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Spectral and Luminescence Properties of Manganese Doped Sodium Lead Alumino Borosilicate Glass System

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Abstract: Na_2O-PbO-Al_2O_3-B_2O_3-SiO_2 glasses mixed with different concentrations of MnO (ranging from 0.3 to 0.9 mol %) was prepared by melt quenching technique. The samples were characterized by X-ray diffraction technique, Optical absorption and Photo luminescence technique. A prepared glass sample is confirmed by X-ray diffraction spectra. The optical absorption spectra of these glasses exhibited a predominant broad band peak at 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 spectral analysis the optical band gap (E_{opt}) and Urbach energy (ΔE) are evaluated. The emission spectra of Mn^{2+} doped titled 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. Various principle physical properties were also evaluated.

Keywords: Borosilicate Glass, Melt Quenching Method, XRD, Optical and Luminescence Studies

Introduction

Borosilicate glasses have wide range of technological applications 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 (Santhan Kumar *et al*., 2013; Wen and Tanner, 2015; Varshneya, 1994; Pfaerder, 1996; Wan *et al*., 2014; Ruengsri *et al*., 2012; Laopaiboon and Bootlomchai, 2015; Bootjomchai *et al*., 2014; Limbach *et al*., 2015).

Sodium alumino borosilicate glass results from the combination of network-forming oxides with the network modifier Na_2O, intermediate oxides, PbO and Al_2O_3. The addition of Na_2O reduces the melting temperature and facilitates the homogenization of the glass system, reducing defects and bubbles (Poli Reddy *et al*., 2014; Serqueira *et al*., 2011; Saini *et al*., 2009; ElBatlal *et al*., 2007). The intermediate oxide, PbO introduced into titled 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 are used as metal seals, ceramic sealants and nuclear radiation shielding windows (Khanna *et al*., 2013; Nagesh *et al*., 1983; Kothiyal, 2004; El-Kameesy *et al*., 2013; Biswas *et al*., 2010; Chen *et al*., 2012) etc. Al_2O_3 can act as network former as well as modifier and enhance the glass forming ability, chemical durability and thermal stability.

An addition of small amount of MnO to borosilicate glasses facilitates the enhancement in mechanical, optical, electrical properties. Manganese ions have strong bearing on the optical, magnetic and electrical properties of glasses. These ions can exist in different valence states with different co-ordinations in glass matrices, for example as Mn^{3+} in borate glasses with octahedral coordination whereas in silicate and germinate glasses as Mn^{2+} with both tetrahedral and octahedral environment. Mn^{3+} and Mn^{2+} ions are well known paramagnetic ions. Mn^{2+} ion 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 have a large magnetic anisotropy due to its strong spin-orbit interaction of the 3d orbital whereas Mn^{2+} ion have small anisotropy

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energy due to its zero orbital angular momentum. 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 optical absorption and luminescence investigations.

Experimental Methods

Na_2O-PbO-Al_2O_3-B_2O_3-SiO_2:MnO doped glasses were prepared by using melt-quenching technique. The dopant ion concentration in the glass composition is between from 0.3 to 0.9 mol%. The chemical compositions of the present study are listed in Table 1. The AR reagents of raw materials (Na_2O, PbO, Al_2O_3, B_2O_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°C for 20 min until a bubble-free liquid is obtained. This liquid was pored on a pre-heated brass mold to the room temperature and subsequently annealed at 400°C for 8 h to reduce thermal stress and increase the mechanical strength of the material. The prepared samples were optically polished. Final dimensions of the samples used for the present study are 1×1×0.1 cm.

Characterization Techniques

The optical absorption spectra were recorded on a JASCO UV-VIS-NIR spectrophotometer (Model V-670) at room temperature in the range 200-2000 nm. 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 10-80^0 (2θ) using Cu K_α radiation having a wavelength of 1.5406 Å at room temperature. The photoluminescence spectra were recorded at room temperature on the fluorescence spectrometer (SPEX Flouorolog-3) using a 450W Xe-lamp as the excitation source.

