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Effect of CdO on physical and optical properties of cadmium silicon bismuthate glasses

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ABSTRACT

Glasses with composition xCdO.ySiO$_2$.yBi$_2$O$_3$ (with *x=0, y=50; x=10, y=45; x=20, y=40* and *x=30, y=35* mol %) were synthesized using conventional melt quenching technique. The variation in physical and optical properties has been studied as a function of CdO content in cadmium silicon bismuthate glasses. The amorphous nature was confirmed by X- ray diffraction. The changes in physical properties like density, molar volume, oxygen packing density (OPD) and crystalline volume (V_c) shows structural modification on addition of CdO. Thermal properties like glass transition temperature (T_g) and crystallization temperature (T_p) were determined by differential scanning calorimetry (DSC) and values of T_g were found to increase while that of T_p decrease as substitution of CdO increases. The fundamental absorption edge has been found out from the optical absorption spectra. The optical band gap, E_g, for indirect allowed transitions has been determined and its value lies between 2.70-2.76 eV and found to decrease with increase in CdO content. The systematic study of structure was carried out by Infrared and Raman spectroscopy which shows that these glasses are made of [BiO_3], [BiO_6] and [SiO_4] structural units and both symmetric as well as asymmetric stretched vibrations of Si-O bonds in SiO_4 were present.

Keywords: Bismuth silicate glasses, FTIR, Glass transition temperature, RAMAN, optical properties.

INTRODUCTION

Bismuthate glasses are of great interest because they can be used to produce glass ceramics, reflecting windows, optical switches, optical waveguides, photovoltaic cells and optoelectronic devices etc.[1] These glasses are suitable materials for investigations related to optical properties and also to structural and electrical properties as stable glasses are formed by them through normal melt quench techniques even in the absence of classical glass former. However, most of the bismuthate glasses reported till now are multi component. It has been noticed due to large polarizibility of bismuth ions its cations in the presence of classical glass-forming cations such as Si^{4+}, B^{3+} P^{5+}, ,etc. may occur in the network of glass in [BiO_3] (3,6) pyramidal units.[2-8] Bismuth silicate glasses are interesting due to their industrial and other applications like low loss fiber optics, active infrared luminescence or as active medium of Raman fiber optical amplifiers and oscillator.[9-10] Batel et al. has observed the IR spectra, thermal properties and density values of bismuth- silicate matrix and found that Bi^{3+} exist in form of BiO_6 and BiO_3 structural units. [11] Yang et al. also reported that Bi_2O_3-SiO_2 glasses has a special property of change of refractive index on exposure to laser which makes them promising candidate for optics.[12]

Nowadays, the glasses having transition metal ion such as CdO, V_2O_5 and ZnO have attained great significance because of their various applications in different fields. Several reports are found on CdO-B_2O_3-SiO_2, SiO_2-ZnO-Bi_2O_3, V_2O_5-Bi_2O_3-B_2O_3, V_2O_5-CdO and V_2O_5-ZnO-Bi_2O_3 systems in literature [8-16] but very few studies are available on glass system CdO-SiO_2-Bi_2O_3. In this paper, work has been done for the synthesis and optical characterization of silicon bismuthate glasses in the presence of cadmium oxide. The optical properties of present glass system were studied by analyzing UV-VIS absorption spectra, Raman and IR spectra. The values of density, molar volume, crystalline volume, transition temperature etc. were also reported.

EXPERIMENTAL DETAILS

The glasses were prepared by normal melt quenching technique. For preparations of studied samples, the weighed quantities of CdO, SiO_2 and Bi_2O_3 were mixed in appropriate proportions to yield about 20 g in a silica crucible. The mixture was melted in an electrical muffle furnace at 1100°C for about 30 min. The melt was swirled frequently to assure homogeneity. The obtained melt was quickly quenched by pouring it between two stainless-steel plates, as a result of which coin-shaped glasses having thickness about 1–2 mm were formed. The glasses so

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obtained were yellow in colour. The XRD plots were obtained to confirm the amorphous nature of glasses using Rigaku Miniflux-II diffractometer at room temperature in the presence of Cu Kα radiation and the values of 2θ lies in the range 10–80° with scanning rate of 2°/min.

