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Optical and luminescence properties of manganese doped sodium lead alumino borosilicate glass system

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Abstract: *Glass system of composition $20Na_2O$-10PbO-(5-x) Al_2O_3-40B_2O_3-25SiO_2: xMnO with ranging from 0.3 to 0.9 mol% has been prepared by melt quenching technique. Further, the samples have been characterized by X-ray diffraction technique (XRD). A number of studies have been carried out, viz., Optical absorption and photoluminescence techniques. Glass formation is confirmed by X-ray diffraction spectra. Thermal analysis has been carried out for the prepared glasses. The optical absorption spectra of these glasses has exhibited a predominant broad band peak at about 21,052 cm^{-1} (475 nm) is identified due to $^6A_{1g}(S) \rightarrow {}^4T_{1g}(G)$ octahedral transition of Mn^{2+} ion. From the optical absorption spectral data, optical band gap (E_{opt}) and Urbach energy (ΔE) are evaluated. The emission spectra of Mn^{2+}: NPABS glasses have shown single and broad emission band at about 600 nm assigned to electronic transition $^4T_{1g}(G) \rightarrow {}^6A_{1g}(S)$ displaying red emission upon excitation at 413 nm. Octahedral coordination of Mn^{2+} ions has been estimated from the positions of emission in luminescence spectra. Various physical properties and optical basicity are also evaluated with respect of the concentration of Mn (II) ions.*

Keywords: *Borosilicate glass, Melt quenching method, XRD, Optical and luminescence properties.*

I. Introduction

Borosilicate glasses have wide range of technological applications due to its chemical and thermal resistances in various fields, such as solar energy technology, optoelectronics, sealing glasses, nuclear waste immobilization and also as construction materials. More over these materials have good optical clarity, for this reason they are used as lens in high quality flash lights and astronomical reflecting telescope in micro electrochemical systems [1-7].

Sodium lead alumino borosilicate glass results from the combination of network-forming oxides B_2O_3 and SiO_2 together with the network modifier oxide Na_2O, intermediate oxides, PbO and Al_2O_3. B_2O_3 increase the glass transparence, SiO_2 is transparent in optical window. The addition of Na_2O reduces the melting temperature and facilitates the homogenization of the glass system, reducing defects and bubbles [8-11]. The intermediate oxide, PbO introduced into borosilicate glasses which results structural changes by strong influence of the local network due to its several properties such as low melting temperature, high density, high refractive index that improves the chemical durability and enhance the resistance against diversification. These materials find applications in borosilicate glasses as metal seals, ceramic sealants and nuclear radiation shielding windows [12-17] etc. Al_2O_3 can act as both network former and modifier and enhance the glass forming ability, chemical durability and thermal stability. Glasses containing transition metal ions have great interest for their usages as photo-conducting devices, magnetic materials, etc [18]. Among transition metal oxides, Manganese ion is particularly an important one because of its optical, magnetic and electrical properties and more no. of applications of glasses. Manganese acts as probe of glass structure since it exhibits different valance states from +2 to +7 (Mn^{2+} ($3d^5$), Mn^{3+} ($3d^4$), Mn^{4+} ($3d^3$), Mn^{5+} ($3d^2$)) in different glass matrices depending on quantitative properties of glass former and modifiers, ion size in glass matrix, field strength and mobility of the modifier cation. An addition of small amount of Mn^{2+} ion to title glasses facilitates the enrichment in mechanical, optical, electrical properties. These ions can exist in different valence states with different coordination in glass matrices, for example as Mn^{3+} in borate glasses with octahedral coordination, whereas in germinate and silicate glasses as Mn^{2+} with both tetrahedral and octahedral environment. Both Mn^{3+} and Mn^{2+} ions are well known paramagnetic ions, while Mn^{2+} and Mn^{4+} are luminescent activators [19]. Mn^{2+}ions have half filled d orbital with d^5 configuration and ^6S as the ground state, for these reasons, the total orbital angular momentum for Mn^{2+} ion is zero. Since the total spin is 5/2, this ion exhibits zero field splitting which is sensitive to the local

environment. The Mn^{3+} ion has a large amount of magnetic anisotropy due to its strong spin-orbit interaction of the 3d orbital whereas Mn^{2+} ion has little amount of anisotropy energy due to its zero orbital angular momentum [20, 21].