Measurements

The density for the glasses was measured by using Archimedes 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:

$$\rho = \frac{w_1}{w_1 - w_2} \times d \tag{1}$$

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

Table 1: Glass compositions of MnO doped NPABS glass system

Glass Code	Na_2O	PbO	Al_2O_3	B_2O_3	SiO_2	MnO
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

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

$$V_M = \frac{M_T}{\rho} \tag{2}$$

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 (Meejitpaisn *et al*., 2012; Kaewjaeng *et al*., 2012; Sreehari Sastry *et al*., 2014). The theoretical values for optical basicity of the glass were estimated using the formula:

$$\Lambda_{th} = \sum_{i=1}^{n} \frac{z_i r_i}{2\gamma_i} \tag{3}$$

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:

$$\gamma_i = 1.36(x_i - 0.26) \tag{4}$$

where, x_i is the Pauling electro negativity of the cation.

Results and Discussion

XRD Studies

From the Fig. 1 the XRD spectra of all the prepared glass samples show no crystalline sharp Bragg's peak, but only a broad hump around lower angle region. This is indication of amorphous nature of the prepared glass samples (Naresh and Buddhudu, 2012).

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 the properties of refractive index. 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 causes enhance in density of the samples, the degree of structural compactness, the modification of geometrical configuration of the glassy network (Singh *et al.*, 2012).

Optical Absorption Studies

The 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:

$$\alpha(\upsilon) = 1/d \ln((1-R)/T) \quad (5)$$

where, 'd' is the thickness of the prepared glass sample, while the absorption coefficient α(υ) as a function of photon energy (hυ) for direct and indirect optical transitions, according to Pankove (1971) is given by:

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

where, 'A' is a constant and E_g is the 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. 2a to 2c (Pal *et al.*, 2011).

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)	29.854	29.629	29.526	29.411
Ion conc. (N_i) x10^{20}	-	0.611	1.228	1.858
Ionic radius (r_i) Å	-	25.8	20.4	17.8
Polaron radius (r_p) Å	-	10.236	8.116	7.081
Refractive index (n_d)	1.653	1.654	1.655	1.656
Optical basicity (Λ_{th})	0.429	0.431	0.432	0.439

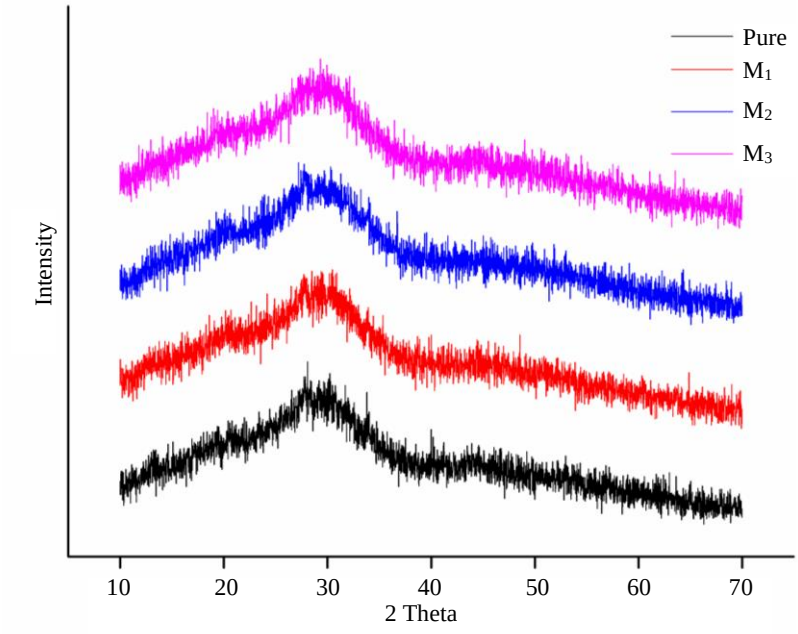

Fig. 1: XRD pattern of MnO doped NPABS glasses

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. Figure 2c shows, the optical spectra of NPABS: 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 (Meejitpaisn *et al*., 2012a; Jaya Raja *et al*., 2014). The optical absorption spectra of Na_2O-PbO-Al_2O_3-B_2O_3-SiO_2: MnO glasses are presented in Table 3.

Photoluminescence Studies

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. Figure 3 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 spectra of Mn^{2+} ion depending on its coordination number, ligand field strength and also on the host composition of the titled glass. 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+} (Rupesh Kumar *et al*., 2013; El-Ahdal *et al*., 2011; Samsudin *et al*., 2016; Peng *et al*., 2016; Naresh and Bhuddhudu, 2013).

