

Structural and physical analysis of borosilicate glasses with Pr³⁺ ions

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Abstract

The present work gives the information about the structural and physical analysis of borosilicate glasses with praseodymium ions. Glass specimens were prepared with the chemical composition $(50-x) \text{B}_2\text{O}_3 - (10+x) \text{SiO}_2 - 10\text{Na}_2\text{O} - 20\text{PbO} - 10\text{ZnO} - 1\text{Pr}_6\text{O}_{11}$ (where $x = 0, 5, 10, 15, 20, 25, 30, 35, 40$) by standard method. The composition of the glass specimens was finalized by EDX. The presence of various stretching and bending vibrations are confirmed with the help of FTIR analysis. The borate network of the present glass samples lies in the wavelength range $500\text{-}1600 \text{ cm}^{-1}$ and confirmed by the TEM image. Anti-symmetric stretching vibrations with three non-bridging oxygens of B – O – B groups are present at $\sim 1488 \text{ cm}^{-1}$. XRD were recorded for confirmation of the amorphous nature of the samples. The SEM image confirmed the XRD results. Various physical parameters have been calculated for knowing the structure of present glass with increasing the SiO_2 concentration. Oxygen Packing Density (OPD) decreases with increase in SiO_2 concentration. Decrease in the value of OPD shows that glass structure is now loosely packed. Absorption spectra of the glass samples were recorded in UV-VIS/NIR region. JO intensity, Racah, Slater-Condon, Lande parameters have been computed. Ω_2 parameter gives information about the glass structure and depends upon the Pr – O bond in the host matrix. Optical energy band gap and Urbach's energy values were calculated for exploring the electronic band structure. Copyright © 2017 VBRI Press.

Keywords: Physical properties, absorption spectra, Urbach's energy, energy and Judd – Ofelt intensity parameters.

Introduction

Praseodymium doped glasses are of great interest due to their potential applications as $1.3 \mu\text{m}$ optical amplifiers [1], memory devices, solid state lasers, solid state – batteries, optical waveguides, optical telecommunication, non-linear optical materials, etc [2]. Generally, glass host is more preferable than crystalline host because of the fact that glass can be produced in large volumes with high optical homogeneity and free of absorbing particle; it possesses low non-linear refractive indices and acts as a good elastic matrix by accepting large concentration of rare earth ions [3]. The rare earth ions doped glasses have attracted a great deal of attention due to their important physical, optical and chemical properties [4]. The electronic energy levels of rare earth ions determine the lasing characteristics of rare earth ions doped materials and are influenced by the host matrix [5].

Now a day's studies on oxide glasses doped rare earth ions are very popular. Among all the oxide glasses, borate based glasses are very important, as B_2O_3 is one of the

most common glasses former and important due to their structural, optical and physical properties [6]. But in crystalline form it is important because of their interesting linear and nonlinear optical properties. Melts with compositions rich in B_2O_3 exhibit rather high viscosity and tend to formation of glasses [7]. The structure of the borate glasses is not a random distribution of BO_3 triangle and BO_4 tetrahedral, but a gathering of these units form well defined and stable borate groups such as diborate, triborate and tetra borate that constitute the three dimensional random network [4]. Borate glasses show properties like high transparency, low melting point, high thermal stability and good glass forming quality. Silicate glasses are thermally stable, chemically durable and optically transparent towards excitation and lasing wavelengths [8] and they have higher phonon energies so up conversion is difficult to be realized in these glasses, resulting in a higher gain in the fibre amplifier. The combination of borate and silicate glass will give a strong host matrix. Borosilicate glasses are expected to be good

materials for optical devices such as lasers and optical fibres.

In this research paper we have taken the combination of borate and silicate i.e. borosilicate as host matrix doped with Pr^{3+} ions have been prepared by melt quenching techniques. The Pr^{3+} doped borosilicate glass specimens were characterized by XRD, FTIR, EDX, SEM and TEM. Physical and optical properties of the present glass specimens were computed with varying concentration of SiO_2 . The optical absorption spectra have been recorded at room temperature in UV-visible and NIR region and various parameters like JO parameter, Racah, Slater – Condon, Lande and bonding parameter were computed.

