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STUDY ON THE ROLE OF MANGANESE IONS ON PHYSICAL AND STRUCTURAL PROPERTIES OF ALUMINO-PHOSPHATE GLASSES BY MEANS OF SPECTROSCOPIC ANALYSIS

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39CdO–10Al2O3-(51-x) P2O5: xMnO (x=0,0.1, 0.2, 0.3 and 0.4 wt.%) glasses were synthesized by melt quenching technique.Various physical parameters of these glass materials were calculated and presented. Amorphous nature of these materials was confirmed from XRD and DTA studies. Various stability factors were calculated and presented from DTA analysis; from the results, the stability of the glass network is observed toincrease with MnO concentration from 0.1 to 0.4 wt. %. IR spectral analysis of these glasses exhibited several symmetrical and asymmetrical bands due to phosphate groups; the observed change in these band intensities with increase in MnO concentration from 0.1 to 0.4 wt. % optical absorption studies of these glasses exhibited an absorption bandwith shifting its band position from 500 to 488 nmwith MnO concentration from 0.1 to 0.4 wt. %, which is an indicative of gradual conversion of Mn2+ ions into Mn3+. EPR spectra of these glasses characterized by two signalsdue to Mn2+and Mn3+ions; observations on these signal intensity variations revealed an increase in stability of the glass network from 0.1 to 0.4 wt. % of MnO concentration.

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INTRODUCTION

Phosphate glass is a class of optical glasses composed of metaphosphates of various metals. Phosphate glasses are useful for applications such as bone transplantation, containment of radioactive wastes, fast ion conductors, laser host materials etc. They exhibit very important physical properties such as low melting temperature, high thermal expansion coefficient, high ultra-violet transmission, low glass transition temperature, low softening temperature, high ionic conductivity and bio compatibility [1-6]. Despite their solubility, the lower processing temperature has led these glasses to be used in applications such as glass to metal seals, low temperature enamels for metals and for optical elements etc. [7-11]. The main component in the phosphate-based glasses, phosphorus pentoxide (P2O5) is highly hygroscopic and shows relatively poor chemical durability; this problem can be addressed by replacing some proportion of phosphorus in the form of stable oxides like Bi₂O₃, Al₂O₃, Fe₂O₃ etc. [12-16]. In the present study, the conditional glass former Al_2O_3 is used to improve the stability of the glass network. CdO is used as a glass modifier; addition of it breaks up the continuous phosphate network and introduces dangling bonds/nonbridging oxygen's (NBOs) [17].

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Manganese, is a class of transition metal, ion is an interesting one because it exists in different valence states in different glass matrices, for example as Mn3+ in borate glass with octahedral coordination whereas in silicate and germanate glasses it exists in Mn^{2+} state with both octahedral and tetrahedral coordination [18]. Further, among different manganese ions, Mn^{2+} and Mn^{3+} are well known paramagnetic ions, Mn^{2+} and Mn^{4+} ions are identified as luminescence activators [19, 20]. The content of manganese in different forms in different valence states exist in the glass depends on the quantitative properties of modifiers and glass formers, size of the ions in glass structure, their field strength, mobility of the modifier cation etc. Manganese ions have been frequently used as paramagnetic probes for exploring the structure and properties of vitreous systems [21]. Hence, the connection between the state and the position of the manganese ion and the physical properties of the glass is expected to be highly interesting. In view of this, it is aimed in this work to establish the valence and the structural role of the manganese ions by means of optical and EPR studies in the glass matrix composed of CdO-Al₂O₃-P₂O₅.

Experimental

In the present study, the glasses were prepared by the meltquenching technique. The starting materials CdO, Al_2O_3 , P_2O_5 and MnO that used for the preparation of the present glass

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systems were with analar grade (larger than 99.9% purity). The chemical composition of these series of glasses is listed below.

