

STRUCTURAL AND OPTICAL MODIFICATIONS IN BOROTELLURITE GLASS BY DYSPROSIUM DOPING

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Abstract

This study investigates the luminescence and physical properties of dysprosium doped borotellurite glass with the composition: $(40-x)\text{TeO}_2 - 35\text{B}_2\text{O}_3 - 5\text{CaO} - 15\text{ZnO} - 5\text{Nb}_2\text{O}_5 - x\text{Dy}_2\text{O}_3$ (while x is 0, 0.1, 0.5, 1.0, 1.5 mol%). The glass samples were synthesized using a conventional melt-quenching technique. Characterization techniques, including X-ray diffraction (XRD), optical absorption, and photoluminescence spectroscopy, were employed to analyze the properties of the prepared glasses. XRD confirmed the amorphous nature of the samples. FTIR measurements revealed structural insights into the borotellurite network with the existence of TeO_4 , BO_4 , and BO_3 . Optical absorption spectra displayed characteristic absorption bands associated with Dy^{3+} ions. Under excitation with the light at 388 nm, the glasses exhibited intense yellow (576 nm) and blue (482 nm) emissions corresponding to the transitions: $^4\text{F}_{9/2} \rightarrow ^6\text{H}_{13/2}$ and $^4\text{F}_{9/2} \rightarrow ^6\text{H}_{15/2}$, respectively. The physical properties, such as density and refractive index were also determined. The results demonstrate the potential of Dy-doped borotellurite glasses as promising materials for optical applications, especially within the development of white light-emitting diodes.

Keywords: Borotellurite Glass; Dysprosium Doping; Luminescence; Optical Properties; White Light-Emitting Diodes

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Introduction

Borotellurite glasses have gained attention in the field of photonics due to their exceptional combination of physical and optical properties, making them ideal for applications such as optical fibers, amplifiers, lasers, and nonlinear optical devices. They, composed of boron oxide and tellurium oxide, are a unique class of materials with a wide range of potential applications. Their mixed structural units, high polarizability, and wide glass-forming region contribute to their desirable properties, including a high refractive index, low phonon energy, and good thermal stability. These properties make them highly attractive for use in optical devices, such as fibers, amplifiers, and lasers, as well as in nonlinear optical devices and radiation shielding.

Ongoing research and development in this area are focused on optimizing the composition and properties of borotellurite glasses to unlock their full potential in various fields. The versatility of these glasses has opened up opportunities for innovation in photonics, energy, medicine, and beyond. Each component of these glasses contributes uniquely to their desirable characteristics.

Tellurium oxide (TeO_2) is a key component due to its high refractive index and low phonon energy, properties crucial for efficient light confinement and minimal signal loss in optical fibers and amplifiers (El-Mallawany, 2002).

Boron oxide (B_2O_3) acts as the glass former, providing structural integrity and enhancing the glass's thermal and chemical stability, which are essential for device longevity (Shelby, 2005).

The addition of network modifiers, such as calcium oxide, zinc oxide, and niobium pentoxide, alters the structure of the glass by introducing non-bridging oxygens. This modification allows researchers to precisely control optical properties like refractive index and dispersion (Elkashef *et al.*, 2015).

Dysprosium (Dy), a rare-earth element, when introduced as Dy_2O_3 , imparts unique luminescent properties to the glass due to the creation of new energy levels within the bandgap. This luminescence is harnessed in various applications like lasers, sensors, and upconversion devices (Elkashef *et al.*, 2015).

Materials and Methods

Glasses with the molar composition $(40-x)\text{TeO}_2 - 35\text{B}_2\text{O}_3 - 5\text{CaO} - 15\text{ZnO} - 5\text{Nb}_2\text{O}_5 - x\text{Dy}_2\text{O}_3$ (where $x = 0.0, 0.1, 0.5, 1.0, 1.5$, and 2.0) were produced using a conventional melt-quenching approach.