Experimental

Materials

The starting materials used in the preparation of borosilicate glasses with Pr^{3+} ions are Silicon dioxide, Borax, Lead carbonate, zinc carbonate and Praseodymium oxide. All of these are of AR grade (99.99% pure) and were procured from E. Merck (India) and Aldrich (USA).

Sample preparation

The Pr^{3+} doped borosilicate glass specimen has been prepared by conventional melt quenching technique. The samples were preheated in electric furnace at 300°C for one hour. After that the temperature is increased slowly up to the working temperature (1000°C) and samples were heated at this temperature for three hours. Homogeneity of the melt was ensured by stirring the samples time to time by Platinum rod. Prepared samples were poured into preheated rectangular brass plate and annealed for 6 hours. Prepared samples were cut and polished for optical measurements. The final composition of glass specimens is $(50 - x) \text{B}_2\text{O}_3 - (10 + x) \text{SiO}_2 - 10\text{Na}_2\text{O} - 20\text{PbO} - 10\text{ZnO} - 1\text{Pr}_6\text{O}_{11}$ where $x=0, 5, 10, 15, 20, 25, 30, 35, 40$.

Characterization of the glass specimen

EDX and XRD of Pr^{3+} doped borosilicate glasses were recorded for characterizing the samples. SEM and TEM of present glasses were recorded by FEI Quanta 200F and Tecnai G^2 20 respectively. FTIR spectrum of the present glass was recorded by Nicolet 6700 FT – IR.

Measurements

The absorption spectra have been recorded at room temperature with the spectrophotometer model Varian carry in UV-VIS/NIR region with the resolution of 0.5 nm. Physical and optical properties of the samples were measured and calculated. Thickness of the glass samples were measured by Vernier callipers and refractive index of the glass samples was measured by using following equation,

$$n_d = 1.57376 + \frac{153.137}{\lambda(\text{\AA}) - 686.2},$$

where, $\lambda(\text{\AA})$ is for peaks.

Results and discussion

Characterization

EDX spectra of the Pr^{3+} doped borosilicate glass has been shown in **Fig. 1(a)**. The various components are present in these spectra which show all the components are present in the final composition, taken initially for preparing the samples.

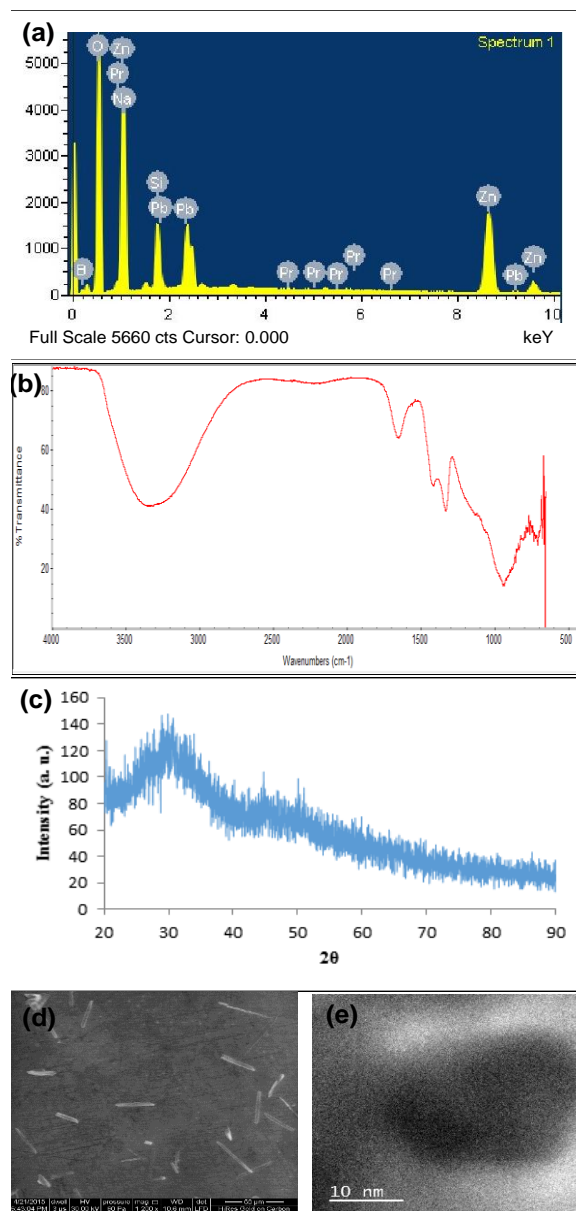


Fig. 1. (a) EDX Spectra of Borosilicate glass with Pr^{3+} ions for E specimen; (b) FTIR Spectrum of Borosilicate glass with Pr^{3+} ions for E specimen; (c) XRD Spectra of Borosilicate glass with Pr^{3+} ions for E specimen; (d) SEM image of borosilicate glass with Pr^{3+} ion for E specimen; (e) TEM image of borosilicate glass with Pr^{3+} ion for E specimen.