Glass Name	Glass Composition
CAPM_0	39CdO-10Al ₂ O ₃ -51P ₂ O ₅
CAPM_1	39CdO-10Al ₂ O ₃ -50.9P ₂ O ₅ : 0.1MnO
CAPM 2	39CdO-10Al ₂ O ₃ -50.8P ₂ O ₅ : 0.2MnO
CAPM ³	39CdO-10Al ₂ O ₃ -50.7P ₂ O ₅ : 0.3MnO
CAPM_4	39CdO-10Al ₂ O ₃ -50.6P ₂ O ₅ : 0.4MnO

The melting temperature of all the glass samples were in the range of 1160-1200 °C. The density d of the glasses was determined by the standard principle of Archimedes with an accuracy of 0.001 g/cm³, by using O-xylene as working liquid at room temperature. The refractive indices n_d of the optically polished glasses was measured using sodium vapour lamp $(\lambda = 589.3 \text{ nm})$ on a precession Abbe's refractometer. The noncrystalline phases of the glasses were confirmed by XRD-7000S. The differential thermal analysis was recorded by differential thermo Gravity (DTG) of model DTG-60H with heating rate of 10 °C per minute in temperature ranges from 25 to 900 °C. FT-IR spectrum was recorded by 8400S model infrared spectroscopy in the wavelength ranging from 400-4000 cm⁻¹ with a resolution of 4 cm⁻¹ by using BEWTeki Raman plus spectrometer and ESR spectra of the samples were recorded at room temperature by E11Z Varian X-band (v = 9.5GMZ) ESR Spectrometer. The optical absorption spectra of the glasses were recorded by 8400S UV-VIS spectrometer at room temperature in wavelength ranging from 300 to 700nm.

RESULTS AND DISCUSSION

Physical Parameters

Various physical parameters of the glasses were determined and presented in Table 1.The density d of the glass samples was determined at room temperature by using Archimedes principle using O-xylene as the immersion liquid. The concentration of manganese ions N_i was calculated from the known densities and compositions of the respective glasses. The refractive index n_d of the glass was measured with an Abbe Refractometer at sodium wavelength (589.3 nm). From the obtained values of N_i , the inter–ionic distance r_i and the Polaron radius r_p of dopantions can be evaluated by the following equations [22-24]:

Inter-ionic distance $r_i(\text{\AA}) = \left[\frac{1}{N_i}\right]^{1/3}$ (1) Polaron radius $r_p(\text{\AA}) = \frac{1}{2} \left[\frac{\pi}{6N_i}\right]^{1/3}$ (2)

Table 1 Various physical parameters of CdO-Al₂O₃-P₂O₅: MnO glasses

WIIO glasses.						
Physical	CAP	CAPM	CAPM	CAPM	CAPM	
Parameters	M_0	_1	_2	_3	_4	
Average molecular weight, <i>M</i>	125. 481	125.41 8	125.36 8	125.35 1	125.31 2	
Density, d (g/cm ³)	5.35 1	5.360	5.362	5.371	5.382	
Molar volume, $V_{\rm M}$	23.4 49	23.398	23.379	23.338	23.283	
Refractive index, n_d	1.55 3	1.572	1.605	1.643	1.692	
Manganese ion concentration $N_{\rm i} ({\rm x10}^{19}$	-	0.672	1.345	2.017	2.691	

ion/cm ³)					
Inter ionic distance, <i>R</i> _i (Å)	-	1.141	0.905	0.791	0.718
Polaron radius, $R_p(Å)$	-	0.352	0.444	0.509	0.560
Electron polarizability, $\alpha_e (x \ 10^{-19} \ ion/cm^3)$	-	0.117	0.061	0.043	0.034
Dielectric constant, ε	2.40 2	2.464	2.560	2.689	2.856
Reflection loss, <i>R</i>	0.04 6	0.049	0.053	0.058	0.065
Molar refractivity, $R_{\rm M}$	3.60 1	3.721	3.892	4.083	4.332

The field strength (F_i) of dopantion in the glass network was described through the oxidation number Z and the polaron radius r_p of the transition metal ions by:

Field strength
$$F_i(\text{cm}^{-2}) = \frac{Z}{r_p^2}$$
 (3)

The theoretical dielectric constant (ε) was calculated from the refractive index of the glass using $\varepsilon = n_d^2$ (4)

The reflection loss from the glass surface was computed from the refractive index by using the Fresnel's formula:

$$R = \left[\frac{(n_d - 1)}{(n_d + 1)}\right]^2 \tag{5}$$

The molar refractivity R_M for each glass was evaluated using the formula:

$$R_{\rm M} = \left[\frac{(n_d-1)}{(n_d+2)}\right] V_m \tag{6}$$

The molar volume of the glass samples was calculated using the formula:

$$V_m = \frac{M}{d} \tag{7}$$

The electronic polarizability α_e was calculated using the formula:

$$\alpha_{e} = \frac{3(n_d^2 - 1)}{4\pi N_i(n_d^2 + 2)}$$
(8) where N_i is the number of

manganese ions per unit volume.

