

Absorption and emission spectral analysis of Pr³⁺: tellurite glasses

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Abstract This paper reports on the results concerning optical absorption and fluorescence properties of 60TeO₂–25ZnO–10BaO–4.5La₂O₃–0.5Pr₂O₃ (Pr³⁺: TZBL) glass. Both electronic (α_e) and vibrational (α_v) band edge cut-off wavelengths of the host glass (TZBL) have been evaluated from the measurement of its UV–Vis and IR transmission spectra. The glass studied has shown 80% transmittance throughout its optical window from 0.366 μm ($\alpha_e = 3.39$ eV) to 6.30 μm ($\alpha_v = 0.197$ eV). The FT-IR transmission spectra of Pr³⁺ doped and also reference tellurite glasses have demonstrated the presence of TeO₄ and TeO₃₊₁ or TeO₃ structural units. The thermal properties of this glass have been investigated from the study of DTA profile. The recorded optical absorption spectra of Pr³⁺: TZBL glass have shown eight absorption bands from 300 nm to 2,500 nm. The fluorescence emission has been observed mainly from ³P₁, ³P₀ and ¹D₂ states to the lower lying states and which are assigned to the transitions of ³P₀ → ³H_{4,5,6}; ³P₀ → ³F_{2,3,4}; ³P₁ → ³H₅ & ¹D₂ → ³H_{4,5} upon excitations at three excitation states of ³P_{0,1,2}. From the time resolved spectra, it is found that ³P₀ level decays faster than ¹D₂ level. The fluorescence decay kinetics of ³P₀ and ¹D₂ levels have been measured and the lifetimes are found to be 21 and 39 μs , respectively.

Introduction

Trivalent rare earth ions doped glasses have been identified as very important class of materials as fluorescent display devices, optical detectors, bulk lasers, fiber amplifiers and waveguide lasers [1–3]. In order to update the glass science and technology, several new optical systems are being brought out from different laboratories in the world from time to time. We are all aware of the fact that the materials with low phonon energies are preferable and such optical systems could significantly encourage in obtaining better and potential emission performance from the dopant luminescent rare earth ions. But, some of the low phonon energy glass hosts such as halide glasses could possess poor chemical durability and chalcogenide glasses essentially require more stringent preparation conditions compared to the oxide glasses. Hence, recently heavy metal oxide (HMO) glasses have been found to be more promising glassy materials for photonic applications with reasonably low phonon energies [4, 5]. Among the HMO glasses, tellurite glasses are more attractive systems with advantages such as low temperature of melting, good corrosion resistance, better thermal stability, low phonon energy, wide transmission region (0.36 μm –6.3 μm), high refractive index and desired rare earth concentration in the matrices [6]. In recent years, tellurite based glasses have extensively been investigated towards the development of fiber amplifiers for use in broadband telecommunication applications [7]. Due to their high refractive index and the presence of lone pair of electrons in electronic structure, those could be functioning as good candidates in the development of certain nonlinear optical devices like optical switches with a large third order nonlinear susceptibility. Thermally poled tellurite based glasses containing zinc or alkaline earth/alkali ions have demonstrated

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a second harmonic generation (SHG) [8, 9]. The binary zinc–tellurite glass systems have been studied widely for their structural details as well as their luminescence properties with Pr^{3+} or Nd^{3+} as the dopant ions due to their technological advantages [10]. Chen et al., have earlier investigated the Er^{3+} doped $\text{TeO}_2\text{--BaO}$ ($\text{Na}_2\text{O}/\text{Li}_2\text{O}$)– La_2O_3 glass for its thermal and spectral properties [11]. It has been reported that, the glass without the alkali has shown a good glass thermal stability and with the inclusion of an alkali in the network stability decreases. Hence, barium-containing glasses are considered as good candidates for optical materials, as it improves the glass stability, chemical resistance, optical refractive index and also it reduces the phonon energy to a certain extent. There has not so far been any information on zinc–barium–tellurite glass system. Earlier, we have reported on the energy transfer mechanisms in the rare earth pairs such as (Eu^{3+} , Nd^{3+}) and (Dy^{3+} , Nd^{3+}) doped in tellurite glasses [12, 13]. In the present work, our main objective is to study the spectral properties of $\text{Pr}^{3+}:\text{TeO}_2\text{--ZnO--BaO--La}_2\text{O}_3$ (TZBL) glass system. Among the rare earth ions, Pr^{3+} has been widely used because of its emission at $1.3\ \mu\text{m}$ so called second telecom window [14]. Apart from this, Pr^{3+} ions have the ability to show four level laser action associated with transitions from $^3\text{P}_0$ state in certain host systems as the potentially active ions for a visible laser generation [15]. The present paper brings out the results pertaining to the studies carried out on different physical, thermal and optical absorption and emission spectra of Pr^{3+} doped $\text{TeO}_2\text{--ZnO--BaO--La}_2\text{O}_3$ (Pr^{3+} : TZBL) glass.

