

INVESTIGATION OF HOLMIUM-DOPED PHOSPHO-TELLURITE GLASS SYSTEMS FOR LASERS

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Abstract

This study explores the properties of Holmium-doped phospho-tellurite glass, with varying concentrations of Ho_2O_3 , measuring the physical, structural, optical, and luminescence characteristics of the glass. Physical properties including density, molar volume, and refractive index are measured and analyzed. XRD is used to confirm the amorphous nature of the glass for structural properties, while FTIR is used to confirm bonding in the glass system. Absorption and luminescence are measured within the wavelength range of 200 - 2000 nm and 400 - 750 nm, respectively. The obtained spectra were analyzed using the Judd-Ofelt theory, which provides insights into the energy levels of Ho^{3+} ion doped in the phospho-tellurite glass matrix. Notably, our analysis of the spectral line corresponding to the $^5\text{F}_4 \rightarrow ^5\text{I}_8$ transition (~ 529.0 nm), showcases a highly stimulated emission cross-section. The calculated oscillator strength, Judd-Ofelt intensity parameters, branching ratio, and other parameters further elucidate the potential of Ho^{3+} ion doped phospho-tellurite glass for efficient laser emission in this glass system.

Keywords: Holmium; Judd-Ofelt; Luminescence; Phospho-Tellurite Glasses

Introduction

Rare-earth-doped glasses have garnered significant attention due to their versatile applications in solid-state lasers, sensors, and fiber optic amplifiers. Among these, holmium-doped glasses stand out for their unique luminescence properties, arising from f-f electronic transitions, which make them promising candidates for photonic applications. The choice of host material plays a critical role in tailoring the optical properties of rare-earth ions, with tellurite and phosphate glasses (TeO_2 and P_2O_5)

being particularly attractive. Their excellent chemical stability, low phonon energy, and ability to form robust glass matrices make them ideal for supporting the incorporation of rare-earth dopants (Lesniak *et al.*, 2019). Sb_2O_3 plays a role in functioning as a network modifier. It reduces the moisture content within the glass structure (Zhang *et al.*, 2008), which helps to overcome the disadvantages of phosphate. Similarly, ZnO enhances the glass's formability and hinders its

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crystallization tendency (Rammah *et al.*, 2020). Doping these glasses with rare earth ions, such as the Ho^{3+} ion, introduces unique luminescence properties due to the presence of f-f electronic transitions within the rare earth ion. This study systematically investigates the influence of Ho_2O_3 concentration on the physical (density, molar volume, and refractive index), structural (amorphous nature confirmation using XRD, FTIR used to find bonding in the glass system), optical properties (absorption), and luminescence properties (excitation, emission, and CIE) of the prepared phospho-tellurite glasses. The Judd-Ofelt theory will be employed to analyze the obtained luminescence spectra, revealing insights into the energy level structure of the Ho^{3+} ion within the glass matrix. Particular focus will be placed on the analysis of the spectral line corresponding to the $^5\text{F}_4 \rightarrow ^5\text{I}_8$ transition (~ 529.0 nm), known for its potential for stimulated emission. The calculated oscillator strengths, Judd-Ofelt intensity parameters, branching ratios, and other relevant parameters will be presented to assess the potential of these Ho^{3+} ion doped phospho-tellurite glasses for achieving efficient laser emission.

Experiment

1. Material preparation

This study explores a new series of holmium-doped tellurite glasses with the composition $(40-x)\text{TeO}_2-30\text{P}_2\text{O}_5-20\text{ZnO}-10\text{Sb}_2\text{O}_3-x\text{Ho}_2\text{O}_3$ (mol%), where x varies by 0.0, 0.1, 0.5, 1.0, and 1.5 (TPZSHx). The preparation process involved weighing all the constituent oxides (TeO_2 , P_2O_5 , ZnO , Sb_2O_3 , and Ho_2O_3) to achieve a total batch weight of 10 grams. These oxides were then thoroughly mixed within a crucible and subsequently melted in an electric furnace at 1100°C with a heating rate of $4.583^\circ\text{C}/\text{min}$ (not holding time). The melt was then poured into a preheated graphite mold and annealed at 300°C for 3 hours. The solidified glasses were cut and polished to attain uniform dimensions of $1.0\text{ cm} \times 1.5\text{ cm} \times 0.3\text{ cm}$ for further characterization, which is shown in Figure 1.