FTIR has been given in **Fig. 1(b)** in the wavelength region $400-4000\text{cm}^{-1}$. FTIR is a powerful tool for defining the functional groups in the present glass samples. This figure gives the information about various stretching

bands present in the glass system. ZnO tetrahedral bond is present $\sim 473\text{ cm}^{-1}$ position. In BO_4 , ν_4 tetrahedral bond is at $\sim 512\text{ cm}^{-1}$, B-O-B bending is present at ~ 708 , penta borate groups are at $\sim 978\text{ cm}^{-1}$, vibrations of pentaborate along with BO_4^- tetrahedral are present $\sim 1078\text{ cm}^{-1}$, \sim stretching of B – O bonds at 1210 cm^{-1} , B – O vibrations of borate group are present at $\sim 1310\text{ cm}^{-1}$ and finally anti symmetric stretching vibrations with three non-bridging oxygens of B – O – B groups are present at $\sim 1488\text{ cm}^{-1}$. Presence of the hydroxyl is the most dominant characteristic in all the infrared groups and for present glass samples; the band around 3406 cm^{-1} is due to the fundamental stretching of O-H group.

XRD spectra of the present glass specimen are shown in **Fig. 1(c)**. As it is seen from this figure that there are no crystalline peaks at lower region in this graph which confirms the amorphous nature of the glass samples. The angular position of the first halo is near about 28.82° .

SEM and TEM of the present glass samples were given in **Fig. 1(d)** and **Fig. 1(e)** respectively. It is clear from the figure that there are no grains, verifying the amorphous nature of the samples. The TEM image verifies the results of the FTIR.

Physical and optical properties

Physical and optical properties of the Pr^{3+} doped borosilicate glass specimens were calculated. Various physical and optical parameters were measured such as thickness, average molecular weight, density polaron radius, inter ionic separation, OPD, molar volume, refractive index etc. Thickness of the glass samples lie between 0.18 to 0.20 cm. Average molecular weight of the present samples were lies in between 106.196 to 110.011 gm. The average molecular weight of the samples decreases with increasing SiO_2 concentration. Density of the glass samples were measured by using Archimedes principle as,

$$\rho = \left(\frac{w_a - w_b}{w_a} \right) \rho_B$$

where, w_a is the weight of sample in air, w_b is the weight of sample in immersion liquid and ρ_B is the weight in toluene. Density and molar volume versus concentration of SiO_2 graph is presented in **Fig. 2(a)** From this fig. we can see that density decreases with increase in SiO_2 concentration while molar volume increases with SiO_2 concentration [7, 9]. The variation in the values of density and molar volume are 6.72 to 6.09 g/cm^3 and 16.371 to 17.437 g/cm^3 respectively. In general density and molar volume shows the opposite behaviour in between and the present work also shows the same behaviour.

Pr^{3+} ion concentration is calculated. Values of Pr^{3+} decreases with increase in SiO_2 concentration and similar graph is shown in **Fig. 2(b)** Similar results were also reported [7]. This concentration was calculated by using the formula,

$$N = \frac{N_A \rho (\text{mol \% of rare earth ion})}{M_{AV}}$$

where, $N_A =$ Avogadro number, ρ is the density and M_{AV} is the average molecular weight. The values of Pr^{3+} ion concentration varies from 3.678 to 3.453 ions/ cm^3 .

