO_4$:Nd^{3+}/Ho^{3+}which appear upconversion and downconversion nanomaterials in luminescence. These nanophosphors emit green and red color light whereas $GdPO_4$: Nd^{3+}/Ho^{3+}downconversion peaks are observed at 460, 550, 672, and 791nm after excitation at 300nm and 532nm. We can observe a non-radiative resonance energy transfer take place along with the P-O CT (Charge Transfer) band of Holmium ions (Ho^{3+}) there by improving emission of Ho^{3+}ions we can also observed that upconversion (UC) nanophosphor $GdPO_4$: Nd^{3+}/Ho^{3+}, this allows for large-scale luminescence creation and improvement. The upconversion emission spectra reveals a clear expectant peak at 550 & 791 nm, 672 and 791 nm of Ho^{3+}ion on high 808 nm laser illumination is observed due to the presence of Nd^{3+}.the method produce well crystalline nanoparticles with a range of a small number of nanometre's to tens of nanometre's as from the experimental study at 300nm & 532nm excitation, $GdPO_4$: Nd^{3+}/Ho^{3+} yielded. A high quantum yield value, indicating that these experimental studies have a wide range of applications for nanophosphor materials.

Keywords: Solvothermal method; Neodymium ion; Holmium ion, Gadolinium ion, Photoluminescence, upconversion, downconversion.

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1. Introduction

Rare-Earth (RE) based materials is constantly growing more attention because of their facile synthesis process, development of various RE doped nanomaterials have been used in various applications extending from solar cell, temperature sensor, spectral convertor, biomedical and high-tech fields. Till now there is an inadequate protocol for the largescale production of dual mode nanomaterials. Interestingly, the RE orthophosphates possess high thermal (upto 2200^0C) and chemical stability, desired optical properties, low solubility and often referred as prominent host for adsorption of nuclear waste. In our existing work, we are reporting solvothermal route for the synthesis of $GdPO_4$: Nd^{3+}/Ho^{3+}dual mode conversion nanomaterial. Due to this method, the size and shape can be tuned, which can enhance the luminescence property Upon CW laser excitation, in addition $GdPO_4$ nanomaterials were used as a host for downconversion(DC)/upconversion(UC) luminescence. This is due to the occurrence of the allowable transition including the charge transfer (CT) process of O^{2-} towards Gd^{3+} which absorbs light from 300nm by an extreme absorption at 532 nm. Based upon UV Light material the emission spectrum shows the sharp peaks at~460 ~550, ~672and ~791 nm peaks of Ho^{3+} ion and same in the case of 300 nm excitation, which shows the downconversion. Here, Nd^{3+} acts as sensitizer which helps in enhancing the Ho^{3+} ion emission intensity in the spectrum. Under near-IR light stimulation near 808 nm, Ho^{3+} emits strongly displays the $GdPO_4$ upconversion emission spectrum at room temperature: Ho^{3+}, Nd^{3+}. A 808nm laser was used to stimulate the sample. In this study, we used a solvothermal method to make $GdPO_4$:Ho^{3+}/Nd^{3+} nanophosphor. Excited at 808

nm (due to Nd^{3+} absorption), 300nm (Indirect P-O Charge Transfer Band, CTB), and 460 nm (due to Ho^{3+} absorption) have been used to study its bright behaviour. The processes of DC, ET efficiency and UC are deliberated. The sensing behaviour of these types of particles will improve its usefulness in optical, display devices, security, and bio-imaging when the functional groups on the manufactured nanomaterials are activated.

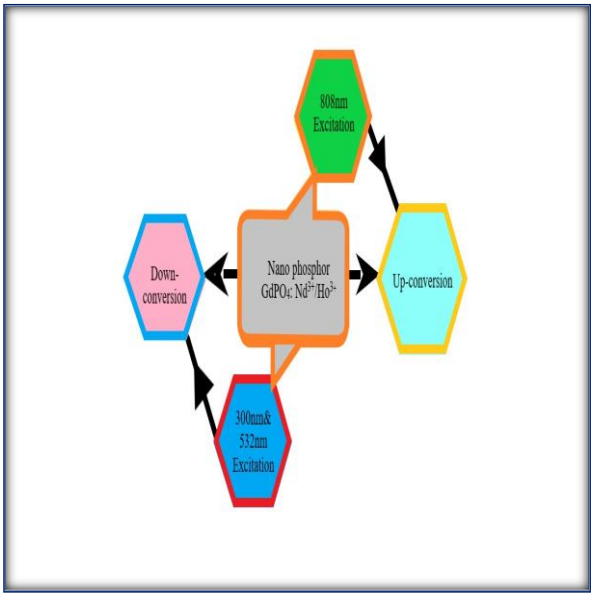

Figure-1: Schematic diagram represents the synthesis and properties studies of $GdPO_4$: Nd^{3+}/Ho^{3+} at 808nm and 300nm & 532nm excitations.