Figure 4 shows CIE diagram of MnO doped in sodium lead alumino borosilicate glasses. In order to inspect the quality of light, Color Correlated Temperature (CCT) values have been calculated from colour coordinates using McCamy empirical formula (Mccamy, 1992):

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

where, $n = (x-x_e)/(y-y_e)$ and the chromaticity epicenters 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 (Lee *et al*., 2006; Murphy Jr, 2014; Ambast *et al*., 2014). 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.

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

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

Table 4: Glass label, chromaticity coordinates (x, y) and Correlated Color Temperature (CCT) for various Mn^{2+}: NPABS Glass systems

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

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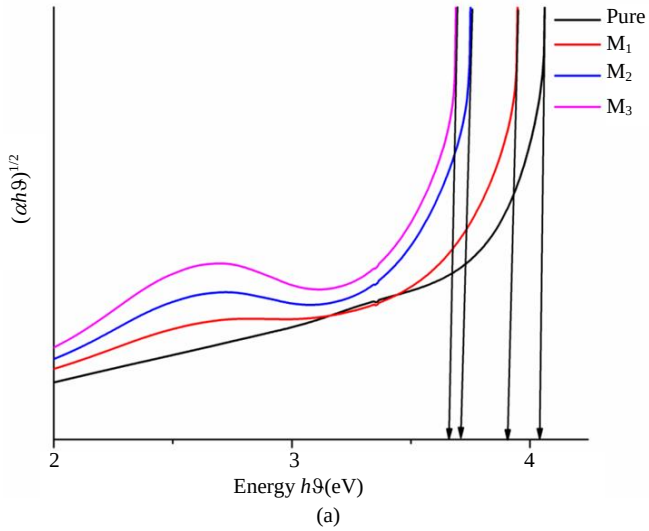
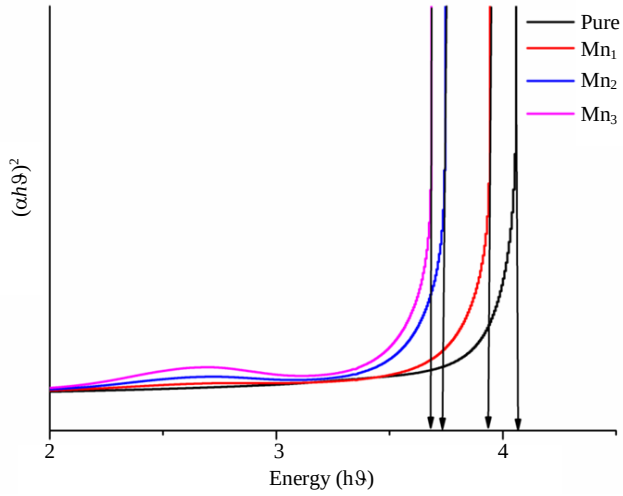
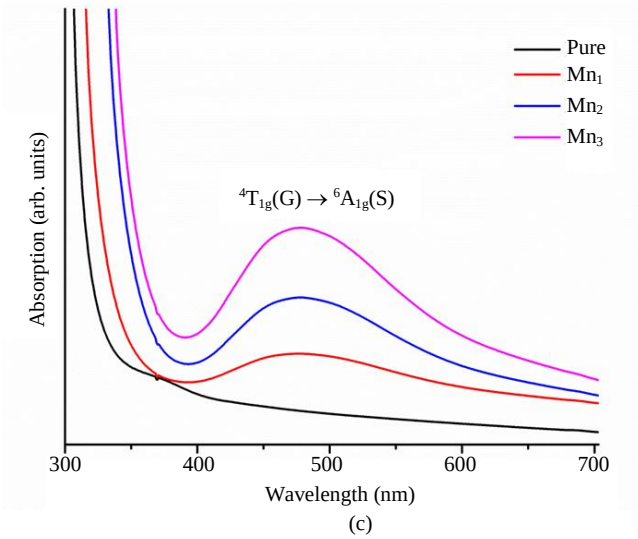


(a)



(b)



(c)

Fig. 2: (a) Indirect plots to evaluate optical band gap of MnO doped NPABS glasses; (b) Direct plots to evaluate optical band gap of MnO doped NPABS glasses; (c) Optical absorption spectra of MnO doped NPABS glasses