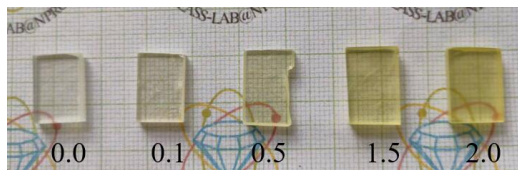


Figure 1. Sample of TPZSHx glasses

2. Measurement

The samples were prepared and Figure 2 shows the schematic characterization that was done for this task. A densitometer (model AND HR-200) was used to weigh the specimens to quantify the samples' density (ρ). An Abbe Refractometer was used to calculate the refractive index (n). The properties of the materials were ascertained by X-ray diffraction (XRD) analysis using a Rigaku SmartLab X-ray Diffractometer. The present generation of phospho-tellurite glasses was subjected to vibrational state analysis using a NicoletTM iSTM5 FTIR spectrophotometer. The UV-Vis-NIR absorption spectra were obtained using a UV-3600 Shimadzu UV-VIS-NIR spectrophotometer. Additionally, the excitation and emission were measured using the Agilent Cary Eclipse fluorescence spectrometer.

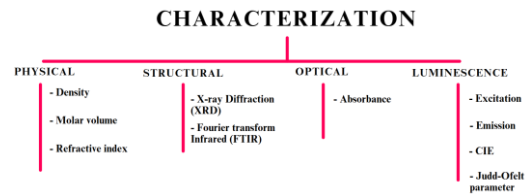


Figure 2. The schematic diagram of the characterization

Results and Discussion

1. Physical properties

Figure 3 provides details on the sample's density, molar volume, and refractive index. In TPZSHx glass, increasing the concentration of Ho_2O_3 (0.0, 0.1, 0.5, 1.0, and 1.5 mol%) increases these physical properties. Specifically, the density and refractive index rise from 4.1456 ± 0.0008 to $4.1603 \pm 0.0012\text{ g/cm}^3$ and 1.6453 ± 0.0002 to 1.6510 ± 0.0003 , respectively. This rise suggests a more compact glass matrix due to the substitution of lower mass TeO_2 with the higher molar mass Ho_2O_3 . As the content of holmium oxide increases, the density values suggest a denser matrix within the three-dimensional glass structure, forming non-bridging oxygens by breaking the bridging oxygen bonds (Mahmoud *et al.*, 2021). Additionally, the molar volume of the glasses shows a steady increase from $36.63 \pm 0.01\text{ cm}^3/\text{mol}$ for the TPZSH0.0 sample to $37.29 \pm 0.01\text{ cm}^3/\text{mol}$ for the TPZSH1.5 sample. This increase in molar volume is likely due to the incorporation of Ho^{3+} ion, which have an atomic radius ($2.47\text{ \AA}$) larger than that of Te ($1.42\text{ \AA}$) (Alsaif *et al.*, 2022).

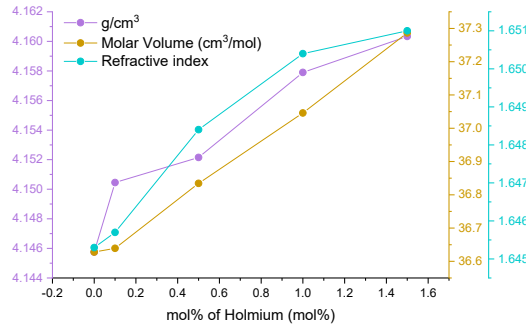


Figure 3. The physical properties of TPZSH glass

2. Structural properties

2.1 X-ray Diffraction (XRD) Pattern: X-ray diffraction (XRD) analysis was performed on all fabricated TPZSH glass samples to investigate their structural characteristics and confirm the material's amorphous nature. The XRD patterns, presented in Figure 4, exhibit a broad hump between 20° and 30° (2θ), characteristic of amorphous materials. This absence of sharp crystalline peaks further validates the amorphous nature of the glasses. (Yasaka *et al.*, 2016)