Density (ρ) of each sample was measured at room temperature (300 K) using the Archimedes method with deionized water as the buoyant liquid. The molar volume of each composition was calculated as V_M = M/D, where M and D are molecular weight and density respectively. The values for glass transition temperature (T_g) for present glasses were measured through differential scanning calorimeter model number (Q600,TA instrument) with heating rate of 10°C/min at 200-1000K temperature range in the atmosphere of inert nitrogen gas (N_2). The IR spectra of present glasses in transmission mode over the range 400-4400 cm^{-1} were recorded by a Perkin-Elmer spectrometer model number BX-II. For this purpose, pellets in form of discs were formed of powered samples by mixing them with dry KBr in proportion of 1:20 by weight and applying pressure of 9-10 tones. The optical transmission spectra were recorded of prepared pellets. The optical absorption spectra of all compositions were recorded at room temperature on a UV-VIS-NIR spectrometer (Cary 5000) over the wavelength range 200-3300nm. The Raman measurements were carried out in the range 100–4000 cm^{-1} at room temperature using a micro Raman system from BRUKER: RFS 27 spectrometer.

RESULTS AND DISCUSSION

3.1 XRD and Physical properties

X-ray diffraction is one of the simplest techniques used by various researchers to establish the amorphous nature of the glass. The diffraction pattern having sharp Bragg peaks corresponds to the crystalline nature of samples whereas broad hump in the diffractogram governs to the amorphous nature**.** The X-ray diffraction (XRD) patterns for xCdO-$y$$SiO_2$-$y$$Bi_2O_3$with $0 \leq x \leq 30$ (in mol%) and $35 \leq y \leq 50$ (in mol%) at room temperature are shown in Figure 1. The absence of sharp Bragg peaks and presence of broad hump in X- ray spectra confirms the amorphous nature of prepared glasses. Thus, these glasses lacks in long range atomic periodicity in their networks. The value of density plays an important role in determining structure of glasses. The value of density has strong co-relations with various parameters like molar volume, glass transition temperature, crystalline volume etc. The density varies from 6.39 g/cm^{-3} to 4.04 g/cm^{-3}, and the molar volume varies from 41.16 cm^{-3}/mol to 55.11cm^{-3}/mol in present glasses as shown in Table 1. The variation in density is opposite to as that of molar volume V_M, i.e., a monotonic decrease is observed with increase in the substitution of CdO in the present glasses. This is expected trend as molecular weight of CdO (128.41 g/mol) is more than SiO_2 (60.08 g/mol) but very less than that of Bi_2O_3 (465.98 g/mol) i.e. heavier oxide is replaced by lighter one. This is also reported by various researchers in their study and observed monotonous changes in these two parameters show the presence of single-phase random network structure in the glass compositions.[16,17]

The volume occupied by crystalline phases in glass network is known as crystalline volume which is given by $V_c = \sum x_i V_i$ where V_i represents the molar volume of i^{th} component in crystalline phase and values of V_i as obtained from literature[10] are 15.76, 25.79 and 52.36 for CdO, SiO_2 and Bi_2O_3 respectively. The variation of V_c and V_M with change in the concentration of CdO content is shown in Figure 2 and the calculated values of V_c is also incorporated in Table 1. As clearly observed from the Table with increase in the substitution of CdO in glass network molar volume increases and crystalline volume decreases which shows that non crystalline i.e. amorphous nature dominates over crystalline. The values of molar volume changes rapidly as we go from CSB1 to CSB2. This may be due to absence of CdO in CSB1.

One more quantity represented in the Table 1 is oxygen packing density (OPD) which governs the tightness in packing of oxide network and is calculated by using formula

$$\mathrm{OPD} = \frac{\mathrm{D}}{\mathrm{M}} \times \mathrm{N}_0$$

here N_0 is the number of oxygen atom per formula unit, D and M corresponds to density and molecular mass.[18] The values of OPD are found to decrease with increase in the content of CdO which is strongly related with decrease in the values of density.

3.2 Differential Scanning Calorimetry (DSC)

DSC is a thermos-analytical technique in which difference in the quantity of heat required to increase the temperature of sample and reference is measured with reference to temperature. During measurement both sample and reference are maintained at almost same temperature and for this purpose the sample undergoes a phase transitions i.e. more or less heat will flow to it which depends upon whether the process is endothermic or exothermic. This technique is widely used to determine various parameters like glass transition temperature, crystallization temperature, melting point etc. The values of glass transition temperature (T_g) plays a important role in determining glass forming tendency and thermal stability of glasses. The glass transition temperature T_g increases with increasing CdO content as reported in Table1 which shows that glass forming tendency and thermal stability also increases with increase in cadmium content. This results contradict to the results obtained from density and molar volume measurements which clearly indicate structure openness. This may be due to the fact that glass transition temperature also depends upon other physical and optical parameters such as type of structural units, bond length, bond strength, optical band gap etc. The DSC thermograph of the prepared glasses is shown in Figure 3. The curve clearly shows an endothermic shift and values of glass transition temperature (T_g) can be obtained by middle of this shift along x-axis.[20] The values correspond to exothermic peaks gives the values of crystallization temperature (T_p). Two exothermic peaks are observed corresponding to sample CSB1 but on addition of CdO in glass matrix this peak reduces to one which again confirms that glassy or amorphous nature of sample increases with increase in CdO content. This result is in agreement with the trends observed in density, crystalline volume etc. The values of crystallization temperature are also shown in Table 1.