The starting materials consisted of high-purity TeO_2 , H_3BO_3 , CaCO_3 , ZnO , Nb_2O_5 , and Dy_2O_3 .

After careful weighing, the raw materials were thoroughly mixed using a planetary ball mill for 30 minutes to ensure a homogeneous blend. This mixture was then placed in porcelain crucibles and inserted into a high-temperature electric furnace.

The furnace temperature was gradually raised to 1150°C at a rate of $5^\circ\text{C}/\text{min}$ and held at this temperature for 30 minutes to facilitate complete melting and mixing of the glass constituents.

After melting, the molten glass was poured onto a preheated graphite plate and then placed in a 350°C annealing furnace for 3 hours to reduce internal stress. The furnace was switched off, allowing the glass samples to gradually cool down to room temperature within the furnace to further minimize stress.

The resulting glass samples were polished to the dimension of $1.0\text{ cm} \times 1.5\text{ cm} \times 0.3\text{ cm}$ using grinding disc of varying grits to achieve optically clear surfaces suitable for subsequent characterization as shown in Figure 1.



Figure 1. The picture of a series of glass samples with varying concentrations of Dy_2O_3 , arranged from left to right in order of increasing concentration (0.0, 0.1, 0.5, 1.0, and 1.5 mol%)

The specific gravity of the refined glass specimens was ascertained by employing Archimedes' principle, with purified water serving as the submersion fluid. Subsequently, the molar volume was computed using this acquired density value and the mean molecular mass (Barde and Waghuley, 2013).

Using a Presidium Refractive Index Meter, the refractive index of the polished samples was determined at room temperature across various wavelengths within the visible and near-infrared spectrum to assess dispersion properties.

The non-crystalline arrangement of the synthesized glass specimens was confirmed through X-ray diffraction (XRD) analysis of pulverized samples. The XRD examinations were conducted utilizing a copper-targeted X-ray tube functioning at

40.0 kV and 30.0 mA, scanning a 2θ range from 10 to 80 degrees.

Fourier-transform infrared (FTIR) spectroscopy was employed to scrutinize the vibrational modes and functional groups present within the glass matrix. FTIR spectra were obtained in the mid-infrared region ($4000\text{--}400\text{ cm}^{-1}$) using an attenuated total reflectance (ATR) accessory.

The fluorescence decay kinetics of Dy^{3+} ions were studied using time-correlated single-photon counting (TCSPC) under pulsed laser excitation. The emission decay curves were analyzed using multi-exponential decay models to extract the fluorescence lifetime within 1000 ms.

UV-Vis-NIR spectrophotometry was utilized to record the optical absorption spectra of the glass samples between 200–1800 nm. The excitation and emission spectra were obtained using a spectrofluorometer with a xenon lamp as the excitation source between 300–500 nm and 400–700 nm respectively. These measurements provided insights into the absorption and luminescence behavior of Dy^{3+} ions within the glass host. The luminescence data can then be used to calculate the CIE chromaticity coordinates and correlated color temperature (CCT) of the light emitted from the samples.

Physical and refractive properties

With the rising concentration of Dy_2O_3 , the specific gravity of the glass generally exhibits an upward trend. This is anticipated since Dy_2O_3 possesses a higher density compared to the fundamental glass composition. However, The result showed, in the Figure 2, that it fluctuates and tends to increase after 1.0 mol%. This fluctuation may be caused by small structural changes influenced how tightly packed the atoms are, or inhomogeneities. Conversely, the molar volume of a lanthanide-doped glass, in most cases, shows a downward trend as the Dy_2O_3 concentration increases. This is an inverse correlation to density, as a denser substance will occupy a smaller volume per mole. However, the result for the samples in this research showed that it provides the upward trend.

The refractive index, however, demonstrates a more complex behavior. It initially ascends with the addition of Dy_2O_3 , reaching a maximum around 0.2 mol%, then gradually descends with further increments in Dy_2O_3 concentration.

