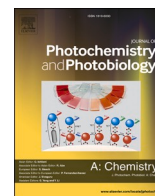




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journal homepage: www.elsevier.com/locate/jphotochemIR radiated Upconversion in Ho & Yb doped MgO-Y₂O₃ for security ink application: Temperature sensing and laser-induced thermal effectsPriyanka Bishnoi^{a,*}, Vaishali Rathi^a, Madan M. Upadhyay^b, Aditya Sharma^a, Kaushal Kumar^b, Ranjeet Brajpuriya^{a,*}, Ankush Vij^{c,*}^a Department of Physics, Applied Science Cluster, UPES, Dehradun 248007, Uttarakhand, India^b Optical Materials & Bio imaging Research Laboratory, Department of Physics, Indian Institute of Technology (Indian School of Mines), Dhanbad 826004, India^c Department of Physics and Astrophysics, Central University of Haryana, Mahendragarh 123029, Haryana, India

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ABSTRACT

This study presents a comprehensive analysis of the novel composition of Ho (0.1–1 mol%) and Yb (0.5–20 mol%) co-doped MgO-Y₂O₃ nanocomposites for upconversion (UC) and downconversion (DC) luminescence applications. The nanocomposites were synthesized using a cost-efficient combustion method. X-ray diffraction (XRD) confirmed stable MgO and Y₂O₃ phases, with peak shifts and unit cell parameter variations correlating with increased dopant (Ho, Yb) concentrations. Transmission electron microscopy (TEM) revealed particle agglomeration along with clear lattice fringes, while selected area electron diffraction (SAED) confirmed well-defined ring patterns characteristic of MgO and Y₂O₃ crystals. Photoluminescence (PL) spectroscopy revealed strong green, red, and near-infrared (NIR) emissions under both 448 nm (downconversion; DC) and 980 nm (upconversion; UC) excitations. Power-dependent PL studies indicated three-photon absorption for green and two-photon absorption for red/NIR emissions. Notably, the intensity of the green emission saturates rapidly at 5 mol% Yb³⁺, while red emission saturation occurs at 15 mol%, indicating efficient energy transfer between Ho³⁺ and Yb³⁺ ions. Power-dependent PL studies unveiled a three-photon absorption mechanism for green emissions and two-photon absorption for red and NIR emissions. Temperature-dependent UC was examined over a range of 298 K to 683 K, demonstrating promising optical temperature sensing capabilities, with maximum sensitivity recorded at $51.3 \times 10^{-4} \text{ K}^{-1}$ at 298 K. Moreover, the nanocomposites exhibited excellent stability under prolonged laser exposure, underscoring their potential for practical applications. Power-dependent tunable colorimetric parameters further highlighted their suitability for warm and cool LED technologies. Finally, the successful demonstration of these nanophosphors as security inks for anticounterfeiting applications opens new possibilities for advanced security solutions.

1. Introduction

Over the past decade, rare earth (RE) ions doped nanophosphors have garnered significant attention. This is primarily due to their applications in the field of luminescence in downconversion (DC), upconversion (UC), and dual-mode. Downconversion involves the conversion of high-energy photons into low-energy photons. The upconversion phenomenon refers to the non-linear process where multiple low-energy photons (NIR) are absorbed and combined to emit the high-energy photons, typically in the visible or UV range [1]. The shielded 4f shells by 5 s and 5p in RE ions make them maintain their distinct nature and display unique transitions of f-f shells [2,3]. As a

result, these ions exhibit specific features such as narrow emission and long lifetimes of excited levels. UC nanophosphors provide a range of distinct advantages compared to downconversion nanophosphors such as negligible noise signal from self-fluorescence, extended decay period, and exceptional upconversion efficiency [4,5]. Additionally, in the biomedical field, the NIR radiation used in the upconversion process easily penetrates the biological tissue and gets less scattered as compared to the UV and visible radiation, allowing the capture of more in-depth images [6]. Among various lanthanides, the Ho³⁺ can produce UC emissions in blue-green and red regions after absorbing NIR radiation due to the presence of high ladder-like energy levels [7–9]. The upconversion efficiency from this kind of emission can be remarkably

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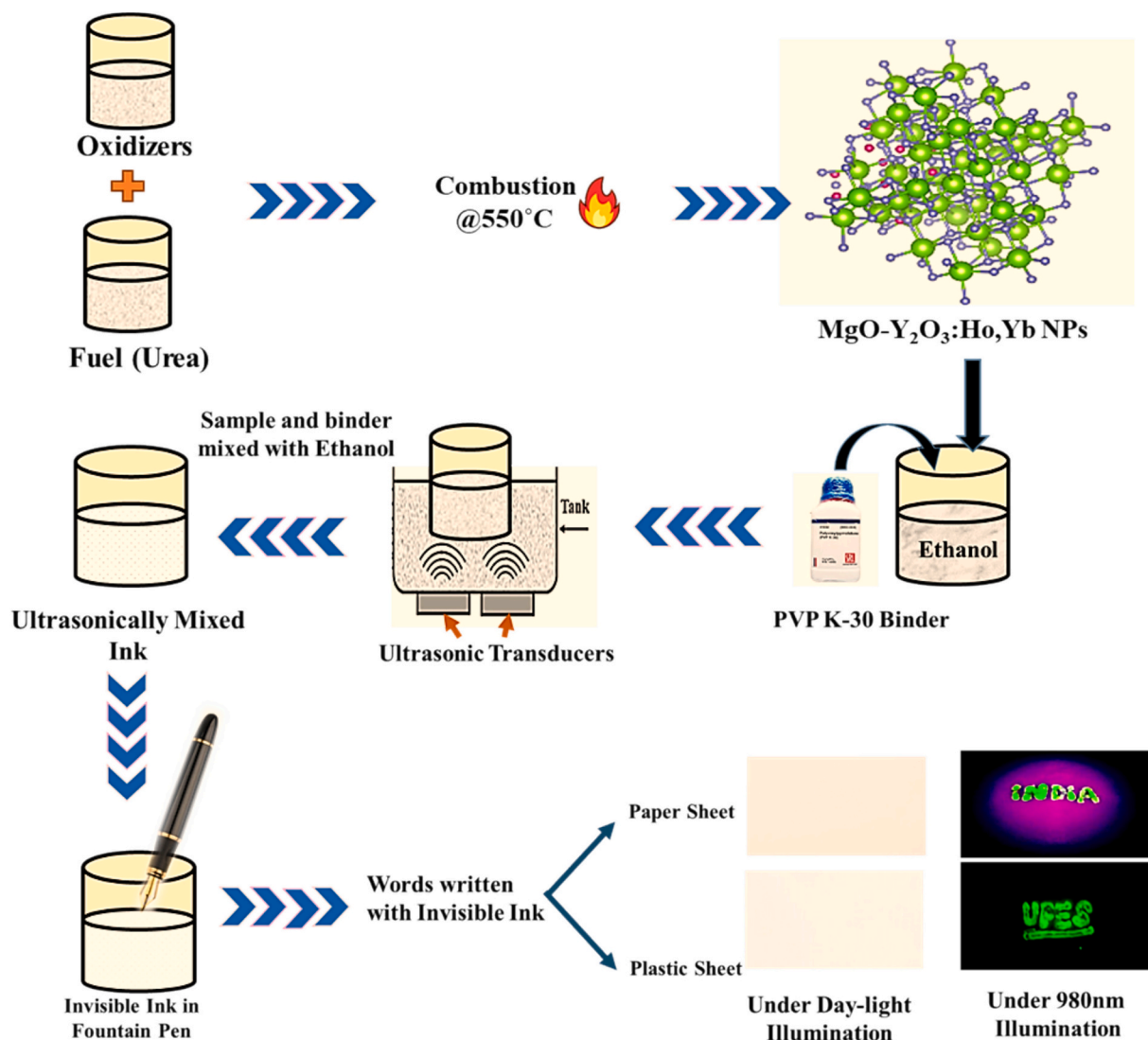


Fig. 1. Synthesis process of $\text{MgO-Y}_2\text{O}_3$ nanocomposites and derived invisible ink for security applications.

enhanced by co-doping of a sensitizer ion which exhibits a strong absorption cross-section in the NIR region and whose excited level matches well with the activator ion, through which it can efficiently transfer energy to the activators. Yb^{3+} is one of the ions that exhibit this behavior by having a large absorption near 980 nm and is used as a sensitizer with different activators as per reported literature [10–12].

Various parameters of the host matrix, such as the phonon frequency of the host lattice and particle size, govern the emission characteristics of RE ions. The inorganic oxides are one of the potential members in luminescent materials due to their ability to emit in the UV and visible region and their capacity to be easily doped with RE ions [13,14]. Several single oxides such as ZnO , Al_2O_3 , Gd_2O_3 , Y_2O_3 , and MgO have been explored from a luminescence perspective. Y_2O_3 is one of the sesquioxide materials that exhibit bixbyite cubic structure and have excellent chemical stability. It has a relatively lower maximum phonon energy (590 cm^{-1}) as compared to other sesquioxides, which helps reduce the multi-phonon relaxation, and further enhances the radiative transitions by minimizing the non-radiative losses [15]. Though Y_2O_3 obeys stable structural properties, RE-doped Y_2O_3 undergoes thermal and mechanical stresses and has been reported to have inferior luminescence efficiency and luminous flux [14]. The low thermal conductivity affects the luminescence efficiency and causes saturation at low concentrations of the dopant. In a recent study, *S. Chen et al.* reported

that the thermal conductivity of Y_2O_3 decreases from 14 W/m/K to 5.9 W/m/K with the doping of Eu ions [16]. Likewise, Ho^{3+} doped Y_2O_3 has been reported for lower thermal conductivity [16]. This thermal quenching, indeed, affects the stability of color quality and lowers the luminescence efficiency, specifically, if used in a field of high-power lighting [17]. An efficient heat dissipation rate in the host is required to circumvent this thermal quenching behavior. To overcome this drawback from such a host material, a contemporary strategy is the introduction of a second stable phase with excellent thermal conductivity, making the phosphor effective and thermally robust for enhancing optical performance [18,19]. $\text{MgO-Y}_2\text{O}_3$ is a new class of composite that has been reported for improved thermal conductivity, which helps to improve the performance of Y_2O_3 in the NIR range emissions [17,20,21]. MgO , which has been introduced as a second phase with Y_2O_3 , is a stable, wide-bandgap material with superior thermal conductivity at room temperature. Compared to the single oxides system, this class exhibits stable grain boundaries and better sinterability, leading to fine crystallites and improved phonon scattering. Further, these features govern the luminescence characteristics by stabilizing excited states, which makes this class more desirable than other oxides that experience higher phonon energy. Also, it does not make a mixed phase with the Y_2O_3 because of its high eutectic point [21–23]. In literature, doping of Er^{3+} , Eu^{3+} , and Ho^{3+} has been

