

# **Crystallographic and Optical Characteristics of Ultraviolet‑Stimulated**  Dy<sup>3+</sup>-Doped Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub> Nanorods

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#### **Abstract**

A ultraviolet-triggered white light-emitting  $Ba_2Gd_{1-x}V_3O_{11}$ :*x*Dy<sup>3+</sup> nanophosphor series has been developed with the aid of urea-fueled solution combustion route, for the frst time. Powder x-ray difraction and Rietveld refnement have been utilized to study the structural features of the synthesized series. Transmission electron microscopy (TEM), feld emission scanning electron microscopy (FE-SEM), and energy-dispersive spectroscopy (EDS) analysis have been employed for morphological and elemental analysis. When triggered by ultraviolet radiation at  $324$  nm, the Dy<sup>3+</sup>-doped nanophosphor exhibited characteristic peaks attributed to emissions due to  ${}^4F_{9/2}$   $\rightarrow {}^6H_{13/2, 15/2}$  transitions. The concentration quenching phenomenon was witnessed for the 3 mol% fluorescent composition in Dy<sup>3+</sup>-doped Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub> nanophosphor. The Auzel's model has been of great usage in determining the values for intrinsic lifetime  $(1.112 \text{ ms})$  and non-radiative rates  $(421.178 \text{ s}^{-1})$ . The efficient and cool conduct of the white light-emitting nanophosphor series has been endorsed by the CIE color coordinates  $(x = 0.256, y = 0.341)$ , correlated color temperature (CCT = 9751 K), and quantum efficiency value (67%). The structural and photoluminescence behavior featured by the trivalent dysprosium-doped  $Ba<sub>2</sub>GeV<sub>3</sub>O<sub>11</sub>$  nanophosphor support its usage in ultraviolet-stimulated lighting devices.

#### **Graphical Abstract**



**Keywords** Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub>:Dy<sup>3+</sup> · WLEDs · non-radiative rate · nanophosphor · photoluminescence

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## **Introduction**

The origin and evolution of life forms on Earth has been heavily dependent upon the Sun, a natural source of light that is well known for shaping the habits of living organisms on the planet. Early humans made a signifcant and remarkable discovery of fre. This major breakthrough led to the manifestation of light in a form other than that of

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natural sources. Human civilizations are known to ignite fres using diferent fuels and exploit this artifcial source of lighting to the fullest. In recent history, artifcial light sources such as gas lamps, electric lamps, incandescent light bulbs, and high-intensity discharge (HID) lamps have been invented. Among these the HID lamps, compact fuorescent lamps (CFLs), high-pressure sodium lamps, and metal-halide lamps are the most advanced and successful types.<sup>[1](#page-12-0)–[3](#page-12-1)</sup> The latter half of the twentieth century witnessed the invention of a solid-state lighting (SSL) technology, far more advanced than its counterparts. This invention, namely, the light-emitting diode (LED), produces light from electricity using a semiconductor material. White light-emitting diodes (WLEDs) are known today for their sturdiness and high-quality light production. The global demand for energy conservation looks at WLEDs as an energy savior. Qualities such as less power usage, long life span, and energy efficiency have made WLEDs quite popular among households, industries, and research units. WLEDs are of great advantage for lighting, displays, and biomedical applications. $4-6$  $4-6$ 

The fabrication process of a WLED plays a crucial role in its resulting properties. On an established note, a yellow light-emitting cerium-doped yttrium aluminum garnet  $(Ce^{3+}$ :YAG) is coated upon a blue light-emitting indium gallium nitride (InGaN) chip. The emitted blue light excites the yellow phosphor and produces white light.<sup>[7](#page-12-4)[,8](#page-12-5)</sup> However, fabrication through this conventional route involves some disadvantages, namely, insufficient red constituent in the emission spectra and low color rendering index (CRI) (Ra  $< 80$ ).<sup>[9,](#page-12-6)10</sup> Another route of fabrication has been followed in order to deal with the abovementioned inadequacies. The ultraviolet/near-ultraviolet (UV/NUV)-emitting chip is used in place of the blue-emitting InGaN chip along with a collection of phosphors with individual red, green, and blue emission. As the three diferent phosphors display diferent decay times, an intense white emission is not observed. $11,12$  $11,12$ Thus, a single white light-emitting phosphor, excitable by UV or NUV radiation, is a good choice to fabricate WLEDs. Consequently, good luminescence, chemical stability, and efficient UV/NUV absorption are some of the qualities that a white light-emitting phosphor should exhibit. $13,14$  $13,14$  Such phosphor materials need to be explored, studied, and potentially used in the fabrication of WLEDs.

An efficient white light-emitting material can be synthesized by packing a stable host and an appropriate luminescent ion together. The metals known as rare earths are well known to absorb UV/NUV radiation in an efective manner. Diferent color emissions can be obtained by switching the rare earth metals or ions as dopants. $15-17$  $15-17$  $15-17$  The nature of the host lattice is crucial to the stability and integrity of the synthesized nanophosphor. Hence, a number of borate-, nitride-,

