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Article

Optical Properties of Tm^{3+} Ions in MO (M= Li, Na and K) – Al_2O_3 – P_2O_5 Glass Systems

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ABSTRACT

The Tm^{3+} ions doped 40 MO – 40 Al_2O_3 – 55 P_2O_5: 1.0 Tm_2O_3 (in mol %) where M = Li, Na and K glasses are synthesized by conventional melt quenching technique. The recorded optical absorption spectral profiles of Tm^{3+}ions MO–Al_2O_3–P_2O_5 Tm_2O_3 glasses have $^3H_6$ ® $^1G_4$, $^3F_2$, $^3F_3$, $^3H_4$, $^3H_5$, $^3F_4$. By performing least square fitting analysis the J-O parameters for the three glass systems are computed and are found to $W_2 > W_4 > W_6$. The luminescence spectra exhibited the bands $^1D_2 \rightarrow {}^3F_3$, $^3H_5$, $^3F_4$ and $^1G_4 \rightarrow {}^3H_6$, $^3F_4$, $^3H_5$, $^3H_4 \rightarrow {}^3F_4$ and $^3F_4 \rightarrow {}^3H_6$. The evaluated lifetimes for the levels $^1D_2$ and $^1G_4$ indicated the highest values for the K_2O mixed glasses. Laser transition is found to be the highest for the blue emission due to $^1G_4 \rightarrow {}^3H_6$ transition among other transitions. The comparison of b value for this transition is found to be the highest for KTm glass. The colour coordinates of Tm3+ ions glasses fall in the blue region.

Keywords: Tm3+ ions, UV-Vis absorption, J-O Theory, Luminescence, CIE Coordinates

Introduction

Tm^{3+} is a promising candidate for optical applications and may be suitable for optical fiber lasers and amplifiers.[1] Tm^{3+} doped glasses due to their optical properties for several applications, such as optical reading, atmospheric sensing, some uses in medical surgery and eye-safe laser radar.[2-5] It is well known that the low chemical resistance and moisture degradation of phosphate glasses impose many restrictions on their commercial exploitation and usefulness. There has been sustained concentration on improving the physical properties and the chemical durability of phosphate glasses by introducing a number of other glass formers and modifiers such as Al_2O_3, MoO_3, Ga_2O_3, In_2O_3, Sb_2O_3, into P_2O_5 glass network.[6-9] It has long been recognized that the addition of $A1_2O_3$ to phosphate glass affects glass properties in ways that imply an increase in the extent of structural polymerization.[10, 11] Incidentally, the vibrational frequency of Al-O stretching in AlO_4 structural units is found to be around 750 cm^{-1} .[12] at where the band due to P-O-P stretching vibrations of PO_4 group is also present.[13, 14] Hence, it is quite likely that Al tetrahedra and P tetrahedra form cross linked AlOP structural units. The presence of such linkages may improve the aqueous durability and other physical properties of these glasses.[15] Depending up on the composition, aluminium ions may also be present with AlO_5 and AlO_6 structural units in the glass network. Compositional variations that change the dominant phosphate structure (from a metaphosphate to a pyrophosphate, or a pyrophosphate to an orthophosphate) are accompanied by a transformation in the dominant Al coordination complicating the effect that $A1_2O_3$ has, on glass properties. Thus the introduction of Al^{3+} ions into phosphate glass network is expected to reduce the thermal expansion coefficient, to increase glass transition temperature T_g, mechanical strength, improve chemical durability and also affects fluorescence properties of the rare earth ions in the glass network profoundly. However, the phosphate glass properties are also dependent on the O/P ratio.

As it is well known that, the incorporation of alkali oxides (viz., Li_2O, Na_2O and K_2O) in the glass structure leads to a disruption of the glass network and promotes the formation of NBO groups. The ionic field strengths I ($I = Z/r^2$ where Z is the charge number and r is the ionic radius) for Li^+, Na^+ and K^+ ions are 2.16 $A°^{-2}$, 1.06 $A°^{-2}$ and 0.56 $A°^{-2}$ respectively, indicating that the network modifier cation, Li^+ has larger ionic field strength than the other two ions. In view of these facts, when aluminium-phosphate glasses are mixed with these network modifying ions there will be structural modifications and local field variations around the rare earth ion dopant in the glass matrix; in other words the symmetry and or covalency of the glass at the rare earth ion should be diverse for changed modifier mixed glasses. Such variations will have strong bearing on various luminescence transitions of the lasing ion. It is with this view the present investigation is undertaken.

