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Optical properties of ternary zinc magnesium phosphate glasses

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Abstract

Ternary phosphate glasses of the system $(ZnO)_{30}(MgO)_x(P_2O_5)_{70-x}$ with x ranging from 5 to 20 mol% were prepared by melt quenching technique. The optical absorption spectra of these glasses were measured at room temperature in the wavelength range between 190 and 1100 nm while the refractive index at wavelength 632.8 nm. The optical absorption indicates that the electronic transition is indirect and associated with phonon-assisted transition. From the absorption spectra, the optical energy band gap (E_{opt}) and Urbach energy (E_U) values for all the glass samples were calculated from their ultraviolet edges. The values of E_{opt} is found to increase from 3.36 to 3.44 eV and values of E_U decrease from 0.47 to 0.29 eV with the increase of MgO content. Variation in these optical parameters, density and molar volume is discussed and correlated with the structural changes within the glassy matrix.

Keywords: C. Optical properties; Phosphate glasses; Refractive index

1. Introduction

Phosphate glasses have several advantages over conventional silicate and borate glasses due to their superior physical properties such as high thermal expansion coefficient, low melting and softening temperatures and high ultra-violet (UV) and far infrared transmissions [1]. Among oxide glasses, phosphate glasses are both scientifically and technologically important materials because of their structural versatility to accept several cation and/or anion exchanges. These features allow the reengineering of glass formulation, which leads to advances in their physical properties and optimizing of processing parameters [2]. Earlier works on alkali metal oxide or a divalent metal oxide phosphate glasses have revealed that the absorption edge fall in the UV region below 400 nm which meet with the requirement for desirable applications in optical system [3].

The study of optical absorption spectra provides a very productive method for investigating optically induced electronic transitions and an insight into the energy gap and band structure of crystalline and amorphous materials [4]. The principle of this technique is that a photon with energy greater than the band gap energy will be absorbed [5,6]. Absorption and transmission in the ultraviolet, visible and infrared regions are important in optical instruments, where the absorption in all three regions can be used to study short-range structure of glasses that is the immediate surrounding of the absorbing atom [7].

Glasses are among the few solids which transmit light in the visible region of the spectrum, and therefore refractive index becomes one of the most important properties in optical glasses. Since glass is the basic elements in all optical instruments, refractive index is a fundamental parameter that strongly relevant to optical devices performance and reliability. Hence, a large number of researchers have carried out investigations to ascertain the relation between refractive index and glass composition. This is because the right selection of glass composition through careful compositional control and determination of the chemical elements that added into the host will control the optical properties and performance of the glass [8].

$BeO - P_2O_5$, $MgO - P_2O_5$ and $ZnO - P_2O_5$ glasses have been identified as glasses that exhibited unusual behaviour in their physical properties (mass density, refraction index) at the metaphosphate composition [9,10]. Thus, it is of interest to investigate the physical, refractive and optical properties of the

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simultaneous admixture of MgO and ZnO into the phosphate compositions, since there has been no attempt to observe the properties in these compositions. In the present investigation, a series of glasses $(ZnO)_{30}(MgO)_x(P_2O_5)_{70-x}$ were prepared with the variation of MgO content while kept the value of 30 mol% ZnO constant. A systematic study has been performed to understand the variation of optical band gap and Urbach energy as a function of composition in mixed zinc magnesium phosphate glasses. Further, other physical parameters namely, refractive index, density and molar volume have also been evaluated as supplement to throw more light to elucidate the structural evolution of the newly prepared glasses.