Table 1 Density (D), Molar volume (V_M), crystalline volume (V_c), excess volume (difference between V_M and V_C , ΔV), oxygen packing density (OPD), glass transition temperature (T_g), crystallization temperature (T_p) and thermal stability (ΔT) for xCdO-$y$$SiO_2$-$y$$Bi_2O_3$ glass system

Sample code	x mol %	y mol %	D g/cc	V_M cc/mol	V_C cc/mol	ΔV	OPD g-atom/l	T_g (ºC)	T_p (ºC)	ΔT (ºC)
CSB1	0	50	6.39	41.16	39.08	2.08	60.73	460	662	202
CSB2	10	45	5.72	43.63	36.74	6.89	53.86	463	670	207
CSB3	20	40	4.92	47.99	34.41	13.58	45.84	469	723	254
CSB4	30	35	4.04	55.11	32.08	23.03	37.20	484	710	226

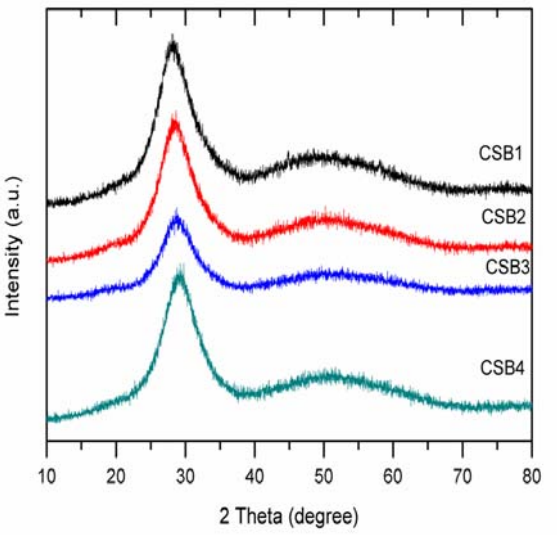


Figure 1. X- Ray Diffraction pattern for xCdO-$y$$SiO_2$-$y$$Bi_2O_3$ glasses at room temperature

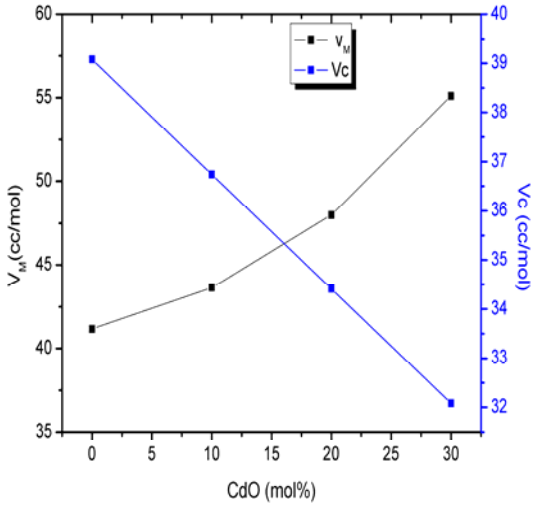


Figure 2. Variation of molar volume V_M and crystalline volume V_c with CdO concentration for xCdO-$y$$SiO_2$-$y$$Bi_2O_3$ (with x=0,y=50; x=10,y=45; x=20,y=40 and x=30, y=35 mol%) glasses

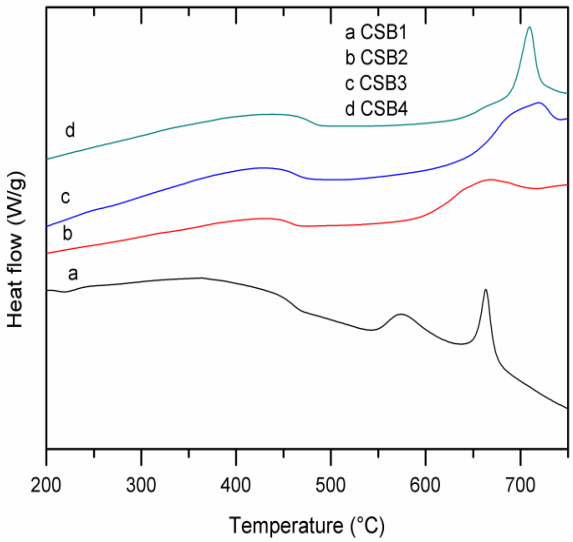


Figure 3. DSC thermographs of xCdO-ySiO_2-yBi_2O_3 glasses at heating rate 10°C/min.