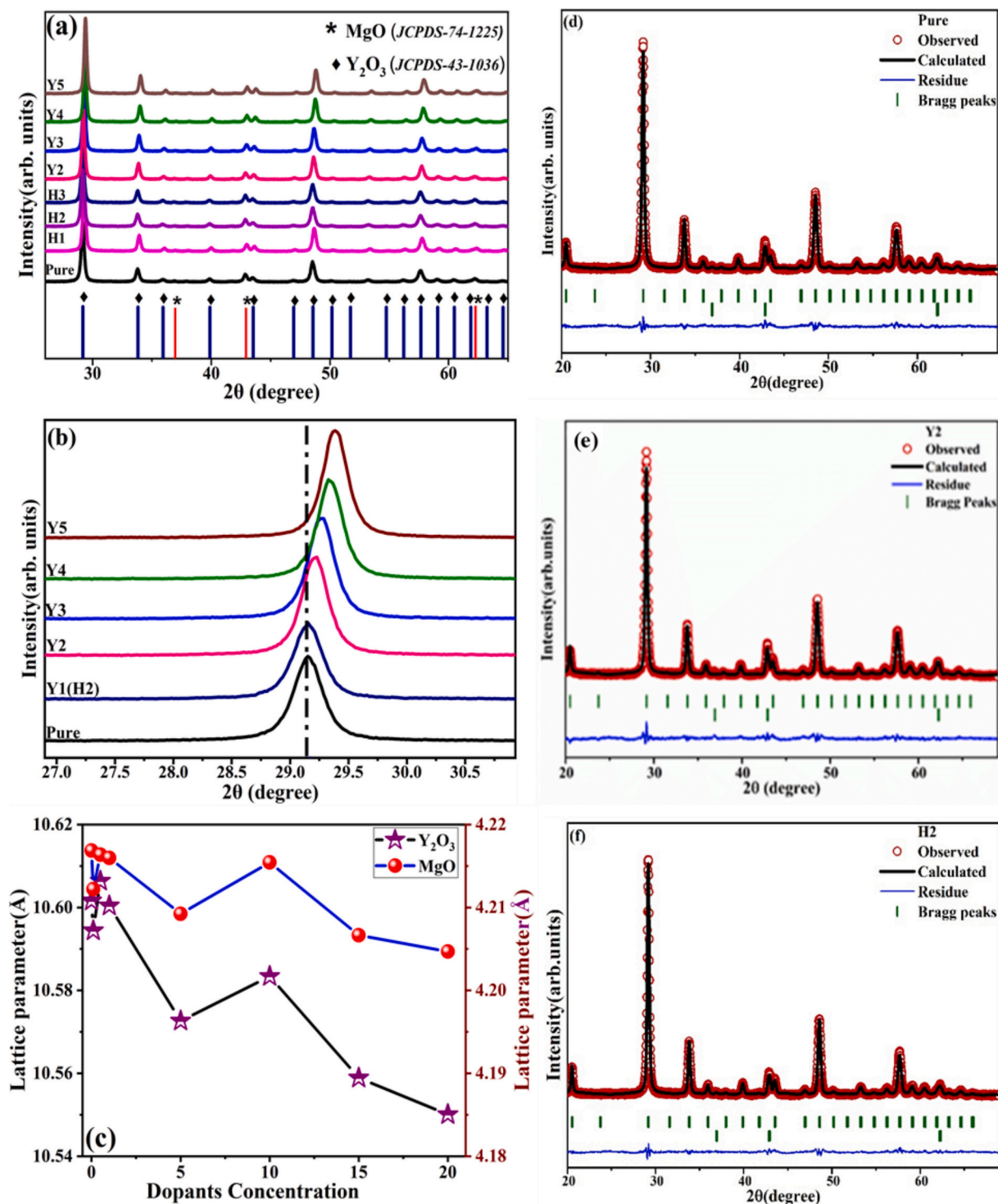


Fig. 2. (a): XRD patterns of Ho, Yb doped MgO: Y₂O₃; (b): enlarged image showing shifting of peaks with dopant concentrations; (c): Variation of lattice parameter of Y₂O₃ and MgO with dopant concentration; (d-f): Reitveld refined patterns of pure, Y2, and H2.

investigated in MgO-Y₂O₃ nanocomposites for different applications [16,17,20].

The spectroscopic characteristics of RE ions in Y₂O₃ do not experience any change with the incorporation of MgO. *Ho Jim et. al* reported that not only the photoluminescence and absorption properties of MgO-Y₂O₃, doped with a high concentration of Er³⁺, have been improved, but also the beam quality, mechanical, and thermal properties were enhanced [15]. Similarly, in Eu³⁺-doped MgO-Y₂O₃, when the ceramics were sintered up to 1800 °C, a quantum yield of ~80 % was achieved

from the prepared ceramics. Although there have been reports on enhanced NIR emissions of MgO-Y₂O₃ ceramic composition investigations on the upconversion luminescence are lacking, specifically doped with Ho, Yb upconverting ions.

Here, in this context, the present work aims to investigate the upconversion, downconversion luminescence, and thermal sensing properties of Ho³⁺, Yb³⁺ doped MgO-Y₂O₃, where Y³⁺ ions of Y₂O₃ are replaced by the doping of Ho³⁺ and Yb³⁺ ions. The present work mechanistically investigated the effect of variation of both the dopants

independently and the possible population routes involved in the UC and DC, such as excited state absorption, ground state absorption, cooperative energy transfer, and back energy transfer involved in the emission. The non-contact thermal approaches have been used to examine the luminescence intensity ratio for thermal sensing characteristics. The laser-induced thermal effect has also been carried out to investigate the stability of samples with time up to 60 min at intervals of 5 min. Moreover, investigations on the efficacy of doped MgO-Y₂O₃ phosphors as security ink for anticounterfeiting applications are attempted in this study for the socio-economic uses of MgO-Y₂O₃ compounds.

2. Experimental details

2.1. Materials

For MgO-Y₂O₃ sample, Magnesium nitrate hexahydrate (MgO (NO₃)₃·6H₂O > 99 %, CDH), Yttrium nitrate hexahydrate (Y₂O₃(NO₃)₃·6H₂O = 99.99 %, CDH), were used as oxidizer and urea (CDH > 99 %) were used as a fuel. In doped samples, apart from nitrates of Magnesium and Yttrium, Holmium nitrate pentahydrate (Ho (NO₃)₃·5H₂O) and Ytterbium nitrate pentahydrate (Yb(NO₃)₃·5H₂O) of purity of 99.99 % were used as oxidizers with the same fuel. Further, for ink formation, commercially available polyvinylpyrrolidonek-30 manufactured by SRL has been used as a resin binder, and ethanol is used as a solvent.

2.2. Synthesis

All the samples were synthesized utilizing the low-temperature solution combustion method. All the precursors were taken as per stoichiometry and kept at magnetic stir in 50 ml distilled water for half an hour to make a homogeneous solution. Then, the mixture was kept in a muffle furnace with a preset temperature of 873 K. The porous and powdery product was obtained after the combustion of the whole mixture and the evaporation of excess water. After cooling down to ambient temperature, the obtained product was crushed using a mortar and pestle. The prepared samples were annealed up to 1073 K for 2 h. The two series have been prepared by varying the concentration of dopants. The pure samples have been labeled as pure in further analysis in the report. In the first series, where Ho³⁺ has been varied with 0.1 mol %, 0.5 mol%, and 1 mol% with Yb³⁺ kept constant at 0.5 mol%, they were labeled as H1, H2, and H3. In other series, where Ho³⁺ is kept constant at 0.5 mol% Yb³⁺ has been varied with 0 mol%, 5 mol%, 10 mol %, 15 mol%, 20 mol% and 25 mol% has been labeled with Y0, Y1, Y2, Y3, Y4, Y5 in throughout the report.

2.3. Synthesis of security ink

The optimized sample Y2 has been used for security ink application. To achieve better writing or drawing patterns, ink's viscosity and dispersion of the sample in solvent without agglomerating is necessary. In the experiment, first, 5 g of the sample is mixed with 10 ml of ethanol in a beaker with a small amount of resin binder in it. The solution was sonicated for 1 h until the particles were completely dispersed in the solvent, and the transparent solution was obtained. This solution was filled in a standard ink pen, then some words and patterns were written on the paper and plastic sheet for further experiments. The synthesis process is shown in Fig. 1.

2.4. Characterizations

Bruker-D8 Advance EcoPro diffractometer, which was irradiated with Cu K α radiation ($\lambda = 1.54056 \text{ \AA}$), has been used for X-ray diffraction measurements. JEOL/JEM-F200 with Thermal Electron Field Emission Electron Gun (TFEG) and OneView CMOS Camera (4 k $\times$ 4 k

pixels) has been used for Transmission electron microscopy (TEM). UV-visible measurements have been carried out by using the Cary 5000 UV-Vis-NIR instrument. Photoluminescence experiments were carried out by using a Horiba Fluoromax+ spectrophotometer with a Xenon lamp and a continuous wave NIR Laser of 980 nm with 2.5 mW power at room temperature. The temperature-dependent upconversion spectra were recorded using the same diode laser, CCD, and optical fiber integrated with a homemade small heater and thermocouple. A potentiometer was used to control the heater temperature.

3. Result and discussion

3.1. X-Ray diffraction

The X-ray diffraction peaks of all the samples are shown in Fig. 2. According to the XRD pattern obtained, it can be verified that the phases match with MgO (JCPDS-74-1225) and Y₂O₃ (JCPDS-43-1036), with the space groups Fm-3 m(225) and Ia-3(206), respectively [15,24]. Notably, no peaks of Yb₂O₃ and Ho₂O₃ were observed in the entire series of samples within the detection range of the employed XRD, ruling out any presence of impurities. For detailed in-depth structural analysis the intense peak at 29.15°(222), 33.78°(040), 48.53°(044), 57.63°(262) of Y₂O₃ and 36.89°(111), 42. 87°(020), 62.19°(022) of MgO were used. It is interesting to note here that with the increase in Ho³⁺ concentration up to 0.5 mol%, the shifting is not observed, which can be due to the similar ionic radii of Y³⁺(1.040 Å) and Ho³⁺(1.041 Å). However, the decrease of FWHM was noted, exhibiting a better crystallinity in the doped samples. Besides this, at Ho³⁺(1 mol%) the maxima slightly shifted towards the higher angle, and the FWHM of Y₂O₃ peaks was enhanced, attributed to the structural perturbations [25]. The anticipated dislocations, caused by dopants, have been evaluated using the formula: $\delta = 1/D^2$, where D is crystallite size, which has been calculated using the Debye-Scherrer formula, $D(\text{crystallite size}) = \frac{k\lambda}{\beta \cos\theta}$, where k is the shape factor, β represents FWHM, λ is source wavelength, and θ represents diffraction angle. It is observed that dislocation density is highest in the H3 sample, likely due to the distortion in the crystal structure of the host due to the dopant, which further also affects the luminescence properties. Similarly, with Yb³⁺ concentration, the peaks prominently shifted towards the higher angles (shown in enlarged Fig. 2 (b)), indicating the shrinkage of crystallite size due to the difference in ionic radii of Y³⁺(1.040 Å) and Yb³⁺(1.008 Å) [17]. The FWHM of peaks first decreases up to Y3 and then again increases, which shows the enhancement in crystalline nature. Due to variation in peak broadening, the dislocation density also shows variation, with the minimum observed in Y2. As the Yb³⁺ concentration increases in the host, the dislocation density increases, which in turn affects the associated spectroscopic characteristics of the host.