The observed trends can be attributed to the structural changes that occur when Dy_2O_3 is incorporated into the glass network. Dysprosium ions (Dy^{3+}) are heavier than the ions typically present in the base glass. When these larger ions are

introduced, they disrupt the glass structure and occupy more space, leading to the observed increase in density and decrease in molar volume (Barde and Waghuley, 2013).

The initial increase in refractive index is likely due to the higher polarizability of the Dy^{3+} ions compared to the base glass ions. This means that the Dy^{3+} ions can more easily respond to an electric field (such as light), leading to a higher refractive index. However, as the concentration of Dy^{3+} ions increases further, the disruption of the glass network becomes more significant. This disruption can lead to a decrease in the overall polarizability of the glass, resulting in the observed decrease in the refractive index at higher concentrations (Abdel-Baki and El-Diasty, 2011).

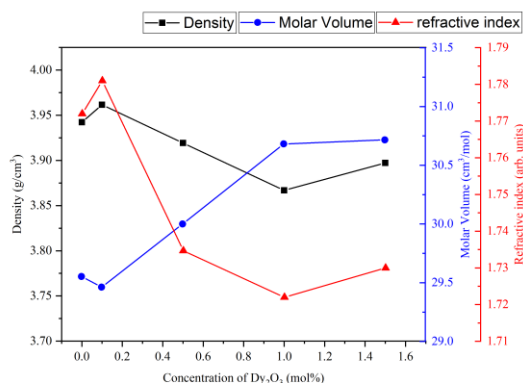


Figure 2. Shows the relationship between the concentration of Dy_2O_3 (dysprosium oxide) in a glass material and its resulting density, molar volume, and refractive index

The XRD patterns exhibit broad peaks, which is characteristic of amorphous materials like glass. This confirms that the samples are indeed glasses and not crystalline structures. The absence of sharp peaks indicates that there are no well-defined, long-range ordered crystal structures within the glass samples.

As the concentration of Dy_2O_3 increases, there are subtle changes in the intensity of the broad peak. This suggests that the addition of Dy_2O_3 is causing slight modifications to the short-range order within the glass network. (Singh *et al.*, 2021).

The XRD data in the Figure 3 indicates that the addition of Dy_2O_3 does not induce crystallization in the glass samples. The glass remains amorphous, even at the highest concentration of Dy_2O_3 . The slight changes in the intensity of the broad peak suggest that the Dy_2O_3 is being incorporated into the glass network, potentially modifying the local environment of the atoms and affecting the short-range order. (Kumar *et al.*, 2017).

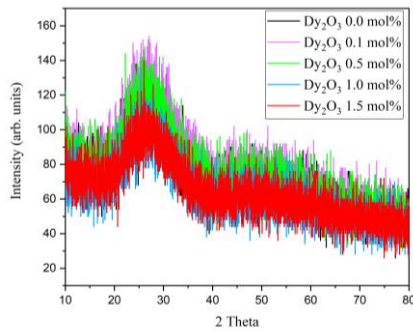


Figure 3. Shows the X-ray diffraction (XRD) patterns of glass samples with different concentrations of dysprosium oxide (Dy_2O_3)

FTIR Spectra

As seen in Figure 4, the FTIR (Fourier-transform infrared) spectra of borotellurite glass samples doped with varying concentrations of Dy_2O_3 (0.0, 0.1, 0.5, 1.0, and 1.5 mol%) reveal insights into their molecular structure and the impact of dysprosium doping.