Experimental studies

Pr^{3+} ions doped tellurite glasses in the composition of $60\text{TeO}_2\text{--}25\text{ZnO}_2\text{--}10\text{BaO}_2\text{--}4.5\text{La}_2\text{O}_3\text{--}0.5\text{Pr}_2\text{O}_3$ have been prepared by means of conventional quenching and annealing methods. The raw materials such as TeO_2 (99.99%) from Alfa, BaCO_3 (99.98%) from EMERCK, ZnO (99.95%) from Fluka, La_2O_3 (99.98%) from IREL and Pr_4O_{13} (99.99%) from Alfa have been used in the glass preparation. Appropriate amounts of chemicals for 15 gm batch were weighed, mixed thoroughly in the Agate mortar and those chemicals mixtures were melted in pure Platinum crucible with lid at $950\ ^\circ\text{C}$ for half an hour in a micro-processor controlled electric furnace. A reference glass (without the dopant Pr^{3+} ions) was also prepared under the similar conditions. The prepared samples were annealed at $350\ ^\circ\text{C}$ temperature for an hour. The densities (d) of the glass samples were measured by following the traditional Archimedes' principle with water as an immersion liquid. The refractive indices at different wavelengths for the reference glass (TZBL) were measured on a precision

spectrometer (Kalnew, Model GMR-1) by adopting a minimum deviation method. The XRD analysis of annealed TZBL reference glass and Pr^{3+} : TZBL glass were done on a X-ray diffractometer type PW710 with Cu K_α radiation ($1.54\ \text{\AA}$) in the 2θ range of $10\text{--}60$ with $0.5\ \text{s}$ per step. FT-IR transmission spectra of both the glasses in the range $2,000\text{--}400\ \text{cm}^{-1}$ were measured using KBr pellet method on a Perkin-Elmer FTIR spectrophotometer model 1615 series with $4\ \text{cm}^{-1}$ resolution. The transmission spectra of TZBL glass sample in the range $200\text{--}1,100\ \text{nm}$ were measured on Perkin-Elmer UV–Vis spectrophotometer model Lambda 20 and in the spectral range $5,000\text{--}1,000\ \text{cm}^{-1}$ on Perkin-Elmer FTIR spectrophotometer model 1615 series. The UV–Vis–NIR absorption spectra of Pr^{3+} : TZBL glass were recorded on a Perkin-Elmer UV–Vis spectrophotometer model Lambda 20 in the wavelength range of $200\text{--}1,100\ \text{nm}$ and on Shimadzu MPC3103 UV–Vis–NIR spectrophotometer from $1,000\ \text{nm}$ to $2,500\ \text{nm}$. The study state emission spectra and also excitation spectra were recorded on a SPEX Fluorolog-2 spectrofluorimeter with $150\ \text{W}$ Xenon lamp as the source of excitation. The time resolved fluorescence spectra and emission transition decay kinetics were measured on the same instrument equipped with 1934D phosphorimeter attachment along with a $50\ \text{W}$ pulsed xenon lamp as the pump source. The system employs Datamax software in acquiring the spectra and the decay curves.

