Comparative analysis of luminescence Property of Tb^{3+} and Er^{3+} Activated Calcium Silicate Phosphor

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Abstract— The silicates of calcium are known for their thermal stability, high temperature strength, low thermal expansion, cheep residence and chemical inertness. Silicate phosphors are used for a fluorescent, a cathode-ray tube, a luminous body, a vacuum ultraviolet excitation light emitting element etc. Calcium Silicate acquires a higher luminous efficiency when it is doped with rare earth activated ions. In present work the silicate is prepared by combustion method at initiating temperature of $700\text{-}800^0$ C, using urea as a fuel and activated by Er^{3+} and $Tb^{3+.}$ The prepared $CaSiO_3:Er^{3+}$ and $CaSiO_3:Tb^{3+}$ phosphor was characterized by X-ray diffract meter (XRD), scanning electron microscopy (SEM), energy dispersive x-ray spectroscopy (EDX), photoluminescence (PL) and thermo luminescence (TL). The chemical composition of the sintered phosphor was confirmed by EDX spectra, The PL spectra indicate that both phosphor exhibit bright green emission and with excellent colour stability. The PL broadness were typically observed in the range of 650-680 nm. The detail analysis of result it is observed the both compositions are promising green emitting phosphor for white light emitting diode (LED) application.

Keywords— X-ray diffract meter (XRD), scanning electron microscopy (SEM), energy dispersive x-ray spectroscopy (EDX), photoluminescence (PL) and thermo luminescence (TL)

I. INTRODUCTION

Recently, nano-sized luminescent materials doped with rare earth (RE) ions have attracted intensive attention not only for their unique physical properties but also for their potential applications in developing novel phosphors and building miniature optoelectronic devices. Because the reduction of particle size can result in remarkable modifications of some of their bulk properties, nano sized phosphors or optoelectronic devices usually exhibit novel capabilities, such as higher luminescent efficiency and better resolution of images in lighting and display[1,2]

The lanthanide ions doped alkaline earth silicates are an important class of phosphorescence materials because of their high quantum efficiency in visible region, long persistence of phosphorescence, good stability, colour purity and good chemical, thermal and radiation resistance [3]. The potential benefit of lanthanide ions as activators has now well established in the field of luminescence [4]. Different activators contribute significantly in tailoring the afterglow properties of phosphors from few seconds to many hours [5]. The emission spectra of lanthanide ions almost remain the same in different host, but the luminescent efficiency,
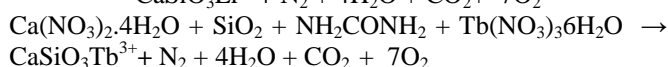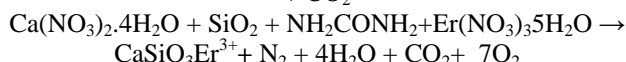
chemical stability and durability largely depends on the physical properties of host selected [6].Erbium ion (Er^{3+})and terbium ion (Tb^{3+}) has been considered as one of the most popular and efficient ions for obtaining infrared to visible up-conversion and broadband emission at 1.5 µm under the 980 nm excitation. The transition, $^{4}I_{15/2} \rightarrow {}^{4}I_{11/2}$ (980nm) of Er^{3+} ion gives poor ground state absorption. Materials with lower phonon energy can effectively restrain the multi-phonon relaxation of the energy level and $CaSiO_3$ powderhas emerged as a candidate due to its good mechanical properties ,microstructure stability and lower phonon energy [7,8]. Analytical grade $Er(NO_3)_3.5H_2O$ can be a promising complement to the $CaSiO_3$ composite due to the f-f transitions within the $^{4}f_{11}$ electronic shell of Er^{3+} ions i.e.$^{4}F_{9/2}$, $^{2}H_{11/2}$ and $^{4}S_{3/2}$ for ground state $^{2}I_{15/2}$ Such transitions occur when Er^{3+} ions occupy a lattice site of no inversion symmetry and are associated with electric dipole.[9,10]. Similarly Tb^{3+} used as an activator in different hosts, and its emissions mainly attribute to the $^{5}D_3$–$^{7}F_6$ (blue) and $^{5}D_4$–$^{7}F_0$(green). With the increasing concentration of $Tb^{3+,}$ the cross relaxation between the $^{5}D_3$ and $^{5}D_4$ energy level takes place owing to the interaction of the Tb^{3+} ions, which results in the enhanced green emission of $^{5}D_4$–$^{7}F_0$[11,12].