2. Experimental Methods

2.1. Chemicals Compounds and Synthesis Method:

Chemicals: As reactants, highly purified Analytical Grade Reagents were utilised.

$Gd(ac)_3).XH_20$ Gadolinium (III) acetate Hydrated, Ammonium dihydrogen phosphate $(NH_4)_2H_2PO_4$,holmium (III) acetate Hydrated $(Ho(ac)_3).XH_20$,Neodynium(III) Acetate Hydrated $Nd(ac)_3.XH_20$,dil HCl, Ethylene glycol , dil NaOH, Deionised water used as precursors.

Synthesis procedure:

Synthesis of GdPO4: Ho^{3+}/Nd^{3+} Nanoparticles:

Using solvothermal method, we have prepared the sample.

The samples that were prepared were as follows: $GdPO_4$:Ho^{3+}(1%), Nd^{3+}(20%) doped $GdPO_4$ luminous nanoparticles ($GdPO_4$:Ho^{3+}, Nd^{3+}) contain 1% Ho^{3+} and 20% Nd^{3+}. This product was made using solvothermal synthesis. In a conventional synthesis, 730.76 mg of $(CH_3CO_2)_3Gd.XH_2O$, 8.7 mg of $(CH_3CO_2)_3Ho.XH2O$, and 178.48 mg of $(CH3CO_2)_3Nd.XH2O$ were added to 5 ml of concentrated HCl and it is heated until dissolved. Metal ions were dissolved in a clean and transparent solution. To eliminate the left over HCl, 10 ml of deionized water was added alternately, followed through heating (80°C) at least five times, the evaporation procedure was repeated. Furthermore, in 10 ml of deionized water, 298.2 mg of $(NH_4)_2HPO_4$was dissolved. In order to come up with a clear solution, 2.64 g of NaOH was dissolved in 10 ml of deionized water. It was added drop by drop to the $(NH_4)_2HPO_4$ solution until it turned translucent. In a 100 ml round bottom flask. The evaporated metal ion solution was shifted, 20 ml of EG added to the mixture and it was refluxed at least 10 minutes at 75 °C before adding the $(NH_4)_2HPO_4$ solution drop wise. While adding $(NH_4)_2HPO_4$ solution to the round bottom flask, a pale-yellow hue was detected; however, following heating at 120°C for 2 hours, the colour was converted from pale yellow to white. As a result, a white precipitate formed, which was allowed to cool at room temperature. The dry powder was collected by centrifuging it at 5000 rpm for 5 minutes, washing it twice with 10 ml acetone, and drying it under an IR lamp. The prepared sample was heated at 900°C for 4 hours. In the same way, different Ho^{3+} concentrations (3, 5, and 7 at. percent) and fixed amounts of Nd^{3+} (10 at. percent) doped $GdPO_4$ nanoparticles were made, and the samples were subsequently annealed at 900°C for 4 h.

2.2 Characterization:

Instrumentation by using synchrotron angle dispersive X-ray diffraction (Source: India) the crystallinity and average crystallite size of the samples were checked. A scanning electron microscope was used to perform microstructural characterizations for particle size measurements and surface morphology (SEM: QUANTA 200). FTIR Spectroscopy was used to investigate the vibrational structure of

the synthesised samples, and UC emission was measured with a Mono Chromator(iHR321, Horiba Jobin Yvon) well-found with a Photomultiplier tube. The samples were excited by the usage of 808 nm radiation from a diode laser. Upconversion research using photoluminescence excitation (PLE). Beyond UV excitation at 300-532 nm, the downconversion emission spectra of $GdPO_4:Ho^{3+}/Nd^{3+}$ is studied using the excitation WL (wavelength) of a Nd:YAG Laser.