For the detailed analysis of structural perturbations, the Rietveld refinement of XRD patterns has been conducted of pure as well as of H2 and Y2 samples using FULLPROF software (Fig. 2 (e-f)). The formation of both the phases of MgO and Y₂O₃ was confirmed through double-phase fitting of patterns in pure and doped samples. The refined parameters suggest that dopant cations occupy both the Y sites (C_{3i} and C₂), resulting in the contraction of the lattice parameter and reduction in bond length of Y₂O₃. The bond length of Mg—O is consistent at 2.418 Å, with doping nullifying the insertion of Ho³⁺ and Yb³⁺ in the MgO lattice. Conversely, the Y₁—O (C_{3i}) decreases from 4.31 Å to 4.30 Å while the bond length of Y₂—O (C₂) decreases from 4.193 Å to 4.190 Å, respectively. At doping of lower concentrations of Yb³⁺ and Ho³⁺ ions, the bond length of both sites of Y experienced a slight reduction, indicating the distribution of dopants in a random way. However, at higher Yb³⁺ concentrations, the Y₁—O bond length shows contraction as compared to the Y₂—O conveying that the Yb³⁺ ions prefer to occupy 8b (C_{3i}) Wyckoff positions in the lattice. These site occupancy preferences further affect the luminescence properties. In other studies, the random

Table 1

Calculated structural parameters for all the compositions.

Parameters		Samples		
		Pure	H2	Y2
Lattice parameter(Å)	Y ₂ O ₃	10.6089	10.6026	10.5704
	MgO	4.2177	4.2167	4.2082
Crysallite size(nm)	Y ₂ O ₃	24.67	23.96	24.88
	MgO	25.13	24.41	21.60
Dislocation density		0.00164	0.00174	0.00161
	Y ₁ (C _{3i})	0.4955	0.4429	0.4465
Occupancy	Y ₂ (C ₂)	0.1409	0.1210	0.1355
	O	0.8668	0.9502	0.8539
	H ₁	-	0.0015	0.0009
	H ₂	-	0.00092	0.0007
	Yb ₁	-	0.0030	0.0265
	Yb ₂	-	0.00076	0.0083
	Mg	1	1	1
	O	0.999	0.9832	0.9482
Bond length(Å)	Y ₁ -O(C _{3i})	2.314	2.312	2.306
	Y ₂ -O(C ₂)	2.193	2.190	2.190
	Mg-O	2.418	2.418	2.418
	R _{wp}	6.9	8.2	6.5
Quality of fit	R _{xp}	5.1	5.2	5.0
	GOF	1.9	2.1	1.6

distribution of lanthanides such as Eu, Gd, and Ho at both the sites of Y has been reported [26–28]. Furthermore, the generation of O2-defects in the MgO lattice was also inferred from the occupancy of O. In previous reports, it has been reported that an increase in distortion in the lattice can increase the O and cationic defects in the host compound [29]. Here, with the variation of both the dopants, variation in the lattice parameter and FWHM of MgO is also observed, showing the creation of different vacancies in the MgO lattice, too. All the refined parameters have been tabulated in Table 1.

The morphology of annealed samples was evaluated by using TEM and SAED analysis. Fig. 3(a-c) shows the TEM images of pure, H2, and Y2 samples at 50 nm resolution. It is observed that the crystals are polyhedron in shape and agglomerated in all three samples. This high-temperature annealing causes grain growth, which reduces the grain boundaries. The particle size of crystals shown in Fig. 3(d-f) remains consistent with the doping concentration and also complements the XRD result. In Fig. 3(g-i), the planar spacing has been derived by using the FFT in ImageJ software. The planar spacing 1.863 Å and 1.506 Å correspond to the (343) and (345) planes of Y₂O₃. In H2 and Y2 planar spacing 1.891 Å and 1.863 Å correspond to the (044) plane of Y₂O₃, while 2.116 Å observed in Y2 corresponds to the (020) plane of MgO, confirms the presence of both the lattices and also matching well with the planar spacing of Y₂O₃ and MgO from XRD patterns. Some moire fringes were also calculated using the formula shown below, which is shown in Fig. 3(g).

$$d_{\text{moire}} = \frac{d_1 d_2}{\sqrt{d_1^2 + d_2^2 - 2d_1 d_2 \cos \phi}}$$

Here, d_1 and d_2 are the d-spacings of primary lattices in the image, and d_{moire} is the spacing for the moire fringes. The angle ϕ is the angle at which both the actual lattices were orientated. The ring patterns observed in SAED, shown in Fig. 3 (j-l), confirm the polycrystalline nature. The observed rings exhibit the planes of Y₂O₃ and MgO in pure and doped samples. No additional planes belonging to the other phases could be detected, confirming the absence of secondary phases in all of the samples.

3.2. UV-Vis absorption spectra

Absorption spectra of all phosphors in the range of 220 nm to 1150 nm have been illustrated in Fig. 4. The absorption in the UV region is attributed to the host. Apart from the host absorption, several other

peaks of Ho³⁺ and Yb³⁺ ions have also been observed. The peak near 450 nm is attributed to the absorption from ⁵I₈ to higher energy levels ⁵G₆/⁵F₁, and ⁵F₃, the other peak around 550 nm is due to the transition from ⁵I₈ to ⁵F₄/⁵S₂, and peaks near 650 nm are contributed by transitions between ⁵I₈ to ⁵F₅ [30,31]. Additionally, the increase in Yb³⁺ ions' peak near the NIR region from 900 nm to 1000 nm is also enhanced, attributed to the Yb³⁺ transition from ²F_{7/2} to ²F_{5/2} [32]. These transitions remarkably increase with the Yb³⁺ concentration, suggesting the strong absorption of Yb³⁺ ions in this region, which further governs the upconversion as well as downconversion mechanism of Ho³⁺ ions.

3.3. Photoluminescence

The impact of Ho³⁺ and Yb³⁺ dopants on DC and UC luminescence in MgO-Y₂O₃ nanocomposite at room temperature was examined by varying both concentrations. Fig. 5 (a) depicts the emission spectra in the range of 500 nm to 800 nm on the excitation of 448 nm at a fixed concentration of Yb³⁺ (0.5 mol%) and different concentrations of Ho³⁺ (0.1, 0.5, and 1 mol%). The spectra consist of three green, red, and NIR emissions ranging from 521 nm to 570 nm, 635 nm to 670 nm, and 735 nm to 775 nm, respectively. The green band is attributed to the transitions from ⁵F₄/⁵S₂→⁵I₈, while red and NIR bands are due to the ⁵F₅→⁵I₈ and ⁵F₄/⁵S₂→⁵I₇ transition of Ho³⁺ ions, respectively [31,33]. The PL spectra conveyed the increase in intensity of NIR and green regions with Holmium concentration up to 0.5 mol%, followed by quenching afterward. However, the intensity in the red region shows a constant increment with an increase in the concentration of Ho³⁺ ions, indicating the increase in populations at the ⁵F₅ level. No emission was observed at wavelengths below 500 nm, implying that first all populations of electrons non-radiatively decayed at ⁵F₄/⁵S₂ and ⁵F₅ from upper levels and then exhibited the radiative transitions to various lower levels. Further to study the influence of Yb³⁺ concentration on luminescence behavior, the Yb³⁺ concentration was increased from 0 to 20 mol% after optimizing the Ho³⁺ concentration at 0.5 mol%, and spectra were collected at room temperature. Fig. 5(c) depicts the emission spectra at various Yb³⁺ concentrations, which shows saturation of intensity at Yb³⁺ 5 mol %. It is interesting to note that the red region, which is enhanced as Ho³⁺ concentration increases, behaves oppositely to the Yb³⁺ concentration. It may be presumed that there is energy transfer (ET) from Ho³⁺ to Yb³⁺ from ⁵F₅ levels of Ho³⁺. A similar kind of transfer from Ho to Yb is also observed in other hosts [32]. The various routes have been described later by using the energy level diagram.

Fig. 5 (b, d) shows the excitation spectra for emission at 547 nm, 667 nm, and 756 nm. Different excitation peaks were observed near 404 nm, 414 nm, 448 nm, 452 nm, 466 nm, and 487 nm. All the peaks observed belong to the Ho³⁺ transitions. The major contribution is from 448 nm to 466 nm, attributed to the transition (⁵I₈→⁵G₆, ⁴F₁). The excitation spectra also complement UV-Vis spectra, which show strong absorption of Ho³⁺ ions near 420 nm, 448 nm, 450 nm, 465 nm, and 480 nm. After absorbing a photon of 448 nm wavelength, electrons from ⁵I₈ jump to the ⁵G₆/⁵F₁ of Ho³⁺. After non-radiative relaxations from higher levels, the electrons will be populated to ⁵F₃ and ⁵S₂/⁵F₄ before showing radiative transitions. The UC luminescence for various Ho³⁺ and Yb³⁺ concentrations has been taken on a 980 nm excitation wavelength.