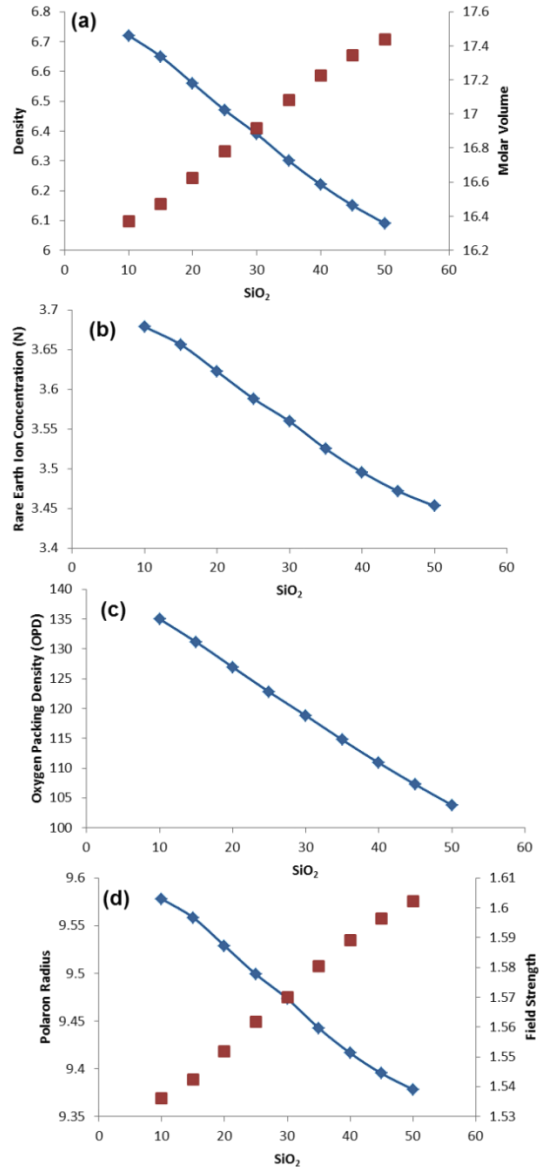


Fig. 2. (a) Variation of density (ρ) and Molar volume (V_m) for borosilicate glasses doped Pr^{3+} ; (b) composition dependence of rare earth ion concentration (N) on SiO_2 contents; (c) variation of Oxygen Packing density with SiO_2 concentration; (d) dependence of Polaron radius and Field strength on SiO_2 concentration.

Oxygen packing density (OPD) is also given for the present glass sample which is the measure of the tightness of the oxide network in the glass sample [7]. OPD is calculated by,

$$O = \frac{1000 * n * \rho}{M}$$

where, n is the no. of oxygen atoms per formula unit, is the molecular weight of the sample. A graph is plotted between OPD and SiO_2 concentration (**Fig. 2(c)**), from this it must be seen that OPD decreases with increase in

SiO₂ concentration and varies in between 134.997 to 103.798 (g-atom/l). Decrease in the value of OPD shows that glass structure is now loosely packed and similar results were reported in other papers [7].

Fig. 2(d) shows the graph between polaron radius and field strength versus SiO₂ concentration. From this graph it would be seen that polaron radius and field strength shows the opposite behaviour with increasing concentration of SiO₂. Polaron radius decreases with increasing concentration of SiO₂ while field strength increases with it. The values of polaron radius and field strength lie in between 9.577 to 9.378 (Å) and 1.536 to 1.602 cm⁻² respectively.

Refractive index, dielectric constant and optical dielectric constant decreases with increase in SiO₂ concentration. The values of all these parameters vary from 1.614 to 1.581, 2.605 to 2.500 and 1.605 to 1.500 respectively. Reflection losses and molar refractivity were also calculated. The value of these parameters lies in between 5.517 to 5.071 and 5.706 to 5.814 cm⁻³. From these data it is observed that reflection losses decrease with increase in SiO₂ concentration while molar refractivity increases with increasing concentration. Electronic polarizability is calculated by using the relation

$$\alpha_e = \frac{3}{4\pi N} R_M$$

The electronic polarizability decreases with increase in SiO₂ concentration and lies in the range 3.019 to 2.711 (ions cm⁻¹).

Absorption studies

Optical absorption spectra of the borosilicate glass doped with Pr³⁺ ion have been recorded in 400-2400 nm at room temperature. Six absorption bands were observed in which four of them are at 442, 470, 480 and 590nm in UV – VIS region and two are in NIR region which are at 1514 and 1920 nm. From **Fig. 3** it is clear that all the glass samples with different concentration of SiO₂ show the similar trend in absorption peaks.