As discussed above, when the aluminium phosphate glasses are mixed with different modifiers, we may expect the structural adjustments and local field variations around Tm^{3+} ion due to variations in the field strength and also in the coordination number of Al^{3+} ions in the glass network. These changes may have strong bearing on various luminescence transitions of Tm^{3+} ions. In the present investigation we have attempted to investigate the influence of three modifier ions (viz., Li^+, Na^+ and K^+) on optical absorption and the fluorescence characteristics of Tm^{3+} ions in aluminium phosphate glasses. The study is further extended to compare the probabilities of various luminescence transitions and to identify appropriate modifier for Tm^{3+} ions for giving maximum luminescence efficiency.

Experimental Procedure

The glasses used for the present study are primed by the melting and quenching techniques. Appropriate amounts of Analar grade reagents of Li_2CO_3, Na_2CO_3, K_2CO_3, $NH_4H_2PO_4$, Al_2O_3 and Tm_2O_3 were methodically diversified in an agate mortar and melted in a crucible at 1000±10 °C for about 1 h until a bubble free liquid was formed. The resultant melt was then cast in a brass mould and subsequently annealed at 350 °C. The furnace used was a PID temperature controlled furnace (Fig. 2.1). The glasses were then ground and optically polished. The approximate final dimensions of the glasses used for the present study are 1 cm x 1 cm x 0.2 cm.

The refractive indices (n_d) of the optically polished glasses was measured using sodium vapor lamp (λ = 589 .3 nm) on a Abbe refractometer (Model NAR-4T) precession refractometer.

The optical absorption spectra of the glasses were recorded using a JASCO Model V-670 UV–VIS–NIR spectrophotometer in the wavelength range 300-2200 nm. The photoluminescence spectra of glasses were recorded on Photon Technology International fluorescence spectrophotometer in UV and NIR regions with a monochrometer and photomultiplier tube for detecting the luminescence response in the appropriate wavelength regions.

Results

Physical Parameters

Table 1.Physical parameters of $MO\text{-}Al_2O_3\text{-}P_2O_5$: Tm_2O_3 glass samples

Glass	Density (g/cm^3)	Conc .of Tm ions N_i (x 10^{20} ions/cm^3)	Inter ionic distance of Tm r_i (A°)	Polaron radius r_p (A°)	Field strength (x 10^{14} cm^{-1})	Refractive index
Li	2.4397	....	....	....	....	1.638
Na	2.5652	....	....	....	....	1.644
K	2.3612	....	....	....	....	1.651
Li-Tm	2.5057	1.31	0.197	7.941	4.76	1.639
Na-Tm	2.5842	1.21	0.202	8.415	4.53	1.646
K – Tm	2.5099	1.07	0.211	8.481	4.16	1.654

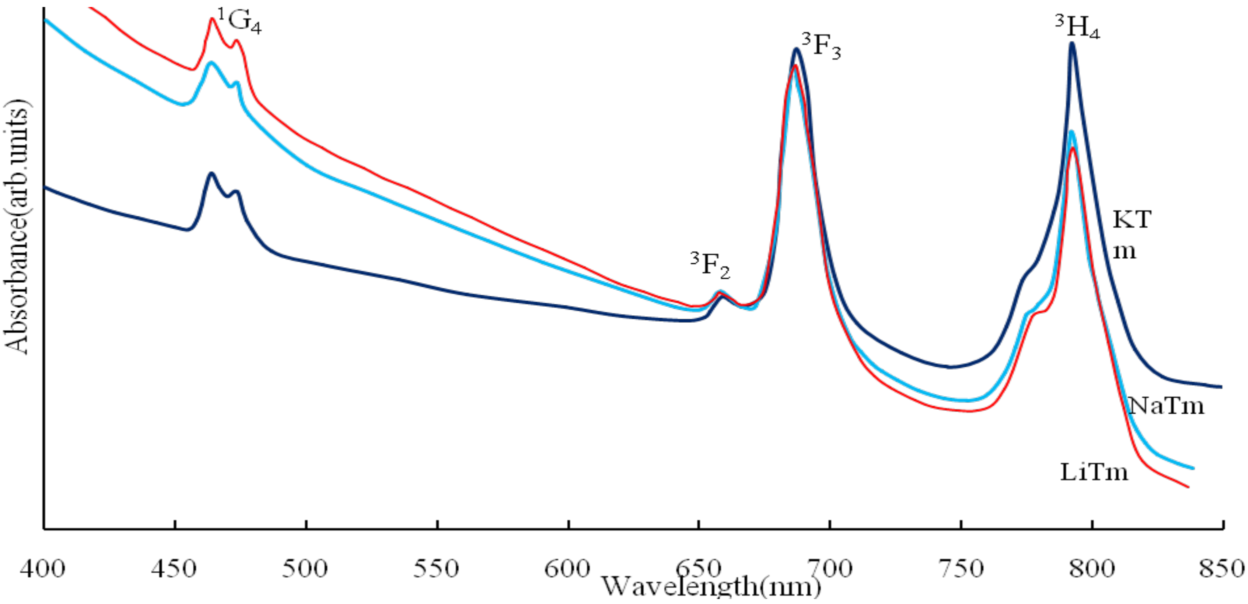

Figure 1.(a) Optical absorption spectra of Tm^{3}+ doped MO-Al_2O_3-P_2O_5 glasses recorded at room temperature in the visible region. All the transitions are from the ground state $^3H_6$