Results and discussion

Amorphous natures of the Pr^{3+} doped and undoped TZBL glasses have been verified from the X-ray diffraction analysis. Figure 1 shows the DTA curves of Pr^{3+} doped and

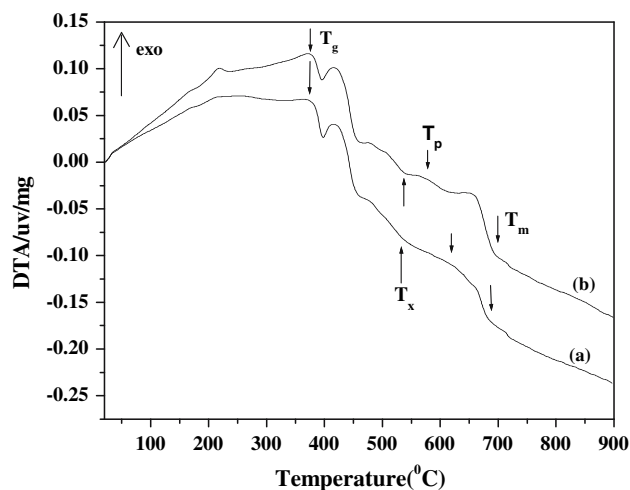


Fig. 1 Differential thermal analysis curves of (a) TZBL and (b) Pr^{3+} : TZBL glasses

undoped TZBL glasses. The glass transition temperatures (T_g) have been determined to be 380 °C for TZBL glass and 379 °C for Pr³⁺: TZBL glass. Hence the substitution of lanthanum for Praseodymium in the network is decreasing the glass transition temperature, which is palpable due to the smaller radius of the dopant ion. The DTA curves of both the glasses exhibit no prominent exotherm peak but there is broad hump with insignificant height is seen in the figure suggesting the slow rate of crystallization of these glasses at temperatures near T_x . The thermal stabilities of the TZBL reference glass and Pr³⁺: TZBL glass have been evaluated from their glass transition temperature (T_g), crystallization onset temperature (T_x) and the melting temperature (T_m) values and the results are listed out in Tables 1 and 2, respectively. The glass stability factor (STF = $T_x - T_g$) for this glass system is comparable to the first commercially available which is reported to be most stable telluride glass fiber of composition (TeO₂–ZnO–Na₂O) [16]. From the measured density (d) and refractive indices (n), many other important physical, optical and nonlinear optical properties have been estimated using standard expressions and the results are presented in Table 1 for the TZBL glass and in Table 2 for the Pr³⁺:

Table 1 Different physical, optical and thermal properties of TZBL (reference) glass

Parameters	Data
<i>Physical and optical properties</i>	
Molecular weight of glass (M g)	147.73
Density (d g cm ⁻³)	5.452
Refractive index (n)	
n_F	2.0629
n_e	2.0369
n_C	2.0135
Electronic band edge energy (α_e eV)	3.39
Vibrational band edge energy (α_v eV)	0.197
Reflection losses (R%)	11.658
Molar refractivity (R_M cm ³)	13.88
Molecular electronic polarisability (α_e Å ³)	5.5
Mean dispersion ($n_F - n_C$)	0.0494
Abbe number (v_e)	20.9899
Non-linear refractive index ($n_2 \times 10^{13}$ esu)	21.915
Non-linear refractive index coefficient ($\gamma \times 10^{-18}$ cm ² /W)	4.51
Nonlinear susceptibility ($\chi_{1111}^3 \times 10^{15}$)	1.184
<i>Thermal properties</i>	
Glass transition temperature (T_g °C)	380
Crystallization initiation temperature (T_x °C)	532
Crystallization peak temperature (T_P °C)	619
Melting temperature (T_m °C)	680
Glass stability factor ($T_x - T_g$)	157
Hurby value ($H = (T_x - T_g)/(T_m - T_P)$)	2.5

TZBL glass. It is well known that, the high refractive index glasses have faster responding nonlinearities over the other organic or semiconducting nonlinear materials and for these materials, the nonlinear coefficient is an important property. Tellurite glasses are such systems widely used for fast optical switches [17]. From Table 1 it is found that the TZBL glass possesses high nonlinear refractive index (n_2) and nonlinear coefficient (γ) and hence which demonstrate its suitability for their use in nonlinear optical device applications.