2. Materials and methods

The glasses were prepared by melt-quenching technique using chemical powders, phosphorus (V) oxide (99.99% purity), magnesium oxide (98% purity) and zinc oxide (99.7% purity) as raw materials. Ternary $(ZnO)_{30}(MgO)_x(P_2O_5)_{70-x}$ glasses were synthesized for $5 \leq x \leq 20$ (x gives the content of MgO in mole basis). Stoichiometric mixture of ZnO, MgO and P_2O_5 were weighed and mixed thoroughly in a 50 cm^3 alumina crucible in appropriate quantities to constitute a 15–20 g batch. The crucible was covered and heated in an electric furnace for about 1 h at a temperature of 673 K to allow the phosphate to decompose and react with other batch constituent before melting occur, ordinarily. The crucible was then transferred to another electric furnace and kept at 1473 K for 2 h. The melt was stirred occasionally every 20 min to ensure homogeneity and proper mixing. Next, each melt was cast into a preheated stainless steel circular-shaped mould and immediately pressed with a stainless steel plate. In this way circular disk-shaped samples with a diameter ≈1.5 cm could be obtained. After casting, each glass was immediately transferred to an annealing furnace, and held at temperature of 673 K for 1 h and slowly cooled to room temperature.

The amorphous nature of these glass samples was established and confirmed using X-ray diffraction (XRD; PANalytical X'pert PRO). The glasses that were successfully prepared were transparent and free of bubbles. The nominal composition of glasses in this study is listed in Table 1 with the designation for the respective mole fraction. Samples for optical property measurements were finely polished in order to obtain smooth and parallel surfaces. The thickness of the glass specimens was measured using a digital micrometer gauge and the average thickness was ≈0.950 mm. These samples were stored in a desiccator and taken out only at the time of measurement of their properties.

Optical absorption spectra were recorded at room temperature using Shimadzu UV-1650 PC spectrophotometer in the wavelength region 190–1100 nm at normal incidence. A software UVProbe calculates and record all measurements.

Refractive index of these glasses was measured using EL X-02C High Precision Ellipsometer equipped with a transmitter units and a receiver unit fixed at the end of adjustable arms at angle of 70°. The transmitter unit consists of a helium–neon laser of 632.8 nm wavelengths as the light source with accuracy of 0.004° for delta (Δ) and 0.02° for psi (Ψ). Measurements were performed at room temperature and the data of the refractive indices of the glasses were obtained by analyzing via the applicable software.

Density (ρ) of the prepared glass samples was measured at room temperature by standard Archimedes method (apparent weight loss) using acetone as the immersion fluid. The sample was first weighed in air, g_a, and then in an immersion liquid (acetone) g_L, with density, $\rho_L = 0.789$ g cm^{-3}. The weighing process was measured by using electronic balance (AND GF-300, ±0.001 g). The density of the sample was then calculated using the relationship $\rho = g_a\rho_L/(g_a - g_L)$. The estimated error was ±0.001 g cm^{-3}.

The molar volume (V_m) of each sample was calculated from the relation $V_m = M_T/\rho$. M_T is the total molecular weight of the glass system taken as $M_T = X_{ZnO}Z_{ZnO} + X_{MgO}Z_{MgO} + X_{P_2O_5}Z_{P_2O_5}$, where X_{ZnO}, X_{MgO} and $X_{P_2O_5}$ are the mole fractions of the constituent oxide and Z_{ZnO}, Z_{MgO} and $Z_{P_2O_5}$ are the molecular weight of the constituent oxide.

3. Results and discussion

The ultra-violet (UV) absorption in insulating materials particularly in disordered materials such as oxide glasses is generally believed to involve the excitation of electrons associated with anions. In general, the prediction of the location of the ultraviolet absorption edge of oxide glasses is substantially complex and in particular such prediction in the case of phosphate glasses is expected to be more complex, since

Table 1
Composition and selected properties of the $(ZnO)_{30}(MgO)_x(P_2O_5)_{70-x}$ glasses.