sulfde-, and vanadate-based host materials are studied and thoroughly analyzed.<sup>18[,19](#page-12-15)</sup> Among the abovementioned materials, the ones comprising a vanadate group are known to form a stable lattice at lower synthesizing temperatures and aid efectively in the luminescence exhibited by the nanophosphor by transfer of energy from vanadate to the dopant unit. $^{20}$  $^{20}$  $^{20}$  These qualities suffice for the vanadate-based host to be considered as an efective host material for the synthesis of nanophosphor. The trivalent rare earth ion  $Dy^{3+}$  is well known for its absorption of radiation falling in the ultraviolet region and emitting bright white light due to its characteristic  ${}^{4}F_{9/2} \rightarrow {}^{6}H_{13/2, 15/2}$  transitions.<sup>[21](#page-12-17)</sup> The above discussion points in favor of doping a vanadate-based host with trivalent dysprosium ions in order to achieve a stable, crystalline, and efficient white light-emitting nanophosphor. According to the literature survey, there are two reports on rare earthdoped  $Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub>,<sup>22,23</sup>$  $Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub>,<sup>22,23</sup>$  $Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub>,<sup>22,23</sup>$  $Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub>,<sup>22,23</sup>$  $Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub>,<sup>22,23</sup>$  but there has not been a single work reported on  $Dy^{3+}$ -doped Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub> nanophosphor. The present report is focused on preparing a homogenous Dy<sup>3+</sup>-doped Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub> nanophosphor series at 900<sup>o</sup>C by means of a time-saving (calcination time  $= 3$  h) solution combustion route. In the literature, the rare earth-doped compositions of  $Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub>$  nanophosphor were synthesized by sol–gel approach constituting 48 h of reaction for gel formation and 3-h-long calcination of the sample in a temperature range of  $600^{\circ}$ C– $800^{\circ}$ C.<sup>22</sup> In the current report, detailed structural, optical, and decay behavior analyses have been performed exclusively. A thorough photometric and decay analysis for trivalent dysprosium-doped  $Ba_2GdV_3O_{11}$ nanophosphor has also been carried out in present report, as original work.

An energy-efficient method of solution combustion has been utilized to prepare the nanophosphor series of  $Ba_2Gd_{1-x}V_3O_{11}$ :*xDy*<sup>3+</sup> (*x* = 0.01–0.09). The Rietveld refinement explored the aspects of coordination of various entities as well as crystal system of the synthesized nanophosphor. The energy transfer phenomenon has been explored via calculations of parameters such as critical distance  $(R<sub>c</sub>)$  of energy transfer. Diferent techniques such as transmission electron microscopy (TEM), feld emission scanning electron microscopy (FE-SEM), and energy-dispersive spectroscopy (EDS) have been employed for morphological plus elemental analysis. The photoluminescence and decay behavior exhibited by the  $Ba_2Gd_{1-x}V_3O_{11}:xDy^{3+}$  nanophosphor have been investigated via excitation emission and decay curves, respectively. The values for radiative rates, quantum efficiency, and non-radiative rates have been attained for a complete series of Ba<sub>2</sub>Gd<sub>1−*x*</sub>V<sub>3</sub>O<sub>11</sub>: $x$ Dy<sup>3+</sup> ( $x = 0.01-0.09$ ) nanophosphor. The CIE *x*, *y* coordinates as well as CCT (K) values confirm the Dy<sup>3+</sup>-doped Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub> nanophosphor as a useful candidate for UV-triggered lighting sources.

#### **Experimental**

#### **Materials and Synthesis**

Solution combustion synthesis was employed to prepare the entire nanophosphor series of  $Dy^{3+}$ -doped  $Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub>$  nanophosphor. The time-saving and energyefficient route led to highly crystalline and homogeneous nanophosphor product. $24-26$  $24-26$  $24-26$  The initial step implied the procurement of highly pure chemicals, namely,  $Dy(NO_3)_{3}$ .  $xH_2O$ , Ba(NO<sub>3</sub>)<sub>2</sub>, Gd(NO<sub>3</sub>)<sub>3</sub>.6H<sub>2</sub>O, NH<sub>4</sub>VO<sub>3</sub>, and urea. The ammonium vanadate and diferent metal nitrates acting as oxidizers were weighed in accordance to the ratio of oxidizer and fuel taken as one. The reactants after proper weighing were dissolved in an appropriate amount of deionized water reserved in a Pyrex beaker (250 mL). The beaker was then placed on a hot plate  $(150^{\circ}C)$  for a time span of 5 min. The heat aided in dissolving the reactants properly. The Pyrex beaker containing the homogeneous solution was subjected to a temperature of 500°C for h inside a muffle furnace. After 0.5 h, the Pyrex beaker was removed from the muffle furnace, and the contents were cooled to room temperature. It was observed that the homogeneous solution had turned into a voluminous powder. The powder, following a fne crushing, was transferred to an alumina crucible and put into a furnace set at 900°C. Later, after 3 h, the alumina crucible was subjected to room temperature, and its content was allowed to cool down. The content was fnely ground to a powder form and saved in a desiccator for characterization procedures. Different compositions of the Ba<sub>2</sub>Gd<sub>1−*x*</sub>Dy<sub>*x*</sub>V<sub>3</sub>O<sub>11</sub> nanophosphor series were prepared via the aforementioned route, as explained by the following chemical equation:

 $2Ba(NO<sub>3</sub>)<sub>2</sub> + (1 - x)Gd(NO<sub>3</sub>)<sub>3</sub> \cdot 6H<sub>2</sub>O +$  $xDy(NO_3)_3$  $xH_2O + 3NH_4VO_3 + 4.33NH_2CONH_2 \rightarrow$  $Ba_2Gd_{1-x}V_3O_{11}$ :*x*Dy<sup>3+</sup> + gaseous products (mixture of  $CO<sub>2</sub>$ , H<sub>2</sub>O, and N<sub>2</sub>)

#### **Methods of Characterization**

The entire  $Ba_2Gd_{1-x}V_3O_{11}$ :*x*Dy<sup>3+</sup> (*x* = 0.01–0.09) nanophosphor series was synthesized via an efficient route of solution combustion. The patterns due to difraction exhibited by the individual compositions were used to analyze the phase purity of the prepared nanophosphors. A Rigaku Ultima-IV labeled powder difractometer working at high resolution with x-rays irradiated from Cu  $K\alpha$ with a 40-mA tube current and voltage of 40 kV was employed to document x-ray difraction (XRD) patterns in a continuous scanning fashion at rate of 2° per minute from an angular range of 10° to 80°. The XRD data of the optimal fuorescent composition was analyzed via Rietveld refnement. In order to do so, a pseudo-Voigt function was applied in the software program General Structure Analysis System (GSAS).