The structure of TeO₂ rich glasses contains basically of three dimensional network of TeO₄ trigonal bipyramidal (tpb) units having two each of oxygens at two equatorial and axial sites with one other equatorial site being occupied by a lone pair of electrons. On the introduction of a modifier, three dimensional network breaks down with the transformation of TeO₄ units into TeO₃₊₁ and TeO₃ units [18]. In order to have an insight into the structural units of glasses studied, FT-IR transmission spectra of TZBL and Pr³⁺: TZBL glasses have been recorded in the range 1,000–375 cm⁻¹ as shown in Fig. 2. The spectra have displayed three bands at 758, 677 and 424 cm⁻¹ and are assigned to stretching vibrations of TeO₃ or TeO₃₊₁, TeO₄ structural units and bending vibrations of Te–O–Te or O–Te–O linkages respectively following the literature reports [19]. According to them, the ratio of 780/675 cm⁻¹ represents the relative concentrations of the TeO₃ and TeO₄ structural units, which is absolutely dependent on the glass composition. From the FT-IR spectra of both glasses, the absence of IR vibrational peaks at 800–780 cm⁻¹ indicates that there are no isolated TeO₃ structural unit in the glass composition studied. Also a highest phonon band at 758 cm⁻¹ has appeared as a shoulder to the main peak at 677 cm⁻¹ which is otherwise found in dominant intensity in some tellurite glass systems of compositions having different other modifiers with almost equivalent molar fractions considered in the present glass composition was explained due to the formation of TeO₃/TeO₃₊₁ pyramids with non-bridging oxygens (NBOs) [19]. The intensity dominance of vibrational band at 677 cm⁻¹ over the band at 758 cm⁻¹ detected in the present glass system could be attributed to be due to the incorporation of ZnO as glass former into a chain like structure with Te₃O₈ groups built by TeO₄ and two TeO₃₊₁ units connected by six coordinated Zn²⁺ ions [20] and the contribution of stretching vibrations due to Zn–O bond are also expected at this wavenumber. The peak at 758 cm⁻¹ as a shoulder to the main peak at 677 cm⁻¹ could be resulted due to the superposition of TeO₃₊₁ and TeO₃ units, which have formed by the distortion of TeO₄ units in the presence of BaO and La₂O₃ as modifiers. This assortment of species in the tellurite glass matrix indicates the presence of a variety of structural sites due to the incorporation of modifiers.

Table 2 Different physical and thermal properties of Pr³⁺: TZBL glass

Characteristic parameters	Data
<i>Physical properties</i>	
Molecular weight of glass (M g)	147.75
Density (d g cm ⁻³)	5.442
Rare earth ion concentration ($N \times 10^{21}$ ions cm ⁻³)	1.1092
Ionic radius (r_p Å)	3.893
Inter ionic distance (r_i Å)	9.660
Field strength ($F \times 10^{-15}$ cm ²)	1.979
<i>Thermal properties</i>	
Glass transition temperature (T_g °C)	379
Crystallization initiation temperature (T_x °C)	539
Crystallization peak temperature (T_P °C)	575
Melting temperature (T_m °C)	694
Glass stability factor ($T_x - T_g$)	160
Hurby value ($H = (T_x - T_g)/(T_m - T_P)$)	1.3

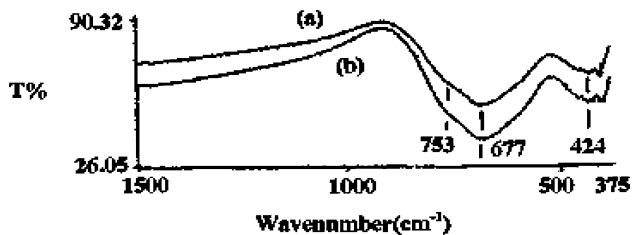
**Fig. 2** FT-IR transmission spectra of (a) TZBL and (b) Pr³⁺: TZBL glass