Fig. 6 (a,b) shows the emission at fixed Yb³⁺ of 0.5 mol% and different Ho³⁺ concentrations from 0.1 to 1 mol%. Like DC, efficient emission was observed in all three regions, attributed to the Ho³⁺ transitions. Here, on excitation at 980 nm, intense green emission with two weak emissions in the red and NIR regions is observed. In case H1, H2, and H3 samples, the highest emission is observed in H2, while quenching is observed afterwards in all three regions. The observed quenching may be due to the decrease in interionic distances, which enhances the non-radiative relaxation in the lattice. The intensity ratio of NIR and red with green is shown in Fig. 6 (c), showing the minimum at 0.5 mol%, where maximum emission is observed, attributed to the minimum non-radiative process allowing decay of excited states via

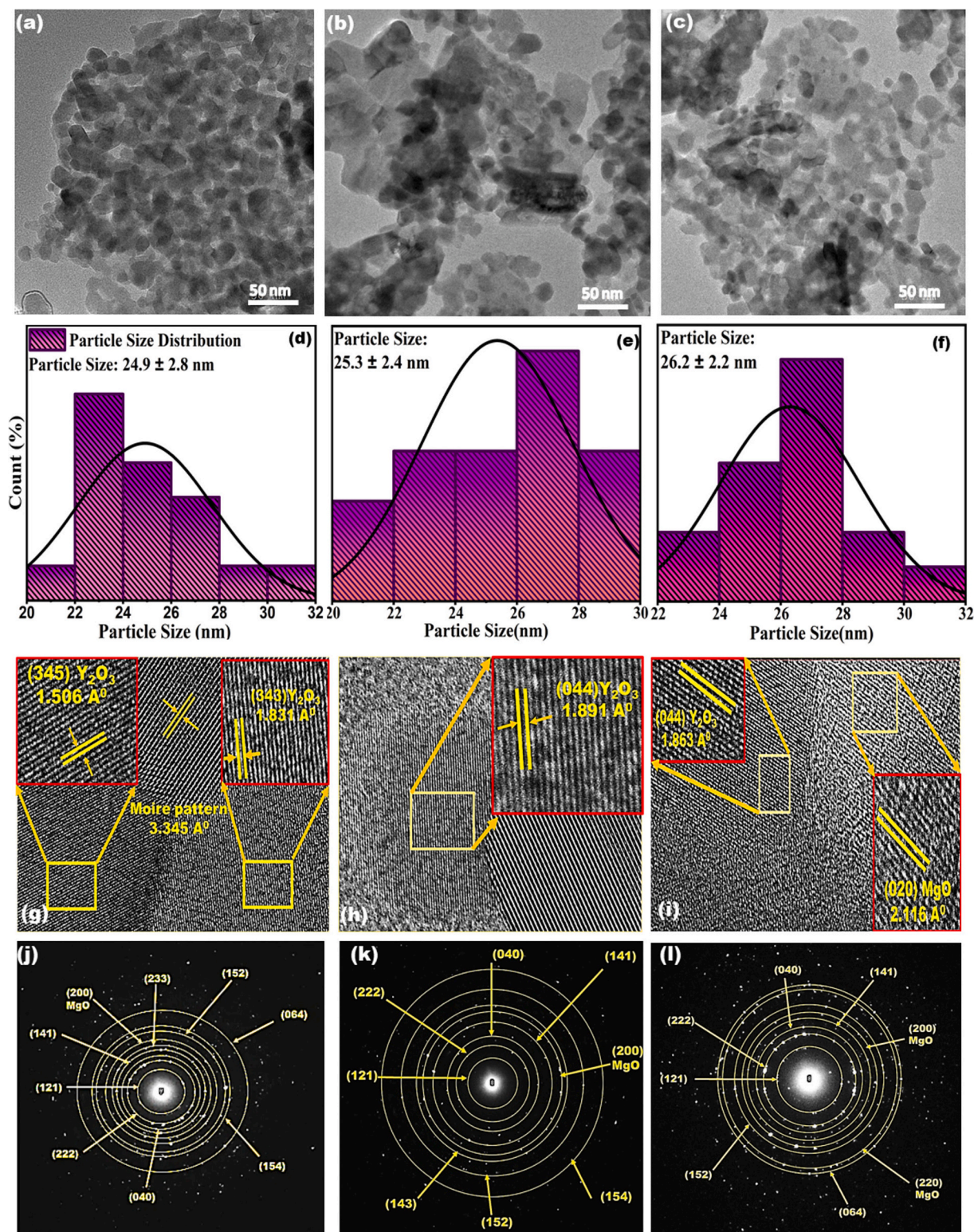


Fig. 3. (a,b,c); TEM images of pure, H₂, Y₂, respectively; (e,f,g); Particle size of pure, H₂, Y₂, respectively; (g,h, i); lattice planes observed in pure, H₂, Y₂ samples; (j,k,l); SAED patterns of pure H₂ and Y₂ samples.

green emission pathways. Furthermore, more detailed mechanism is discussed below with the help of the energy level diagram.

Further, the effects of Yb³⁺ concentration on UC luminescence taken at the fixed concentration of Ho³⁺ of 0.5 mol% is shown in Fig. 6 (d,e). The emission is observed in all three regions, with the strongest emission in the green region. The emission in NIR and the green region shows

quenching after Yb³⁺ of 5 mol%, but the red region intensity is enhanced up to 15 mol% of Yb³⁺, after which it saturates. It exhibits that non-radiative migration from 5F₄/ 5S₂ and 5I₇ via different cross-relaxation from Ho to Yb level reduces the green and NIR emission. These cross-relaxation populated the 5F₅ level, which causes an increase in the red region. The intensity ratios of red to green first decrease on the

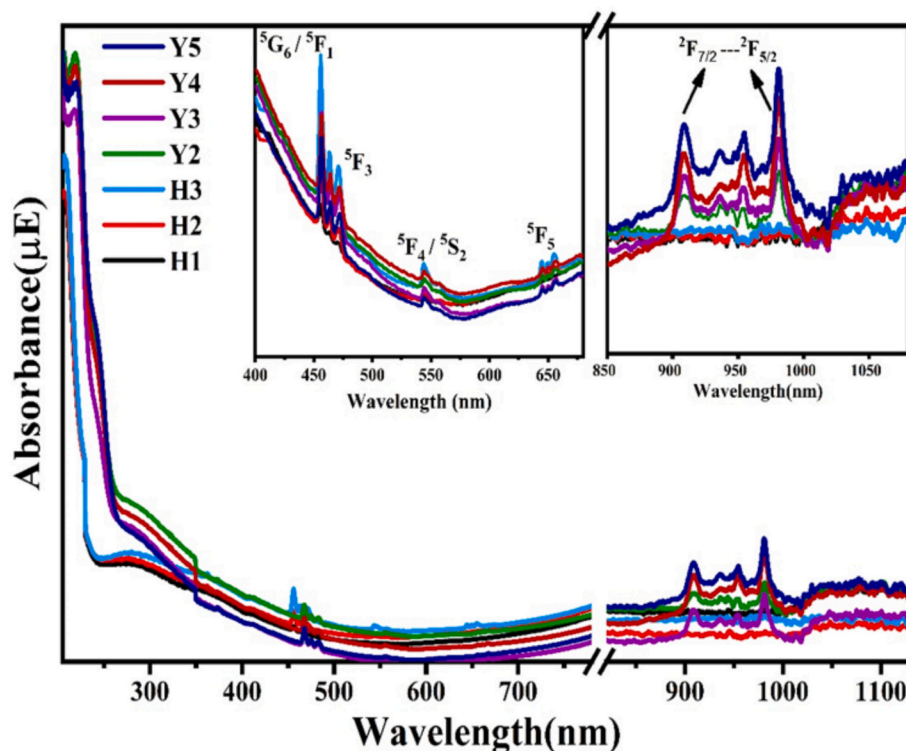


Fig. 4. UV-Vis spectra of Ho Yb doped MgO: Y₂O₃ nanocomposites.

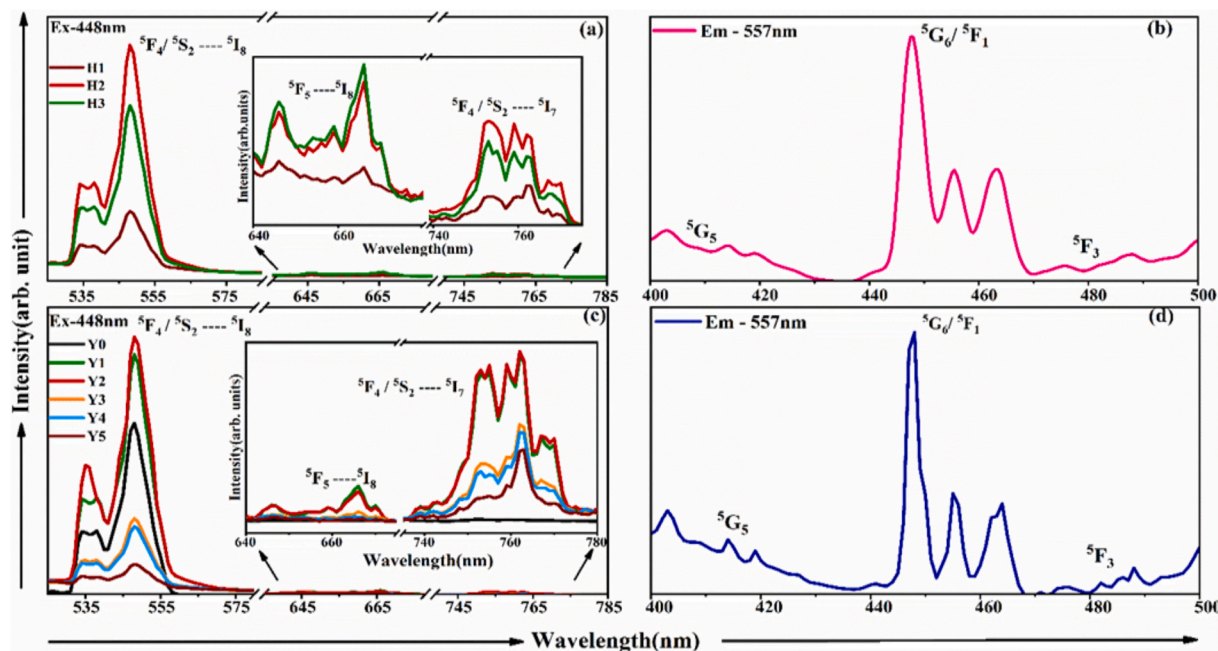


Fig. 5. (a) Emission spectra of Ho³⁺ concentration on excitation of 448 nm; (b) Excitation spectra of H2 sample; (c) Emission of Yb³⁺ concentration at excitation of 448 nm; (d) Excitation spectra of Y2 sample.

introduction of Yb³⁺ ions and increase after 5 mol%. As discussed above, the cross relaxation causes the decrease in green emission, while the red remains strong, causing the ratio to increase. On the other hand, while the intensity ratio of NIR to green constantly decreases after a 5 mol% concentration of Yb³⁺ shown in Fig. 6(f), attributed to the continuous non-radiative relaxation from the ⁵I₇ level. This will also be discussed with an energy level diagram. For further analysis of the UC mechanism, the emission spectra of prepared phosphors have been studied by

varying the input pump power of a 980 nm laser. The non-linear process is involved in the upconversion mechanism where the intensity is proportional to the power of input radiation represented below $I \propto P^n$. Here, I represents the UC intensity, P represents the pump power density (mW), and n is the number of photons involved in the UC process. Fig. 7(a, b) illustrates the behavior of spectra of H2 and Y2 with input power, as both phosphors show the optimum emission in DC as well as in the UC process in the whole series. The intensity of both phosphors increases as