An FEI Tecnai TF20 labeled transmission electron microscope recorded the TEM and high-resolution transmission electron microscopy (HRTEM) micrographs of  $Ba_2Gd_{1-r}V_3O_{11}$ :*x*Dy<sup>3+</sup> nanophosphor. The surface of the synthesized sample was analyzed via feld emission scanning electron microscopy (FE-SEM) using an FEI Quanta 200 consisting of an intense feld-emission gun (FEG) as the source and an Everhart–Thornley detector. The sample surface was scanned with a high-energy electron beam in a raster scan pattern. The elemental analysis was performed using the Oxford EDS system IE 250 X Max 80 with a silicon drift detector (SDD). A fuorescence spectrophotometer marked as Hitachi F-7000 was used to study the photoluminescence spectra of the Dy<sup>3+</sup>-doped Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub> nanophosphor. A wavelength scan at a rate of 1200 nm/min was run at a 400-V photomultiplier tube (PMT) voltage, and 5 nm was the slit width to record the excitation and emission spectra of the Ba<sub>2</sub>Gd<sub>1−*x*</sub>V<sub>3</sub>O<sub>11</sub>: $x$ Dy<sup>3+</sup> ( $x$  = 0.01–0.09) nanophosphor series. The decay curves were recorded by a time scan at a chopping speed of 40 Hz and a response time of 2 ms with abovementioned conditions of voltage and slit width. The color coordinates  $(x, y)$  of the optimal luminescent composition of  $Ba_2Gd_{0.97}Dy_{0.03}V_3O_{11}$  were conveyed via the CIE 1931 chromaticity diagram. The *u'*, *v'* coordinates and CCT values were also calculated.

### **Results and Discussion**

#### **Structural Studies**

An energy-efficient method of solution combustion was followed to formulate a homogenous and crystalline Ba<sub>2</sub>Gd<sub>1−*x*</sub>V<sub>3</sub>O<sub>11</sub>: $x$ Dy<sup>3+</sup> ( $x$  = 0.01–0.09) nanophosphor series. The XRD patterns for both pure host and doped composition were obtained by x-ray investigation and detected to be in close accord to that of standard JCPDS card no. 45-0097 for  $Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub>$  pure host, as depicted in Fig. [1.](#page-3-0) The trivalent dysprosium ion is supposed to replace the trivalent gadolinium ion on doping owing to similar charges and ionic radii. This assumption can be reasoned by calculating the radius percentage difference  $(\Delta_r)$  by the following equation:

<span id="page-2-0"></span>
$$
\Delta_r = \frac{R_m(CN) - R_d(CN)}{R_m(CN)} \times 100\tag{1}
$$

In Eq. [1,](#page-2-0)  $\Delta_r$  stands for the percentage difference in radius,  $R<sub>m</sub>(CN)$  signifies the effective ionic radii corresponding to

host ions,  $R_d(CN)$  represents the effective ionic radii analogous to dopant ions. Also, *CN* symbolizes the coordination number of the concerned ion. The coordination exhibited by both Dy<sup>3+</sup> and Gd<sup>3+</sup> ions in Ba<sub>2</sub>Gd<sub>1−*x*</sub>Dy<sub>*x*</sub>V<sub>3</sub>O<sub>11</sub> nanophosphor is seven  $(CN = 6)$ . In the present case, the magnitude for radius percentage difference  $(\Delta_r)$  comes out to be 2.77%. Also, the approved value of  $\Delta_r$  is less than 30%.<sup>[27](#page-12-22)</sup> Therefore,  $Dy^{3+}$  dopant ions replace  $Gd^{3+}$  in the host lattice. The assumptions of  $Ba^{2+}$  or  $V^{5+}$  ions being replaced by dopant ions cannot be approved due to dissimilarity in



<span id="page-3-0"></span>**Fig. 1** XRD pattern of  $Ba_2Gd_{1-x}Dy_xV_3O_{11}$  ( $x = 0 - 0.09$ ) nanophosphor system along with the standard data of  $Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub>$  host lattice.

their charge, size, coordination, and large value of radius percentage diference.

Rietveld refinement was performed on XRD data of  $0.03Dy^{3+}$ :Ba<sub>[2](#page-3-1)</sub>Gd<sub>0.97</sub>V<sub>3</sub>O<sub>11</sub> nanophosphor (Fig. 2). The refned graph consists of a regular red line (experimental data set), an irregular square-shaped line (observed data set), and a navy-bluish line exhibiting the diference between the previously mentioned lines. The pattern was refined up to  $\gamma$  2 = 2.076. Different parameters relative to lattice, namely  $a = 12.3932(12)$  Å,  $b = 7.7317(7)$  Å,  $c =$ 11.1786(11)  $\AA$ , and  $V = 1042.51(11) \AA^3$ , were obtained and listed in Table [I.](#page-4-0) There was an observed increase in density of the doped composition due to increase in formula weight and a subsequent decrease in volume on replacing  $Gd^{3+}$  ions (larger ionic radii) with dopant  $Dy^{3+}$  ions (smaller ionic radii). The ion types and the respective occupancy are depicted in Table [II.](#page-5-0) There are two types of Ba atomic sites (Ba1 and Ba2), one type of Gd/Dy site (Gd1/Dy1), and three types of V sites (V1, V2 and V3). Ba1, Ba2, and Gd/Dy bear a coordination number of ten, 11, and six, respectively (Fig. [3\)](#page-4-1). There are present both  $VO_4^{3-}$  and  $V_2O_7^{4-}$  groups in Ba<sub>2</sub>Gd<sub>1−*x*</sub>Dy<sub>*x*</sub>V<sub>3</sub>O<sub>11</sub> nanophosphor. All V sites were found to have a coordination number of four and coordinated in a tetrahedral fashion. The homogenous powder form of synthesized nanophosphor may consist of agglomerated particles, and hence, the average crystallite size of the particles of synthesized  $Dy^{3+}$ :Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub> nanophosphor was estimated using the renowned Scherrer equation<sup>28</sup> as follows:



<span id="page-3-1"></span>**Fig. 2** Rietveld refinement of Ba<sub>2</sub>Gd<sub>0.93</sub>Dy<sub>0.03</sub>V<sub>3</sub>O<sub>11</sub> nanophosphors.  $\chi^2 = 2.076$ ,  $R_{wp}$  (%) = 11.87, and  $R_p$  (%) = 7.81.