XRD studies

Fig.1 represents X-ray diffraction patterns of MnO doped CdO-Al₂O₃-P₂O₅ glasses. XRD patterns of powder samples were recorded at room temperature using Phillips X-ray generator (Model XRD-7000 S) in 2θ ranges from 10° - 80° . In all glass samples, the observation shows that, the broad peaks (absence of sharp peaks), is characteristics of glass structure with in the resolution limit of the XRD.



Study on the Role of Manganese Ions on Physical And Structural Properties of Alumino-Phosphate Glasses By Means of Spectroscopic Analysis

DTA studies

Fig. 2 represents differential thermal analysis (DTA) traces of pure and MnO doped CdO-Al₂O₃-P₂O₅ glasses recorded in the temperature range 30-1000 °C. DTA traces show an inflection due to the glass transition temperature $T_{\rm g}$ in the region from 492 to 524°C followed by a well-defined exothermic effect due to crystallization temperature T_c between 698 to 750 °C and melting temperature $T_{\rm m}$ due to endothermic effect ranging from 945 to 956 °C. From the data of DTA traces various stability parameters were calculated and presented in Table 2. The difference between crystallization temperature $T_{\rm c}$ and transition temperature $T_{\rm g}$ is an indication of thermal stability of glasses against crystallization. The variation of parameters $T_{\rm g}/T_{\rm m}$, $(T_{\rm c}-T_{\rm g})/T_{\rm g}$, $(T_{\rm c}-T_{\rm g})/T_{\rm m}$ and Hruby's parameter $K_{\rm gl} = (T_{\rm c}-T_{\rm g})/T_{\rm m}$ $T_{\rm g}$ /($T_{\rm m}$ - $T_{\rm c}$), which give information on the stability of the glass, with the concentration of MnO is plotted as an inset in the Fig. 2. All the traces show an increasing trend from 0.1 up to 0.4wt. % of MnO; suggesting an increase in stability of the glass network.



Table 2 Data on DTA analysis of CdO-Al2O3-P2O5: MnOglasses.

Glasses	$T_{g}(^{\circ}C)$	$T_{c}(^{\circ}C)$	$T_{\rm m}(^{\rm o}{\rm C})$	$T_{\rm g}/T_{\rm m}$	$(T_{c}-T_{g})/(T_{m}-T_{c})$	$(T_{c}-T_{g})/T_{g}$	$(T_{c}-T_{g})/T_{m}$
CAPM_0	493.0	698	945	0.522	0.829	0.415	0.216
CAPM_1	499.0	712	953	0.524	0.883	0.426	0.223
CAPM_2	505.5	723	953	0.530	0.945	0.430	0.228
CAPM_3	511.5	732	954	0.536	0.993	0.431	0.230
CAPM_4	523.6	750	956	0.547	1.099	0.432	0.236

IR spectral studies

Fig.3(a) represents the room temperature recordings of IR spectra of the pure and MnO doped CdO-Al₂O₃-P₂O₅ glasses. The glass matrix with increasing concentration of MnO exhibits different IR bands corresponding to phosphate and aluminate structural groups. The observed band positions in this work are almost similar to those reported in literature [25-29]. The data on various band positions from the IR spectra of these glasses was presented inTable 3.

Table 3 Data on various band positions from the IR spectra of
CdO-Al2O3-P2O5: MnO glasses.