This article deals with the compression of structural and luminescence property of rare earth doped $CaSiO_3$:$Er^{3+}$and $CaSiO_3$:Tb^{3+} phosphor prepared by combustion synthesis method. Structural and functional group analyses by X-ray diffraction, SEM and EDX were also discussed. The Luminescence properties were discussed by TL and PL behaviour.

II. EXPERIMENTAL PROCEDURES

2.1. Synthesis

$Er^{3+}$and Tb^3doped $CaSiO_3$ phosphor were prepared by combustion synthesis. The starting material include calcium nitrate ($Ca(NO_3)_2$.$4H_2O$; Merck), silica fumes (SiO_2, 99.9% surface area 200 m^2/g), Erbium nitrate $Er(NO_3)_3$.$5H_2O$, terbium nitrate $Tb(NO_3)_3$$6H_2O$ and urea (NH_2CONH_2).Stoichiometric composition of the metal nitrates (oxidizers) and urea(fuel) were calculated using the total oxidizing and reducing valences of the components, which serve as the numerical coefficients so that the equivalence ratio is unity and the heat liberated during combustion is at a maximum .Mixture was placed over a muffle furnace heated to a temperature of 700-800^0 C. Gaseous products such as oxides of carbon and nitrogen are released as the mixture undergoes dehydration and ignition. Once ignited, the combustion propagates on its own without the need of any external heat. The silicate in a foamy form was obtained finally assuming total combustion of the redox mixture for the synthesis of $CaSiO_3$ could be written as:

$Ca(NO_3)_2$.$4H_2O$ + SiO_2 + NH_2CONH_2→$CaSiO_3$ + N_2 + H_2O + CO_2

$Ca(NO_3)_2$.$4H_2O$ + SiO_2 + NH_2CONH_2+$Er(NO_3)_3$$5H_2O$ → $CaSiO_3Er^{3+}$+ N_2 + $4H_2O$ + CO_2+ $7O_2$

$Ca(NO_3)_2$.$4H_2O$ + SiO_2 + NH_2CONH_2 + $Tb(NO_3)_3$$6H_2O$ → $CaSiO_3Tb^{3+}$+ N_2 + $4H_2O$ + CO_2 + $7O_2$

2.2. CHARACTERIZATION

X -ray diffraction of the prepared phosphor was recorded in a wide range(10° – 70°) of Bragg angle 2θ using a Bruker D8 advanced X-ray diffraction measuring instrument with Cu target radiation (λ = 0.154056nm). The thermo luminescence (TL) data were collected using a Thermo luminescence Reader (Integral- Pc Based) Nucleonix TL 1009I. Er3+and Tb3+doped $CaSiO_3$ were prepared at temperature of700-800°C. Photoluminescence (PL) data were collected using by RF- 5301PC SHIMADZU spectro fluoro photometer (RF-5301PC).Emission and excitation spectra were recorded using a spectral slit width of 1.5nm.Surface morphology and EDX analysis performed with the help of SEM (JEOL-JSI microscope) fitted with EDX.

III. RESULTS AND DISCUSSION

3.1 XRD Analysis

In order to determine the phase structure, crystalline size, lattice constant powder XRD analysis has been carried out.