<span id="page-4-1"></span>**Fig. 3** Unit cell view of Ba<sub>2</sub>Gd<sub>1−*x*</sub>Dy<sub>*x*</sub>V<sub>3</sub>O<sub>11</sub> nanocrystals along with the coordinative environment of various cations.

<span id="page-4-0"></span>Table I Crystal structure data and goodness of fit for  $Ba_2Gd_{0.97}Dy_{0.03}V_3O_{11}$  phosphor system as compared to  $Ba_2GdV_3O_{11}$ host matrix

$Ba_2Gd_{0.97}Dy_{0.03}V_3O_{11}$	$Ba_2GdV_3O_{11}$
760.88	760.72
Monoclinic	Monoclinic
P21/c(14)	P21/c(14)
12.3932(12)	12.3400
7.7317(7)	7.7420
11.1786(11)	11.2490
90 103.277	90 103.300
1042.51(11)	1045.86
4	4
4.84752	4.8308
mP68	mP68
e17	e17

$$
D = \frac{0.941\lambda}{\beta \cos \theta} \tag{2}
$$

In the abovementioned equation, *D* stands for the average crystallite size,  $\lambda$  symbolizes the x-ray wavelength set for the difractometer (= 0.1540562 nm), and β represents the full width of the difraction line at an intensity value half of the maximum for the most intense peak, whereas θ signifes that angle where the most intense peak is positioned. The average size of crystallite corresponding to the Ba<sub>2</sub>Gd<sub>1−*x*</sub>V<sub>3</sub>O<sub>11</sub>:*x*Dy<sup>3+</sup> (*x* = 0.01–0.09) nanophosphor was estimated to be 34 nm.

The synthesized trivalent dysprosium-doped nanophosphor was analyzed through HRTEM, and the results are depicted in Fig. [4a](#page-5-1) and b. The HRTEM result consists of planes with interplanar distance of 0.311 nm that equates to a diffraction plane of (122) Ba<sub>2</sub>Gd<sub>1−*x*</sub>V<sub>3</sub>O<sub>11</sub>:*x*Dy<sup>3+</sup> nanophosphor. The TEM (Fig. [4](#page-5-1)b) and FE-SEM (Fig. [4](#page-5-1)c) results represent rod-like structures with a diameter in the nano range. The EDS) analysis result provided a graph with peaks corresponding to the elements present in the platinum-coated sample of prepared nanophosphor  $Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub>:Dy<sup>3+</sup>$  (Fig. [5\)](#page-6-0). There was no other peak than the respective elements, and hence, the synthesized nanophosphor is free from any kind of impurity. The nanophosphor was synthesized through solution combustion route and that is known for homogenous product formation.<sup>[29](#page-12-24)[,30](#page-12-25)</sup> In order to analyze the uniform distribution of various chemical components, elemental mapping was performed. Figure [6](#page-6-1) indicates a homogenous nature of the synthesized  $Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub>:Dy<sup>3+</sup>$  nanophosphor via elemental mapping.

<span id="page-5-0"></span>**Table II** Various ion types and occupancy in  $Ba_2Gd_{0.97}Dy_{0.03}V_3O_{11}$ nanophosphor





<span id="page-5-1"></span>**Fig. 4** (a) HRTEM, (b) TEM, and (c) FE-SEM images of  $Ba_2Gd_{1-x}Dy_xV_3O_{11}$  nanophosphor.

#### **Photoluminescence Analysis**

Different mol% compositions of the  $Ba_2Gd_{1-r}Dy_rV_3O_{11}$  $(x = 0.01 - 0.09)$  nanophosphor series were prepared by solution combustion method, and the respective excitation/emission spectra and decay curves were documented on a fluorescence spectrophotometer using a running wavelength and time scan, respectively. The excitation spectra of  $Ba<sub>2</sub>Gd<sub>1-x</sub>Dy<sub>x</sub>V<sub>3</sub>O<sub>11</sub>$  nanophosphors at emission



<span id="page-6-0"></span>

wavelength of 573 nm are displayed in Fig. [7](#page-7-0). There is an intense band centered at 324 nm that is attributed to the absorption of host. As the wavelength proceeds towards 500 nm, multiple other bands arise in the excitation spectra due to characteristic  $f-f$  transitions of dopant  $Dy^{3+}$  ions. These include four small bands centered at 351 nm, 365 nm, 389 nm, and 427 nm and two broad peaks at 453 and 474 nm. The most intense band with maxima at 324 nm arises in the excitation spectra due to  ${}^{1}A_{1} \rightarrow {}^{1}T_{1,2}$  transition in the  $VO_4^{3-}$  group of host. The  $VO_4^{3-}$  unit of the host comprises a ground-state term  $({}^{1}A_{1})$ , two singlet excitedstate terms  $({}^{1}T_{1}, {}^{1}T_{2})$ , and two triplet excited-state terms  $({}^{3}T_{1}, {}^{3}T_{2})$ .<sup>[31,](#page-12-26)32</sup> A broad band at 324 nm indicates that the singlet excited states  $({}^{1}T_{1}, {}^{1}T_{2})$  are closer in energy; hence, rather than two separate peaks, a broad band arises due to  ${}^{1}A_{1} \rightarrow {}^{1}T_{1,2}$  transitions. Furthermore, the small bands at 351 nm, 365 nm, 389 nm, and 427 nm are attributed to  ${}^{6}H_{15/2} \rightarrow {}^{6}P_{7/2}$ ,  ${}^{6}P_{5/2}$ ,  ${}^{4}I_{13/2}$ , and  ${}^{4}G_{11/2}$  transitions, respectively, whereas the broad peaks centered at 453 and 474 nm arise due to  ${}^{6}H_{15/2} \rightarrow {}^{4}I_{15/2}$  and  ${}^{6}H_{15/2} \rightarrow {}^{4}F_{9/2}$  characteristic transition of  $Dy^{3+}$  ions.<sup>[33](#page-12-28)</sup> In the excitation spectra from 200 to 500 nm, the charge transfer band due to  ${}^{1}A_1$  $\rightarrow$  <sup>1</sup>T<sub>1,2</sub> transition is the most intense. This excitation at Fig. 5 EDS analysis of Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub>:Dy<sup>3+</sup> nanophosphor. 324 nm brands the synthesized nanophosphor as suitable