3.3 Structural properties

FTIR and Raman are two spectroscopic techniques used by various researchers to study the structure of glasses. The major difference between these two techniques is the type of molecular transition occurring in them. The transition which involves change in the polarizibility of molecule during vibration are Raman active while for a molecule to show IR transition there must be change in its dipole moment. Symmetric molecules can't show IR transition since they do not undergo change is dipole moment which is foremost condition for a molecule to be IR active. Our present samples were also analyzed these two spectroscopic techniques; the results are shown in Figure 4 and 5 respectively. The FTIR spectrum of the xCdO-ySiO_2-yBi_2O_3 glasses contains three vibrational bands at 496-505 cm^{-1}, at 895-913 $cm^{-1}$and at around 1630 cm^{-1}. The first broad band at 496-505 cm^{-1} is observed which shift to higher wave number (505 cm^{-1}) with increase in cadmium content or decrease in bismuth content. This band is formed due to Bi-O bonds in [BiO_6] octahedra and increase in the degree of distortions is the reason of this observed shifting to higher wave number.[19,21] Dimitriev and Mihallova also assigned this shifting of the band from lower (485 cm^{-1}) to higher wave number (523 cm^{-1}) is to the changes occurs in local

symmetry of highly distorted [BiO_6] polyhedral, as decrease in Bi_2O_3 content.[22] Some authors suggest that this band originate due to the Si-O-Si bending vibration .Thus, the band at around 496 cm^{-1} may be because of the combination of Bi vibration in [BiO_6] and the Si-O bend vibrations.[22] A broad band in the range 895-913 cm^{-1} is assigned to Si-O-Si asymmetric vibrations in SiO_4 tetrahedra and also due to Bi-O vibration in [BiO_3].[23]The presence of peak at 840 cm^{-1} is not observed in present system which suggests formation of Cd^{2+} in tetrahedral co ordination (i.e.CdO_4) is absent. The band at around1630 cm^{-1} is observed in the spectra for all synthesized glasses is because of adsorption of water in the powdered samples.[23-24]

Along with IR spectroscopy Raman spectroscopy also proves a important technique to obtain information regarding structure of molecules. Sometimes a transition forbidden in IR is allowed in Raman spectra which enhance its utility. For obtaining precise information we have deconvoluted Raman spectra. Figure 6 shows the deconvoluted Raman spectra for CSB1 glass. Similar curves are obtained for other glasses of present system with very little difference in peaks. The Gaussian distribution is used to deconvolute broad peak into six peaks. The Raman spectrum of present glasses consist of four predominant bands in the region 70-140 cm^{-1}, 200-250 cm^{-1}, 360-470 cm^{-1} and 910-1010 cm^{-1}. As clearly observed from the Figure 5 Raman intensity increases as substitution of CdO increases which suggests that CdO plays the role of network modifier in present system which is also supported by FTIR analysis. For all synthesized compositions peaks at nearly 75cm^{-1}, 120 cm^{-1} are observed and is attributed to the vibrations of motions of Bi^{3+} cations in [BiO_6] and [BiO_3] polyhedral.[25-26] As content of CdO increases in glass matrix these bands are accompanied by increase in intensity. The band 200-250 cm^{-1} centred at 204 cm^{-1} can be attributed to the Bi-O-Bi and Bi-O vibrations in [BiO_6] octahedral.[22,27] A shift towards higher wave number (204 cm^{-1} to 235 cm^{-1}) is observed as content of CdO increases in glass matrix. The band centred at around 360 cm^{-1} is associated with the Bi-O-Bi and Bi-O stretching vibrations of [BiO_6] units.[22,27] Some authors also relate this band to the asymmetric bending vibrations of Si-O-Si in SiO_4 units[28-29] which is also confirmed by FTIR spectroscopy. The band centred around 910 cm^{-1}, 920 cm^{-1} (for CSB1), 924 cm^{-1} (for CSB2), 927 cm^{-1} (for CSB3) and 925 cm^{-1} (for CSB4) is related SiO_4 tetrahedral symmetric stretching vibrations[28] in the units having two non bridging oxygen atoms (NBOs) per silicon which is also supported by presence of presence of band in the range 895-913 cm^{-1} due to Si-O-Si asymmetric vibrations in SiO_4 tetrahedra in IR spectra.[25] Similar results are obtained in other cadmium oxide based glasses.