The XRD patterns of CaSiO3:Er3+ and of CaSiO3:Tb3+for different mole% of Er3+ and Tb3+ in CaSiO3 are shown in Fig.1(a & b). The position and intensity of diffraction peaks of the prepared CaSiO3:Er3++ and of CaSiO3:Tb3+phosphor were matched and found to be consistent with the standard XRD pattern (COD card No. 96-900-6941). The phase structure of theCaSiO3:Er3 phosphor is alkamanite type structure which belongs to the tetragonal crystallography with space group P421m (113 space number and D32d space group), this structure is a member of the melilite group and forms alayered compound. CaSiO3 monoclinic phase is present in large abundance along with small trace of Ca2SiO4 a sorthorhombic phase. The crystal field symmetry andhence the effect of field on the shifting of emission linesis strongly dependent on the relative contents of these two phases. The average crystallite size was calculated from the XRD pattern using Debye Scherrer relation $D = \frac{k\lambda}{\beta cos\theta}$ where D isthe crystallite size for the (hkl) plane, λ is the wavelength of theincident X-ray radiation [CuKa (0.154 nm)], b is the full with that half maximum (FWHM) in radiations, and θ is the corresponding angle of Bragg diffraction. For CaSiO3:Er3++ (Fig. 1a)sharper and isolated diffraction peaks such as 2θ = 24.15 (1 1 1), 29.04 (2 1 0), 31.26 (21 1), 36.44 (3 1 0), 38.93 (3 0 1), 44.55(2 1 2) were chosen for calculation of the crystallite size. Based on the Debye-Scherrer's formula, the crystallite size is ~79 nm, 76nm, 70 nm,69 nm, 66 nm, 65 nm was calculated, respectively and the average crystallite size is ~70.00 nm.

For CaSiO3:Tb3+(Fig. 1b)) X-ray diffraction pattern show desolated diffraction peaks such as 2θ 25.1 (1 1 1), 28.00 (2 1 1), 30.16 (20 1), 35.24 (2 1 0), 37.83 (3 1 1), 43.15(2 1 2) were chosen for calculation of the crystallite size. Based on the Debye-Scherrer's formula, the crystallite size is ~80nm, 76nm, 71 nm,69 nm, 68 nm, 64 nm was calculated, respectively and the average crystallite size is ~68.50 nm. On close examination of these profiles, it can be noticed that Tb3+dopet calcium silicate is there is a slight sharpening of the broad peak with an increase in Tb3+ concentration in the glasses signifying the structural changes.
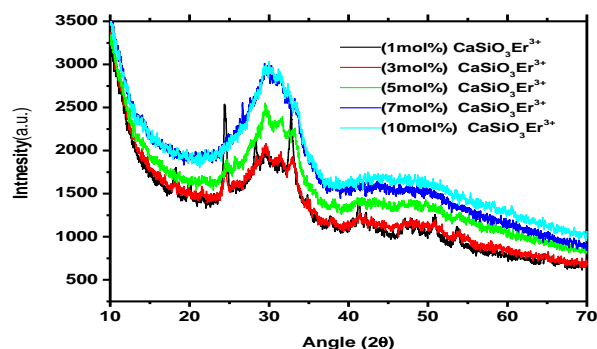

Fig. 1a: XRD patterns of $CaSiO_3$:Er^{3+} phosphor at different mole % of Er

3.2 Scanning Electron Microscopy (SEM)

It is known that the luminescence characteristics of phosphor particles depend on the morphology of the particles, such assize, shape, size distribution, defects, and so on. The surface morphology of the $CaSiO_3:Er^{3+}$ and $CaSiO_3:Tb^{3+}$phosphor is shown in fig. 2i.and fig 2 ii. at different magnification. The surface morphology of the particles was not uniform and they aggregated tightly with each other. From the SEM image, it can be observed that the prepared sample consists of particles with different size distribution. In addition, there are some big aggregates is also present due to high temperature heat treatment. The surface morphology conclude that $CaSiO_3:Tb^{3+}$phosphor is more amorphous then $CaSiO_3:Er^{3+.}$