<span id="page-6-1"></span>**Fig. 6** The elemental mapping of  $Ba_2Gd_{1-x}Dy_xV_3O_{11}:Dy^{3+}$  nanophosphor.

for usage in UV-directed WLEDs.<sup>[34](#page-13-0)</sup> The characteristic  $f$ –*f* transitions attributed to  $Dy^{3+}$  ions arise from the groundstate  ${}^{6}H_{15/2}$  in the excitation spectra (Fig. [7\)](#page-7-0). A report on doped  $Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub>$  indicated excitation in the nearinfrared region (NIR) with wavelength of 978 nm. Also, the  $Yb^{3+}$  acts as a sensitizer enhancing absorption of the NIR radiation, whereas the low concentrations of emitters such as  $Er^{3+}$ , Ho<sup>3+</sup>, and Tm<sup>3+</sup> with 2, 1, and 0.5 mol% compositions exhibit characteristic *f*–*f* transitions that led to emission in green, orange, and blue regions. $^{22}$  $^{22}$  $^{22}$ 



<span id="page-7-0"></span>**Fig. 7** Excitation spectra of  $Ba_2Gd_{1-x}Dy_xV_3O_{11}$  ( $x = 0.01-0.09$ ) nanophosphor studied at  $\lambda_{em} = 573$  nm.

The emission spectra displayed by the  $Dy^{3+}$ -doped  $Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub>$  nanophosphor series on excitation by 324-nm wavelength are represented in Fig. [8](#page-7-1) along with the emission curve of  $Ba_2Gd_{0.97}Dy_{0.03}V_3O_{11}$  in the inset. The emission spectra encompass two intense peaks as the wavelength proceeds from 400 to 700 nm. A peak at 484 nm arises in the blue zone, whereas the peak centered at 573 nm lies in the yellow zone of the concerned spectra. The former peak is attributed to magnetic dipole type of  ${}^{4}F_{9/2} \rightarrow {}^{6}H_{15/2}$  transition, and the latter is credited to  ${}^{4}F_{9/2} \rightarrow {}^{6}H_{13/2}$  which is an electric dipole transition.<sup>[35](#page-13-1),36</sup> The yellow-to-blue integrated photoluminescence emission intensity ratio for respective compositions of the Dy<sup>3+</sup>-doped Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub> nanophosphor series is listed in Table [III](#page-8-0). It can be witnessed from the table that the ratio comes out to be closer to unity and bears only small variation. Therefore, diferent mol% compositions show stability in color display. The emission spectra bear no peak or band because of the  $VO<sub>4</sub><sup>3-</sup>$  group of the host. So it indicates that the excitation wavelength of 324 nm after being absorbed by the vanadate group is transferred nonradiatively to the  $Dy^{3+}$  dopant ions. It aids to the increment in the intensity of the characteristic emission demonstrated by  $Dy^{3+}$  ions. Also, there occurs a continuous increase in the emission intensity of Ba<sub>2</sub>Gd<sub>1−*x*</sub>V<sub>3</sub>O<sub>11</sub>: *x*Dy<sup>3+</sup> nanophosphor up to  $x = 0.03$  and a decrement afterwards, as displayed in Fig. [10.](#page-9-0) The concentration quenching phenomenon plays a role in the increment in the emission intensity values up to the optimal fuorescent composition followed by a decline in the same. The various excitation and emission transitions and



<span id="page-7-1"></span>**Fig. 8** Emission spectrum of Ba<sub>2</sub>Gd<sub>1-*x*</sub>Dy<sub>*x*</sub>V<sub>3</sub>O<sub>11</sub> ( $x = 0.01-0.09$ ) phosphor at  $\lambda_{ex} = 324$  nm.

their corresponding wave numbers are listed in Table [IV](#page-8-1). The Dy<sup>3+</sup>-doped Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub> nanophosphor on excitation at 324 nm exhibited various radiative and non-radiative processes of energy transfer. Figure [9](#page-8-2) displays an energy level diagram and is a depiction of all these processes occurring among quantum mechanical term states. The diagram comprises the energy level of the vanadate group of the host and dopant  $Dy^{3+}$  ion. The VO<sub>4</sub><sup>3-</sup> group consists of <sup>1</sup>A<sub>1</sub> term as ground state, singlets  ${}^{1}T_{1}$  and  ${}^{1}T_{2}$  along with triplets  ${}^{3}T_{1}$  and  ${}^{3}T_{2}$  as excited states. The nanophosphor, on being supplied with 324 nm of excitation wavelength, exhibited energy absorption by the vanadate group of the host  $({}^{1}A_{1} \rightarrow {}^{1}T_{1,2})$  transition) and a successive transfer of this energy to dopant  $Dy^{3+}$ ions. The emission spectra are deprived of any band credited to the host. Hence, there is no energy transmission among singlet  ${}^{1}T_{1,2}$  states to triplet  ${}^{3}T_{1,2}$  states in the VO<sub>4</sub><sup>3–</sup> group. A complete transfer of energy occurs in a non-radiative fashion among  ${}^{1}T_{1,2}$  states of the vanadate group and term states of dopant  $Dy^{3+}$  ions. The  ${}^{6}H_{15/2}$  ground state of trivalent dysprosium ion populates  ${}^{6}P_{7/2}$ ,  ${}^{6}P_{5/2}$ ,  ${}^{4}I_{13/2}$ ,  ${}^{4}G_{11/2}$ ,  ${}^{4}I_{15/2}$ , and  ${}^{4}F_{9/2}$  term states on excitation of 351, 365, 389, 427, 453, and 474 nm of wavelength, respectively. These excited term states relax non-radiatively to another excited term state:  ${}^{4}F_{9/2}$ . Two emission peaks emerge at 484 and 573 nm due to radiative relaxation of the  ${}^{4}F_{9/2}$  quantum mechanical state to  ${}^{6}H_{15/2}$  and  ${}^{6}H_{13/2}$  term states. The intensity of these emission