Thus, from the analysis of Raman and FTIR spectroscopy of present glass system it is observed that present glass system consists of [BiO_6], [BiO_3] and [SiO_4] structural units and formation of tetrahedral coordination i.e. CdO_4 is absent in this system. The present study also shows that in this glass system symmetric and well as asymmetric stretched vibrations of SiO_4 exists and symmetric vibrations dominates with increase in cadmium oxide content which may be attributed due to replacement of larger bismuth oxide molecules by smaller cadmium oxide molecules. This results into increase in the compactness of glass network which is also observed by increase in values of T_g as content of CdO increases and decrease in the value of V_c and density.

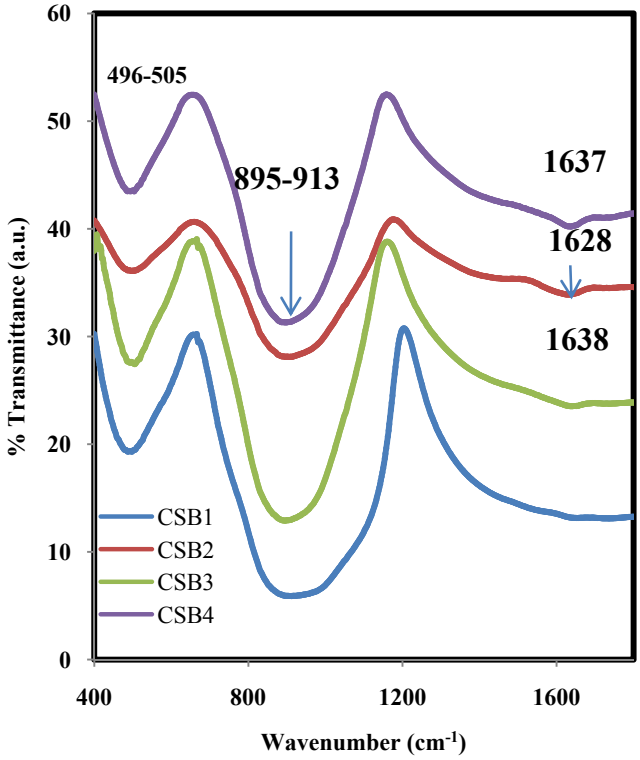


Figure 4. FTIR spectra of $xCdO\text{-}ySiO_2\text{-}yBi_2O_3$ glass samples.

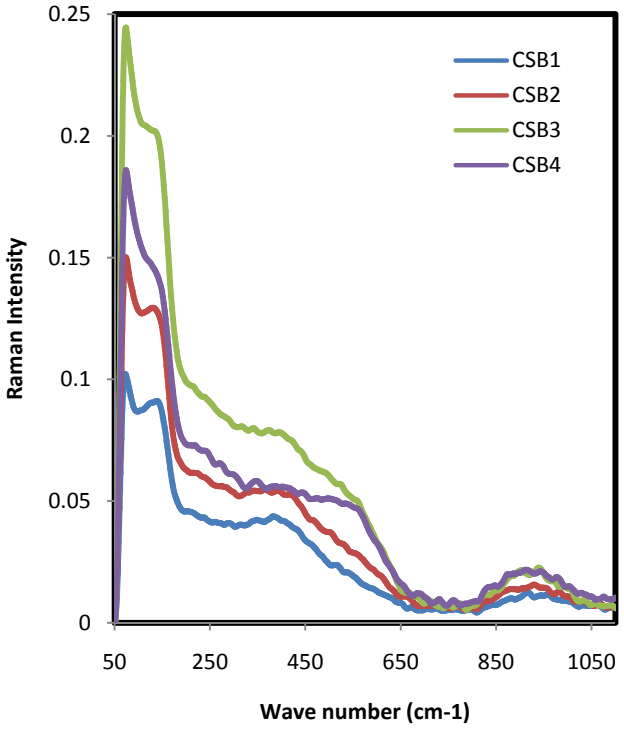


Figure 5. Raman spectra of $xCdO\text{-}ySiO_2\text{-}yBi_2O_3$ (with *x=0,y=50; x=10,y=45; x=20,y=40* and *x=30, y=35* mol%) glasses.