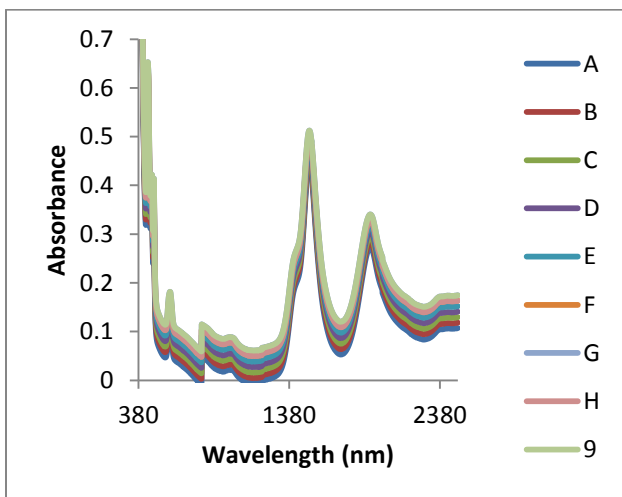


Fig. 3. Absorption spectrum of borosilicate glasses with Pr³⁺ ion in UV – VIS – NIR region at room temperature.

There is no sharp edge (*i.e.* Urbach’s edge) in this graph which indicates that present glass samples are amorphous in nature. These absorption spectra have been used in the calculation of optical energy band gap and Urbach’s energy. The optical energy band gap is calculated by using the Tauc’s relation, which is,

$$\alpha(\omega) = [B(\hbar\omega - E_g)^n]/\hbar\omega$$

where, B is the band tailing parameter, E_g is the optical energy band gap, n depends on the type of the transition *i. e.* direct or indirect transitions. Value of optical band gap is calculated by extrapolating the linear part of the **Fig. 4** and lies in between 0.938 to 0.942.

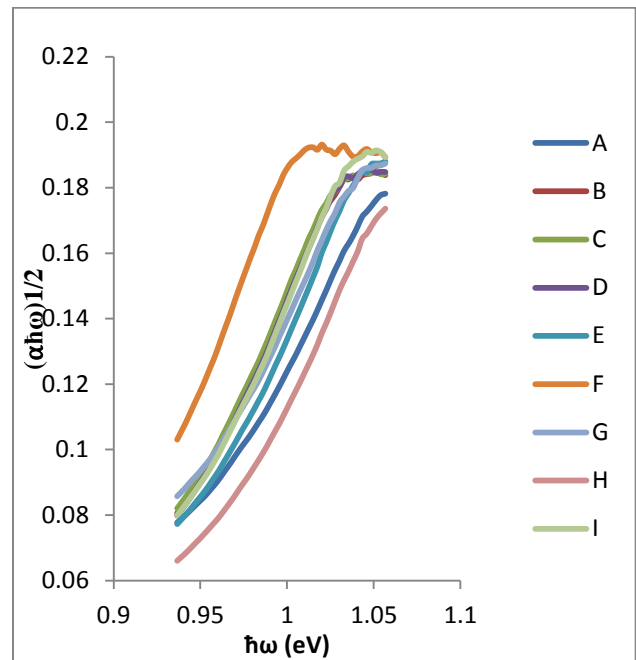


Fig. 4. Optical band gap of Borosilicate glass with Pr³⁺ ions.

The absorption co-efficient α(ν) near the band edge exhibit an exponential dependence on the photon energy (hν) in the lower absorption region {α(ν) < 10⁴cm⁻¹} and obeys the empirical relation [10],

$$\alpha(\nu) = \alpha_0 \exp(h\nu/\Delta E)$$

where, α₀, a constant and ΔE is the Urbach’s energy defined as width of the tail states which extend into the forbidden band gap. The Urbach’s energy is used to identify the disorderliness degree in amorphous materials.

The value of Urbach’s energy is calculated from **Fig. 5** by extrapolating the linear part and taking its reciprocal. The Urbach’s energy values are found to be in the range 1.068 – 1.077 eV. Generally, Urbach’s energy values vary inversely with optical band gap values. The lower values of Urbach’s energy indicate that prepared glass sample possess minimum defects and less disorderliness which in turn leads to structural rearrangement.