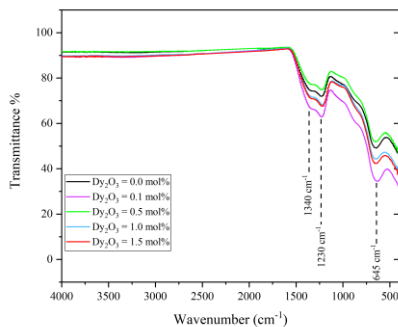


Figure 4. FTIR Spectra of Dy_2O_3 -Doped Borotellurite Glass Samples

Bands around 1230-1340 cm^{-1} : These overlapping bands are associated with B-O stretching vibrations in the borate (B_2O_3) network. The slight fluctuation in FTIR-transmittance with varying Dy_2O_3 concentration suggests that dysprosium may be disrupting or modifying the borate network structure. (Shaaban *et al.*, 2008; Rada *et al.*, 2010; Fudzi *et al.*, 2017).

Band around 645 cm^{-1} : This band could be attributed to Te-O vibrations in the tellurite (TeO_2) network. The slight shift and change in intensity of this band with varying Dy_2O_3 concentrations may indicate that the dysprosium ions are also interacting

with and influencing the tellurite network. (Fudzi *et al.*, 2017).

The FTIR spectra suggest that the addition of Dy_2O_3 to the borotellurite glass affects both the borate and tellurite networks. The dysprosium ions appear to interact with and modify these networks, as indicated by the changes in the vibrational bands associated with B-O and Te-O bonds. The presence of hydroxyl impurities is consistent across the samples, implying that it is likely a result of the glass preparation process rather than the Dy_2O_3 doping.

Table 1. Assignment of infrared transmission bands of glass samples with different molar fraction of Dy_2O_3

Wave no.	Assignments
645	The TeO_4 structural unit is invariably present within all vitreous materials containing tellurium oxide. (Fudzi <i>et al.</i> , 2017).
1230	Non-symmetrical stretching vibration of the B-O bond within planar BO_3 units. (Shaaban <i>et al.</i> , 2008; Rada <i>et al.</i> , 2010; Fudzi <i>et al.</i> , 2017).
1340	Balanced stretching vibration of the B-O bond within planar BO_3 groups. (Shaaban <i>et al.</i> , 2008; Rada <i>et al.</i> , 2010; Fudzi <i>et al.</i> , 2017).

The Absorbance Spectra

The absorbance spectra in the Figure 5 show the absorption of light by dysprosium (Dy^{3+}) ions in a glass matrix at different concentrations of Dy_2O_3 (0.0, 0.1, 0.5, 1.0, and 1.5 mol%). Each curve represents the absorbance of light at different wavelengths for a specific Dy_2O_3 concentration.

Several distinct absorption peaks are observed in the spectra, each corresponding to a specific electronic transition within the Dy^{3+} ion. The wavelengths of these peaks are labeled on the graph: 449 nm, 807 nm, 907 nm, 1098 nm, 1281 nm, and 1690 nm analogous to the transitions from the state $^6\text{H}_{5/2}$ to $^4\text{I}_{5/2}$, $^6\text{F}_{5/2}$, $^6\text{F}_{7/2}$, $^6\text{F}_{9/2}$, $^6\text{F}_{11/2}$, and $^6\text{H}_{11/2}$ respectively. The absorbance of the absorption peaks increases with the increasing concentration of Dy_2O_3 . This indicates that a higher concentration of Dy^{3+} ions in the glass leads to a greater absorption of light at those specific wavelengths. The position of the absorption peaks remains relatively constant regardless of the Dy_2O_3 concentration. This suggests that the electronic transitions responsible for the absorption peaks are not significantly affected by the concentration of Dy^{3+} ions.