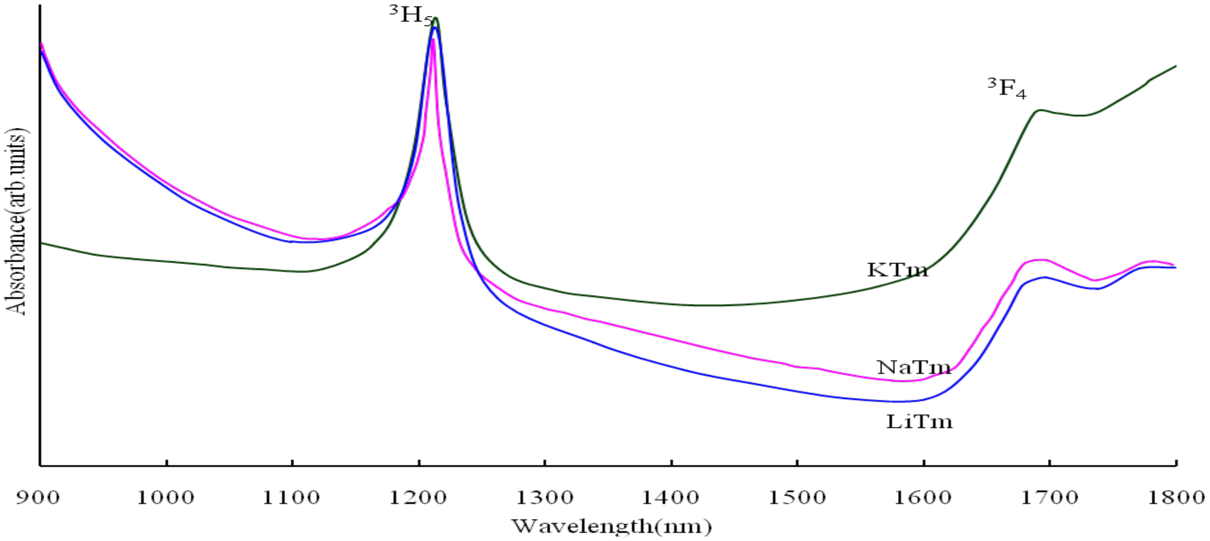

Figure 1.(b)Optical absorption spectra of Tm^{3+} doped MO-Al_2O_3-P_2O_5 glasses recorded at room temperature in the NIR region. All the transitions are from the ground state $^3H_6$

Optical Absorption

In the present glass system indicate at room temperature (Figs. 3.1 a & b) in the wavelength range 300-2100 nm, have exhibited the following absorption bands: $^3H_6 \rightarrow {}^1G_4$, $^3F_2$, $^3F_3$, $^3H_4$, $^3H_5$ and $^3F_4$. $^3H_6 \rightarrow {}^3H_5$, $^3F_4$ transitions are found to be in the IR zone, $^3H_6 \rightarrow {}^1G_4$ in the blue zone where as $^3H_6 \rightarrow {}^3F_2$, $^3F_3$, $^3H_4$ are in orange and red spectral zones. As shown in the Fig. 3.1 due to strong absorption of the host glass in the ultraviolet range, the absorption bands at wavelength shorter than 400 nm, i.e., transitions to the multiplets above $^1G_4$, could not be observed. The observed oscillator strengths (OS) of the absorption transitions are estimated from the absorption spectra in terms of the area under an absorption peak.

Further, the NIR band $^3H_6 \rightarrow {}^3H_5$ is found to be relatively sharper. The difference of modifier oxide in the glass matrix does not alter the spectral positions of the absorption bands but the absorption strength under given peak is found to be slightly higher for K_2O mixed glasses followed by NaTm glasses.