Sample code	Composition (mol%)			Density, ρ (g cm^{-3}) (±0.001 g cm^{-3})	Molar volume, V_m (cm^3 mol^{-1}) (±0.001 cm^3 mol^{-1})	E_{opt} (eV) (±0.02 eV)	E_U (eV) (±0.01 eV)
	ZnO	MgO	P_2O_5				
CZ 05	30	5	65	3.228	36.768	3.37	0.47
CZ 08	30	8	62	3.118	37.008	3.36	0.39
CZ 10	30	10	60	3.045	37.309	3.39	0.40
CZ 13	30	13	57	2.937	37.643	3.41	0.34
CZ 15	30	15	55	2.905	37.358	3.41	0.32
CZ 18	30	18	52	2.815	37.469	3.43	0.34
CZ 20	30	20	50	2.712	38.143	3.44	0.29

structural changes occur with change in the composition [3]. The absorption coefficient $\alpha(\omega)$ below and near the fundamental absorption edge of each curve in Fig. 1 was calculated at wavelength intervals of 1.0 nm from the relation $\alpha(\omega) = (1/d)\ln(I_o/I_t)$ where d is the thickness of each sample, I_o and I_t are the intensities of incident and transmitted radiation, respectively, and $\ln(I_o/I_t)$ corresponds to absorbance, A.

The typical optical absorption spectra for ternary $(ZnO)_{30}(MgO)_x(P_2O_5)_{70-x}$ glasses with $x = 5$, 8, 10, 13, 15, 18, and 20 mol% as a function of wavelength are shown in Fig. 1. All these glasses exhibit an optical absorption band in the visible-near infrared region and a fundamental optical absorption edge in the ultraviolet (UV) region. It is observed that there is no sharp absorption edge and this is the characteristic of the glassy state. From the results presented, the absorption edges shift back and forth as the mole fraction increased. This observation has been detected before in phosphate glasses [11,12]. The absorption edge shifts back and forth with increasing alkaline earths content in phosphate glasses are most likely related to the structural rearrangement of the glass network and modifier and the relative concentrations of the various fundamental units as well [13]. It is attributed to the changes in the bonding that take place in the glassy network as the metal content increased [11].

For the high absorbing region, the absorption coefficient $\alpha(\omega)$ follows a power law given by Davis and Mott [14] as shown below:

$$\alpha(\omega) = \frac{B}{\hbar\omega}(\hbar\omega - E_{\text{opt}})^n \tag{1}$$

where B is a constant, E_{opt} is the energy of the optical band gap, $\hbar\omega$ is the photon energy of the incident radiation, and n is an index which can have values of 1/2, 3/2, 2 and 3, depending on the nature of the inter-band electronic transitions for the absorption [15]. For n equals to 1/2 is associated with allowed direct transitions, 3/2 is for direct forbidden transition, 2 is for allowed indirect transitions and 3 is for forbidden indirect transitions. The other region of the absorption edge at lower values of $\alpha(\omega)$ is often described by a relationship developed by Urbach [16]. He assumed that in this region, the absorption coefficient was an exponential function of the photon energy, $\hbar\omega$ and therefore

$$\alpha(\omega) = \alpha_o \exp\left(\frac{\hbar\omega}{E_U}\right) \tag{2}$$

where α_o is a constant and E_U is the Urbach energy.

Graphs of optical absorption coefficient, α, against photon energy, $\hbar\omega$, of the studied glasses were plotted and shown in Fig. 2 by using Eq. (1). The exponential dependence of the absorption coefficient as a function of the incident photon energy suggests that the Urbach rule is obeyed, and indicates the formation of a band tail [17].

Optical absorption in solids and liquids occur by various mechanisms. The photon energy can be absorbed by either lattice (or phonon) or by electrons where the transferred energy is conserved. The lattice (or phonon) absorption normally occurs in the infrared region of the spectrum due to atomic vibration. While glass generally absorbing light in the ultraviolet due to the interband electronic transition in interior molecules and provide further information about the electron states [18,19].

From these spectra, the slight increase of the optical absorption coefficient (α) at lower photon energies is due to the lattice absorption via atomic vibration. The onset of rapid change in α is the results of transition of a valence electron of an oxygen ion in the glass network to an excited state, i.e., from the top of the valence band to the bottom of the conduction band. In other words, the pronounced optical absorption occurs when the frequency of light is near the nature frequency of free oscillation of the electron.