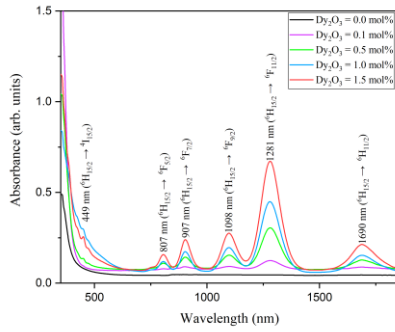


Figure 5. The absorption of light by dysprosium (Dy³⁺) ions in a glass matrix at different concentrations of Dy₂O₃ (0.0, 0.1, 0.5, 1.0, and 1.5 mol%)

Emission spectra

The luminescence spectra illustrated in Figure 6 reveal the light emission of dysprosium (Dy³⁺) ions embedded within a glass matrix at different concentrations of Dy₂O₃ (0.1, 0.5, 1.0, and 1.5 mol%). These spectra were generated under excitation at a wavelength of 388 nm.

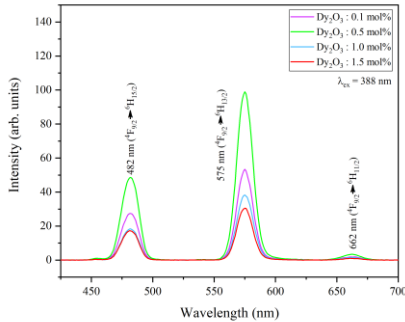


Figure 6. The emission spectra of dysprosium (Dy³⁺) ions in a glass matrix at varying concentrations of Dy₂O₃ (0.1, 0.5, 1.0, and 1.5 mol%)

Three distinct emission bands are evident at roughly 482 nm, 575 nm, and 662 nm. These bands signify characteristic electronic transitions of Dy³⁺ ions as they relax from the excited state ⁴F_{9/2} to the ground states ⁶H_{15/2}, ⁶H_{13/2}, and ⁶H_{11/2}, respectively.

The intensity of the emission peaks increases with increasing Dy₂O₃ concentration from 0.1 mol% to 0.5 mol%. However, at 1.0 mol%, the intensity decreases, indicating concentration quenching. This phenomenon occurs when the high concentration of Dy³⁺ ions leads to increased nonradiative energy transfer between the ions, reducing the overall luminescence. For additional

insights, it is directly influenced by the Dy-Dy inter-ionic distance. This distance determines the probability of non-radiative energy transfer between neighbouring Dy³⁺ ions, such as resonant energy transfer or cross-relaxation. When the inter-ionic distance is small, these non-radiative processes become more likely, leading to a decrease in luminescence intensity as the excitation energy is dissipated as heat. The distance depends on critical concentration (N_c) for each sample which can be calculated from the equation (Auzel *et al.*, 2003):

$$N_c = \frac{\text{mol}\%}{100} \frac{\rho}{M} (2N_A) \quad (1)$$

where: mol% is the mole percent of Dy₂O₃. ρ is the density of the glass in g/cm³. M is the molar mass of Dy₂O₃ (373.0 g/mol). N_A is Avogadro's number (6.022 × 10²³ ions/mol).

The critical distance (R_c) or the average Dy-Dy inter-ionic distance for each sample was then calculated using the relation:

$$R_c = \left[\frac{3}{4\pi N_c} \right]^{\frac{1}{3}} \quad (2)$$

where: R_c is the critical distance (or average Dy-Dy inter-ionic distance) in nanometers (nm). N_c is the critical concentration of Dy ions in ions per cubic centimeter (ions/cm³).

Both calculated values are shown in Table 2. The drop in emission for the 1.0 mol% doped sample indicates that the inter-ionic distance of 1.24 nm is significantly small, leading to the concentration quenching effect.

Table 2. The critical concentration and the critical distance of glass samples with different molar fractions of Dy₂O₃

mol% of Dy ₂ O ₃	Density g/cm ³	N _c ions/cm ³	R _c nm
0.1	3.96167	1.28 × 10 ¹⁹	2.65
0.5	3.91925	6.33 × 10 ¹⁹	1.56
1.0	3.86690	1.25 × 10 ²⁰	1.24
1.5	3.89729	1.89 × 10 ²⁰	1.08

The peak at 575 nm (⁴F_{9/2} → ⁶H_{13/2}) is the most intense for all concentrations, suggesting this transition is the most favorable radiative relaxation pathway for Dy³⁺ ions in this glass matrix.