<span id="page-8-0"></span>**Table III** Y/B ratio, CIE 1931 chromaticity coordinates, and CCT of  $Ba_2Gd_{1-x}Dy_xV_3O_{11}$  ( $x = 0.01-0.09$ ) nanophosphors

$Ba_2Gd_{1}$ , D $y_r V_3 O_{11}$ $(mol\%)$		Y/B ratio CIE coordinates (x, y)	(u', v')	CCT(K)
1	0.59	0.231, 0.304	0.149, 0.442 14,189	
$\overline{c}$	0.63	0.240, 0.315	0.152, 0.450 12,330	
$\mathcal{E}$	0.77	0.256, 0.341	0.155, 0.466 9751	
$\overline{4}$	0.71	0.240, 0.334	0.147, 0.460	11.195
5	0.65	0.239, 0.321	0.150, 0.453	11.987
7	0.66	0.232, 0.319	0.146, 0.451	12,804
9	0.67	0.226, 0.323	0.141, 0.453 13,016	

<span id="page-8-1"></span>**Table IV** Diferent types of transitions occurring in photoluminescence spectra of  $Ba_2Gd_{1-x}Dy_xV_3O_{11}$  (*x* = 0.01–0.09) nanophosphors

peaks increases up to 3 mol% composition and decreases afterwards, which is quite similar to that of its  $Eu^{3+}$ -doped counterpart.<sup>23</sup> In Ba<sub>2</sub>Gd<sub>0.97</sub>Dy<sub>0.03</sub>V<sub>3</sub>O<sub>11</sub> nanophosphor, the critical distance  $(\mathring{A})$  of energy transfer was calculated by the following equation $37$ :

<span id="page-8-3"></span>
$$
R_c = 2\left[\frac{3V}{4\pi x_c N}\right]^{1/3} \tag{3}
$$

The abovementioned equation comprises  $R_c$  (the critical distance),  $V$  (the volume capacity related to the unit cell),  $x_c$ (optimal fuorescent composition), and *N* (replaceable num-ber of cations per unit cell). In Eq. [3,](#page-8-3) putting  $V = 1042.51$  $\AA^3$ ,  $x_c = 0.03$ , and  $N = 4$ , the value of  $R_c$  was obtained as 25.50 Å. For exchange interactions to occur, the condition of  $R_c \leq 5$  Å must be met. As far as the obtained value of critical distance, 25.50 Å is considered in the present case; the exchange interactions do not play a role in energy migration.



<span id="page-8-2"></span>**Fig. 9** Energy level diagram for  $Dy^{3+}$ -doped  $Ba_2GdV_3O_{11}$  nanophosphor.





<span id="page-9-0"></span>**Fig. 10** Variation of photoluminescence emission intensity as a function of dopant concentration in Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub>:Dy<sup>3+</sup> nanophosphor.

Furthermore, the absence of any kind of overlap of bands among excitation and emission spectra rules out the possibility of energy transfer by means of radiative reabsorption. The multipolar interactions led by energy transfer have been indicated by Dexter's theoretical model. $^{27}$  The intensity corresponding to the yellow emission at 573 nm displays a decrement post the optimal fuorescent composition of 3 mol% as shown in Fig. [10.](#page-9-0) This trend is attributed to the phenomenon of concentration quenching and has been explained in the following manner by an equation given by Huang: $38$ 

$$
\log\left(\frac{I}{x}\right) = -\frac{s}{d}\log\left(x\right) + \log\left(f\right) \tag{4}
$$

In Eq. [4,](#page-9-1) the ratio of the intensity of the emission peak and respective molar concentration of  $Dy^{3+}$  ions ( $x \ge 0.03$ , the optimal luminescent composition is 3 mol%) is represented by *I*/*x*; a constant independent of *x* is denoted by *f*, the dimensions of the sample under study are given by  $d = 3$ , whereas the multipolar interactions in action are represented by a constant *s*. The interactions are dependent on a value specific to *s*. The constant *s* offers a value of six, eight, and ten for dipole–dipole, dipole–quadrupole, and quadrupole–quadrupole interactions to be in action, respectively. Figure [11](#page-9-2) conveys a graph between log (*I*/*x*) and log (*x*) that results into a straight line with a value of − 2.11 for the slope. The multipolar interaction constant *s* is estimated to bear a value of 6.33 that is closer to the *s* value of six for dipole–dipole (d-d) interactions. Therefore, the transfer of energy plus concentration quenching taking place in  $Dy^{3+}$ -doped Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub> nanophosphor were found to be directed by dipole–dipole interactions rather than from exchange interactions, as witnessed in the reported in litera-ture on Eu<sup>3+</sup>-doped Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub>.<sup>[23](#page-12-19)</sup>

The Ba<sub>2</sub>Gd<sub>1−*x*</sub>Dy<sub>*x*</sub>V<sub>3</sub>O<sub>11</sub> ( $x = 0.01-0.09$ ) nanophosphor series after excitation undergoes radiative de-excitation



<span id="page-9-2"></span>**Fig. 11** Plot of log (*I/x*) as a function of log (*x*) in Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub>:Dy<sup>3+</sup> nanophosphor.