The numerical values of the OS are calculated. The theoretical OS have been evaluated using the modified

Judd-Ofelt theory as prospective in mentioned.[16, 17] Within this framework, the oscillator strength of an electric dipole transitions between two energy levels were calculated as

$$f_{cal} = \frac{8\pi^2 mc\nu\chi}{3h(2J+1)} \sum_{\lambda=2,4,6} \left[1+2R_\lambda\left(E_J-2E_f^0\right)\right]\Omega_\lambda$$
$$\left\langle f^N[\gamma,S,L]J\left\|U^\lambda\right\|f^N[\gamma',S',L']J'\right\rangle^2$$

where $\chi = \frac{\left(n^2+2\right)^2}{9n}$ is the local Lorentz field correction factor with n being the index of refraction, E_f^0 ≈17800 cm^{-1} is the energy of the barycenter of the Tm^{3+} $4f^{12}$ electron configuration[17] R_λ are the additional parameters caused by inter-configurational interaction (the 4f and 5d configurations).

The best fit was obtained when $R_2 = R_6 = -5\times10^{-5}$; $R_4 = 5\times10^{-5}$. The determined intensity parameters both for all the three glasses are furnished in Table 1. The negative values of the R_2 and R_6 are necessary, since most of the considered transitions are originating from the states below the energy of the barycenter of the Tm^{3+} $4f^{12}$ electron configuration. The quality of fitting is determined by the root mean squared deviation (RMS) represented by

$$RMS = \sqrt{\frac{\sum_{i=1}^{N}\left(f_{\exp}^{i} - f_{calc}^{i}\right)^2}{N-3}}$$

are corresponding values are given in Table 2. The value of rms ‘Δf’ listed in Table 2, is in the typical error range of the J–O fitting and indicates the good agreement between the experimental and calculated results.

In the present glass system of JO parameters trend $W_2 > W_4 > W_6$ .The compilation of the data on W_l parameters of Tm^{3+} ion in various other glass matrices (Table 3) indicated similar trend.

Table 2.The absorption band energies and the oscillator strengths for the transitions of Tm^{3+} ion in $MO-Al_2O_3-P_2O_5$ glasses

Absorption band	Energy (cm^{-1})	LiTm (10^{-6})		NaTm (10^{-6})		KTm (10^{-6})	
		f_{exp}	f_{calc}	f_{exp}	f_{cal}	f_{exp}	f_{cal}
$^3H_6 \rightarrow {}^3F_4$	5984	3.263	3.194	3.325	3.195	3.592	3.668
$^3H_5$	8251	3.412	2.993	3.527	3.605	3.583	3.706
$^3H_4$	12610	8.013	8.005	7.948	8.064	8.164	8.504
$^3F_3+{}^3F_2$	14556	7.367	7.270	7.381	7.084	7.493	7.385
$^1G_4$	21552	0.911	0.941	1.169	1.206	1.182	1.191
r.m.s.=		$\pm 0.195 \times 10^{-6}$ $W_2 = 3.45 \times 10^{-20}$ $W_4 = 3.15 \times 10^{-20}$ $W_6 = 2.61 \times 10^{-20}$		$\pm 0.159 \times 10^{-6}$ $W_2 = 3.02 \times 10^{-20}$ $W_4 = 2.18 \times 10^{-20}$ $W_6 = 1.61 \times 10^{-20}$		$\pm 0.172 \times 10^{-6}$ $W_2 = 2.64 \times 10^{-20}$ $W_4 = 1.59 \times 10^{-20}$ $W_6 = 1.01 \times 10^{-20}$	

Table 3.A comparison Table of $\Omega_\lambda x10^{20}$ (cm^2) parameters for a number of other glass systems containing Tm^3+ ions

Glass System	Ref	W_2	W_4	W_6	Comparison
$LiO-Al_2O_3-P_2O_5$: Tm_2O_3	Present work	3.45	3.15	2.61	$W_2>W_4>W_6$
$NaO-Al_2O_3-P_2O_5$: Tm_2O_3	Present work	3.02	2.18	1.61	$W_2>W_4>W_6$
$KO-Al_2O_3-P_2O_5$: Tm_2O_3	Present work	2.64	1.59	1.01	$W_2>W_4>W_6$
TeO_2-WO_3	[18]	8.6	2.7	2.3	$W_2>W_4>W_6$
$ZrF_4-ZnF_2-AlF_3-BaF_2-YF_3$	[19]	3.11	1.47	0.50	$W_2>W_4>W_6$
TeO_2–$CdCl_2$ glass	[20]	0.181	0.169	0.012	$W_2>W_4>W_6$

Luminescence Spectra

The emission spectra Fig 3.2(a) of $MO-Al_2O_3-P_2O_5$ glasses (excited at 460 nm corresponding to $^3H_6 \rightarrow {}^1G_4$ transition) have exhibited six emission bands in the visible region originated from $^1D_2$ and $^1G_4$ levels: $^1D_2 \rightarrow {}^3F_3$, $^3H_5$, $^3F_4$, $^1G_4 \rightarrow {}^3H_6$, $^3F_4$, $^3H_5$