For the present phosphate glasses, Eq. (2) can be readjusted to represent the linearity between $(\alpha\hbar\omega)^{1/2}$ and $(\hbar\omega - E_{\text{opt}})$ as follow:

$$(\alpha\hbar\omega)^{1/2} = B(\hbar\omega - E_{\text{opt}}) \tag{3}$$

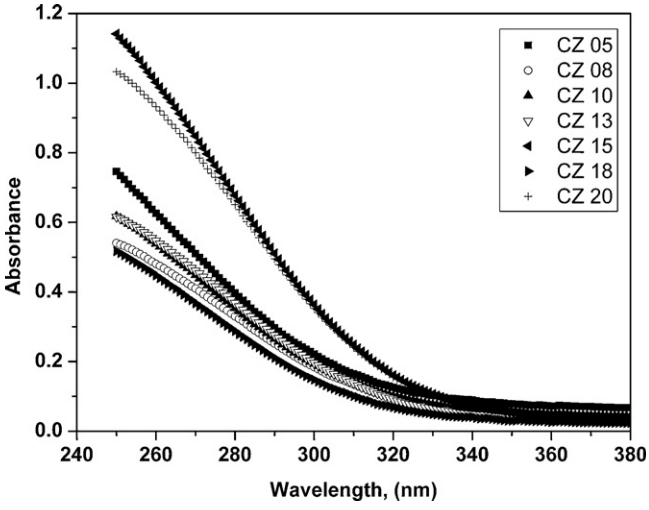

Fig. 1. The optical absorbance as a function of wavelength in the UV region of $(ZnO)_{30}(MgO)_x(P_2O_5)_{70-x}$ glasses.

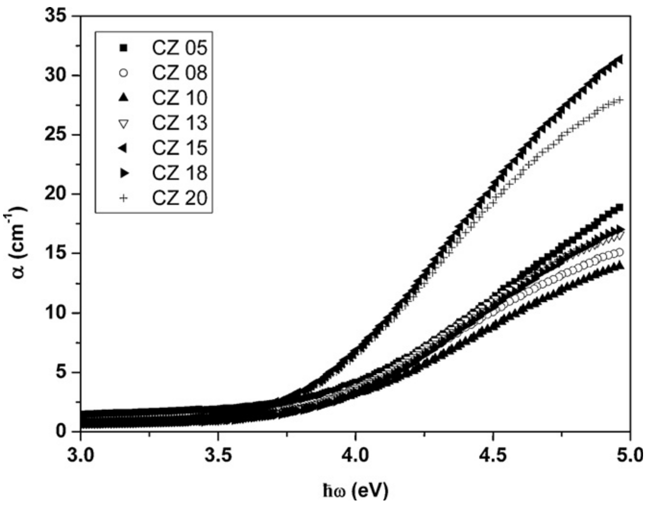

Fig. 2. Optical absorption coefficient, α, plotted against photon energy, $\hbar\omega$, for $(ZnO)_{30}(MgO)_x(P_2O_5)_{70-x}$ glasses.

The index value in Eq. (3) indicates the indirect optical transition that may associated with transitions from the top of the valence band to the bottom of the conduction band, where the interactions with lattice vibrations (phonon) take place, i.e., phonon assisted. The optical energy band gap (E_{opt}) values can be obtained from the curves plot between $(\alpha\hbar\omega)^{1/2}$ and $\hbar\omega$ as shown in Fig. 3. Least square fitting method was used to fit the data on the linear region of the curves in Fig. 3. The E_{opt} values were determined from the curves by extrapolating of the linear region of the plot to $(\alpha\hbar\omega)^{1/2} = 0$. Values of E_{opt} are tabulated in Table 1 with estimated probable error of ±0.02 eV.