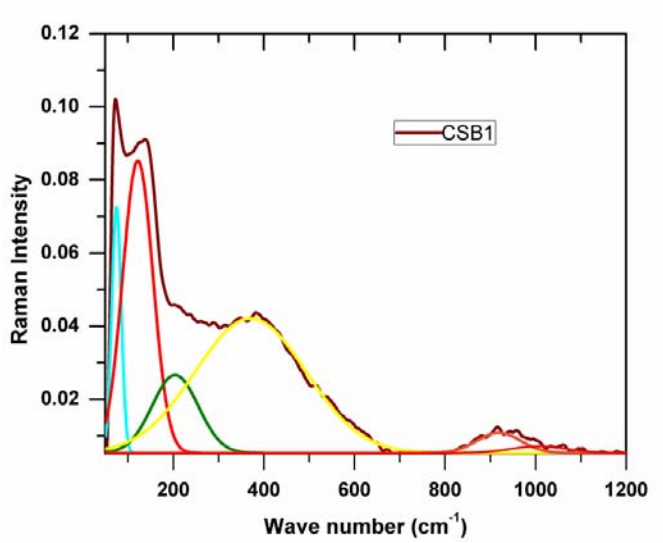


Figure 6. Deconvoluted Raman plot of sample CSB1 with R^2= 0.992

3.4 Optical properties

The absorption spectra is very important tool to find out various information like optical band gap, cut-off wavelength, band tailing parameter, refractive index and polarizibility etc.

The optical absorption and transmission spectra of polished glasses at room temperature in the region 200-3200 nm is recorded and Figure7 shows optical absorption spectra in the spectral range 380-600 nm. It is clearly observed that these glasses possess large optical transmission window and no sharp absorption edge is observed which confirms the glassy nature, also confirmed by IR and XRD. The values of cut off wavelength (λ_{cutoff}) has been calculated from Figure 7 and are reported in table 2. The cut off wavelength (λ_{cutoff}) is found to decrease with increase in the content of CdO which shows that glass network became tightly packed or compact which is also clear by values of T_g.

The absorption coefficient α(ν) was determined from relation[30]:

$$\alpha(\nu) = \left(\frac{1}{d}\right) \ln\left(\frac{I_0}{I_t}\right) \qquad (1)$$

where I_0 is the intensity of incident beam, I_t represent intensity of transmitted beams and d corresponds to the thickness of glass sample under study. The factor $\ln(I_0/I_t)$ corresponds to absorbance, A. The cut-off wavelength (λ_{cutoff}) and the optical band gap (E_g) were calculated for each sample and are shown in Table 2.

The data for Figure 8 (Tauc's plots) were obtained from the relation given by Davis and Mott[31]:

$$\alpha(\nu) = \frac{B(h\nu - E_g)^r}{h\nu} \qquad (2)$$

where E_g represents optical band gap and r corresponds to the index which can have different values depending on interband transition. The value 2 is assigned to the indirect allowed transitions, 3 is used for indirect forbidden transitions, 1/2 and 1/3 refers to direct allowed and direct forbidden transitions, respectively. The symbol B is a constant is known as band tailing parameter and hν is the energy of incident photon.

The value of E_g has been determined from the linear region of curves by extrapolating them to meet the hν axis at $(\alpha h\nu)^{1/2}$=0.

The calculated values of both optical band gap (E_g) and band tailing parameter (B) for r=2 (indirect allowed) and for r=3 (indirect forbidden) are portrayed in Table 2. The variation as clearly observed from the table is non linear which shows that dual role of network former as well as network modifier is played by CdO in glass matrix. However there is overall decrease in values of E_g. This may be due to structural changes occurs in glass matrix on addition of CdO.[32] This decrease can be explained in terms of increase in concentration of non bridging oxygen (NBOs). [17,33-34] These non bridging oxygens are very energetic and contribute more in the valence band and as a result of this valence band shifts a bit upward which in turn reduces the optical band gap.[35-37]

The values of refractive index for synthesized glasses have been calculated with the help of optical band gap using relation given by Dimitrov and sakka [38-39]:

$$\frac{n^2 - 1}{n^2 + 2} = 1 - \sqrt{\frac{E_g}{20}} \qquad (3)$$

The calculated value of linear refractive index of all the glass samples is given in Table2. It is observed that 'n' increases with an increase in the CdO content in the present glass system and lies in the range from 2.465-2.483. This is because of increase in the concentration of (NBO) atoms and polarity of such atoms is higher than that of bridging oxygen atoms.