The emission spectra demonstrate the luminescence properties of Dy³⁺ ions in the glass host. The different peaks reveal the various energy levels and transitions involved in the emission process. The concentration dependence of the peak intensity highlights the importance of optimizing the

Dy₂O₃ concentration to achieve maximum luminescence efficiency.

From the Table 3 (Ichoja *et al.*, 2018), the scenario for electrons can be illustrated. First some of them were excited from the ground state $^6H_{15/2}$ to the excited states $^4I_{13/2} + ^4F_{7/2}$ by the light of wavelength 388 nm. After their lifetime passed by, they eventually relaxed to the lower energy state $^4F_{9/2}$ with a non-radiative process. From that state they went on to the lower energy states: $^6H_{15/2}$, $^6H_{13/2}$, and $^6H_{11/2}$ by emitting the light of wavelength 482 nm, 575 nm, and 662 nm, respectively.

Table 3. Observed transitions in Dy³⁺ doped glass samples

Transition	Wavelength (nm)
$^6H_{15/2} \rightarrow ^4I_{13/2} + ^4F_{7/2}$	388 (excited)
$^4F_{7/2} \rightarrow ^4G_{11/2}$	Non-radiative
$^4G_{11/2} \rightarrow ^4F_{9/2}$	Non-radiative
$^4I_{13/2} \rightarrow ^4F_{9/2}$	Non-radiative
$^4F_{9/2} \rightarrow ^6H_{15/2}$	482 (emitted)
$^4F_{9/2} \rightarrow ^6H_{13/2}$	575 (emitted)
$^4F_{9/2} \rightarrow ^6H_{11/2}$	662 (emitted)

Excitation spectra

The excitation spectra presented in the Figure 7 depict the luminescence intensity of Dy³⁺ ions in borotellurite glass samples as a function of the excitation wavelength. In essence, it shows how efficiently different wavelengths of light can excite the Dy³⁺ ions to higher energy states, from which they subsequently emit light (luminescence) at a fixed emission wavelength ($\lambda_{em} = 575$ nm).

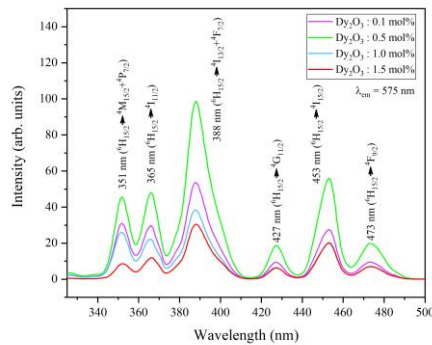


Figure 7. The excitation spectra of dysprosium (Dy³⁺) ions in a glass matrix at varying concentrations of Dy₂O₃ (0.1, 0.5, 1.0, and 1.5 mol%)

The spectra display several distinct peaks, each representing a specific excitation wavelength that effectively promotes Dy³⁺ ions to different excited states. The peaks are labeled with the corresponding electronic transitions within the Dy³⁺ ion.

The variations in peak intensities suggest that some excitation wavelengths are more effective than others in stimulating Dy³⁺ ions to specific excited states. The most intense peak is located around 388 nm, corresponding to the $^6H_{15/2} \rightarrow ^4I_{13/2} + ^4F_{7/2}$ transition.

The intensity of the excitation peaks increases with increasing Dy₂O₃ concentration from 0.1 mol% to 0.5 mol%. However, from 1.0 mol% to 1.5 mol%, the intensity tends to decrease,

Fluorescence decay curves

The graph displays the fluorescence decay curves for dysprosium (Dy³⁺)-doped materials with varying concentrations of Dy₂O₃ (0.1, 0.5, 1.0, and 1.5 mol%). The y-axis shows the logarithm of the normalized intensity, and the x-axis represents time (in milliseconds).