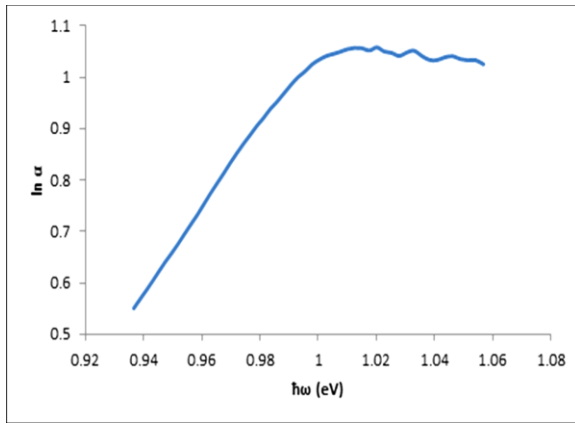


Fig. 5. Representative spectrum of $\ln \alpha$ plotted against photon energy, $\hbar\omega$, for E glass specimen.

From absorption spectra various energy parameters *i.e.* Slater – Condon (F_k), Racah (E^k), Lande (ζ_{4f}) and Bonding parameter ($b^{1/2}$) have been computed by partial regression method and collected in **Table 1**. For the present glass sample relation between F_k parameters is $F_2 > F_4 > F_6$. The values of $F_4/F_2 \sim 0.138$ and $F_6/F_2 \sim 0.015$ which is nearly same as reported values [11]. The ratios of E^1/E^3 and E^2/E^3 are 9.890 and 0.0517 respectively and almost equal to the hydrogenic ratio [12]. The computed values of nephelauxetic ratio (β') and bonding parameter ($b^{1/2}$) are 0.94626 and 0.163916 respectively (**Table 1**).

The intensity of the absorption bands can be expressed in terms of oscillator strengths. The experimental oscillator strengths can be obtained by integrating the area of each band in the absorption spectra following the expression given below [10],

$$f_{exp} = \frac{2.303mc^2}{N\pi e^2} \int \varepsilon(\nu)d\nu = 4.318 \times 10^{-9} \int \varepsilon(\nu)d\nu$$

where $\varepsilon(\nu)$ is the molar absorptivity (in cm^{-1}) of the corresponding transition, c is the velocity of light, ‘ m ’ and ‘ e ’ are the mass and charge of the electron and N is the Avagadro’s number. According to Judd – Ofelt theory, the calculated oscillator strength of an electric dipole transition from ground state to excited state can be calculated by using the equation,

$$f_{cal} = \left[\frac{8\pi^2 m c \nu}{3h(2J+1)} \right] \left[\frac{(n^2+2)^2}{9n} \right] \times \sum_{\lambda=2,4,6} \Omega_{\lambda} (\psi J \| U^{\lambda} \| \psi' J')^2$$

where, n is the refractive index, Ω_{λ} ($\lambda = 2, 4, 6$) are the Judd – Ofelt intensity parameters which are used to examine the nature of the metal – ligand bond and $\| U^{\lambda} \|^2$ are the reduced square matrix elements of the unit tensor operator of rank $\lambda = 2, 4$ and 6 respectively and J is the angular momentum of the ground state. The Judd – Ofelt parameters are obtained for the all glass samples using the spectral intensities of observed absorption bands and doubly reduced matrix elements using least – square fit approximation. The Judd – Ofelt parameters for the present glass system have been presented in **Table 2**. These parameters show the trend $\Omega_6 > \Omega_4 > \Omega_2$. Similar results were reported in literature [13, 14].

Table 2. Judd – Ofelt intensity of the Borosilicate glasses with Pr^{3+} ion.

Glass system	Ω_2 (10^{-20})	Ω_4 (10^{-20})	Ω_6 (10^{-20})	Trend	Ω_4/Ω_6
A	1.6454	5.2386	7.4320	$\Omega_6 > \Omega_4 > \Omega_2$	0.7049
B	0.9022	5.4198	7.5118	$\Omega_6 > \Omega_4 > \Omega_2$	0.7215
C	0.9036	5.3578	7.4899	$\Omega_6 > \Omega_4 > \Omega_2$	0.7153
D	1.0072	5.6047	7.6840	$\Omega_6 > \Omega_4 > \Omega_2$	0.7294
E	0.7458	6.4569	7.5224	$\Omega_6 > \Omega_4 > \Omega_2$	0.8583
F	0.6799	5.1632	7.1323	$\Omega_6 > \Omega_4 > \Omega_2$	0.7239
G	0.6892	6.0706	7.3272	$\Omega_6 > \Omega_4 > \Omega_2$	0.8285
H	1.1030	6.8017	8.1427	$\Omega_6 > \Omega_4 > \Omega_2$	0.8353
I	0.9358	6.3063	7.7699	$\Omega_6 > \Omega_4 > \Omega_2$	0.8116