The objectivity of the present investigation is to have a comprehensive understanding over the local environment of manganese ion in Na_2O-PbO-Al_2O_3-B_2O_3-SiO_2 glass system, by a systematic study of various physical parameters, coupled with spectroscopic (Optical absorption and Photoluminescence) investigations.

II. Materials And Methods

2.1. Glass Preparation

MnO doped Na_2O-PbO-Al_2O_3-B_2O_3-SiO_2 glasses were prepared, using melt-quenching technique. The content of manganese in the glass composition is varied from 0.3 to 0.9 mol%. The details of chemical compositions chosen for the present study are listed in **Table1**. The analytical grade reagents of raw materials (Na_2CO_3, PbO, Al_2O_3, H_3BO_3, SiO_2 and MnO of 99.99 % purity) were powdered and thoroughly mixed in an agate mortar. The mixture was taken in a silica crucible placed in an automatic temperature controlled furnace at a temperature 1200^0C for 20min. The bubble-free molten material was quenched on a pre-heated brass mold to the room temperature and subsequently annealed at 400^0C. The prepared samples were optically polished. Final dimensions of the samples used for the present study are 1 cm x 1 cm x 0.1 cm.

Table1 Glass compositions of MnO doped NPABS glass system

Glass code	Na_2O (mol%)	PbO (mol%)	Al_2O_3 (mol%)	B_2O_3 (mol%)	SiO_2 (mol%)	MnO (mol%)
Pure	20	10	5.0	40	25	-
M_1	20	10	4.7	40	25	0.3
M_2	20	10	4.4	40	25	0.6
M_3	20	10	4.1	40	25	0.9

2. 2. Characterization techniques

The optical absorption spectra were recorded on a JASCO UV-VIS-NIR spectrophotometer (Model V-670) at room temperature in the range 200-1400nm. The X-ray powder diffraction pattern of prepared glass samples were recorded using on XRD-6100 SHIMADZU X-ray diffractometer in the scanning range of $10\text{-}80^0$ (2θ) using Cu K_α radiation having a wavelength of 1.5406 Å at room temperature. The photoluminescence spectra (PL) were recorded at room temperature on the fluorescence spectrometer (SPEX Flouorolog-3) using a 450W Xe-lamp as the excitation source. By using Archimedes's principle, the density of the glasses was determined to an accuracy of ±0.001 by means of O-xylene (99.99 % pure) as the buoyant liquid. The refractive index of the glasses was measured using Abbe's Refractometer and mono- bromonaphthalene as the contact layer.

2. 3. Measurements

The density for the glasses was measured by using Archimedes's principle with O-xylene as immersion liquid. The weights of the prepared glass samples were measured in air and O-xylene using a 4- digit sensitive microbalance. Then the density (ρ) was determined from the relation

$$\rho = \frac{W_1}{W_1 - W_2} \text{x } d \qquad (1)$$

where 'w_1'is the weight in air, 'w_2' is the weight in O-xylene and 'd' is the density of O-xylene

The corresponding molar volume (V_M) was calculated using the relation

$$V_M = \frac{M_T}{\rho} \qquad (2)$$

where M_T is the total molecular weight of the multi-component glass system

The refractive index was measured at λ=589.3 nm on Abbe's Refractometer with monobromonaphthalene as the constant layer between the sample and prism of a refractometer by using sodium

vapor lamp as the source and average molecular weight (M), Optical basicity have been evaluated [22-24]. The theoretical values for optical basicity of the glass were estimated using the formula

$$\Lambda_{th}=\sum_{i=1}^{n}\frac{Z_i r_i}{2\gamma_i} \quad (3)$$

where n is total number of cations present, Z_i for oxidation number of the i^{th} cation, r_i ratio of number of i^{th} cation to the number of oxides present and γ_i basicity moderating parameter of the i^{th} cation. The basicity moderating parameter γ_i was calculated from the following equation

$$\gamma_i = 1.36(x_i-0.26) \quad (4)$$

where x_i is the Pauling electro negativity of the cation

III. Results and discussion

3.1. X-ray diffraction (XRD) Studies

From the **Fig. 1** the XRD pattern of all the glass samples shows no sharp Bragg's peak, but only a broad diffuse hump around lower angle region. This is indication of amorphous nature within the resolution limit of XRD instrument [25].