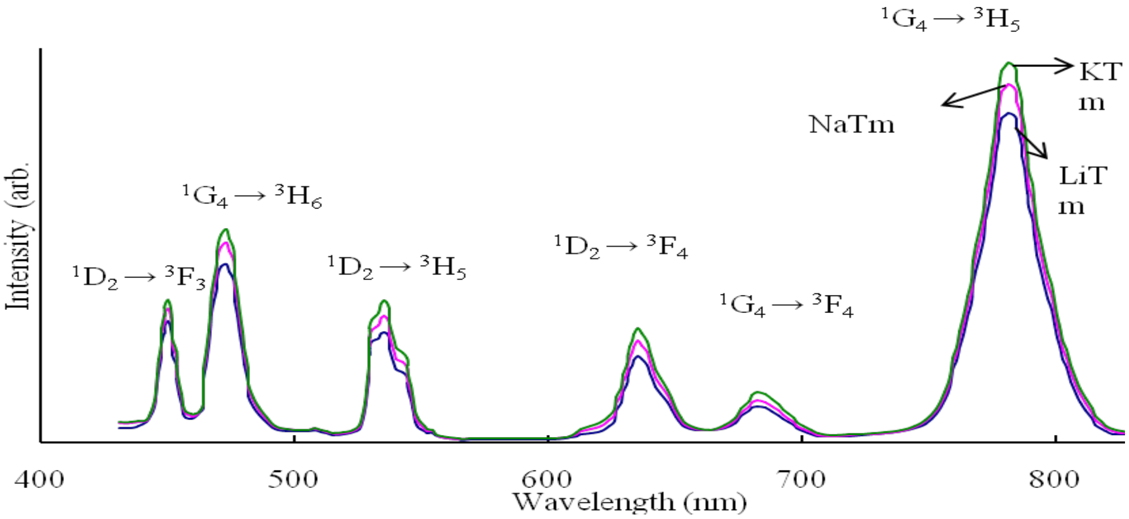

Figure 2.(a) Photoluminescence spectra of MO-Al_2O_3-P_2O_5: Tm^{3+} glasses recorded at room temperature in visible region (λ_{exc} = 460 nm)

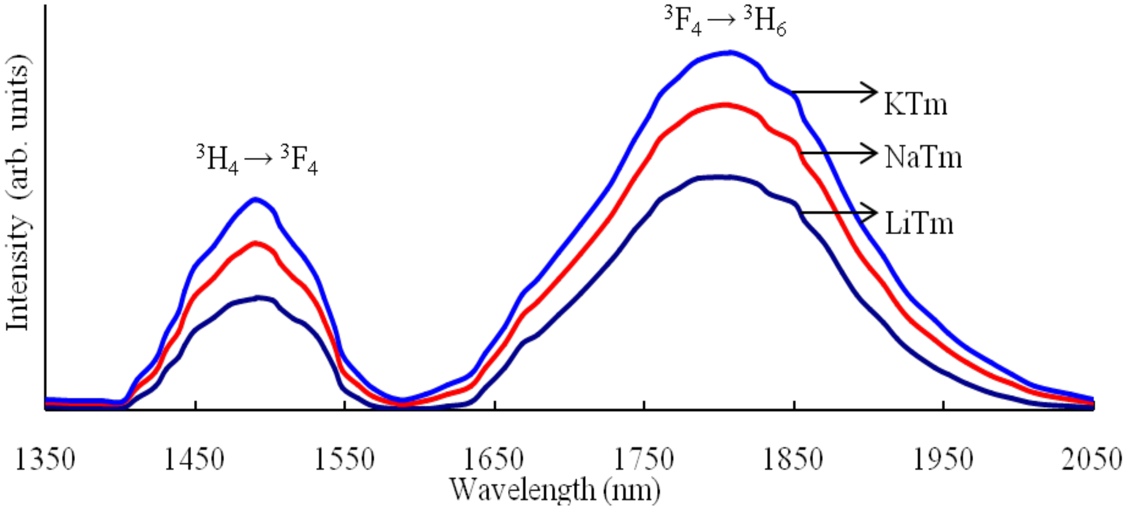

Figure 2.(b) Photoluminescence spectra of MO-Al_2O_3-P_2O_5: Tm^{3+} glasses recorded at room temperature in NIR region (λ_{exc} = 720 nm)

When pumped by 720 nm, two well resolved bands viz., $^3H_4 \rightarrow {}^3F_4$ and $^3F_4 \rightarrow {}^3H_6$ in the NIR region have been obtained Fig. 3.2 (b). The energy level diagram containing the observed absorption and emission transitions of Tm^{3+} ion for K_2O mixed glass is shown in Fig. 3.2 (c).