As can be seen in Fig. 4 the optical energy band gap (E_{opt}) for the glasses was found to increase linearly from 3.37 to 3.44 eV (refer Table 1) as the content of MgO increases from 5 to 20 mol%. It is generally accepted that absorption edge depends on the oxygen bond strength in the glass-forming network [20]. Any change of oxygen bonding in glass network such as the formation of NBOs changes the absorption characteristics. Thus, the shift of E_{opt} to higher energies for the present study could be related to the increases of the cross-linking density in the glassy matrix. Such polymerization process is contributed by Mg^{2+} ions which probably develops strong P–O–Mg–O–P chains linking the phosphate tetrahedral within the glassy matrix and strengthen the oxygen bond strength. Moreover, the ionicity of oxygen ions decreases on account of converting the NBO ions to BO, and lower the top of the valence band resulting in the increment of band gap energy. Since the BO could binds excited electrons more tightly than NBO, therefore the higher energy is required to excite an electron from BO than from the charged NBO. As a result of this, the E_{opt} increases with increasing of MgO concentration.

The observed variation in the E_{opt} with increase in mole percentage of MgO in the present study is explained by considering the change in the glass structure as a result of expansion of the glass network. This explanation is in line with the results shown in Table 1 which shows a decreasing trend in density and its molar volume increases correspondingly with increase in MgO content. Clearly density is an effective tool to explore the degree of structural compactness [21]. Such modification of the geometrical configurations of the glass network leads to rearrangement of the structure with addition of MgO in vitreous network and reflected as well in the variation of molar volume with MgO concentration.

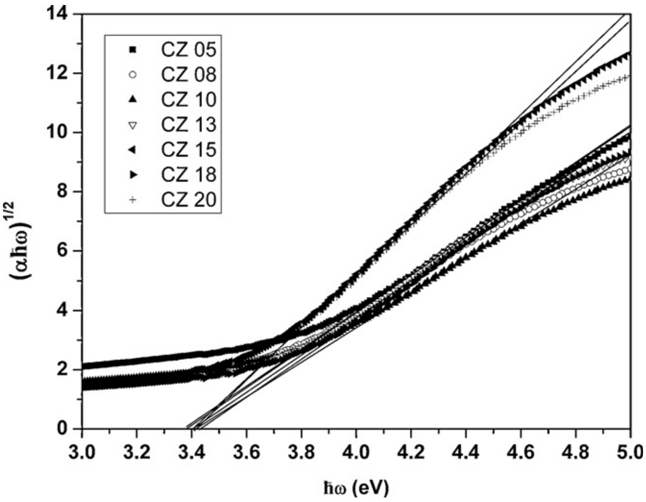

Fig. 3. The $(\alpha\hbar\omega)^{1/2}$ as a function of photon energy, $\hbar\omega$, for $(ZnO)_{30}(MgO)_x(P_2O_5)_{70-x}$ glasses.

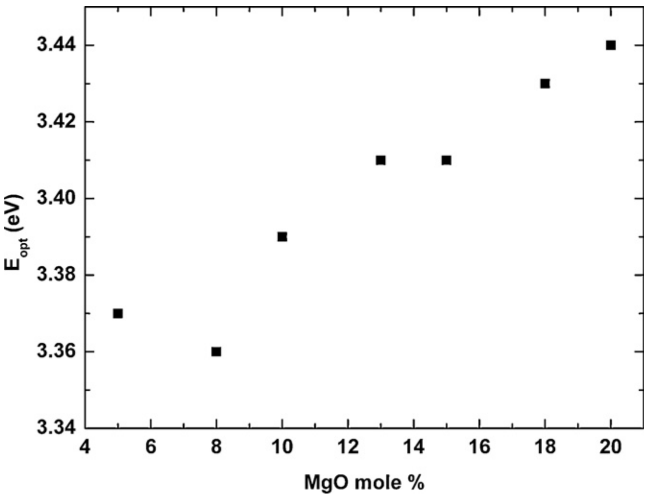

Fig. 4. Variation of E_{opt} with the mol% content of MgO.