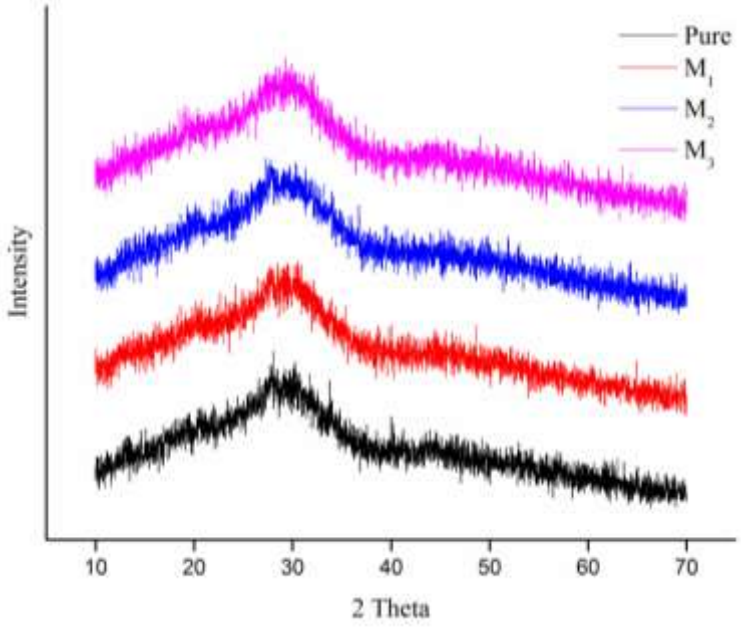

Fig. 1 XRD pattern of MnO doped NPABS glasses

3.2. Physical parameters

The physical properties of prepared glasses are very interesting and provide useful information regarding the structure and transmission mechanism due to transport of ions. The density of glass is one of the most important properties in manufacturing glass production and it is required for calculating other properties such as refractive index, elastic properties and thermal conductivity. The measured values of density and physical parameters such as dopant ion concentration (N_i), mean separation (r_i), refractive index and optical basicity of these prepared glasses are given in **Table 2**. The progressive introduction of MnO has caused the enhance in density of the samples, the degree of structural compactness, the modification of geometrical configuration of the glassy network [26].

Table 2 Physical parameters of MnO doped NPABS glass system

Parameters	Sample Code			
	Pure	M_1	M_2	M_3
Density (ρ) gcm^{-3}	2.960	2.981	2.989	2.999
Molar volume (V_m) cm^3mol^{-1}	29.854	29.629	29.526	29.411
ion conc. (N_i) $x10^{20}$ions cm^{-3}	-	0.611	1.228	1.858
Ionic radius (r_i) A^o	-	25.8	20.4	17.8
Polaron radius (r_p) A^o	-	10.236	8.116	7.081
Field strength (F_i) $x10^{14}$ cm^2	-	2.862	4.554	5.983

Refractive index (n_d)	1.653	1.654	1.655	1.656
Optical basicity (Λ_{th})	0.429	0.431	0.432	0.439
Reflection loss (%)	3.195	3.203	3.211	3.219
Molar refractivity	10.928	10.859	10.837	10.809
Dielectric constant	2.732	2.735	2.739	2.742
Electronic polarizbility ($x10^{-22}$)	-	14.355	7.163	4.762

3.4. Optical absorption Studies

The fundamental optical band gap of these glasses has been computed based on their optical absorption spectra for understanding their optically induced transitions. There are two types of transitions, which can occur at the fundamental absorption edge of glass materials. They are direct and indirect transitions. In both cases, electromagnetic waves interact with the electrons in the valence band, which rise across the fundamental band gap to the conduction band. The optical absorption coefficient, α, of a material can be evaluated from the optical transmittance and reflectance using the relation

$$\alpha = \frac{1}{d}\frac{\ln(1-R)}{T} \quad (5)$$

where d is the thickness of the prepared sample, while the absorption coefficient α(υ) as afunction of photon energy (hυ) for direct and indirect optical transitions, according to Pankove [27] is given by

$$\alpha h\upsilon = A(h\upsilon - E_g)^n \quad (6)$$

where A is a constant and E_g is the optical band gap energy and the exponent 'n' take the values ½ or 2 for allowed direct or indirect transitions, respectively. To estimate the optical energy band gap values for direct or indirect transitions $(\alpha h\upsilon)^{1/2}$ and $(\alpha h\upsilon)^2$ as a function of hυ have been plotted. The respective values of the band gap energies can be obtained by extrapolating the linear portion of the plot for $(\alpha h\upsilon)^{1/2}=0$ for indirect transition, the optical band for indirect transition values varies from 4.040-3.664 eV and $(\alpha h\upsilon)^2=0$ for direct transitions, whose values vary from 4.053-3.656 eV. The optical band gap energies decrease with the increase of manganese ion concentration. Also Urbach energy values increases from 0.249-0.275 eV with the increase of manganese ion concentration as shown in **Fig. 2** (a), (b) and (c) [28].