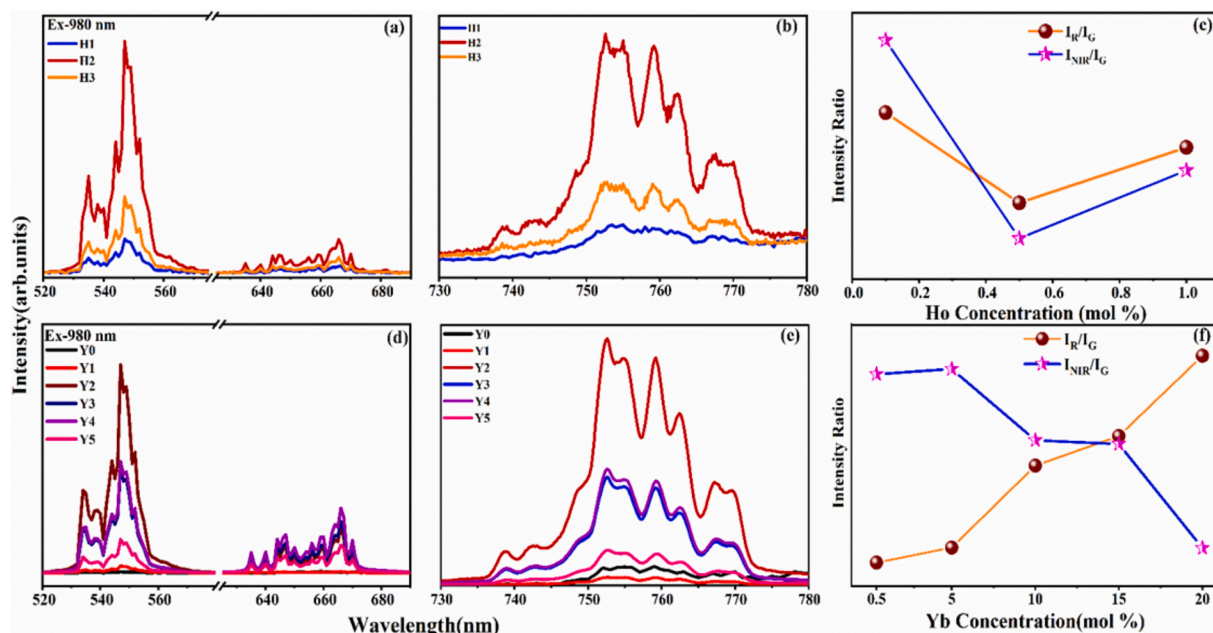


Fig. 6. (a) Emission spectra of Ho^{3+} series in visible region under excitation of 980 nm; Emission of Ho^{3+} series in NIR region; (c) Intensity ratio of red and NIR with green emission in Ho^{3+} concentration; (d) Emission spectra of Yb^{3+} series in visible region under the excitation of 980 nm; (e) emission spectra in NIR region; (f) Intensity ratio of red and NIR with green emission in Yb^{3+} concentration. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

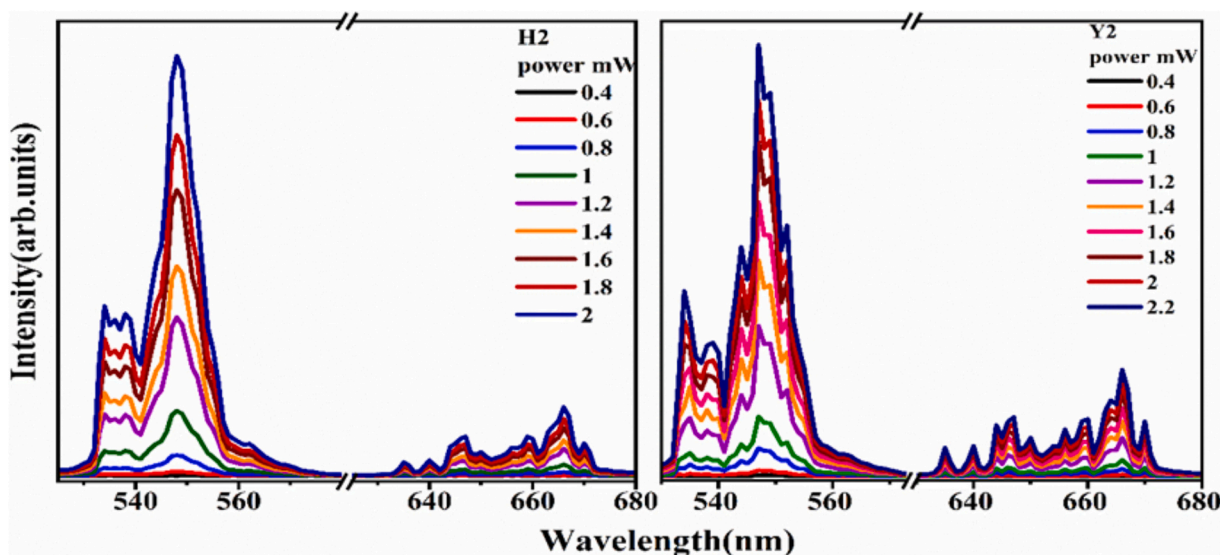


Fig. 7. (a) Intensity variation with the function of input pump power of sample H2; (b) Intensity variation of Y2 sample with input pump power.

Table 2

Slope value (n) obtained by fitted experimental data of the integrated intensity of green, red, and NIR emission.

Sample	Green	Red	NIR
Y1	2.1	1.9	2.3
Y2	2.2	2.1	1.7
Y3	2.2	2.0	2.1
Y4	1.4	2.2	2.4
Y5	2.3	2.1	2.4

the input power increases indicating the stability of the samples. After evaluating the slope values, by taking a logarithmic plot between the integrated intensity and input power, the number of photons involved in

the UC process has been summarized in Table 2 and for H2 and Y2 samples the graph is presented in Fig. 8. It is observed that the value of n is close to 2 for green, red and NIR emission. This involvement of photons may be due to the energy transfer looping and, energy back transfer populating the intermediate levels. Similar values of n have also been reported in other rare earth doped samples [34–36].

To gain a deeper understanding of the different processes involved in UC/DC mechanisms, such as ground state absorption (GSA), excited state absorption (ESA), energy transfer (ET), and cross-relaxation (CR), Fig. 9 shows an energy level diagram. In the case of a single doped with Ho^{3+} ions, the weak green, red, and NIR emission is observed due to weak absorption of Ho^{3+} ions near 980 nm. After absorbing the photon of 980 nm, Ho^{3+} ions jump to the $^5\text{I}_6$ levels denoted as GSA in the band diagram. After absorbing simultaneously another photon, the electrons

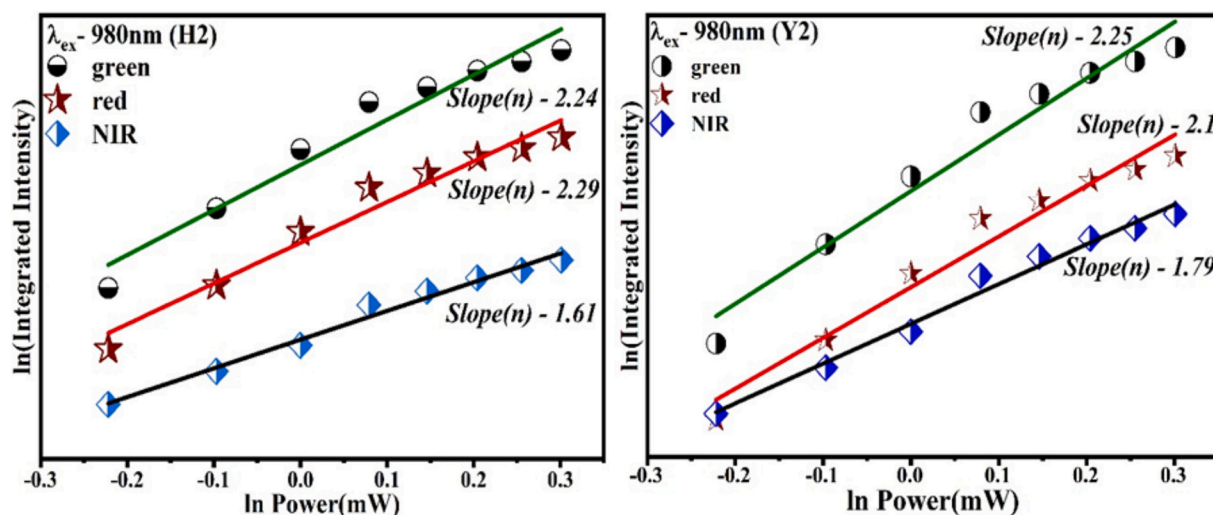


Fig. 8. Log plots between integrated intensity and input pump power for H2 and Y2 samples.

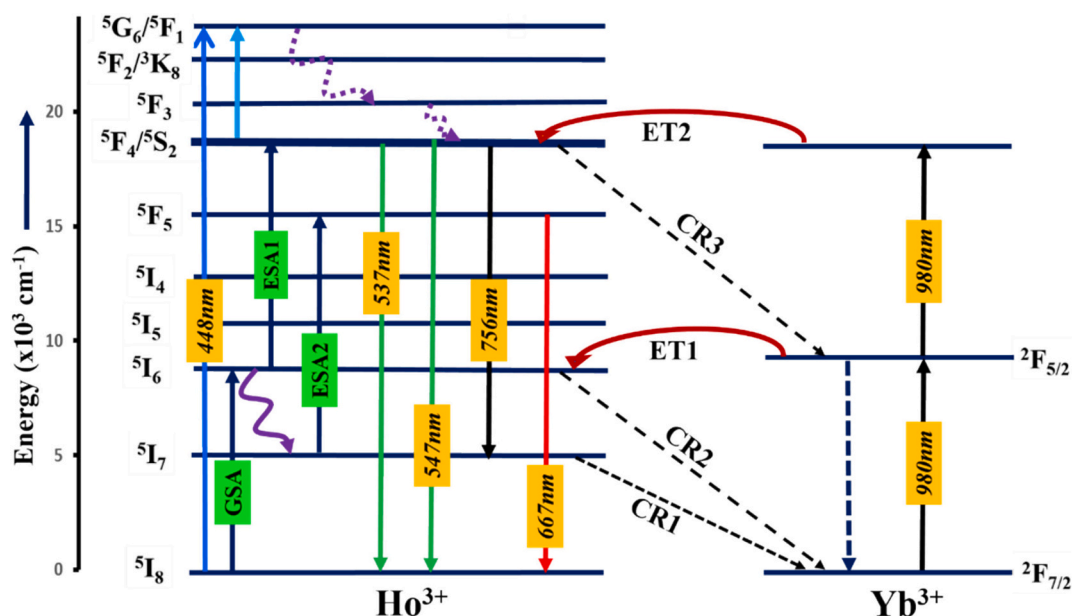


Fig. 9. Energy level schematic diagram of Ho, Yb-doped MgO: Y₂O₃. The solid blue lines represent the absorption at various levels (GSA ground state absorption; ESA- excited state absorption); Curvy dotted lines denoted non-radiative transition involved; Black dashed lines denoted cross-relaxation (CR1, CR2, CR3) while red solid line denoted as (ET) energy transfer between Ho to Yb ions. Green, Red, and Black solid lines represent the various emissions involved in up/down conversion luminescence. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