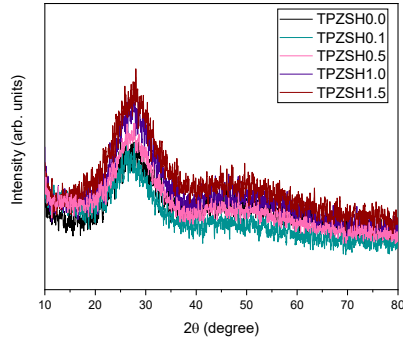


Figure 4. X-ray diffraction (XRD) pattern of TPZSH glasses

2.2 Fourier transform Infrared (FTIR):

Fourier transform infrared (FTIR) spectroscopy was used to examine the structural properties of the sample, focusing on its functional groups. Measurements were recorded over the range of $400\text{--}3500\text{ cm}^{-1}$, as illustrated in Figure 5. The detected bands and their corresponding vibrational modes are listed in Table 1. Tellurite exhibits a single structural configuration, consisting of trigonal bipyramidal units (TeO_4), with stretching modes of TeO_4 trigonal bipyramids featuring bridging oxygen appearing at 623 cm^{-1} (Satya Gopal Rao *et al.*, 2021). Phosphate, on the other hand, consists of various structural configurations. The first band at 501 cm^{-1}

corresponds to the vibration mode of P-O, while the other band around 901 cm^{-1} is attributed to the symmetric vibration of $(\text{PO}_4)^{3-}$ (Elkhoshkhany *et al.*, 2019). The inclusion of P_2O_5 poses a drawback for the glass, as phosphate leads to moisture absorption by the glass, resulting in peaks around 1634 cm^{-1} and in the $2000\text{--}3500\text{ cm}^{-1}$ range. All of the peaks are not consistent with the addition of holmium to the glass as in this literature (Dahiru *et al.*, 2024).

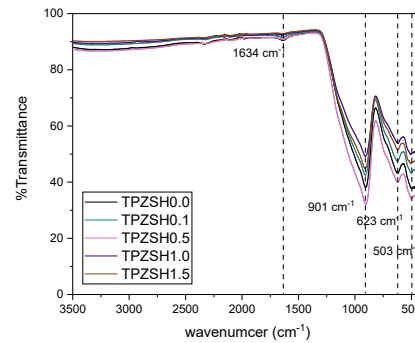


Figure 5. FTIR transmittance spectra of TPZSH glass

Table 1. Assignments of FTIR

Band position	Assignments	Reference
503 cm^{-1}	vibration mode of P-O	(Elkhoshkhany <i>et al.</i> , 2019)
623 cm^{-1}	stretching vibrations of Te-O bonds in TeO_4 (tbp) units	(Satya Gopal Rao <i>et al.</i> , 2021)
901 cm^{-1}	symmetric vibration of $(\text{PO}_4)^{3-}$	(Elkhoshkhany <i>et al.</i> , 2019)
$1634, 2000\text{--}3500\text{ cm}^{-1}$	OH group	(Selvi <i>et al.</i> , 2017)

3. Optical properties

3.1 Absorbance spectra: In the $200\text{--}2,000\text{ nm}$ region, Figure 6 illustrates the absorbance spectra of TPZSH glasses. With an increase in Ho_2O_3 concentration, a slight enhancement in absorbance is observed. The absorbance spectra feature 8 peaks at distinct transition states (from the $^5\text{I}_8$ to high states), as depicted in Figure 6: wavelengths of 418 nm ($^5\text{G}_5, ^3\text{G}_5$), 452 nm ($^5\text{G}_6$), 484 nm ($^5\text{F}_3$), 538 nm ($^5\text{F}_4$), 643 nm ($^5\text{F}_5$), 897 nm ($^5\text{I}_5$), 1168 nm ($^5\text{I}_6$) and 1955 nm ($^5\text{I}_7$). As shown in Figure 6, the absorbance positions remained unchanged while their intensity increased approximately linearly with the increase in Ho^{3+} ion concentration. This phenomenon is attributed to the shielding effect of the outermost electrons on the 4f electrons of the rare earth (RE) ions. Notably, the $^5\text{I}_8 \rightarrow ^5\text{G}_6$ transition, occurring around 452 nm , is

identified as a hypersensitive transition. This transition follows the selection rules $|\Delta S| = 0$, $|\Delta L| \leq 2$, and $|\Delta J| \leq 2$, which make it highly sensitive to the local environment of the Ho^{3+} ion (Rani *et al.*, 2020).