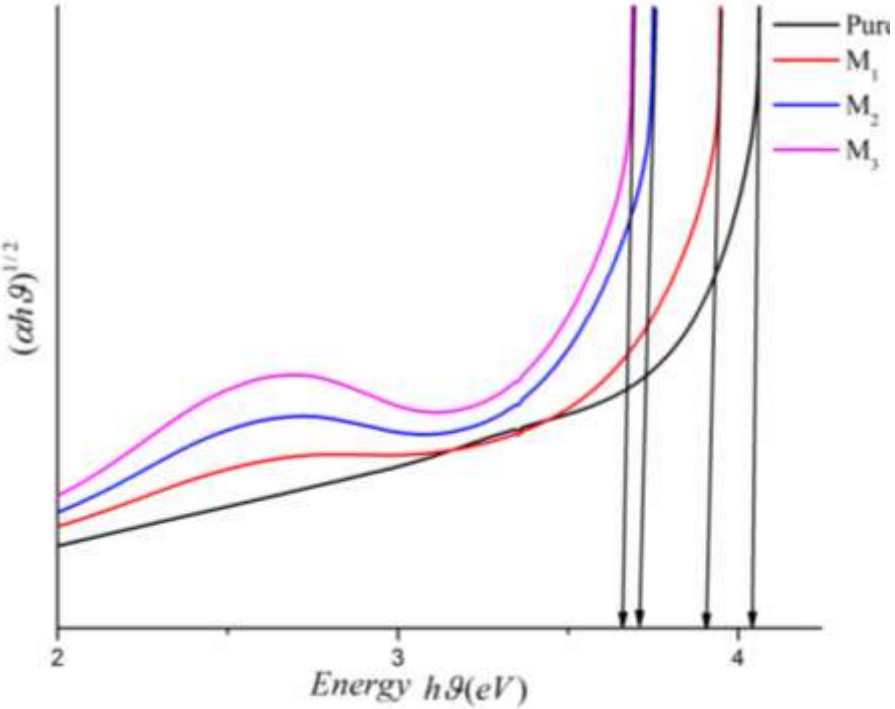

Fig. 2(a) Indirect plots to evaluate optical band gap of MnO doped NPABS glasses

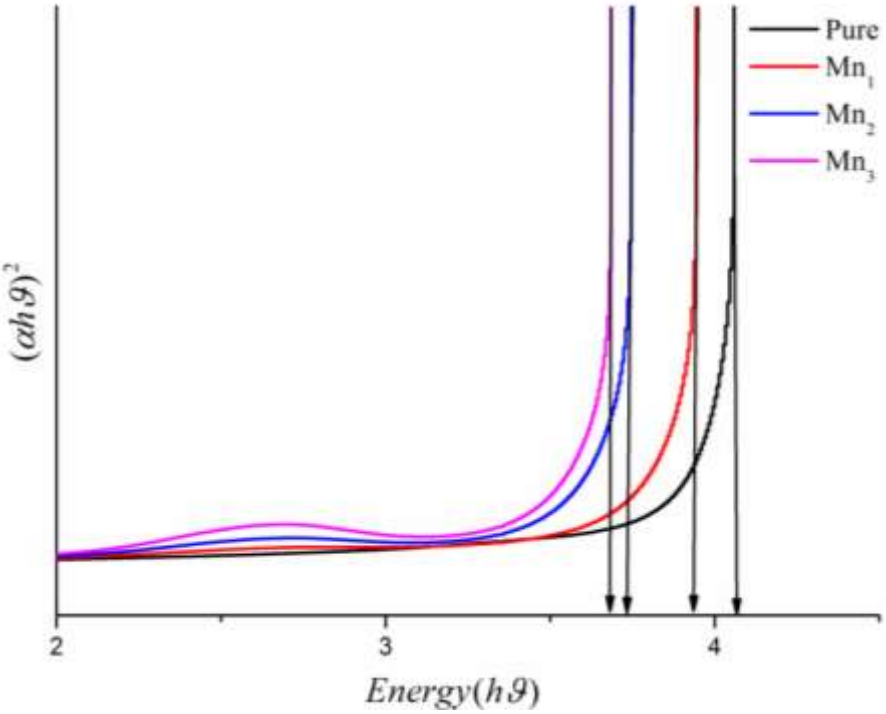

Fig. 2(b) Direct plots to evaluate optical band gap of MnO doped NPABS glasses

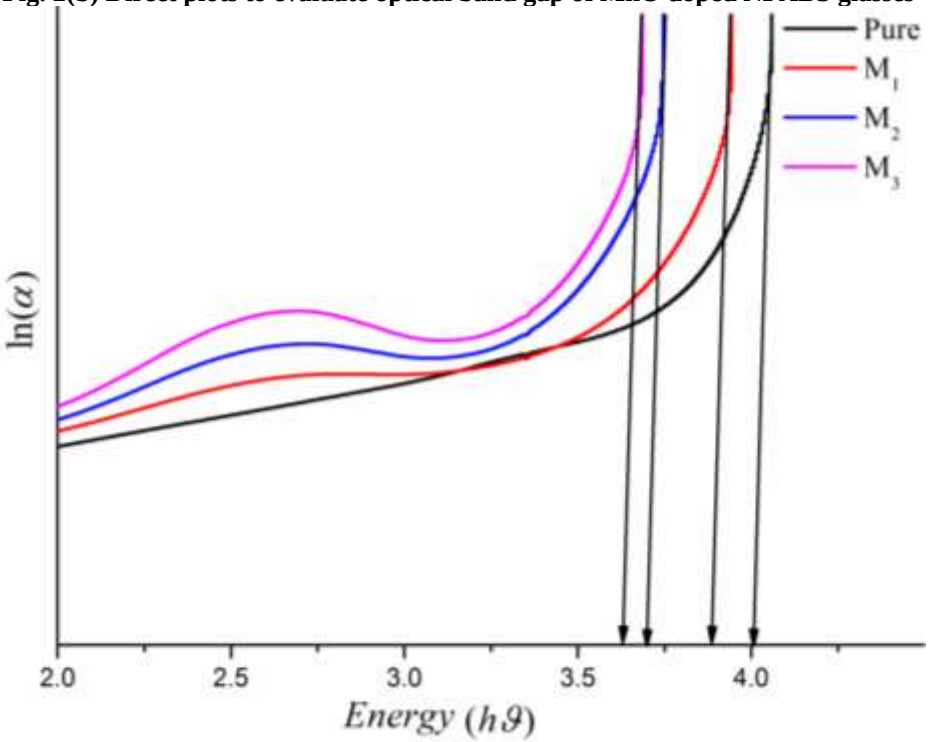

Fig. 2(c) Urbach plots to evaluate optical band gap of MnO doped NPABS glasses

The absorption spectra of transition metal ions are influenced by the nature of the host matrices into which those ions are accommodated owing to the excitation spectra of 3d electrons. The absorption spectra of transition metal ions are fairly broader and sensitive to the changes in coordination and symmetry. Due to the presence of various oxidation states, each of the states can give an increase to different absorption spectra which can be explained by the application of ligand field theory. **Fig. 3** shows, the optical spectra of Na_2O-PbO-Al_2O_3-B_2O_3-SiO_2: MnO glasses in the wavelength region 300-700 nm. The absorption edge appeared at 305 nm for pure glass M_0 is shifted slightly towards to higher wavelengths with increasing concentration of MnO. The

spectrum of these glasses has exhibited a broad absorption band at 480 nm corresponding to the transition $^6A_{1g}(S) \rightarrow {}^4T_{1g}(G)$ of Mn^{2+} ions. This band is identified due to the octahedral transition of Mn^{2+} ions [22, 29]. In general, the Mn^{2+} ($3d^5$) complexes with five unpaired electrons are expected to occupy octahedral positions in the glass network. The summary of data on the positions of bands in the optical absorption spectra of Na_2O-PbO-Al_2O_3-B_2O_3-SiO_2: MnO glasses are presented in **Table 3.**