If the glass behaves as a quasi-intrinsic semiconductor, then the Fermi energy will be in the middle of the band gap and the electrical energy gap would be given by twice the electrical conduction activation energy, E_σ, and would be expected to be equal to E_{opt} [22]. This is consistence with the results of many workers on oxide and chalcogenide glasses. However, as clearly observed from Table 1 the values of E_{opt} are considerably and about three times larger than E_σ. The values of E_σ have already been reported in previous work [23]. Such results may ascribed to the electronic activation is not across the whole mobility gap but is possibly from one or more trapping levels to the conduction band or from bonding states to a trapping level [24].

For the absorption in the region of lower photon energy of the edge, the Eq. (1) gradually changes into an exponential dependence, frequently obeys the empirical Urbach law [13]:

$$\alpha(\omega) = \alpha_o \exp\left(\frac{\hbar\omega}{E_U}\right) \tag{4}$$

where α_o is a constant and E_U is the Urbach energy which is interpreted as the width of the localized state in the normally forbidden band gap, i.e., band tailing. E_U is attributed to the optical electronic transition between a localized band tail and an extended band [25].

Fig. 5 corresponds to plots of $\ln\alpha(\omega)$ as a function of incident $\hbar\omega$ for the different composition of the studied glasses. The Urbach plots were linear for all the glass samples studied verifying Urbach law. The values of Urbach energy (E_U) were calculated by determining slopes of the linear regions of these curves and taking their reciprocals. The values of E_U were

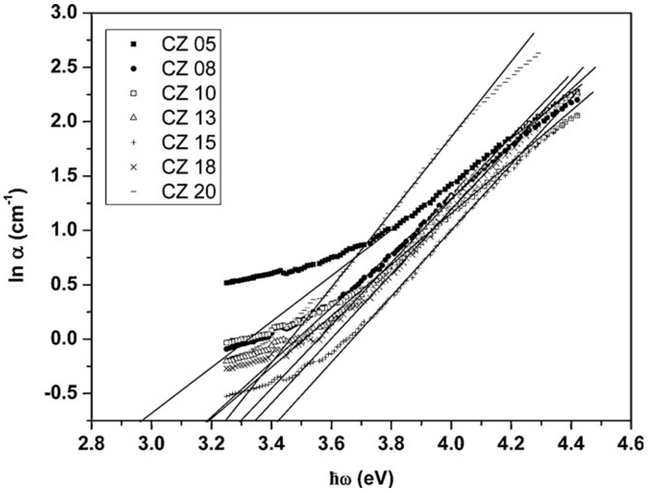

Fig. 5. The $\ln \alpha$ as a function of photon energy, $\hbar\omega$, of $(ZnO)_{30}(MgO)_x(P_2O_5)_{70-x}$ glasses.

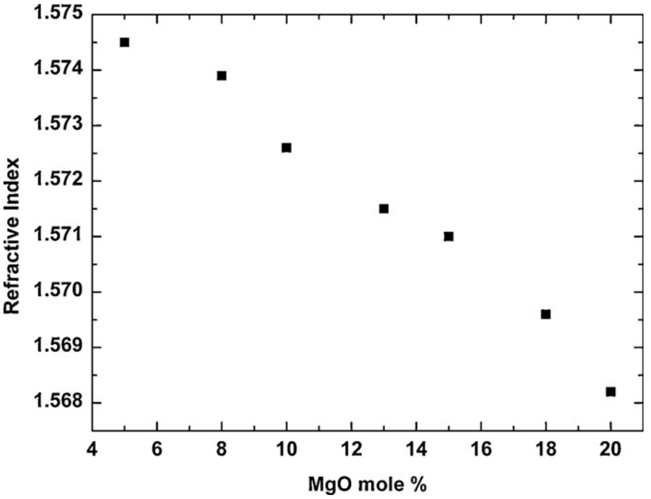

Fig. 7. Variation of refractive index versus MgO mol% for CZ glass system.

determined and presented in Table 1 with estimated probable error of ±0.01 eV.