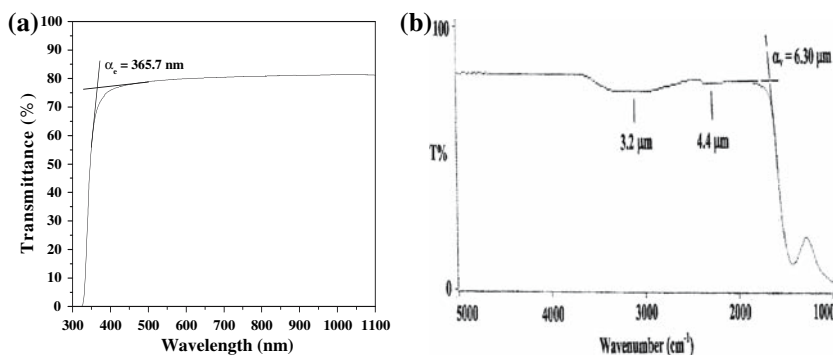
Figure 3a, b shows the transmission characteristics of a reference glass (TZBL glass) in the UV–Vis–NIR range. From these transmission curves, electronic (α_e) and vibrational (α_v) absorption edge wavelengths have been estimated as shown in Fig. 3a, b and the results are given in Table 1. It is clear from this figure that, the TZBL glass has above 80% transmission in the entire range of optical window between 0.366 μm and 6.30 μm . The vibrational absorption edge at 6.30 μm (1,587 cm^{-1}) in the IR transmission spectrum of TZBL glass has been resulted from the overtones of the Te–O stretching vibrations at 800–600 cm^{-1} as noticed in its recorded FTIR spectrum. The absorption peaks detected in IR transmission curve at 3.2 μm (3,128 cm^{-1}) and 4.4 μm (2,328 cm^{-1}) are due to stretching vibrations of hydroxyl (OH^-) groups. Earlier in the literature, many tellurite glasses exhibited vibrational bands due to OH^- groups in the range 2,300–3,500 cm^{-1} and they were assigned to be as follows: the band at $\sim 3,500$ cm^{-1} is due to hydrogen bond free OH^- groups, the band at $\sim 3,000$ cm^{-1} is due to weak hydrogen bonding between NBO and OH^- groups and the band at $\sim 2,500$ cm^{-1} is due to the strong hydrogen bonding of OH^- to NBO [21, 22].

Accordingly, in the present glass, the observed bands at 3,128 and 2,328 cm^{-1} are considered due to weakly and strongly hydrogen bonded OH^- groups, respectively. And it can be stated that, the hydrogen bond free OH^- groups are absent as there is no band observed at around 3,500 cm^{-1} . In the rare earth doped glasses, OH^- group act as fluorescence quenching sites, which promote non-radiative decay of emitting level and ultimately decreasing the emission efficiency of the material. More is the absorption coefficient of OH^- (α_{OH^-}), higher would be its influence on the fluorescence quenching. The absorption coefficient of OH^- (α_{OH^-}) which is determined based on the application of a standard procedure made available in literature [23] from the recorded IR transmission spectrum of the glass investigated has been found to be 0.81 cm^{-1} which is certainly a lower value and therefore it could be considered to show a negligible effect on the fluorescence efficiency of the dopant ion in the glass matrix studied here. The recorded optical absorption spectrum of Pr³⁺: TZBL glass in two wavelength ranges 375–650 nm and 875–2,500 nm are presented in Fig. 4a, b. The glass has revealed eight bands four each in UV–Vis and NIR regions from ground state ($^3\text{H}_4$) to various higher energy states of Pr³⁺ ions. They are assigned to the transitions of $^3\text{H}_4 \rightarrow ^3\text{F}_2, ^3\text{F}_3, ^3\text{F}_4, ^1\text{G}_4, ^1\text{D}_2, ^3\text{P}_0, (^3\text{P}_1, ^1\text{I}_6)$ and $^3\text{P}_2$ depending upon their peak energies. Similarly, the excitation spectrum recorded by monitoring the emission wavelength at 615 nm has been revealed three peaks corresponding to the transitions from ground state to $^3\text{P}_0, (^3\text{P}_1, ^1\text{I}_6)$ and $^3\text{P}_2$ levels as shown in Fig. 5. The room temperature fluorescence emission spectrum (475–750 nm) of Pr³⁺: TZBL glass excited by three wavelengths of $^3\text{P}_0, ^3\text{P}_2$ and $(^3\text{P}_1, ^1\text{I}_6)$ bands separately are shown in Fig. 6a, b, c. The emission bands have been labeled as summarized below:

Transition	Wavelength	Color	Significance	Emission type
$^3\text{P}_0 \rightarrow ^3\text{H}_4$	(490 nm)	Blue	High	Three level
$^3\text{P}_0 \rightarrow ^3\text{H}_5$	(536 nm)	Green	Medium	Four level
$^3\text{P}_1 \rightarrow ^3\text{H}_5$	(532 nm)	Green	Medium	Three level
$^1\text{D}_2 \rightarrow ^3\text{H}_4$	(604 nm)	Red	High	Three level
$^3\text{P}_0 \rightarrow ^3\text{H}_6$	(615 nm)	Red	High	Four level
$^3\text{P}_0 \rightarrow ^3\text{F}_2$	(646 nm)	Red	High	Four level
$^1\text{D}_2 \rightarrow ^3\text{H}_5$	(688 nm)	Deep red	Low	Four level
$^3\text{P}_0 \rightarrow ^3\text{F}_3$	(703 nm)	Deep red	Low	Four level
$^3\text{P}_0 \rightarrow ^3\text{F}_4$	(730 nm)	Deep red	Low	Four level

Based on the literature reports, the emission band at 532 nm has been assigned to the transition of $^3\text{P}_1 \rightarrow ^3\text{H}_5$, this band has been detected even when its lower laying level $^3\text{P}_0$ is excited upon. Thus, it is clear that the emission from $^3\text{P}_1$ level is resulting due to its thermal population as

Fig. 3 (a) UV–Vis transmission spectra of TZBL glass. (b) NIR transmission spectra of TZBL glass TZBL glass



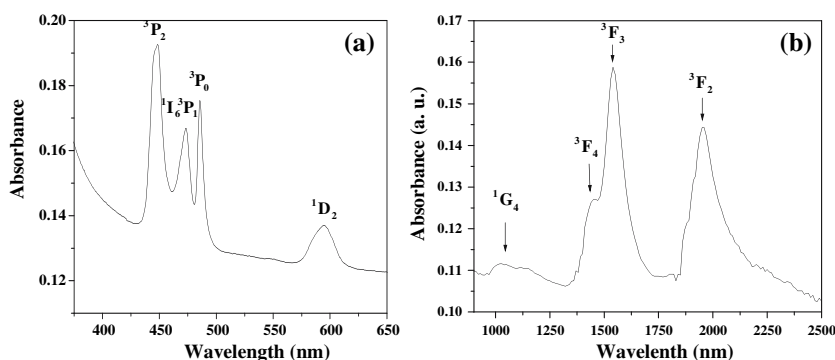
the energy gap between this level and lower lying 3P_0 state is only around 650 cm^{-1} [24]. Among the measured emission transitions, $^3P_0 \rightarrow ^3H_6$ and 3F_2 transitions are in the red region and which are more intense. They occur due to the participation of four energy levels, when 3P_2 and (3P_1 , 1I_6) levels are excited which can be seen in partial energy diagram of Pr^{3+} : TZBL glass as depicted in Fig. 7. It is well known that, four level emission is more efficient than three level emission mechanism in the view of practical laser applications. It is observed from the fluorescence spectra that the relative intensities of these two red emission transitions vary with the change in excitation wavelengths. For example, their ratio ($I(^3P_0 \rightarrow ^3H_6)/I(^3P_0 \rightarrow ^3F_2)$) are 1.15, 1.03 and 0.89, respectively when excited at 484, 443 and 468 nm, respectively. In the fluorescence spectrum, the intense red emission at 600–630 nm could be due to both $^3P_0 \rightarrow ^3H_6$ and $^1D_2 \rightarrow ^3H_4$ transitions with dominant emission from 3P_0 level. To verify this, time resolved emission spectra have been carried out for Pr^{3+} : TZBL glass with an excitation at 444 nm ($^3H_4 \rightarrow ^3P_2$) at varied delay times. The time resolved spectra are shown in Fig. 8a, b, c, d and e. It is noticed from this figure that, with an increase in the time delay from 0.01 ms to 0.1 ms, the emission from 3P_0 decreases and at longer delay times, emission from 1D_2 level alone could become possible. This provides a good support upon overlay of both the emission transitions $^3P_0 \rightarrow ^3H_6$ and $^1D_2 \rightarrow ^3H_4$ in the peak at wavelength

600–630 nm region. This time resolved spectra also used to investigate the time evaluation of the fluorescence from the emitting levels 3P_0 and 1D_2 . It is observed that, 3P_0 level decays faster than 1D_2 and which has been substantiated from the measured decay curves of emission bands as shown in Fig. 9 and from which lifetimes of the 3P_0 and 1D_2 emission bands are 21 and 39 μs , respectively.