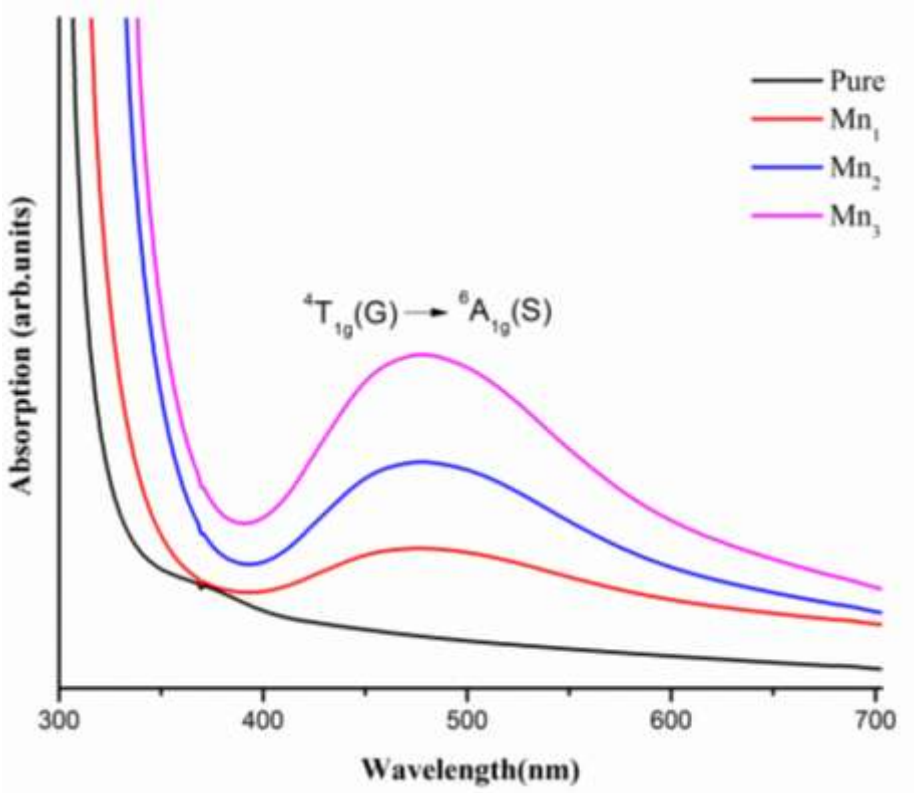

Fig. 3 Optical absorption spectra of MnO doped NPABS glasses

Table 3 Summary of data on optical absorption of MnO doped NPABS glass system

Sample code	Cut-off wavelength (nm)	Band position (nm) $^6A_{1g}(S) \rightarrow {}^4T_{1g}(G)$	Band gap energy (eV)		
			Direct	Indirect	Urbach (ΔE)
Pure	305	----	4.053	4.040	0.249
M_1	313.5	474.5	3.959	3.911	0.257
M_2	330	476	3.722	3.709	0.271
M_3	336	477	3.656	3.664	0.275

3.7. Photoluminescence studies (PL)

Luminescence characteristics are very sensitive and the complex property mainly depends on the spin and parity forbidden transitions of electronic configuration and also on the local structure of luminescent species which is affected by the surrounding matrix. **Fig. 4** shows emission spectra of sodium lead alumino borosilicate (NPABS) glasses. The excitation spectrum exhibiting a couple of bands at 367 nm and 413 nm is assigned to d-d transitions of $^6A_{1g}(S) \rightarrow {}^4T_{2g}(D)$ and $^6A_{1g}(S) \rightarrow {}^4T_{1g}(D) + {}^4E_{1g}(G)$. From the excitation spectrum, the band at 413 nm has been chosen to measure emission spectra of MnO: NPABS glasses. Upon exciting at 413 nm, manganese ions in ground are excited to upper ligand field status there upon they relax non radiatively from

these localized states to $^4T_{1g}(G)$ state through $^4E_{1g}(D)$, $^4T_{2g}(D)$, $(^4E_{1g}, ^4A_{1g})(G)$ and $^4T_{2g}(G)$ intermediate energy levels and decay radiately to $^6A_{1g}(S)$ state via phonon emission generating a broad red emission band around 600 nm assigned to a spin forbidden transition of $^4T_{1g}(G) \rightarrow ^6A_{1g}(S)$ with a inversion symmetry. The emission spectra of Mn^{2+} doped glasses are broad indicating broad distribution of Mn^{2+} sites and sensitive to change in coordination and symmetry. Generally, the emission color of Mn^{2+} depending on its co-ordination number, ligand field strength and also on the host composition. Mn^{2+} ion in tetrahedral environment exhibits emission in green region while Mn^{2+} ion in octahedral environment is in the red region. In the present case, spectral position of emission band peaking at 600 nm exhibiting red emission having six coordination number with strong ligand field strengths confirms the octahedral site symmetry of Mn^{2+} [21, 30-32].