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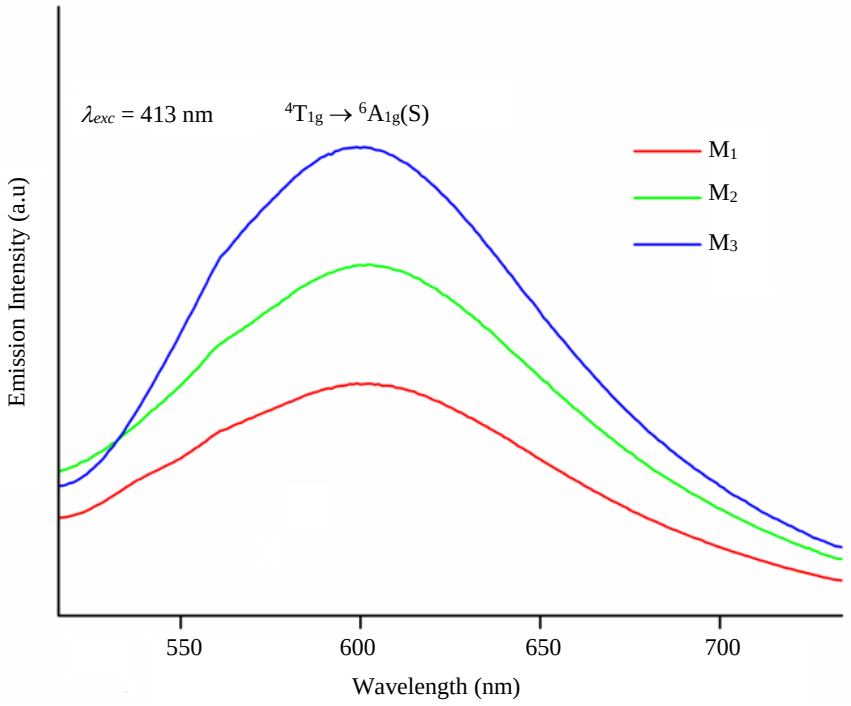

Fig. 3: Emission spectra of MnO doped NPABS glasses

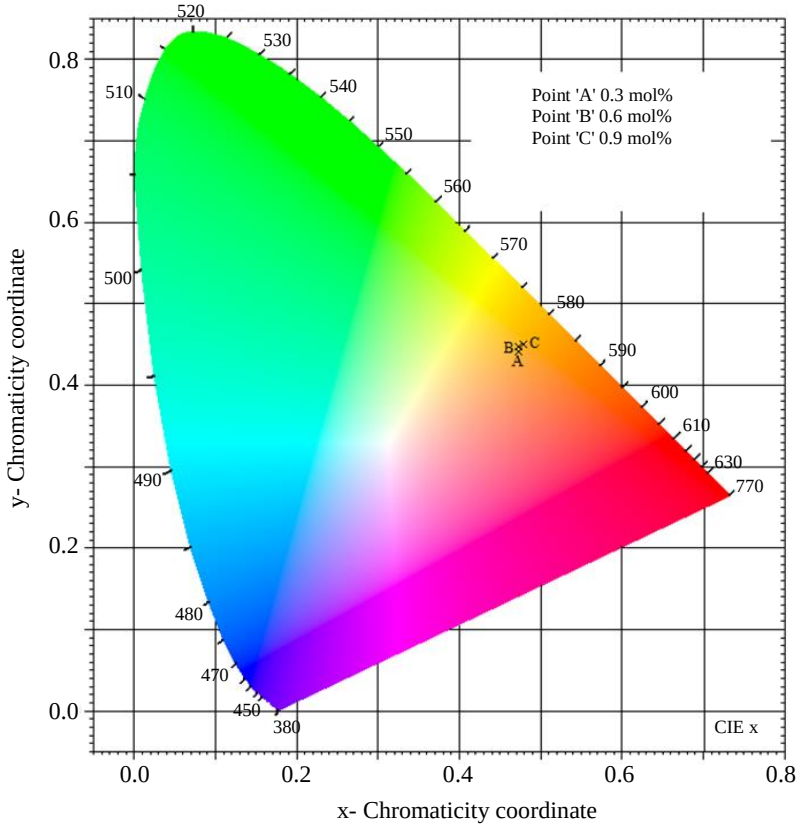

Fig. 4: 1931 CIE color chromaticity diagram of the Mn^{2+} doped NPABS glasses

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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Author's Contributions

All authors equally contributed in this work.

Ethics

This is my own work, after the publication of this paper; if any ethical issues maybe arise I will have answered.

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