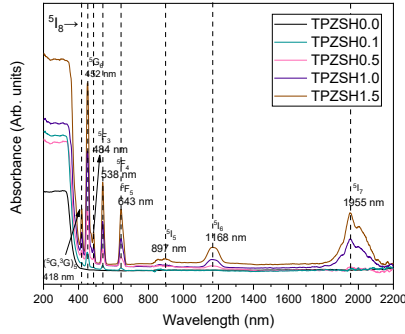


Figure 6. The absorbance spectra of TPZSH glass

4. Luminescence properties

4.1 Excitation and emission spectra: The excitation spectra of TPZSH glasses, shown in Figure 7, were recorded within the 300-500 nm wavelength range while monitoring an emission wavelength of 529 nm. The spectra display typical bands at approximately 322, 335, 352, 370, 387, 415, 435, 455, and 478 nm, corresponding to the $^5\text{I}_8 \rightarrow ^5\text{G}_2$, $^3\text{K}_6$, $(^5\text{F}, ^3\text{F}, ^5\text{G})_2$, $^3\text{K}_7$, $^5\text{G}_4$, $(^5\text{G}, ^3\text{G})_4$, $^5\text{F}_1$, $^5\text{G}_6$, and $^5\text{F}_3$ transitions, respectively (Carnall *et al.*, 1968). Among these transitions, the $^5\text{I}_8 \rightarrow ^5\text{G}_4$ transition at 387 nm is the most prominent and is used as the excitation wavelength for recording the emission spectra. Figure 8 presents the emission spectra of the glasses recorded at room temperature in the visible regions, upon excitation at 387 nm. The spectra reveal a single emission band of Ho^{3+} ions in the visible region at around 529 nm, which corresponds to the green emission due to the $^5\text{F}_4 \rightarrow ^5\text{I}_8$ transition (Babu & Ratnakaram, 2016), which excitation and emission spectra are described in the diagram in Figure 9. The peak intensity at 529 nm increases from TPZSH0.1 to TPZSH1.0 and then decreases for TPZSH1.5, indicating a concentration quenching effect. After a 1.0 mol % decrease, the interaction between the host glass matrix and Ho^{3+} ion strengthens. As the concentration of Ho_2O_3 increases, the interion distance between Ho^{3+} ions decreases, resulting in the fluorescence being extinguished rather than incorporated into the phospho-tellurite network. At low concentrations (approximately 1.0 mol%), the fluorescence intensity increases due to the interaction of the Ho^{3+} ion with the nonbridging oxygen (NBO) group. However, at higher concentrations (1.5 mol%), the

number of NBOs gradually decreased with increasing Ho^{3+} ions because the connection between phospho-tellurite groups and oxygen through holmium ions decreased. This reduction in interconnecting bonds results in increased coordination of bridging oxygen with Ho_2O_3 (Rani *et al.*, 2020).