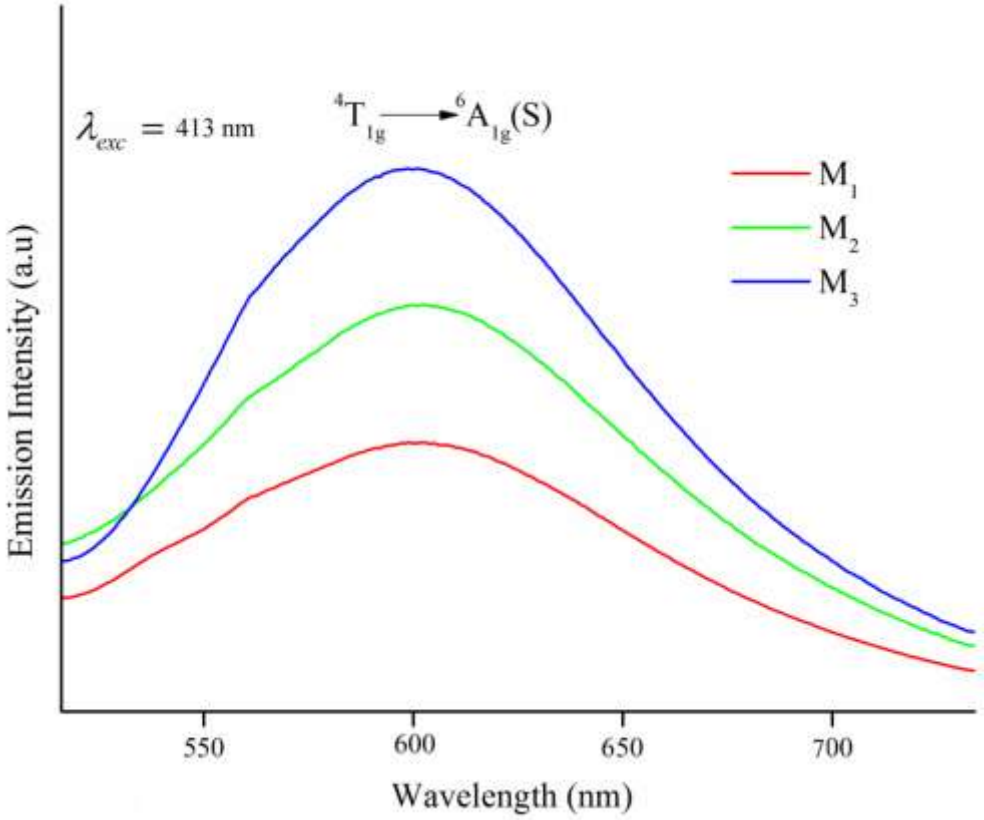

Fig. 4 Emission spectra of MnO doped NPABS glasses

Fig. 5 shows CIE diagram of MnO doped in sodium lead alumino borosilicate glasses. In order to inspect the quality of light, colour correlated temperature (CCT) values have been calculated from colour coordinates using McCamy empirical formula [33]:

$$CCT = - 437n^3 + 3601n^2 - 6861n + 5514.31 \quad (7)$$

where $n = (x - x_e)/(y - y_e)$ and the chromaticity epicenter is at $x_e = 0.3320$ and $y_e = 0.1858$, (x, y) are the calculated coordinates of prepared samples. The evaluated CIE coordinates are denoted as (x = 0.4759, y = 0.4477), (x = 0.4749, y = 0.4488) and (x = 0.4879, y = 0.4508) respectively. CCTs of MnO doped NPABS glasses range between 2770-2707 K respectively. Generally, CCT value greater than 5000 K indicates the cold white light used for commercial lighting purpose and less than 5000 K indicates the warm white light used for home appliances [34-36]. The color intensity coordinates of CIE are mentioned in **Table 4** along with CCT values, CIE coordinates also acclaim that these glasses are suitable for red emission. Hence, MnO doped NPABS glasses emits a near warm white light emission.