Glasses	O-P-O Units (cm ⁻¹) Band 1	PO4 ³⁻ Units (cm ⁻¹) Band 2	P-O-P Units (cm ⁻¹) Band 3	AlO ₄ units (cm ⁻¹)
CAPM_0	1214	995	896	726
CAPM ¹	1210	1005	903	726
CAPM ²	1207	1020	907	726
CAPM_3	1204	1033	911	726
CAPM_4	1198	1047	915	726

For this specific network of glasses, the IR transmission spectra recorded for glasses (Fig. 3) exhibit the following conventional bands due to:

- The O-P-O symmetric stretching vibrations in the region of 1198-1214 cm⁻¹(band 1).
- The asymmetrical stretching of PO_4^{3-} groups in the region between 995-1047 cm⁻¹ (band 2).
- The asymmetrical bending vibrations of P-O-P groups in the region between 896-915 cm⁻¹ (band 3).
- The vibrations of AlO_4 groups in the regionbetween 700-750 cm⁻¹.



From the IR-Spectra, it has been observed that, with increase of MnO concentration from 0.1 to 0.4 wt. % in the glass matrix, the frequency of the band 1 (symmetrical band) shifts towards lower frequency with increase in intensity. Whereas bands 2 and 3 (symmetrical bands) shift towards higher frequency with decrease in intensity; indicating a decrease in the degree of asymmetric stretching and increase in symmetrical stretching in phosphate units, which further shows an increase in polymerization/stability of the glass network. However, there is no observed change in the band position of AlO₄ groups at any concentration of MnO in the glass network. Fig. 3(b) showsthe variation of symmetrical and asymmetrical band intensities for pure and MnO doped CdO-Al₂O₃-P₂O₅ glasses.

EPR studies

Fig. 4 shows the EPR spectra of CdO-Al₂O₃-P₂O₅:MnO glasses recorded at room temperature. The spectra are characterized by two intense resonance signals; one of them iscentered at around g = 2.01 (signal 1) with six line hyperfine pattern, which is a characteristic of isolated Mn²⁺ ions, and the other is centered at g = 4.3 (signal 2), which is a characteristic of isolated Mn³⁺ ions.



The observed spectra of these samples are similar to those reported for manganese ions doped in various glass systems[30-36]. With the increase of MnO concentration from 0.1 to 0.4 wt. %, the intensity of the signal 2 is found to increase at the expense of the intensity of the signal 1, which is an indicative of the conversion of Mn^{2+} ions into Mn^{3+} ions.The EPR spectra of Mn^{2+} , in general, can be analyzed using the spin-Hamiltonian of the form:

$$H = \beta(g_{xx}B_xS_x + g_{yy}B_yS_y + g_{zz}B_zS_z) + (A_{xx}S_xI_x + A_{yy}S_yI_y + A_{zz}S_zI_z) + (DS_z^2 - \frac{1}{2}S(S+1)) + E(S_x^2 - S_y^2).$$
(9)

Where *g* is the isotropic factor, β is the Bohr magneton, *B* is the external magnetic field, S is the vector operator of the electron spin momentum and A is the hyperfine interaction parameter, I is the vector operator of nuclear spin momentum and D is the zero field splitting parameter. Most Mn²⁺ complexes are octahedral, and have a high spin arrangement with five unpaired electrons. The d^5 electronic configuration has a ${}^6S_{5/2}$ ground state in the free atom and possesses zero orbital angular momentum and hence an ESR signal is expected with g value very close to the free electron value 2.0023 [36]. Signal at g \sim 2.01 due to Mn²⁺ ionsshows an octahedral symmetrical environment with the nature of the bonding is dominantly ionic [37, 38]. The probable local structure to occur for Mn³ in these glasses is as a MnO₆ octahedron entwined with a PO₄ tetrahedron MnPO₉ where all the oxygen's are bridging and charge compensated by a pair of Cd²⁺ ions. The presence of such complexes obviously increases the stability of the glass network possibly with covalent environment. In the present glass system, with increase of MnO concentration from 0.1 to0.4 wt. %, there is an observed conversion of Mn²⁺ions into Mn³⁺ ions, which is an indicative of increased covalent nature over that of ionic. Further, this suggests that the glass polymerization increases with improved stability with increase in MnO concentration from 0.1 to 0.4 wt. %in the glass matrix.

Optical absorption studies

Fig. 5 represents optical absorption spectra of CdO-Al₂O₃-P₂O₅: MnO glasses. The spectra of MnO doped glasses exhibit two absorption bands resulted from Mn²⁺ transition (around 500 nm)⁶ $A_{1g}(S) \rightarrow {}^{4}T_{1g}(G)$ and Mn³⁺ transition (around 490 nm)⁵ $E_{g} \rightarrow {}^{5}T_{2g}$.