3. Result and Discussion

3.1 XRD Sample Study:

We can observe the XRD form of the nanophosphor material $GdPO_4$: 1 at. percent Ho^{3+} and 20 at. percent Nd^{3+} codoped $GdPO_4$ (which can be annealed at 900°C and which was referred to as $GdPO_4:Ho^{3+}/Nd^{3+}$) is shown in Figure-2, strong and Sharp peaks arise in the diffraction patterns, which are constant with the usual Monoclinic Phase. The absence of an impurity peak indicates that the dopants are evenly distributed throughout the host lattice. In the XRD pattern, the peak locations of two of the greatest intensity peaks, here the XRD patterns show and the diffraction patterns occurred are well matched with the tetragonal structure of standard (pure $GdPO_4$). Because their ionic radii are similar on the basis of CN 9, Nd^{3+} and Ho^{3+} ions were substituted at Gd^{3+} sites of the $GdPO_4$ lattice. Where nine O^{2-} ions encircle the Gd^{3+} ion, Gd^{3+} was coupled to nine O^{2-} ions, generating a pentagonal interpenetrating tetrahedral polyhedron in the nanophosphor. Which has the monoclinic structure of $GdPO_4$ (PITP).

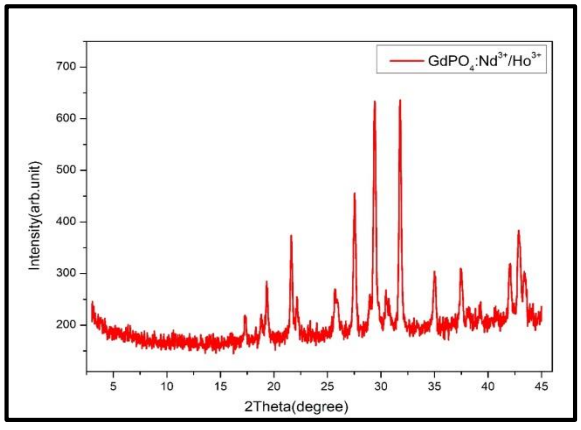

Figure-2: XRD pattern of $GdPO_4:Nd^{3+}/Ho^{3+}$ samples respectively.

3.2 SEM Study:

Here Figure-3 indicate the SEM image for $GdPO_4:Ho^{3+}/Nd^{3+}$nanophosphor material annealed at 900°C. Which appearances the uneven shapes of nanoparticles (a large number of sponge shape along with a rare no.of (cone , spherical shapes). The usual size got from spherical particles is ~50nm. The individual basic compositional images of Gd, P, Nd, Ho, and O were revealed in Figure-3.

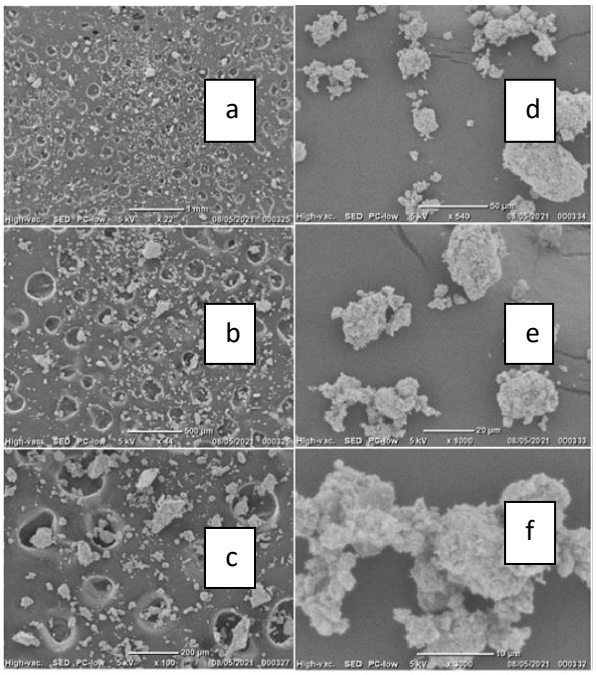

Figure-3: SEM images of $GdPO_4:Ho^{3+}/Nd^{3+}$

3.3 FTIR:

The vibrational structure of the produced materials was investigated using $1cm^{-1}$ resolution spectroscopy of FTIR (SHIMADZU spectrophotometer) Figure-4. shows what we're talking about.

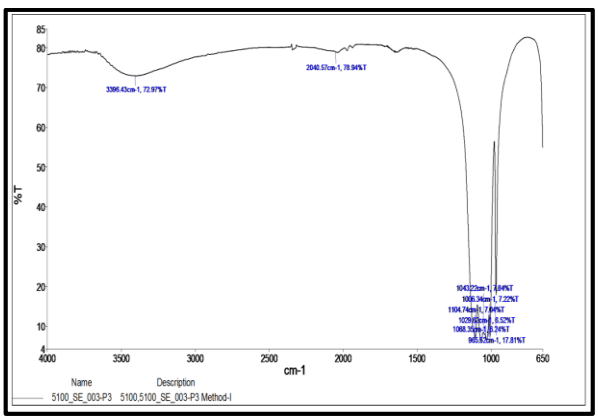

Figure-4: FTIR of $YPO_4:Ho^{3+}/Nd^{3+}$

3.4 Photoluminescence Study:

Photoluminescence of $GdPO_4$: Ho, Nd:

UC Study: Anti-stokes luminescence causes upconversion luminescence, this is an optical process in which low energy photons are absorbed and high energy photons are emitted (Multiphoton).