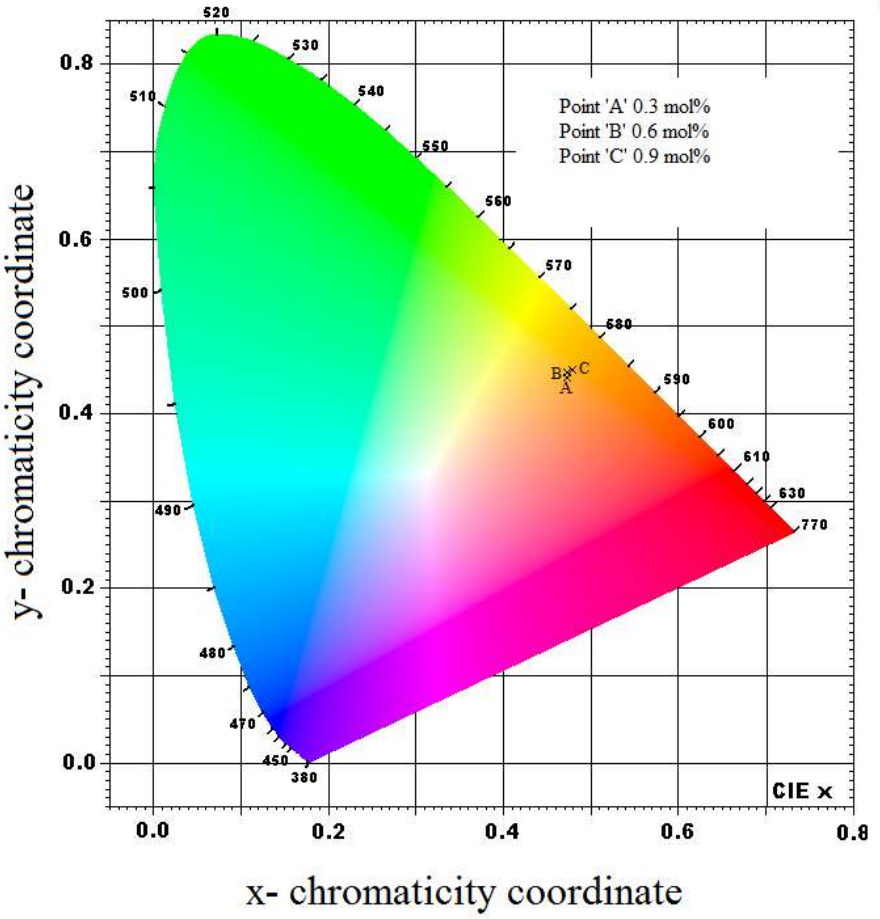

Fig. 5 1931 CIE color chromaticity diagram of the MnO doped NPABS glasses

Table 4 Glass label, chromaticity coordinates (x, y) and correlated color temperature (CCT) for various Mn^{2+} doped NPABS glass system

Glass label	Chromaticity		CCT (K)
	x	y	
$NPABSM_1$	0.4759	0.4477	2770
$NPABSM_2$	0.4749	0.4488	2743
$NPABSM_3$	0.4879	0.4508	2707

IV. Conclusion

The conclusion drawn from studying various properties of Na_2O-PbO-Al_2O_3-B_2O_3-SiO_2 glasses doped with manganese ions are as follows:

Amorphous nature of the samples is confirmed by the broad diffused haloes in XRD pattern. The density and refractive index of the samples are found to increase with increasing concentration. Optical absorption spectra of these glasses exhibits a predominant broad band peak at about 475 nm is identified due to $^6A_{1g}(S) \rightarrow {}^4T_{1g}(G)$ octahedral transition of Mn^{2+} ion. MnO doped NPABS glasses have displayed a broad red emission band at 600 nm assigned to a spin forbidden transition of $^4T_{1g}(G) \rightarrow {}^6A_{1g}(S)$. The CIE chromaticity color coordinates calculated from emission spectra of Mn^{2+} doped NPABS glasses show that the glasses emit warm white light. The band position of manganese emission confirms Mn^{2+} state in octahedral position having six coordination numbers with strong crystal field strength. From this observation it is concluded that the Mn^{2+} ions predominately occupy octahedral positions in this glass network.

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