The radiated properties strong-minded by Using J-O parameters W_l.The spontaneous emission probability A, the total emission probability A_T connecting all the middle terms, the radiative life time (t_r) of the excited states and the fluorescent branching ratio for various transitions and the stimulated emission cross sections of the consistent fluorescent levels have also been check out using the classic equations and are conferred in Table 4.The comparison of the spectra of the three glasses indicate that the intensity and the half width of the band due to $^1D_2 \rightarrow {}^3F_4$ transition are significantly higher for K_2O mixed glass than that observed in Li_2O and Na_2O mixed glasses. The comparison of lifetimes for the $^1G_4$ level (Table 4) for the three glasses indicates higher value for the K_2O mixed Al_2O_3–P_2O_5 glass.

Colour Coordinates: Fig.3.3 shows the colour coordinates of MO-Al_2O_3-P_2O_5: Tm^{3+} doped glasses from their Vis emission spectra for studied concentrations, which fall in the blue region. Therefore, these glasses can be used in the amalgamation of blue LEDs.

Discussion

Generally, the structure of the simple phosphate based glasses is vulnerable on O/P ratios and the fraction of Q tetrahedral of phosphate. For single P_2O_5 glass O/P = 2.5 and the glass network is enhance of Q^3 tetrahedra with the

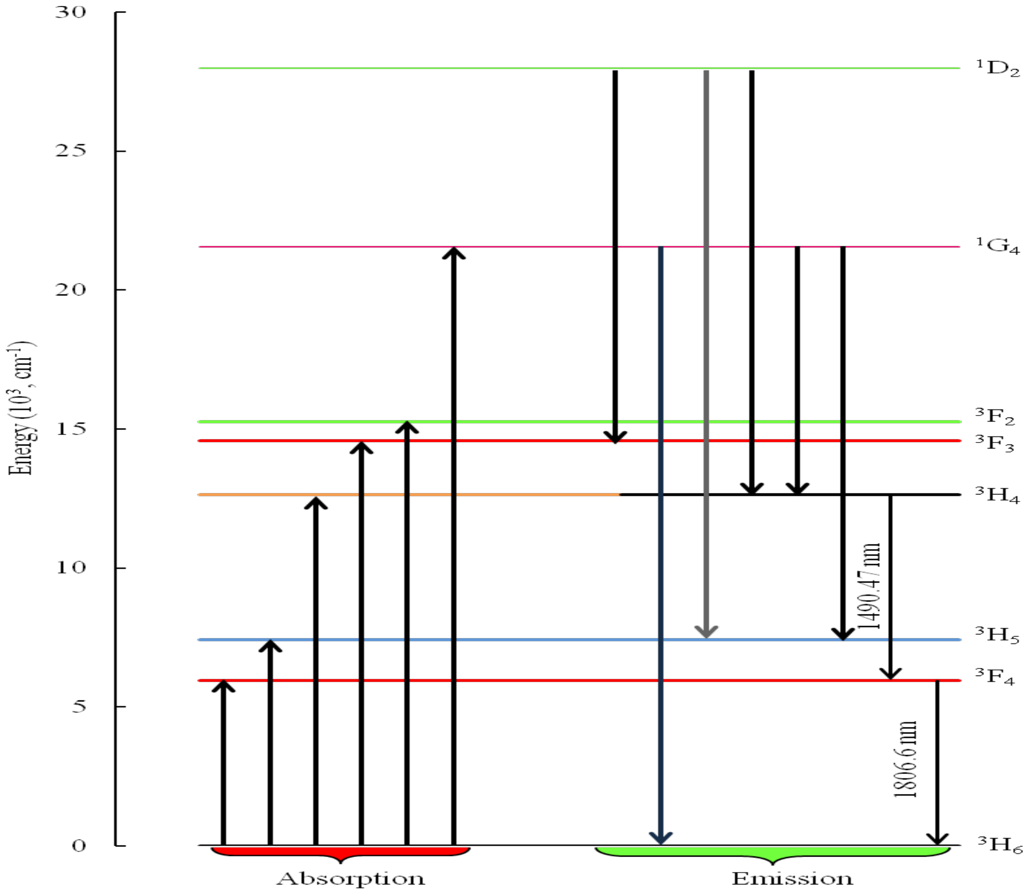

Figure 2.(c) Energy level diagram containing the observed absorption and emission transitions of Tm^{3+} ion for K_2O mixed Al_2O_3-P_2O_5 glass