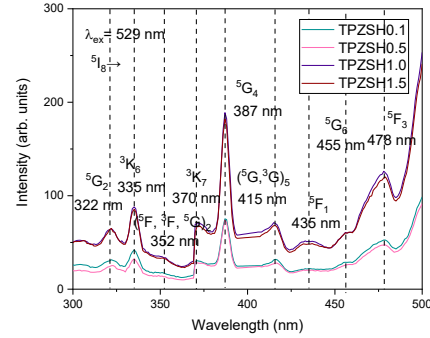


Figure 7. The excitation spectra of TPZSH glass

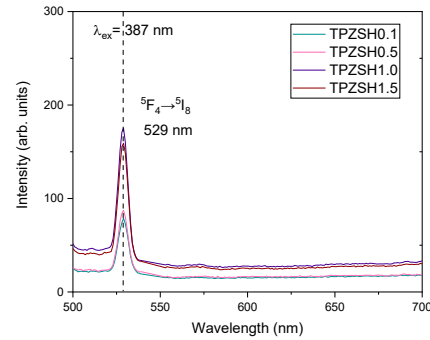


Figure 8. The emission spectra of TPZSH glass

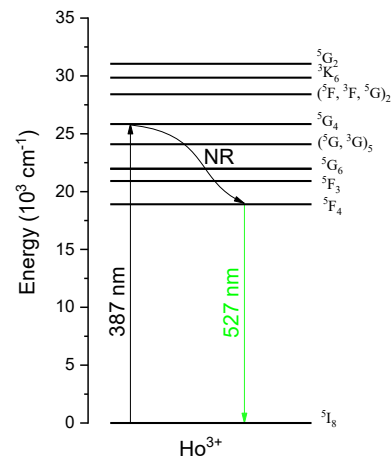


Figure 9. The energy level diagram of TPZSH glass

4.2 CIE 1931 chromaticity: Colorimetry involves the quantitative measurement of color. In the tricolor color system color is measured using tristimulus values or mathematical functions derived from these values. The color coordinates for the TPZSH1.0 glass were determined from the fluorescence spectrum under 387 nm excitation using a CIE 1931 system, with the results shown in Figure 10. The CIE 1931 coordinates for TPZSH1.0 glass fall within the green region, corresponding to the $^5F_4 \rightarrow ^5I_8$ transition at the 529 nm emission wavelength. This suggests that the glass materials investigated in this study are well-suited for creating compact, high-quality green solid-state light sources, with promising applications in laser technology.

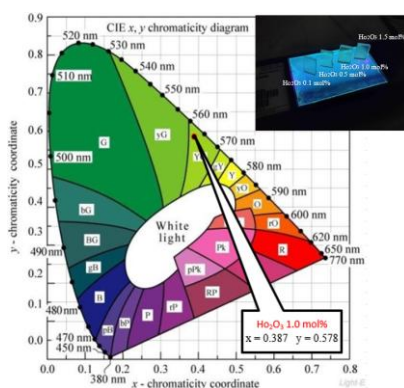


Figure 10. The CIE 1931 diagram of TPZSH1.0 glass

3 Judd Ofelt (JO) parameter: The experimental and calculated oscillator strength values (f_{exp} , f_{cal}) for TPZSH1.0 glass were determined using relevant expressions from the literature (Yamsuk *et al.*, 2020) with individual values listed in Table 2. The oscillator strengths for the hypersensitive $^5I_8 \rightarrow ^5G_6$ transition were higher

compared to other transitions listed in Table 2. A small difference between the experimental and calculated oscillator strengths was observed. The root mean square deviation ($\delta_{\text{rms}} = 0.312$) was used to gauge the overall quality of fit between the estimated and experimental oscillator strengths. Furthermore, by computing the Judd-Ofelt intensity parameters Ω_x ($x = 2, 4, 6$), it is possible to assess the characteristics of the local structure, site symmetry, and bonding between RE ions and the surrounding ligands. These parameters, along with their reported values, are shown in Table 3 and were calculated using a least squares fitting analysis of the experimental spectral intensities (f_{exp}) with doubly squared reduced matrix elements, as described in Ref. (Ofelt, 1962). The Ω_2 parameter is particularly sensitive to the local environment of RE ions, often providing insights into the bonding type, polarizability of ligand ions, and asymmetry of the coordination structure. Deviations from inversion symmetry contribute to Ω_2 because $f \rightarrow f$ transitions of RE ions are parity-forbidden (Rani *et al.*, 2020). Table 3 illustrates the relationship between the parameters Ω_4 and Ω_6 with the viscosity and rigidity of the host matrices, respectively. Moreover, higher values of Ω_2 suggest an increased level of covalency between Ho^{3+} ions and oxygen ions, thereby enhances the asymmetry around the immediate surroundings of Ho^{3+} ions. Larger Ω_2 values typically indicate better laser performance and are generally associated with a higher degree of covalency and optical basicity within the glass system. Additionally, Table 4 enumerates other important factors, including branching ratios (β) and spontaneous transition probabilities (A_{rad}), stimulated emission cross-sections (σ), which were evaluated using the Ω_x values. The transition probabilities represent the probability per unit of time that an atom or molecule in an excited state will