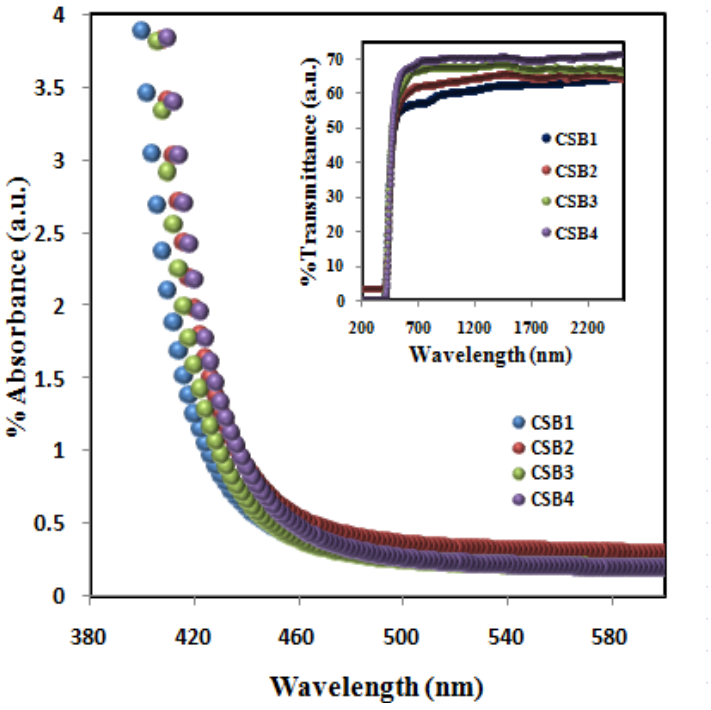


Figure 7 Optical absorption and transmission spectra for all glass compositions.

Table 2 : Cut-off wavelength ($\lambda_{cut\text{-}off}$), optical band gap (E_{opt}), band tailing (B), refractive index (n) and Urbach energy (ΔE) for xZnO·ySiO$_2$·yBi$_2$O$_3$ glasses.

Sample Code	$\lambda_{cut\text{-}off}$ (nm)	E_g (eV)		B (cmeV)$^{-1/r}$		n	ΔE (eV)
		$r = 2$	$r = 3$	$r = 2$	$r = 3$		
CSB1	415	2.76	2.61	51.62	13.66	2.465	0.154
CSB2	413	2.71	2.56	29.35	9.45	2.480	0.157
CSB3	411	2.73	2.63	47.12	14.52	2.474	0.132
CSB4	409	2.70	2.55	30.93	9.74	2.483	0.148

The optical band gap data is also helpful to determine band structure and band energy gap both in case of crystalline as well as amorphous materials. The optical absorption sometimes photons having energy greater than band gap energy get absorbed which leaves behind an absorption edge with exponential increase in the values of absorption co-efficient, α(ν). The increase in the values of α (ν) is related to hν is determined using Urbach rule[40] which is as follows.

$$\alpha(\nu) = \alpha_0 e^{\frac{\nu}{\Delta E}} \qquad (4)$$

here α_0 is a constant and ΔE is the Urbach energy. The Urbach's plot (i.e. $h\nu$ vs. ln(α)) for sample CSB1 is shown in Figure 9; the reciprocal of slope of Urbach plot gives the value of Urbach energy, ΔE. From the values of Urbach energy the information regarding the disorder effects in both crystalline and amorphous materials can be obtained. Due to short range order or lack of long range in amorphous materials there occurs tailing of density of states which can be obtained from the values of urbach energy. It is associated with the transitions between localised tail states near valence band to the extended states in the conduction band. The obtained values of Urbach energy (ΔE) lies in the range 0.132 to 0.157 eV and are displayed in Table 2. This broad width is due to well known phonon assisted indirect electronic transitions as reported by various researchers in literature.[41] Greater the value of ΔE, greater will be the tendency of material to convert weak bonds into defects.