The band at 500 nm is arises from intra-configurational transitions due to Mn^{2+} ions [39, 42]. These detected bands due to Mn^{2+} ionswere obscured, beyond 0.1 wt. % of MnO concentration, with a presence of a new broad absorption band with a maximum at about 490 nm due to Mn^{3+} ions [43, 44]. With increase of MnO concentration from 0.1 to 0.4 wt. %, there is an observed shift of band positionsfrom 500 to 488 nm; indicating the gradual conversion of Mn^{2+} ions into Mn^{3+} ions. Fig. 6 presents Urbach plot of CdO-Al₂O₃-P₂O₅: MnO glasses.



The optical band gap E_{opt} can be determined from the Urbach plot $(\alpha hv)^{1/2}$ versus hv, which is related by the equation.

$$\alpha(v) = \alpha_0 \left[\frac{hv - E_{opt}}{hv} \right]^2.$$
(10)

Where, α_0 is a constant related to the extent of the band tailing, and E_{opt} is the optical band gap energy [45]. The absorption coefficient, $\alpha(v)$, can be determined near the absorption edge of different photon energies for all glass samples. With increase of MnO concentration from 0.1 to 0.4 wt. %, the optical band gap is observed to increase, whereas the Urbach energy is found to decrease; is an indicative of improvement in the insulating character and hence the stability of the glass network. Table 4 presents the data on optical absorption, calculated optical band gaps, and Urbach energies of CdO-Al₂O₃-P₂O₅: MnO glasses. Finally, the observed band positions and the calculated optical band gaps show an improvement in the glass network and an insulating character with increase in MnO concentration from 0.1 to 0.4 wt. %. A similar trend was also observed from DTA, IR, and EPR studies of CdO-Al₂O₃-P₂O₅: MnO glasses.

Table 4 Data on various band positions from the opticalabsorption spectra of CdO-Al2O3-P2O5: MnO glasses.

Glass	Band edge λ (nm)	Band positionλ (nm) due to Mn ²⁺ transitions ⁶ A _{1g} (S)→ ⁴ T _{1g} (G)	$\begin{array}{c} \text{Band} \\ \text{position } \lambda \\ (nm) \text{due to} \\ \text{Mn}^{3+} \\ \text{transition} \\ {}^5\text{E}_g \rightarrow {}^5\text{T}_{2g} \end{array}$	Optical band gap energy <i>E</i> _{opt}	Urbach energy ∆ <i>E</i> (eV)
CAPM_0	320.5	-	-	2.56	0.61
CAPM_1	315.7	500	-	2.44	1.03
CAPM_2	308.0	497.5	-	2.52	0.89
CAPM_3	301.5	-	490	2.53	0.67
CAPM_4	300	-	488	2.75	0.55

CONCLUSIONS

The conclusions drawn from the study of various physical and spectroscopic properties of CdO-Al₂O₃- P_2O_5 : MnO glasses are summarized as follows:

- i. Study on the DTA traces recorded in the temperature range 30-1100 $^{\circ}C$ indicate an improved glass network with increase of MnO concentration from 0.1 to 0.4 wt. % .
- ii. The IR spectra recorded at room temperature exhibited n increase in intensity of symmetrical band (band 1) at the expense of intensities of asymmetrical bands (bands 2 and 3) with increase in MnO concentration from 0.1 to 0.4 wt. %; the observed trend shows an increase in the stability of the glass network.
- Study on the EPR spectra recorded at room temperature revealed a gradual conversion of Mn²⁺ ions into Mn³⁺ with increase in MnO concentration from 0.1 to 0.4 wt. %, which indicates an increased covalent environment of manganese ions and further shows an increase in the stability of the glass network.
- iv. Study on optical absorption spectra revealed an increase in the optical bandgap and an increase in accumulation of Mn³⁺ions at the expense of Mn²⁺ions with increase in MnO concentration from 0.1 to 0.4 wt. %; the observed trend shows an increase in insulating character and further the stability of the glass network.
- v. Finally, the results from various studies made on CdO-Al₂O₃-P₂O₅: MnO glasses reveal an improved stability of the glass network with increase in MnO concentration 0.1 to 0.4 wt. %.

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