Table 4.Various radiative properties of Tm^{3+} doped MO-Al_2O_3-P_2O_5 glasses

Sample	**Initial state and calculated radiative lifetime**	**Final state**	**Radiative probability A (s^{-1})**	**Branching ratio b%**	**Emission cross section s^E x 10^{-20} (cm^2)**
LiTm	$^1D_2$ t = 0.051 ms	$^3F_3$	617.99	3.13	0.154
		$^3H_5$	125.62	0.635	0.032
		$^3F_4$	8097.73	40.95	3.58
			A_T = 19774.54		
	$^1G_4$ t = 0.550 ms	$^3H_5$	654.54	36.01	0.396
		$^3F_4$	170.94	9.403	0.086
		$^3H_6$	737.13	40.55	0.112
			A_T = 1817.96		
NaTm	$^1D_2$ t = 0.066 ms	$^3F_3$	516.26	3.407	0.129
		$^3H_5$	79.43	0.525	0.02
		$^3F_4$	6939.05	45.79	3.06
			A_T = 15155.3		

	$^1G_4$ t = 0.774 ms	$^3H_5$	435.75	33.71	0.264
		$^3F_4$	112.35	8.70	0.056
		$^3H_6$	565.54	43.75	0.086
			A_T = 1292.843		
KTm	$^1D_2$ t = 0.083 ms	$^3F_3$	440.62	3.642	0.11
		$^3H_5$	51.43	0.425	0.013
		$^3F_4$	6023.03	49.78	2.66
			A_T = 12098.12		
	$^1G_4$ t = 1.042 ms	$^3H_5$	300.15	31.26	0.182
		$^3F_4$	76.55	7.98	0.039
		$^3H_6$	452.36	47.12	0.069
			A_T = 959.99		

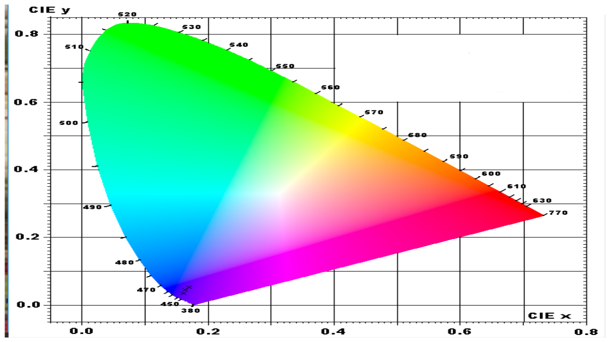

Figure 3.Chromaticity diagram of $MO-Al_2O_3-P_2O_5$: Tm^{3+} glasses

bridging oxygens; the fourth oxygen, is doubly bonded to the phosphorus atom.

Hence truncated chains in which a fraction of the phosphate tetrahedra retain three unshared oxygen corners produce the structure. These tetrahedra can be inscribed as $[POO_{2/2}O]^-$ with two bridging oxygens and $[POO_{1/2}O_2]^{2-}$ with one bridging oxygen.

The glass formation with the combination of Al_2O_3 and P_2O_5 is possible only if the phosphate network contains $[POO_{2/2}O]^-$ and $[POO_{1/2}O_2]^{2-}$ structural groups [21].

Alkali oxides are well-known modifiers and may enter the glass network by breaking P–O–P bonds and introduce bonding defects. Earlier NMR studies on aluminum phosphate glasses have indicated that aluminium ions occupy both tetrahedral (AlO_4) and octahedral (AlO_6) sites [22]:

$$2Al_2O_3 \rightarrow [Al^{3+}]_o + 3[AlO_{4/2}]_t$$

Following equation of AlO_4 tetrahedrons may enter the glass network and alternate with PO_4:

$$Al_2O_3 + P_2O_5 \rightarrow 2[AlO_{4/2}^-]_t + 2[PO_{4/2}^+]_t$$

Number of Researchers are also pointy out that the glass structure Al(6) leads when Al_2O_3 is existing in low concentrations and Al(4) forms only at higher $Al_2O_{3,}$ content.[23, 24]

Isotropic bonding of each Al(4) contributes 0.75 valance units to its four nearest neighboring oxygens, Al(6) contributes 0.5 valance units to its six nearest neighboring oxygens, and P(4) contributes 1.25 valence units to its four nearest neighboring oxygens. The alkali-earth cation (Ba^{2+}) have coordination spheres that are less well defined and contribute fewer valance units (typically 0.1 to 0.4) to their nearest neighboring oxygens. If the nearest neighboring sum significantly exceeds +2.0, the oxygen is over bonded and the structure is unstable; if the nearest neighboring valance units sum is less than 2.0, the oxygen is under bonded and the structure is also unstable unless modifying cations are available to neutralize the site. Hence, octahedral Al is not stable at the AlPO, stoichiometry since it contributes only 0.5 valence units to a POAl bond, leaving that oxygen under bonded by 0.25.