Table 2. The experimental and estimated oscillator strengths (f_{exp} & f_{cal}) of TPZSH1.0 glass

Transition $^5I_8 \rightarrow$	$f_{\text{exp}} (\times 10^{-6})$	$f_{\text{cal}} (\times 10^{-6})$
$(^5G_5, ^5G_6)$	1.9533	2.0032
5G_6	16.6947	16.6973
5F_3	0.9952	1.5311
5F_4	3.9422	3.512
5F_5	2.7241	2.9306
5I_5	0.3514	0.241
5I_6	0.8643	1.3489
5I_7	1.9538	1.8246

Table 3. JO parameters (Ω_x) of TPZSH1.0 glass

Glass code	JO parameter			Reference
	$\Omega_2 (\times 10^{-20})$	$\Omega_4 (\times 10^{-20})$	$\Omega_6 (\times 10^{-20})$	
TPZSH1.0	4.07	1.64	2.23	Glass sample
K	3.86	3.65	1.25	(Babu & Ratnakaram, 2016)
BaPbAlFBHo1.0	3.83	3.22	2.95	(Rani <i>et al.</i> , 2020)
BCBaSrHo(0.5)	2.87	3.66	3.11	(Khan <i>et al.</i> , 2023)

Table 4. Radiative transition probability (A_R), emission cross-section (σ), calculated branching ratios (β_R) of TPZSH1.0 and comparison with other work

Code name of samples	Transition $^5F_4 \rightarrow$	λ (nm)	A_{rad} from J-O (s^{-1})	σ ($\times 10^{-20} cm^2$)	β_R	
					Exp.	Cal from J-O
TPZSH1.0	5I_8	529	4182	4.180	1	0.84
BCBaSrHo(0.5) (Khan <i>et al.</i> , 2023)		542	4185	1.400	0.23	0.30
Lithium potassium (x = 10) (Ratnakaram <i>et al.</i> , 2008)		540	2697	2.559	-	-
sodium-potassium (x = 10) (Ratnakaram <i>et al.</i> , 2008)		542	3818	2.078	-	-
15PbO (Sooraj Hussain <i>et al.</i> , 2006)		537	2635	-	-	0.83

spontaneously decay to a lower energy state while emitting a photon. These probabilities need to be high values. High transition probabilities indicate a greater likelihood of spontaneous emission, which is crucial for determining the radiative properties of the material. The stimulated emission cross-section (σ) quantifies the probability of stimulated emission occurring when an excited atom or molecule interacts with an incident photon. A larger stimulated emission cross-section indicates a higher probability of stimulated emission, which is important for the efficiency and gain of laser materials. All the results demonstrate strong potential for laser applications as each parameter value compares favorably with those from other studies.

Conclusions

The TPZSH glasses have been synthesized and their properties (physical, structural, optical, and luminescence) have been studied. The physical properties show that density, molar volume, and refractive index values increase with the concentration of Ho_2O_3 . Although normalized density increases molar volume decreases but overall density and molar volume increase with the incorporation of Ho^{3+} ions, which have a larger atomic radius than Te. The structural properties indicate an amorphous nature in XRD and peaks of tellurite and phosphate in FTIR that confirm that tellurite is not lost in the glass system (as the melting point of tellurite is lower than the experimental used). Optical properties show that peak absorption increases with the concentration of Ho_2O_3 following the Beer-Lambert law. Luminescence properties indicate the maximum intensity of emission spectra in TPZSH1.0 glass with a CIE of x = 0.387 and y = 0.578, corresponding to green emission. Considering the JO parameters, TPZSH1.0 glass exhibits the highest values of Ω_2 , radiative transition probability (A_R), and radiative cross-section (σ) compared to other glass compositions, suggesting

that tellurite glass is a promising candidate for laser applications. Overall, the results suggest that the sample glass has potential for development and application in laser media.

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