The Ω_2 parameter gives information about the glass structure and depends upon the Pr – O bond in the host matrix. The Ω_4 and Ω_6 corresponds to the rigidity of the medium. The ratio of Ω_4 and Ω_6 parameters is known as spectroscopic quality factor (χ) and given in the **Table 2** along with the intensity parameters. It is the measure of emission strength of the glasses and used to identify the optical quality of the prepared glasses [10]. Also the spectroscopic quality factor is related to the rigidity of the glass system and for present glass samples its values lie in between 0.7 to 0.9. The values of spectroscopic quality factor show that the prepared glasses are rigid in compare to other glasses [15].

Table 1. Various energy parameters of the Borosilicate glass with Pr^{3+} ions.

PARAMETERS	FREE IONS	A	B	C	D	E	F	G	H	I
$F_2(\text{cm}^{-1})$	305	304.771	304.782	304.787	304.782	304.760	304.794	304.754	304.760	304.765
$F_4(\text{cm}^{-1})$	51.88	42.074	42.075	42.076	42.075	42.072	42.077	42.071	42.072	42.073
$F_6(\text{cm}^{-1})$	5.321	4.605	4.605	4.605	4.605	4.605	4.605	4.605	4.605	4.605
$\zeta_{4f}(\text{cm}^{-1})$	730.5	910.757	907.174	907.174	907.174	914.280	901.782	916.041	914.280	912.518
$E^1(\text{cm}^{-1})$	4728.92	4474.580	4474.744	4474.825	4474.744	4474.419	4474.923	4474.338	4474.419	4474.500
$E^2(\text{cm}^{-1})$	24.75	23.406	23.407	23.408	23.407	23.406	23.408	23.408	23.406	23.406
$E^3(\text{cm}^{-1})$	478.1	452.401	452.418	452.426	452.418	452.385	452.436	452.377	452.385	452.393
F_4 / F_2	0.14	0.138	0.138	0.138	0.138	0.138	0.138	0.138	0.138	0.138
F_6 / F_2	0.02	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015	0.015
E^1/E^3	9.89	9.891	9.891	9.891	9.891	9.891	9.891	9.891	9.891	9.891
E^2/E^3	0.05	0.052	0.052	0.052	0.052	0.052	0.052	0.052	0.052	0.052
β'	0.946	0.946	0.946	0.946	0.946	0.946	0.946	0.946	0.946
$b_{1/2}$	0.164	0.164	0.164	0.164	0.164	0.164	0.164	0.164	0.164

Conclusion

Present Praseodymium doped borosilicate glasses with varying concentration of SiO₂ were prepared by standard melt quenching technique. The samples were analyzed by XRD, FTIR, EDX, SEM and TEM. The amorphous nature of the samples was confirmed by XRD and SEM images. FTIR gives the information about the various functional groups in prepared glass samples and B-O-B network of the prepared glass is present in the wavelength range 500 – 1600 cm⁻¹ and further confirmed by TEM image. Physical and optical properties of the samples were calculated. Density, refractive index, Pr³⁺ ion concentration, OPD decreases with increasing SiO₂ concentration while field strength, inter ionic separation, molar volume increases with increasing concentration of SiO₂. Decrease in the value of OPD with SiO₂ concentration shows that structure is loosely packed. Optical band gap and Urbach's energy are also calculated for the present glass. Various energy interaction parameters viz. Slater – Condon, Racah, Lande, Bonding and Judd- Ofelt parameters were also computed for all the different concentration of SiO₂. Judd – Ofelt intensity parameters shows the general trend $\Omega_6 > \Omega_4 > \Omega_2$. The Ω_2 parameter defines the glass structure and depends upon the Pr – O bond in the glass host. Spectroscopic quality factor (Ω_4/Ω_6) is defines the rigidity of the glass samples and its values show that the prepared glasses are rigid in comparison to other glasses.

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