Conclusions

In summary, it is concluded that we have developed greenish-yellow colored Pr^{3+} doped tellurite glass in the composition of $60\text{TeO}_2\text{--}25\text{ZnO}\text{--}10\text{BaO}\text{--}4.5\text{La}_2\text{O}_3\text{--}0.5\text{Pr}_2\text{O}_3$ and analyzed. Glass thermal properties, FT-IR, optical absorption and time resolved fluorescence spectra have systematically been studied in evaluating the properties of this optical glass. The measured spectrum from UV–Vis to the IR spectra have shown the glass has been transmitting from 365.7 nm ($\alpha_c = 3.39\text{ eV}$) to 6,300 nm ($\alpha_v = 0.197\text{ eV}$) with a 80% transmission within the optical window. The FT-IR transmission spectra of Pr^{3+} doped and also un-doped tellurite glasses have demonstrated the presence of TeO_4 and TeO_{3+1} or TeO_3 structural units. It is believed that, the ZnO presence could be participating in the glass network as six coordinated Zn^{2+} ions. The glass stability factor (GSF) estimated from glass transition (T_g), crystallization onset (T_x), peak (T_p) and melting (T_m)

Fig. 4 (a) Visible and (b) NIR absorption spectra of Pr^{3+} : TZBL glass



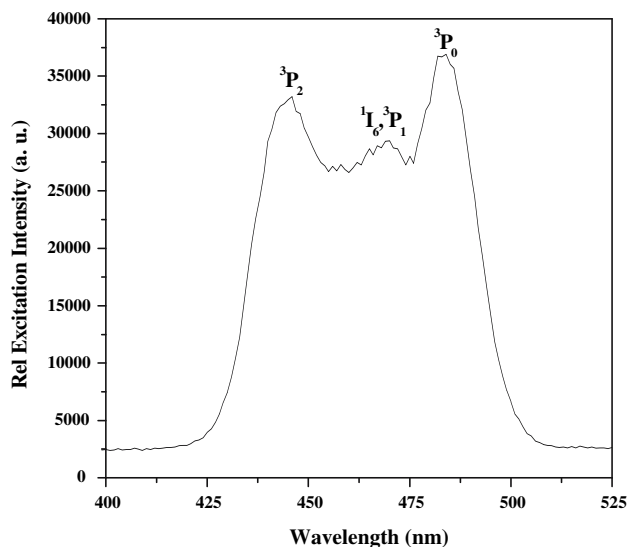


Fig. 5 Excitation spectrum of Pr³⁺: TZBL glass

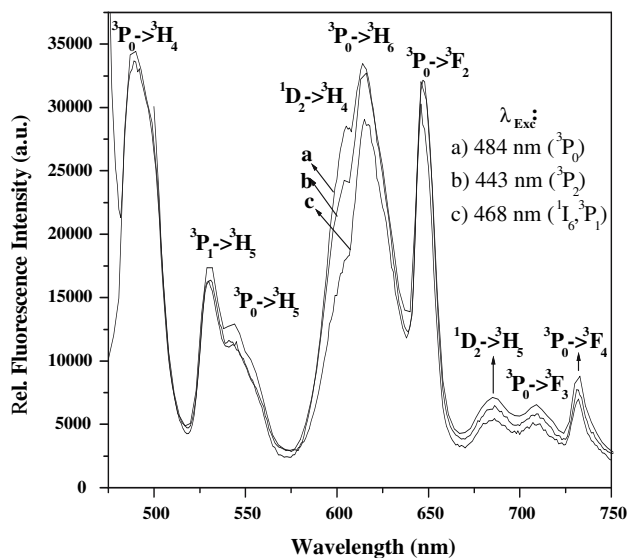


Fig. 6: Visible fluorescence spectra of Pr³⁺: TZBL glass at different excitation wavelengths