Each curve exhibits an exponential decay behavior, indicating that the fluorescence intensity decreases over time following an exponential function. This is typical for spontaneous emission processes.

The fluorescence lifetime (τ) is the time it takes for the intensity to decay to 1/e (about 37%) of its initial value. The lifetimes are provided in the legend for each concentration:

1. 0.1 mol% Dy₂O₃: $\tau = 0.424$ ms
2. 0.5 mol% Dy₂O₃: $\tau = 0.363$ ms
3. 1.0 mol% Dy₂O₃: $\tau = 0.278$ ms
4. 1.5 mol% Dy₂O₃: $\tau = 0.233$ ms

The fluorescence lifetime as shown in the Figure 8 decreases with increasing Dy₂O₃ concentration. This phenomenon is known as concentration quenching, where interactions between Dy³⁺ ions at higher concentrations lead to non-radiative decay pathways, reducing the overall fluorescence lifetime.

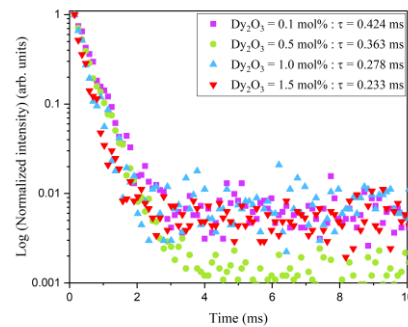


Figure 8. The fluorescence decay curves for dysprosium (Dy³⁺)-doped materials with varying concentrations of Dy₂O₃ (0.1, 0.5, 1.0, and 1.5 mol%)

CIE chromaticity diagram and correlated color temperature

The CIE chromaticity diagram is a two-dimensional representation of color, with each point on the diagram representing a unique color. The x and y coordinates of a point on the diagram indicate the relative amounts of red and green light in the color. The luminance data were used to calculate the coordinates which appeared to be $x = 0.3734$, $y = 0.4070$ falling into the white-light region in the diagram as shown in Figure 9.

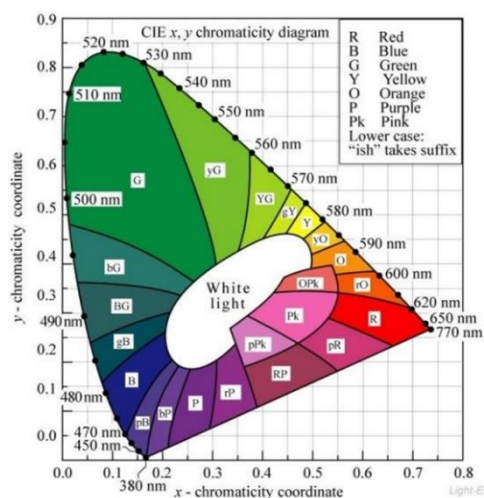


Figure 9. CIE chromaticity diagram for curves for dysprosium (Dy^{3+})-doped materials

The correlated color temperature (CCT) of a light source is a measurement of its color appearance, with higher CCT values indicating a cooler (bluer) appearance and lower CCT values indicating a warmer (redder) appearance. From the calculation, the CCT value is 4363.474 K corresponding to bright yellow color (Marzouk *et al.*, 2017).

Conclusions

In conclusion, this research successfully prepared and characterized Dy-doped borotellurite glasses using the melt-quenching technique. The structural analysis revealed the amorphous nature of the glasses and the influence of Dy_2O_3 on the borate and tellurite networks. The optical properties, including density, refractive index, absorption, and emission spectra, were investigated, demonstrating the potential of these glasses for photonic applications. The observed concentration quenching highlights the importance of optimizing Dy_2O_3 concentration for maximum luminescence efficiency. These

findings contribute to the understanding of rare-earth-doped borotellurite glasses and their potential for various optical and photonic devices, particularly in the development of white light-emitting diodes.

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