get excited to ⁵F₄ and ⁵S₂ levels denoted as ESA1, and there they decay to the ⁵I₆ and ⁵I₇ showing the green and NIR emissions respectively. Apart from these two emissions, the red emission arises by another pathway from ⁵I₆, electrons first non-radiatively decay to ⁵I₇, and from there, after absorbing another photon, get excited to ⁵F₅ (denoted as ESA2) and radiatively decay back to ⁵I₈. When Yb³⁺ is introduced along with Ho³⁺ ions in the host, the intensity in all regions is enhanced abruptly as Yb³⁺ has a large absorption near 980 nm cross-section and exhibits the transition from ²F_{7/2} to ²F_{5/2} [37,38]. Then, Yb³⁺ ions transfer their energy to Ho³⁺ ions via ET1 to ⁵I₆, as the energy of ⁵I₆ matches well with the ²F_{5/2} of Yb³⁺ [39]. Simultaneously, Yb³⁺ ions, after absorption of two photons, transfer energy (ET2) to Ho³⁺ ions, which enhances the population at ⁵F₄ and ⁵S₂ and causes intense green emission. As the concentration of Yb³⁺ increases, the green and NIR emission saturation is observed first. Back energy transfer (BET) from Ho³⁺ to Yb³⁺ is possible via ⁵F₄/⁵S₂ → ²F_{5/2}, ⁵I₇ → ²F_{7/2}, and ⁵I₆ → ²F_{7/2} denoted as CR1

CR2 and CR3. The ⁵F₄/⁵S₂ and ⁵I₆ populations declined fast through CR1 and CR3 at Yb³⁺ more than 5 mol%. Then, the energy from ²F_{5/2} is again re-transferred to the ⁵I₆ via ET1 due to populations, which causes the increase of red emission. Thus, the intensity of green decreases while red increases, and the ratio I_r/I_g also increases, indicating that level ⁵F₄/⁵S₂ depopulates faster than ⁵F₅ as the concentration of Yb³⁺ increases [32]. Apart from this, the increment in concentration of Ho³⁺ ions also causes the saturation of intensity after 0.5 mol%, indicating the concentration quenching caused by the decrease in the inter-ionic distances between Ho³⁺ ions, increasing the energy reabsorption. The ratio of red intensity to green decreases and then increases at 1 mol%, showing the parallel cross-relaxation between ⁵F₅ → ⁵I₇ and ⁵S₂ → ⁵F₄ → ⁵I₆ affects the population at excited levels, causing a decrease in intensities as well as the ratio, as per earlier reported literature [30,33,40,41].

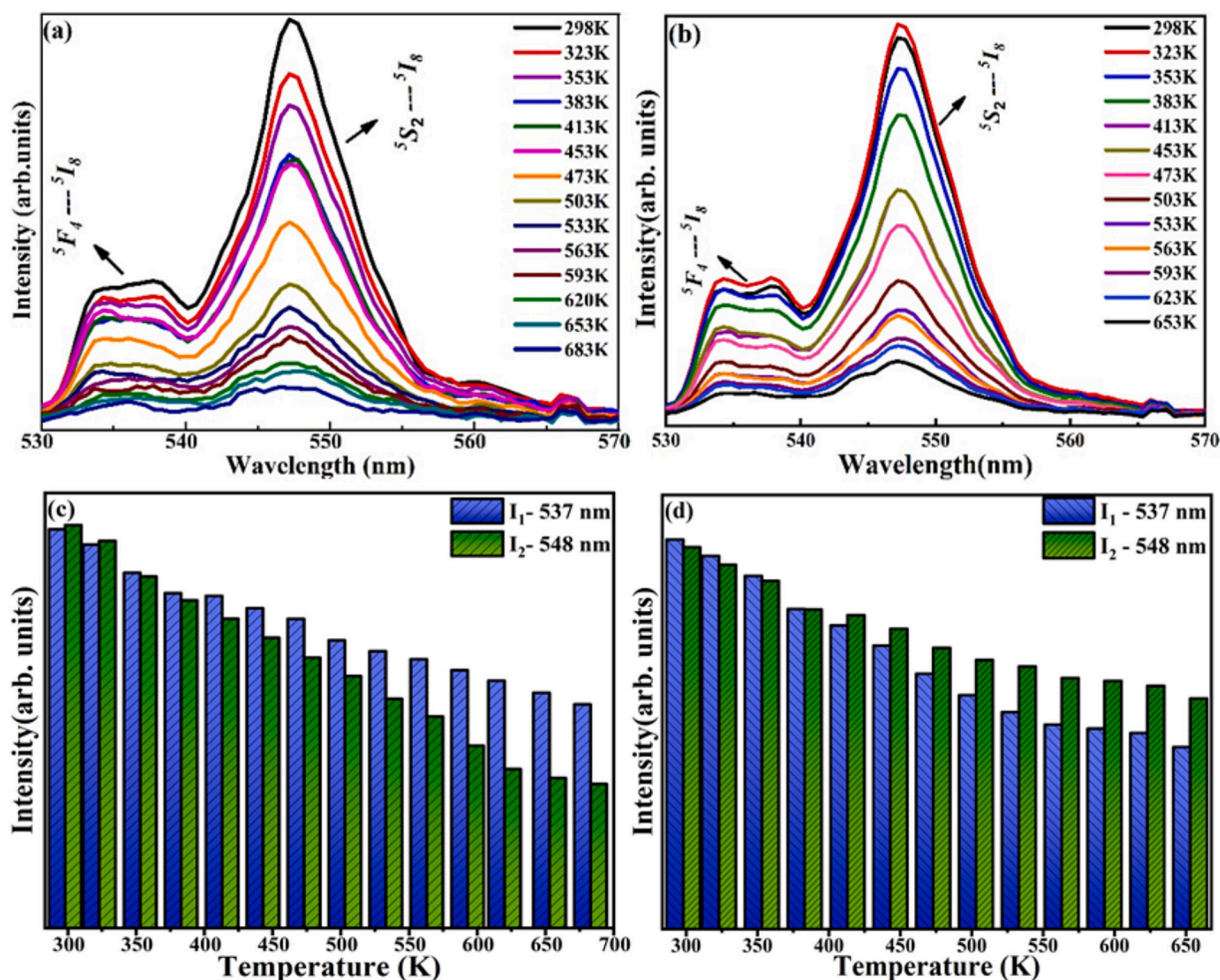


Fig. 10. (a) Temperature-based up conversion spectra of H2 sample under excitation wavelength of 980 nm; (b) Temperature-based up conversion spectra of Y2 sample under excitation of 980 nm; (c) variation of I_1 (537 nm) and I_2 (548 nm) at different temperatures of H2 sample; (d) Variation of I_1 (537 nm) and I_2 (548 nm) at different temperature from 298 K to 683 K.

3.4. Optical temperature sensing study and laser-induced thermal effect based on intensity ratio

Due to the prolonged necessity of luminescent materials, which typically cause an increase in temperature or operation at elevated temperatures, understanding their temperature-sensing properties becomes increasingly essential. However, the traditional contact measurements are inadequate to monitor the accurate internal temperature of these nanophosphors. Therefore, it is imperative to identify a non-contact technique for measuring the internal temperature. For luminescence materials to be more effectively used in practical situations, it is essential to comprehend the influence of temperature on their associated spectroscopic properties. In the present report, the authors investigated the upconversion mechanism of Ho^{3+} ions in H2 and Y2 across the temperature range from 298 K to 683 K.

The levels 5F_4 and 5S_2 of Ho^{3+} ions, which lie with a separation of 305 cm^{-1} , [42,43] follow the Boltzmann distribution in the thermal equilibrium state. This temperature-dependent behavior of the green emission can be further analyzed for exploring the temperature sensing study by probing the intensity ratio between the upper level (5F_4) and lower level (5S_2) at different temperatures. The relationship between two intensities is described by Eq. (1) [33,44]:

$$LIR(T) = \frac{I_u}{I_l} = B \exp\left(-\frac{\Delta E}{kT}\right) \quad (1)$$

Here, I_u , I_l are the intensity of upper and lower levels, respectively. B is constant, T is temperature, ΔE is the separation between two energy levels, and k is the Boltzmann constant. Fig. 10(a, b) shows that the peak positions for both samples remain unchanged across the temperature range from 298 K to 683 K, but quenching in overall green emission occurs with an increase in temperature. As the temperature of the sample increases, the overall PL intensity decreases. The decrement in PL intensity attributed to the enhanced lattice vibration at elevated temperatures causes the increase in number of phonons resulting in non-radiative relaxations and compete with the radiative transitions. This competitive behavior enables the activators to relax to lower energy level without emitting photon [35,36,45–47]. Here, it can be seen from Fig. 10(c, d) that the intensity I_2 (548 nm) decreases more intensively than I_1 (537 nm). It is observed that both bars decline, but green decreases more rapidly than the blue one, which causes an increase in the ratio of both intensities. This may be due to the cross-relaxation between 5F_4 to 5S_2 levels, causing the slower decrement in upper levels as compared to the lower level with increasing temperature. In the case of Y2, where Yb^{3+} concentration is higher than H2, the two distinct Stark components are also visible at 535 nm and 537 nm (see Fig. 10(c)). With the increase in temperature, the intensity remains constant up to 323 K, while a further increase in temperature causes a rapid decrease in the population of 5S_2 , while the population of 5F_4 remains almost the same up to 423 K and then slowly declines afterward. This pattern, resulting from cross-relaxation of population between thermally coupled levels, also causes an increase in luminescence intensity ratio, which shows the