It depends on the host and codopant concentrations, Ho^{3+} can emit green or red emissions. The downconversion(DC) and upconversion(UC) emission spectra of $GdPO_4$: Ho^{3+},Nd^{3+} are shown in Figures 4&5.

Interestingly, we made $GdPO_4$:0.01Ho^{3+}/0.2Nd^{3+} nanophosphor material from Ho^{3+} doped $GdPO_4$ (Ho^{3+} = 1at.%) and Nd^{3+}doped $GdPO_4$ (20at.%). For UC and DC luminescence experiments, the optimised nanophosphor material is further investigated. Figure-5 shows the upconversion Emission Spectra for $GdPO_4$:Ho^{3+}/Nd^{3+} (1 at. percent Ho^{3+} and 20 at. percent Nd^{3+}) at dissimilar laser powers beyond 808nm excitation[12]. The emission bands at 550 nm (G = green), 672nm, (R= red) and 791nm NIR(R= red) in the UC spectra are caused by ($^5F_4$,$^5S_2$→$^5I_8$), ($^4G_{7/2}$→$^4I_{13/2}$) and ($^4G_{7/2}$→$^4I_{15/2}$) electronic transitions of the Ho^{3+} ion respectively. At 808nm excitation, Nd^{3+}ions are operating as sensitizers, with a greater absorption fraction than Ho^{3+}.At 808nm excitation, the absorption percentage of Nd^{3+} is 11.6* 10-20 cm^2[(16)]. The number of photons in a single photon Graph-5. Excitation spectra of $GdPO_4$: Ho^{3+}/Nd^{3+} (1 at. percent Ho) at emission = 550 nm, 672nm and 791nm showing the P-O CTB and the Ho^{3+} peak at 456 nm, and emission spectra at excitation = 300 and 532 nm, exhibiting. The P-O and Ho^{3+} peaks. The unconverted emission intensity changes as $I\alpha P_n$, where I represent the unconverted intensity, P represents the laser input power, and n is the number of photons participating in the unconverted emission bands. When we plot a graph of Ln(I) versus Ln(P), we see that G and R have slopes of n = (2.3 and 2.2), respectively. This indicates that both G and R lights emit UC as a result of the two-photon process. The R/G ratio grows with the laser input strength, as shown in the spectra (Figure-5) $2\pi r^2$ (here r = 1 mm) governs the laser beams focal point area when it hits the centre.

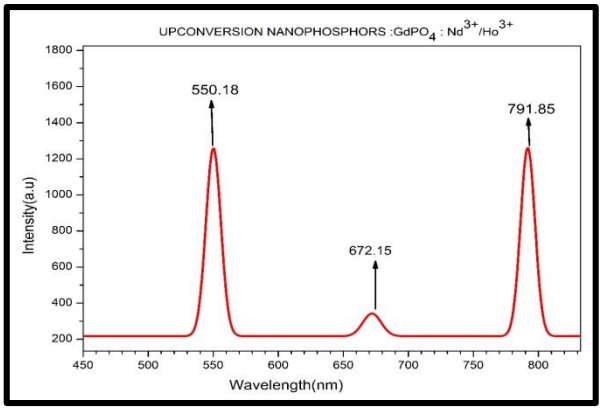

Figure-5: Emission spectra of $GdPO_4$: Ho^{3+}/Nd^{3+} (1 at.%Ho) excited at 808 nm

DC Study:

Downconversion: It is the process of lower-energy radiative light (E_{em}) being released after light (E_{exc}) has been absorbed. Which is mentioned as the stokes shift. Beyond UV excitation at ~300 nm, ~530nm the DC emission spectrum of $GdPO_4$:Ho^{3+}/Nd^{3+} (1 at. % Ho) which is shown in (Figure-6) 456nm, the green (~550 nm), red ~672nm and NIR (~791 nm) emission bands associated with ($^5F_4$,$^5S_2$→$^5I_8$), ($^4G_{7/2}$→$^4I_{13/2}$) and ($^4G_{7/2}$→$^4I_{15/2}$) ET'S of the Ho^{3+} ion, correspondingly are detected [(15,16)]. The emission spectra are monitored at different excitation wavelengths at ~300 and 532 nm. The Emission peaks attributable to Ho^{3+} are shown for each excitation. The Emission intensity of Ho^{3+} is weaker when it is directly excited at 532 nm ($^4I_8$→$^4G_6$) than when it is indirectly excited at 300 nm. This is owing to Ho^{3+} transitions having a low absorption cross section [(1,2)]. The broad emission band (410-532 nm) connected with PO_4^{3-}, as well as peaks of Ho^{3+}, is visible when excitation is at 300 nm. The emission peaks attributable to Ho^{3+} are shown