<span id="page-9-3"></span><span id="page-9-1"></span>**Fig. 12** The luminescence decay curve for 573-nm  $({}^{4}F_{9/2} \rightarrow {}^{6}H_{13/2})$ emission of  $Ba_2Gd_{1-x}Dy_xV_3O_{11}$  ( $x = 0.01–0.09$ ) nanophosphors.

from its  ${}^{4}F_{9/2}$  state. In order to analyze the radiative/relaxation process, the normalized PL intensity associated with the transition, namely,  ${}^{4}F_{9/2} \rightarrow {}^{6}H_{13/2}$ , at an excitation wavelength of 324 nm was plotted with respect to time (ms). These plots are also known as decay curves. The curves corresponding to the Ba<sub>2</sub>Gd<sub>1−*x*</sub>V<sub>3</sub>O<sub>11</sub>:  $xDy^{3+}$  ( $x =$ 0.01–0.09) nanophosphor series are displayed in Fig. [12](#page-9-3) along with time-correlated normalized PL intensity of  $Ba_2Gd_{0.97}Dy_{0.03}V_3O_{11}$  in the inset. The decay curves displayed by the prepared nanophosphor series ft quite well in the mono-exponential framework of decay function, as given below:

$$
I = I_0 \exp\left(-t/\tau\right) \tag{5}
$$

<span id="page-10-0"></span>**Table V** Photoluminescence lifetime, non-radiative rates, and quantum efficiencies of the <sup>4</sup>F<sub>9/2</sub> level in Ba<sub>2</sub>Gd<sub>1-*x*</sub>Dy<sub>*x*</sub>V<sub>3</sub>O<sub>11</sub> (*x* = 0.01– 0.09) nanophosphors

$Dy^{3+}$ concen- tration $(mol\%)$	Average lifetime (ms)	Non-radiative rates $(s^{-1})$	Ouantum efficiency $(\%)$
$\mathbf{1}$	$0.801 \pm 0.131$	349.158	72
$\mathcal{L}$	$0.763 + 0.087$	409.915	68
$\mathcal{F}$	$0.757 \pm 0.096$	421.178	67
4	$0.487 \pm 0.026$	1153.377	44
$\overline{5}$	$0.389 \pm 0.017$	1670.387	35
	$0.367 + 0.009$	1821.685	33
$\mathbf Q$	$0.356 \pm 0.010$	1907.746	32

In the aforementioned equation, the mono-exponential function consists of the PL intensity at time *t* denoted by *I*, the PL intensity at time  $\theta$  represented by  $I_0$ , and the average lifetime displayed by  $\tau$ . The values for average lifetime related to Ba<sub>2</sub>Gd<sub>1−*x*</sub>Dy<sub>*x*</sub>V<sub>3</sub>O<sub>11</sub> ( $x = 0.01-0.09$ ) nanophosphor series are summarized in Table [V](#page-10-0), and the values exhibited a decrease from  $0.801 \text{ ms } (1 \text{ mol\%)}$  to  $0.356 \text{ ms } (9 \text{ mol\%})$ . This decrement is attributed to non-radiative means of transfer of energy in the synthesized nanophosphor. The diferent compositions of  $Ba_2Gd_{1-x}Dy_xV_3O_{11}$  ( $x = 0.01-0.09$ ) nanophosphor indicate the mono-exponential decay conduct (Fig. [12\)](#page-9-3); therefore,  $Dy^{3+}$  ions inhabit a single type of emission center in  $Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub>$  host.

In the trivalent dysprosium-doped  $Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub>$  nanophosphor, the values of PL lifetime display a decreasing trend with a raise in concentration from  $x = 0.01$  to  $x = 0.09$ . This conduct was analyzed through Auzel's model via Eq. [6](#page-10-1):

$$
\tau(c) = \frac{\tau_R}{1 + \frac{c}{c_o} e^{-N/3}}\tag{6}
$$

In Eq. [6](#page-10-1), the average PL lifetimes correlated to diferent Dy<sup>3+</sup> proportions are displayed as  $τ(c)$ ;  $τ<sub>R</sub>$  is the intrinsic lifetime relative to the state that is emitting,  $c<sub>o</sub>$  is a constant, whereas *N* represents the overall number of phonons that have been generated. The average PL lifetime values were induced and ftted in Eq. [6,](#page-10-1) and the total number of phonons  $(N = 20.00 \pm 0)$  drawn out during relaxation of <sup>4</sup> $F_{9/2}$  state in a non-radiative way to ground state have been estimated. In Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub> host prototype, the obtained value of *N* is analogous to the energy gap in addition to the phonon energy.<sup>23</sup> The greater the number of phonons, the larger will be the rate of radiative decay in the synthesized nanophosphor. Additionally, a lifetime value (1.112 ms) intrinsic to the  ${}^{4}F_{9/2}$  state was attained (Fig. [13\)](#page-10-2).

The value attained for the intrinsic lifetime was utilized for the estimation of quantum efficiency  $(\phi)$  for the  ${}^{4}F_{9/2}$  state analogous to variously doped proportions of



<span id="page-10-2"></span>**Fig. 13** Dependence of photoluminescence lifetime of the  ${}^{4}F_{9/2}$  state on the doping concentration of  $Dy^{3+}$  through Auzel's model.