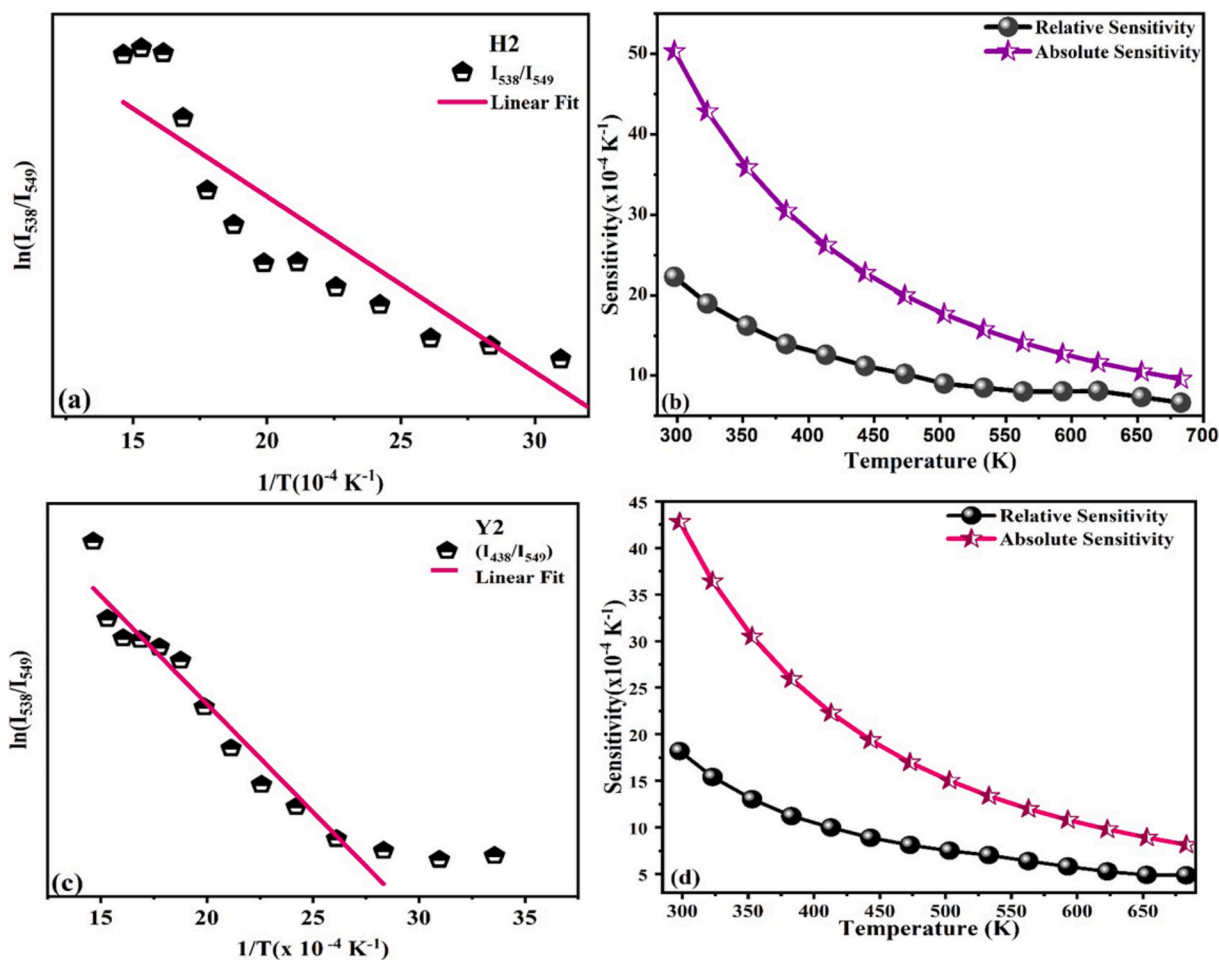


Fig. 11. (a) Intensity ratio of green emission of H2 with temperature; (b) Sensitivity plot with temperature for H2 sample; (c) Intensity ratio of green emission of Y2 with temperature; (d) Sensitivity plot of Y2 sample with temperature. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Table 3

Temperature Sensitivity of different materials doped with lanthanides and values obtained in the present study.

Material	Temperature range(K)/ max temperature	Sensitivity(absolute/ relative) $\times 10^{-4} \text{ K}^{-1}$	Reference
KLu(WO ₃) ₂ : Ho, Tm, Yb	273–523	S(R) -61.0	[50]
NaBiTiO ₃	163–613	S(R) -31	[51]
Y ₂ O ₃ :Ho,Yb	13–450	S(R) -116.8(13K)	[29]
Ba ₃ Y ₄ O ₉ :Ho/ Tm, Yb	298–573	S(A) -17.0	[52]
Y ₂ O ₃ :Ho, Yb, Tm	13–450	S(R) -130(13 K)	[29]
CaTiO ₃ /ZnO: Er, Yb	Max(temp)311	S(R) -10.5	[44]
YNbO ₄ :Er,Yb	293–723	S(A) -83.80	[53]
BaTiO ₃ :Ho,Yb	12–300	S(R) -2.0	[41]
ZnGa ₂ O ₄ :Ho, Yb, Bi	300–600	S(R) -13.6/S(A)-14.9	[33]
Y ₂ O ₃ :Er,Yb	93–613	S(A)-44	[51]
SrF: Er	273–373	S(R)- 93 %	[49]
MgO/Y ₂ O ₃ : Ho, Yb(H2)	298–683	S (A)- 51.34	Present study
MgO/Y ₂ O ₃ : Ho, Yb	298–683	S(R)- 22.24	Present study
MgO/Y ₂ O ₃ : Ho, Yb(Y2)	298–683	S (A)- 42.74	Present study
MgO/Y ₂ O ₃ : Ho, Yb	298–683	S(R)- 18.18	Present study

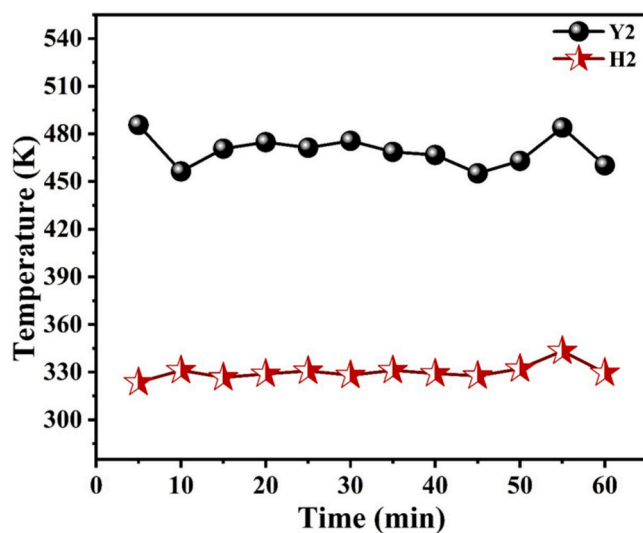


Fig. 12. Variation of internal temperature of H2 and Y2 nano phosphors on the time under 980 nm exposure.

exponential temperature dependence and is employed for further temperature sensing measurements. The $\Delta E/k$ and B have been calculated by fitted curves shown in Fig. 11 (a, c). For H2, $\Delta E/k$ was 447 ± 13.2 ,

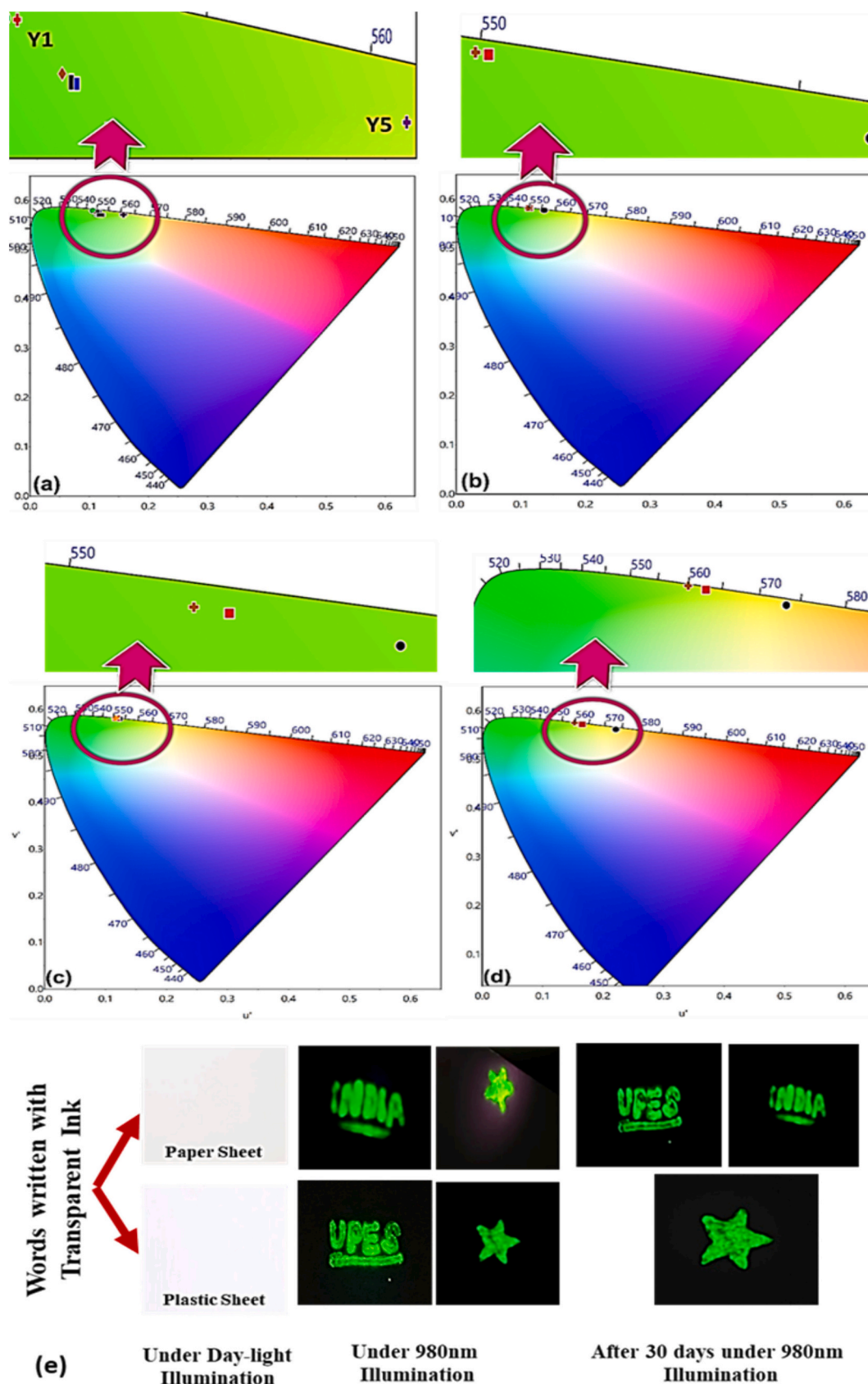


Fig. 13. (a) CIE parameters of all samples; (b-d) CIE parameter of H2, Y2, and Y5 with power variation (black solid circle represents coordinates for lowest pump power and red solid square is coordinates at highest pump power); (e) Under the irradiation of 7 W 980 nm the handwritten letters visibility on paper and plastic sheet and stability of Ink after 30 days. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Table 4
Quantum yield of sample at excitation of 448 nm.