for each excitation. The emission intensity of Ho^{3+} is weaker when it is directly excited at 532nm ($^4I_8$→$^4G_6$) than when it is indirectly excited at 300 nm. This is owing to Ho^{3+} transitions having a low absorption cross section. In the case of excitation at 300 nm the broad emission band (410–532 nm) associated with PO_4^{3-} is detected, as well as peaks of Ho^{3+}. At 300 nm, there is a large absorption cross section because of the permitted transition of the P-O CTB. As a significant number of excited photons of P-O are De-excited and the excited

energy is transferred from P-O to Ho^{3+}, the radiative rate of Ho^{3+} increases. The emission band of the P-O CTB and the absorption peaks of Ho^{3+} are overlapping. This is known as the ET from PO_4^{3-} to Ho^{3+} resonance. Figure shows $GdPO_4$:Ho^{3+}/Nd^{3+} monitoring at 550nm emission and 672nm and 791nm peaks also emitted because of Nd^{3+} through the highest at 300 nm and 532nm excitation this has to do with the P-O CTB transition that is permitted. Sharp peaks with low intensities owing to Ho^{3+} have been recorded at 456, and 550 nm, 672nm and 791nm [1,2] different amount of Ho^{3+} are doped into the $GdPO_4$ host at the given concentration of Nd^{3+} to see the concentration-dependent luminescence (20 at. percent). $GdPO_4$:Ho^{3+}, Nd^{3+} (x at. percent = 1, y at. percent = 20) displays emission peaks of Ho^{3+} when excited at 300 and 532nm. The light intensity diminishes when the Ho^{3+} ion concentration rises above 1% (Figure-6). The concentration quenching effect is the key cause [24, 31]

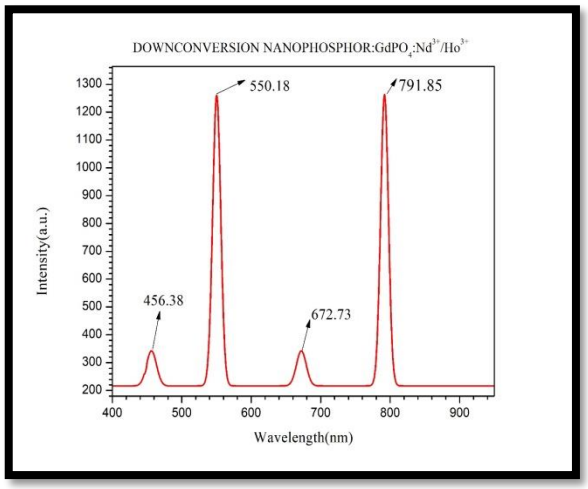

Figure-6: Excitation spectrum of $GdPO_4$: Ho^{3+}/Nd^{3+} (1 at.% Ho) excited at 300 and 532 nm

4. CONCLUSIONS:

In conclusion, solvothermal method is used to successfully synthesised $GdPO_4$: $0.01Ho^{3+}/0.2Nd^{3+}$nanophosphor material. After preparation, the sample is annealed for 4 hours at ~900°C which increase crystallinity and also eliminate organic contents and H_2o molecules. The monoclinic structural phase with space group I41/amd was confirmed by XRD. Under 808nm excitation, GdPO4:$0.01Ho^{3+}/0.2Nd^{3+}$ produces prominent upconverted green and red colour bands at 550 nm ($^5F_4$, $^5S_2 \rightarrow {}^5I_8$) 672nm ($^4G_{7/2} \rightarrow {}^4I_{13/2}$) and791nm ($^4G_{7/2} \rightarrow {}^4I_{15/2}$) of Ho^{3+}. A broad emission peak at 460nm, 550nm, and 672nm, 791nm, ($^5F_4$, $^5S_2 \rightarrow {}^5I_8$), ($^4G_{7/2} \rightarrow {}^4I_{13/2}$) and ($^4G_{7/2} \rightarrow {}^4I_{15/2}$) Ho^{3+} characteristic peaks are detected at 300-532 nm excitation. With a downconversion rate the emission band results primarily from ligand to metal week CT (P-O) charge transfer band. Two-photon absorption causes the green and red bands, according to the UC study.

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