obtained from the DTA measurements reveal that the glass examined here possess good thermal stability. Both the measured absorption and emission spectra of the Pr³⁺ glass have been assigned to the appropriate electronic transitions. From the time resolved spectra, it is found that ³P₀ level decays faster than ¹D₂ and at shorter delay times, ³P₀ emission dominates while with an increase in delay time, ¹D₂ emission becomes more effective. The fluorescence lifetimes of ³P₀ and ¹D₂ levels have been computed from the measured decay kinetics and are found to be 21 and 39 μs, respectively. The emission from ³P₁ level has been attributed to the thermalization of the closely lying lower state namely ³P₀.

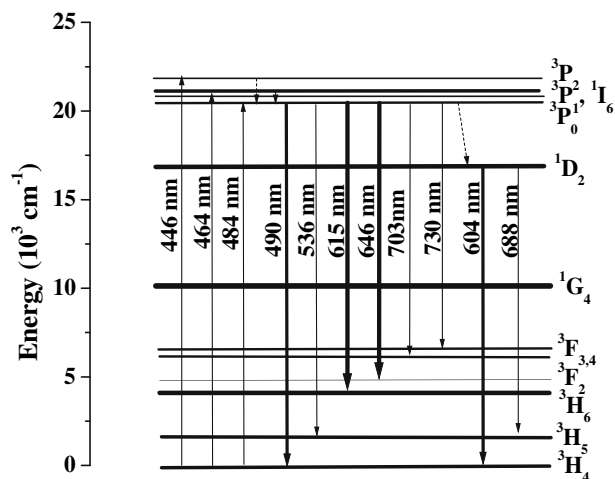


Fig. 7 Partial energy level diagram and emission mechanism in Pr³⁺: TZBL glass

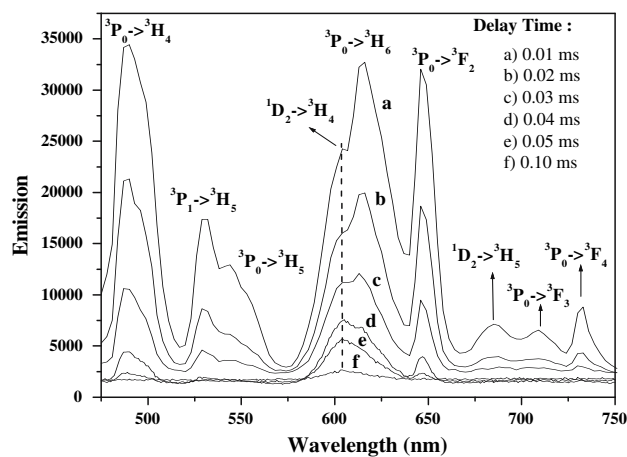


Fig. 8 Time resolved emission spectra of Pr³⁺: TZBL glass with $\lambda_{exc} = 444$ nm

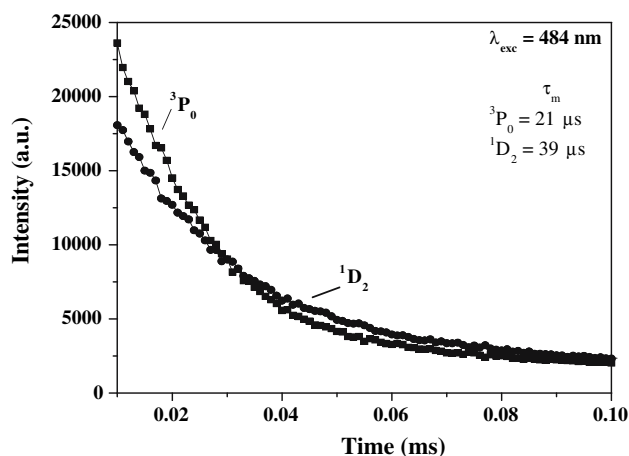


Fig. 9 Fluorescence Decay curves of ³P₀ and ¹D₂ levels of Pr³⁺: TZBL glass

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