In glass and amorphous materials there exists a band tailing in the forbidden energy band gap. According to the model of density states in amorphous solids proposed by Mott and Davis [26], the model assumes that the higher the disorderness in the non-crystalline material, the greater is the density and the tail of the localized states in the energy gap. The presence of these localized states in the energy gap is a direct result of the presence of chemical impurities, vacancies, and broken and dangling bonds [27]. Thus, the extent of band tailing or known as Urbach energy (E_U) is a measure of the disorder in the material [28].

From Fig. 6, Urbach energy (E_U) decreases progressively from 0.47 to 0.29 eV as the MgO content increased from 5 to 20 mol%. The tendency to decrease in E_U with increasing of the MgO contents is ascribed to the tendency for an increase in the orderness in studied glasses as a consequence of polymerization which likely decrease the density and the tail of the localized states in the energy gap upon substitution of P_2O_5 by MgO up to 20 mol%.

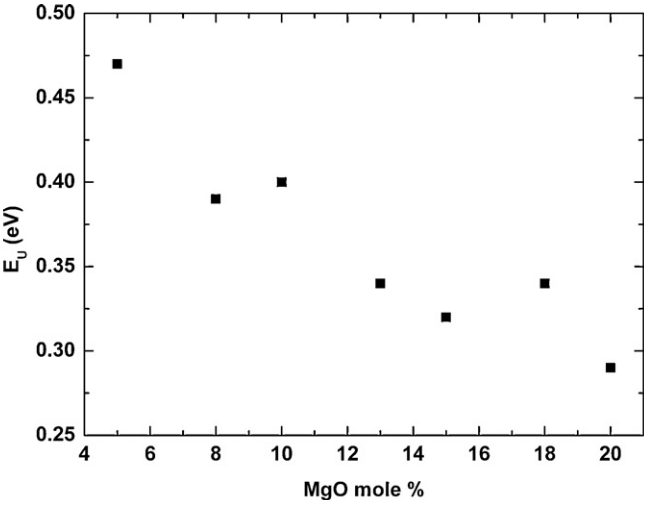

Fig. 6. Variation of E_U with the mol% content of MgO.

Davis and Mott [14] reported that the presence of high density of localized states in the band structure is responsible for lower optical energy band gap. This is in agreement that the values of E_U with its composition dependence show an in inverse trend to the values of E_{opt} as depicted in Table 1.

The compositional dependence of refractive indices of the glasses depicted in Fig. 7 shows that the refractive index is greater than unity and decreases monotonically in between 1.58 and 1.56 with the addition of MgO at the expense of P_2O_5 contents. Since the refractive index of a glass is determined by the interaction of light with the electron clouds of the constituent atoms and ions of the glass, increases in either electron density or polarizability of the ions increase the refractive index. Thus the incorporation of Mg^{2+} ions into the glassy network will enhance the structural polymerization and this development will gradually reduces the number of NBOs which is more polarizable than BOs and increases the degree of cross-linking in the glass skeleton. Hence, light will propagate through the phosphate network with less impedance, leading to lower refractive index as MgO concentration increase.

4. Conclusions

The optical properties of ternary zinc magnesium phosphate glasses have been studied in the range of visible and ultraviolet spectrum. From the optical absorption spectral, absorption edge falls in the UV region below 400 nm. The values of E_{opt} and E_U of the glass system were found to be strongly and systematically compositional dependent and sensitive to the addition of MgO. Values of the E_{opt} range from 3.36 to 3.44 eV while the E_U range from 0.47 to 0.29 eV, showing increase in structural orderness of glass as a result of incorporation of MgO as a part of the network structure. The variation of refractive index was attributed to the increase of continuity of the glassy matrix and such network modification is further supported with the variation of the density and molar volume.

Acknowledgements

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