 $Ba_2Gd_{1-x}Dy_rV_3O_{11}$  ( $x = 0.01-0.09$ ) nanophosphor series. The quantum efficiency  $(\phi)$  can be calculated with the help of intrinsic lifetime value in the following manner $39$ :

<span id="page-10-3"></span>
$$
\phi = \frac{\tau}{\tau_R} = \frac{A_R}{A_R + A_{nR}}\tag{7}
$$

The intrinsic lifetime value can also be utilized to calculate the rates of relaxation specifcally by non-radiative means.<sup>[40](#page-13-6)</sup>

<span id="page-10-4"></span>
$$
\frac{1}{\tau} = \frac{1}{\tau_R} + A_{nR} \tag{8}
$$

<span id="page-10-1"></span>The above Eqs. [7](#page-10-3) and [8](#page-10-4) consist of the average lifetime value denoted as  $\tau$  and the intrinsic lifetime value represented as  $\tau_R$ ; the rate of relaxation particularly by non-radiative means is shown as  $A_{nR}$ , and by radiative means is represented by  $A_R$ . In the present case of  $Ba_2Gd_{1}Dy_rV_3O_{11}$  (*x* = 0.01–0.09) nanophosphors, the non-radiative rates (*s*−1) and quantum efficiency values  $(\%)$  for the emitting state are listed in Table [V.](#page-10-0)

The chromaticity parameters *x*, *y* have also been computed so as to analyze the color behavior of the synthesized nanophosphor series of  $Dy^{3+}$ -doped  $Ba_2Gd_{1-x}V_3O_{11}$  $(x = 0.01 - 0.09)$ . The MATLAB software package was used to process the emission spectra of diferent mol% compositions, and the respective color coordinates (*x*, *y*) have been obtained and listed in Table [III.](#page-8-0) The  $x (= 0.256)$ and *y* (= 0.341) coordinates of  $Ba_2Gd_{0.97}Dy_{0.03}V_3O_{11}$ nanophosphor are conveyed by means of the CIE 1931 chromaticity diagram in Fig. [14](#page-11-0) with the UV radiationtriggered color displayed by the optimal fuorescent composition in the inset. The *x*, *y* coordinates of other doped proportions are near the valued of  $Ba_2Gd_{0.97}Dy_{0.03}V_3O_{11}$ nanophosphor, and it emits white color that makes it suitable for UV-triggered WLEDs.



<span id="page-11-0"></span>**Fig. 14** CIE chromaticity diagram for  $Ba_2Gd_{0.97}Dy_{0.03}V_3O_{11}$  nanophosphor.

Furthermore, *u'*, *v'* coordinates and the CCT values were estimated for the optimal luminescent composition of  $0.03Dy^{3+}$ :Ba<sub>2</sub>Gd<sub>0.97</sub>V<sub>3</sub>O<sub>11</sub> by means of Eqs. [9,](#page-11-1) [10](#page-11-2) and [11,](#page-11-3) respectively.<sup>41</sup>

$$
u' = \frac{4x}{12y - 2x + 3} \tag{9}
$$

$$
v' = \frac{9y}{12y - 2x + 3} \tag{10}
$$

In abovementioned Eqs. [9](#page-11-1) and [10](#page-11-2), *x* and *y* signify the coordinates of  $Ba_2Gd_{0.97}Dy_{0.03}V_3O_{11}$  nanophosphor. The values for *u'* and *v'* come out to be 0.155 and 0.466, respectively.

$$
CCT = -437n^3 + 3601n^2 - 6861n + 5514.31\tag{11}
$$

In Eq. [11](#page-11-3), the ratio of  $(x - x_c)$  and  $(y - y_c)$  is represented by *n*; *x* and *y* are related CIE coordinates for  $Ba_2Gd_{0.97}Dy_{0.03}V_3O_{11}$ , whereas  $x_c$  (= 0.3320) and  $y_c$  (= 0.1858) stand for chromaticity epicenters. The CCT study of  $Ba_2Gd_{0.97}Dy_{0.03}V_3O_{11}$  has been conveyed through Fig. [15.](#page-11-4) The *u'*, *v'* and CCT values of more than 5000 K (indicating cool light) $42,43$  $42,43$  for the complete nanophosphor series  $Ba_2Gd_{1-x}Dy_xV_3O_{11}$  ( $x = 0.01-0.09$ ) are listed in Table [III](#page-8-0). The CCT was estimated to be 9751 K, turning it into a white light emitter for illumination intent.



<span id="page-11-4"></span>**Fig. 15** CCT study of  $Ba_2Gd_{0.97}Dy_{0.03}V_3O_{11}$  nanophosphor.

#### **Conclusions**

<span id="page-11-1"></span>Urea-fueled solution combustion was employed to synthesize mono-phased Dy<sup>3+</sup>-doped  $Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub>$  nanocrystalline series. The XRD patterns of the synthesized nanophosphors are in accordance to that of standard host. The Rietveld refnement indicates a crystal system (monoclinic) and space group symmetry (*P21/c*). The FE-SEM results ratify the synthesis of rod-shaped nanocrystals, whereas the absence of any impurity was confrmed by EDS analysis. The ultraviolet excitation at 324 nm leads to  ${}^{4}F_{9/2} \rightarrow {}^{6}H_{13/2, 15/2}$  transitions that are characteristic to dopant  $Dy^{3+}$  ions. The dipole–dipole (d-d) interactions were found to be accountable for the phenomena of concentration quenching post optimal luminescent composition. The intrinsic lifetime (1.112 ms) and non-radiative rates (421.178  $s^{-1}$ ) were attained by analyzing the monoexponential conduct of decay curves related to the prepared nanophosphor compositions. The CIE coordinates *x*, *y* (0.256, 0.341) along with correlated color temperature value (9751 K) support the candidature of  $Dy^{3+}$ -doped  $Ba<sub>2</sub>GdV<sub>3</sub>O<sub>11</sub>$  as a white light-emitting component having good quantum efficiency  $(67%)$  to be used in lighting devices.

<span id="page-11-3"></span><span id="page-11-2"></span>**Acknowledgment** One of the authors, Ms. Priya Phogat, gratefully recognizes the University Grants Commission (UGC), New Delhi, India, for granting support to the research work fnancially in the form of senior research fellowship (SRF, Award No. 128/ CSIR-UGC NET DEC 2017).

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