Thus the atomic arrangement at short and intermediate range in the glass network can be considered as a decisive factor for the emission characteristics of the glass network. Following the order of the ionic size or radius of the three modifier ions ($r_{Li+} < r_{Na+} < r_{K+}$), one can understand that the replacement of Li_2O successively by Na_2O and K_2O, causes to increase the degree of disorder in the glass network. Thus, the change of the modifier oxide is expected to cause structural modifications at the lasing ion site in aluminium phosphate glass network.

Additionally, the variations in the attentiveness of various structural units of phosphate, aluminium oxide and linkages between them are also expected to amend the crystal field around lasing ions in the glass network. Overall, a higher degree of structural depolymerization in the glasses mixed with K_2O as modifier is expected when compared with other two glasses for the reasons mentioned above.

The rare earth ions that occupy different coordination sites with non-centro symmetric potential contribute significantly to W_2. Even with similar coordination, the modifications in the distortion at these ion sites may lead to a distribution in the crystal field. The variations in the sites with non–centro symmetric potential (that may arise due to the influences of the dielectric of media, the environment of the rare earth ion and nephelauxetic effect) lead to changes in W_2 value. Among the three J–O parameters, the parameter W_2 is related to the covalency and structural changes in the vicinity of the Tm^{3+} ion (short–range effect) and W_4 and W_6 are related the long-range effects and are strongly influenced by the vibrational levels associated with the central rare earth ions bound to the ligand atoms. The comparison of W_2 parameter for the three glass shows slightly lower value for glass mixed with K_2O.

In the present context, this may be understood as follows: the larger the size of the modifier ion, the larger is the average distance between Al–O–P, P–O–P chains causing the average Tm–O distance to increase. Such increase in the bond lengths produces weaker field around Tm^{3+} ions leading to a low value of W_2 for the glass mixed with K_2O. Additionally, the dissimilarities in the concentration of phosphate groups with different number of non-bridging oxygens, as discussed above, also play an important role in the variation of value of W_2. (ii) The covalency amid the Tm^{3+} ion and the ligand oxygen ion also contribute to W_2. Generally oxide glasses this is close to the radial overlapping integral of the wave functions between 4f and admixing levels, e.g. 5d, 5g and the energy denominator between these two energy terms.

The Judd-ofelt parameters of W_2 and W_4 are dominant for $^1G_4 \rightarrow ^3H_6$ laser transition of blue region with mixed with Tm^{3+} ions [18]. $^3H_6 \rightarrow ^3F_{2,3}$ transitions which are in conventional used as the pumping level for blue up adaptation emission from the $^1G_4$ level are dependent on W_6. Branching ratio 'b' of the blue emission due to $^1G_4 \rightarrow ^3H_6$ transition is found to be the highest for K_2O mixed glasses. The value of b defines the quantum efficiency of the transition consistent to the laser transition; the higher value of b obtained for KTm glass clearly suggests that the growth of the ionic radius of the alkali modifier oxide causes the enhancement of luminescence efficiency.

The evaluated lifetimes t_c for the levels $^1D_2$ and $^1G_4$ (Table 3) indicate the highest value for KTm glass. This observation suggests the phonon losses in this glass are lower and facilitate for higher luminescence efficiency as experiential.

Conclusions

The Tm^{3+} ions doped 40 MO – 40 Al_2O_3 – 55 P_2O_5: 1.0 Tm_2O_3 (in mol %) where M = Li, Na and K glasses are synthesized by conservative melt quenching technique. The recorded optical absorption spectral profiles of Tm^{3+}ions MO–Al_2O_3–P_2O_5 Tm_2O_3 glasses have $^3H_6$ ® $^1G_4$, $^3F_2$, $^3F_3$, $^3H_4$, $^3H_5$, $^3F_4$. By performing least square fitting analysis the J-O parameters for the three glass systems are computed and are found to $W_2 > W_4 > W_6$. The luminescence spectra exhibited the bands $^1D_2 \rightarrow ^3F_3$, $^3H_5$, $^3F_4$ and $^1G_4 \rightarrow ^3H_6$, $^3F_4$, $^3H_5$, $^3H_4 \rightarrow ^3F_4$ and $^3F_4 \rightarrow ^3H_6$. The evaluated lifetimes for the levels $^1D_2$ and $^1G_4$ indicated the highest values for the K_2O mixed glasses. Laser transition is found to be the highest for the blue emission due to $^1G_4 \rightarrow ^3H_6$ transition among other transitions. The comparison of b value for this transition is found to be the highest for KTm glass. The colour coordinates of $Tm3^+$ ions glasses fall in the blue region.

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