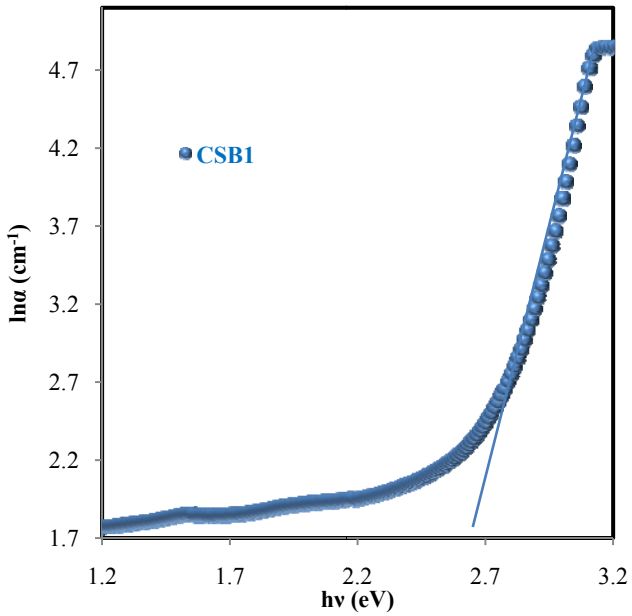
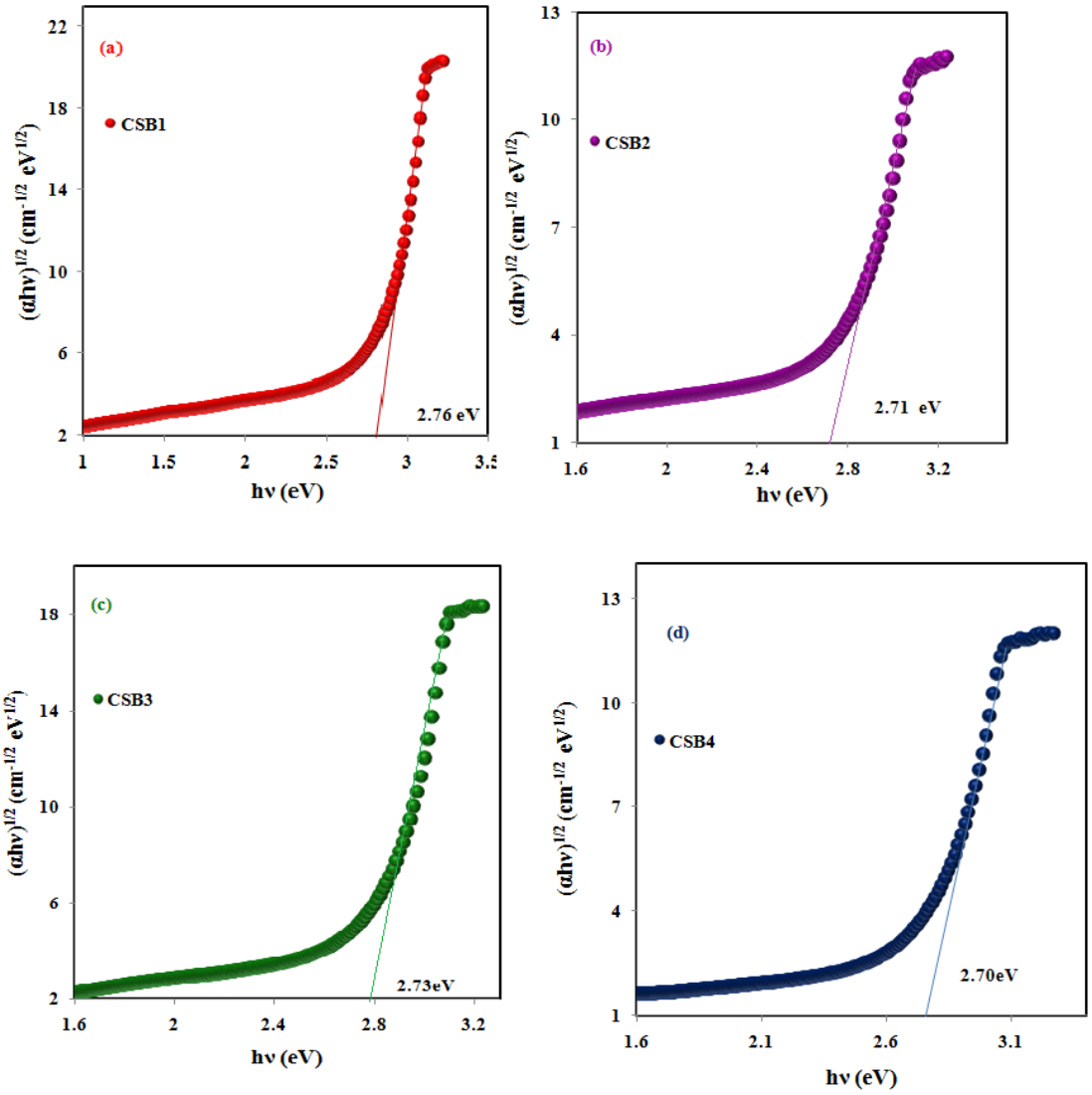


Figure 9 Urbach energy plot for sample CSB1

CONCLUSIONS

The physical, structural and optical properties of xCdO-ySiO$_2$-yBi$_2$O$_3$ (with x=0, y=50; x=10, y=45; x=20, y=40 and x=30, y=35 mol %, CSB1-4) glasses have been studied and following conclusions are drawn:

- The presence of broad spectrum in X-ray di□ractograms confirms the amorphous nature of studied glass system. The variations observed in various physical parameters like density, oxygen packing density, crystalline volume etc. shows that CdO has a strong influence on glass matrix.



- The IR and Raman spectra reveals that the glass network built of [BiO_6], [BiO_3] and [SiO_4] structural units and formation of tetrahedral coordination i.e. CdO_4 is absent in present glass system.
- A non-linear variation in optical band gap and refractive index indicate that dual role is played by Cd^{2+} ions in glass matrix.
- The presence of phonon-assisted indirect electronic transitions is predicted by broad width of absorption tail (ΔE).

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