Sample	Quantum yield
H1	1.89
H2/Y1	20
H3	2.3
Y0	9.8
Y2	14.6
Y3	14.1

Table 5
CIE parameters of all phosphors under 980 nm and parametric analysis with power variation.

Sample	x, y	Purity (%)	CCT(K)
H1	(0.2954, 0.6555)	87.1	6074
H2	(0.3000, 0.6897)	98.8	5967
H3	(0.2879, 0.6805)	95.6	6158
Y2	(0.3082, 0.6819)	98.6	5854
Y3	(0.3346, 0.6557)	98.2	5479
Y4	(0.3422, 0.6482)	98.1	5367
Y5	(0.3710, 0.6195)	97.9	4929

and for Y2 the value was 380 ± 10.1 , which is nearly equal to the theoretical value, i.e., 438.84. These values were used for further sensor sensitivity calculation, which can be expressed as follows [48,49]:

$$S_{Abs}(T) = \frac{\partial(LIR)}{\partial T} = B \frac{\Delta E}{kT^2} \exp\left(-\frac{\Delta E}{kT}\right) \quad (2)$$

$$S_{Rel}(T) = \left(\frac{S_{Abs}}{LIR}\right) = \frac{\Delta E}{kT^2} \quad (3)$$

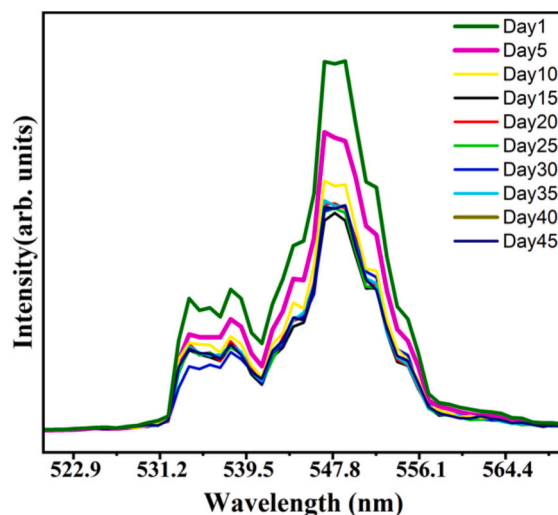
From the observed value of B and $\Delta E / K$, sensing parameters were calculated, and variations of sensitivities with temperature were shown in Fig. 11(b and d). At 298 K the S_{Abs} of H2 and Y2 sample were $51.3 \times 10^{-4} K^{-1}$ and $42.74 \times 10^{-4} K^{-1}$, respectively. At 683 K these values were $9.58 \times 10^{-4} K^{-1}$ and $8.6 \times 10^{-4} K^{-1}$, respectively. The relative sensitivity (S_{Rel}) at 298 K was $22.24 \times 10^{-4} K^{-1}$ and $18.18 \times 10^{-4} K^{-1}$ for H2 and Y2, respectively, and the S_{Rel} decreased up to 6.68×10^{-4} and 4.98×10^{-4} at 683 K, respectively. The results of the present work have been compared with the literature and summarized in Table 3. It is observed that at lower temperatures the sensitivity is better and also at lower concentrations of Yb^{3+} the sensitivities are higher suggesting that the rate of radiative transitions varies with the rare earth ions doping concentration, specifically with the Yb^{3+} ions. Consequently, the doping concentration of ions must be optimized properly for specific applications to attain effective results.

Furthermore, to assess the practical stability of prepared phosphors under the input laser exposure, the input laser effect was also measured to check the stability of samples with time up to 60 mins. The input power density may cause the thermal effect which can increase the temperature of samples and can further affect the luminescence characteristics. The H2 and Y2 were continuously exposed to an NIR laser with an input power density for 60 mins and the spectra were collected at intervals of 5 min. The internal temperature of phosphors has been

derived by using Eq. (1). Calculated results shown in Fig. 12 indicate that the temperature remains relatively constant throughout the exposure, suggesting that both samples achieve thermal equilibrium efficiently. However, it has also been observed that the temperature of Y2 is higher than H2 indicating that an increased concentration of Yb leads to an increase in internal temperature and a more pronounced thermal effect from input power.

3.5. Colorimetric analysis, quantum yield and anti-counterfeiting application

The colorimetric parameters were calculated by using a CIE diagram using OSRAM software and shown in Fig. 13. The CIE parameters, purity, and CCT are summarized in the Table 5. The color purity of all the samples is greater than 96 % and highest in the Y1 and Y2 samples. With the increase in Yb^{3+} , all the colors move towards the yellowish color due to an increase in red contribution with Yb^{3+} enhancement. The correlated color temperature (CCT) is above 5000 K making all the phosphors as natural color emitters except for Y5 where the value is below 5000 K. It suggests that with an increase in concentration of Yb^{3+} , the emitted colors shift towards warm light. Another interesting result is obtained by analyzing the colorimetric parameters with the variation of input power of different samples and parameters are summarized in Table 6. The parameters for H2, Y2, and Y5 have been summarized in the Table 4. It is observed that at minimum power all the samples emit in the yellow region and with an increase in power, it shifts towards a greener region shown in Fig. 13. Also at the highest concentration of Yb^{3+} , the CCT is 3394 K at low input which is warm light and varies with power to 4929 K. It shows that all the samples move towards natural light with an increase in input power. The stability of these materials suggests that the specific composition of the prepared phosphors has the potential to be employed after collective optimization of concentration and input power for different applications in warm and cool LEDs. Furthermore,

**Fig. 14.** Emission spectra of ink taken at excitation of 980 nm up to 45 days.**Table 6**
Parametric analysis with power variation.

Power (mW)	Y1			Y2			Y5		
	x, y	Purity	CCT (K)	x, y	Purity	CCT (K)	x, y	Purity	CCT (K)
0.6	(0.3421, 0.6443)	97	5368	(0.3201, 0.6690)	98.1	5688	(0.4594, 0.5186)	93	3394
1	(0.3015, 0.6882)	98.7	5947	(0.3103, 0.6797)	98.8	5825	(0.3885, 0.5980)	96.5	4643
2	(0.3000, 0.6897)	98.8	5967	(0.3082, 0.6819)	98.6	5854	(0.3710, 0.6195)	97.9	4929

the quantum yield of samples has been recorded by using Xenon lamp at excitation of 448 nm. Quantum yield (QY) is the ratio of number of photons absorbed and number of photons emitted by the sample. Here, BaSO₄ has been used as the reference. Highest QY is observed for Y2 sample i.e. 20 %. The quantum yield of all samples summarized in Table 4.

Finally, this report presents the novel use of MgO-Y₂O₃ (Ho³⁺, Yb³⁺) as the security ink for anti-counterfeiting applications, which were not reported in the literature. The synthesis of ink has been described in the synthesis section. The spectra of prepared ink has been taken upto 45 days and no shifting and decaying has been observed (see Fig. 14). With the prepared ink some patterns and letters were written on paper, plastic, and card which were not visible in daylight shown in Fig. 12 (e). On the irradiation with 980 nm NIR laser after tuning it to 7 W the patterns and letters INDIA, UPES, and pattern of star illuminated with intense green light. Irradiating the letters again after 1 month no fading was observed confirming the stability of fabricated ink for application purposes. The obtained findings demonstrate that the prepared phosphors will provide a novel approach to fabricating cost-effective material for advanced security ink applications.

4. Conclusion

In the present report, a comprehensive study on MgO - Y₂O₃ nanocomposites co-doped with Ho (0.1, 0.5, 1 mol%) and Yb (0.5, 5, 10, 15, 20 mol%) has unveiled the promising results on DC and UC luminescence. It has been investigated that dopants mainly occupy the lattice positions of the Y₂O₃ compound and facilitate structural perturbations. The morphology of doped samples remains invariant under the doping concentration variations. The successful synthesis of dual-phase nanocomposites with controlled morphology, along with the mechanism of UC and DC luminescence demonstrates the composition's versatility in optical applications. The in-depth analysis of DC and UC luminescence, with optimized concentration at Ho (0.5 mol%) Yb (5 mol%), conveys that emission in green, red, and NIR regions strongly depends on both the dopant concentrations. The power-dependent luminescence mechanism revealed the involvement of three to four-photon for green emissions and two to three-photon for red and NIR emissions. Additionally, the temperature sensing studies show that absolute and relative sensitivity for H2 samples were $51.3 \times 10^{-4} \text{ K}^{-1}$ and $22.24 \times 10^{-4} \text{ K}^{-1}$ respectively, and the same for Y2 were $42.74 \times 10^{-4} \text{ K}^{-1}$ and $18.18 \times 10^{-4} \text{ K}^{-1}$ at 298 K. Further, both the optimized nano phosphors easily reached thermal equilibrium when irradiated with prolonged 980 nm input power showing stability. The tunable colorimetric properties, with adjustable correlated color temperature and color purity >98 %, make them a potential candidate for warm and cool LEDs. The successful demonstration of the fabricated security ink of the MgO-Y₂O₃ nano phosphors and the ability to create invisible patterns obtained from ink that illuminate under NIR excitation, coupled with long-duration stability paves the way for their practical implementation in specific industrial applications.

CRediT authorship contribution statement

Priyanka Bishnoi: Writing – review & editing, Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Vaishali Rathi:** Writing – review & editing, Methodology, Investigation, Data curation. **Madan M. Upadhyay:** Writing – review & editing, Resources, Methodology. **Aditya Sharma:** Writing – review & editing, Supervision, Resources, Methodology, Investigation, Formal analysis. **Kaushal Kumar:** Writing – review & editing, Resources. **Ranjeet Brajpuriya:** Writing – review & editing, Visualization, Supervision, Resources, Methodology, Formal analysis. **Ankush Vij:** Writing – review & editing, Writing – original draft, Validation, Supervision, Resources, Methodology, Investigation, Formal analysis, Conceptualization.

Author statement

The results reported in the manuscript are original and have been written by the stated authors, who are all aware of its content and approved its submission. It has not been published previously. It is not under consideration for publication elsewhere. No conflict of interest exists. If accepted, the article will not be published elsewhere in the same form, in any language, without the written consent of the publisher.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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