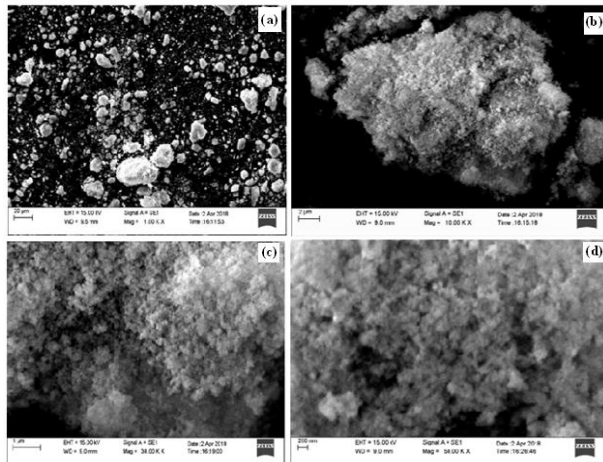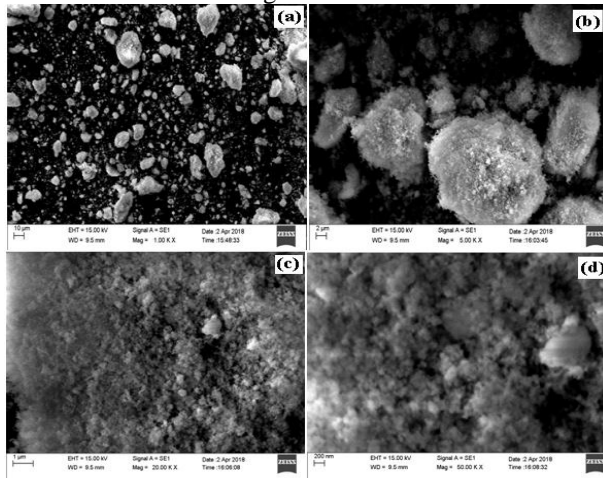**Fig. 2(i) :**SEM image of $CaSiO_3:Er^{3+}$ phosphor with different magnification

Fig. 2(ii) :SEM image of $CaSiO_3:Tb^{3+}$phosphor with different magnification.

3.3. Energy dispersive X-ray spectroscopy (EDX)

The chemical composition of the powder sample has been measured using EDX spectra as shown in fig 3(i) and 3(ii)It is a standard procedure for identifying and quantifying elemental composition of sample area as small as a few nano meters. In EDX spectra, the presence of Ca, Si, O, Er and Tb, intense peak are present which preliminary indicates the formation of $CaSiO_3:Er^{3+}$ and $CaSiO_3:Tb^{3+}$aphosphor respectively. As well as the existence of Erbium and terbium is clear in their corresponding EDX spectra. There appeared no other emission apart from calcium (Ca), silicon (Si) and oxygen (O) in the EDX spectra of$CaSiO_3:Er^{3+}$and $CaSiO_3:Tb^{3+}$ phosphor. The elements presents in the Weight% and Atomic% also determined which is represented in table 1.
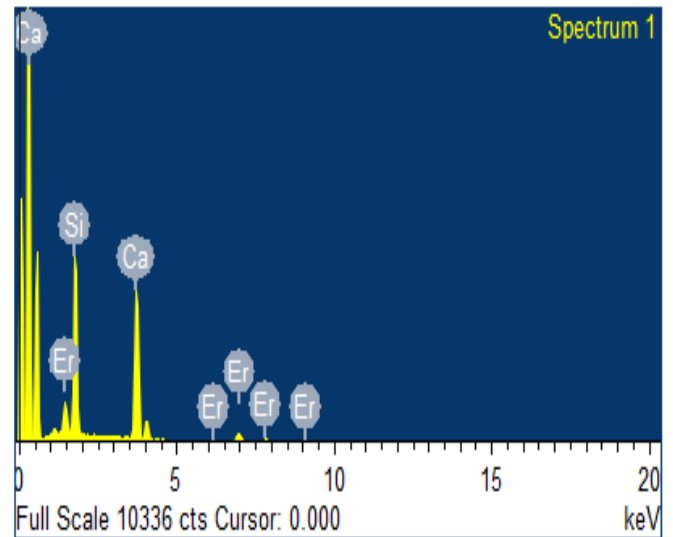

Fig. 3(i): EDX spectra of $CaSiO_3:Er^{3+}$ phosphor.

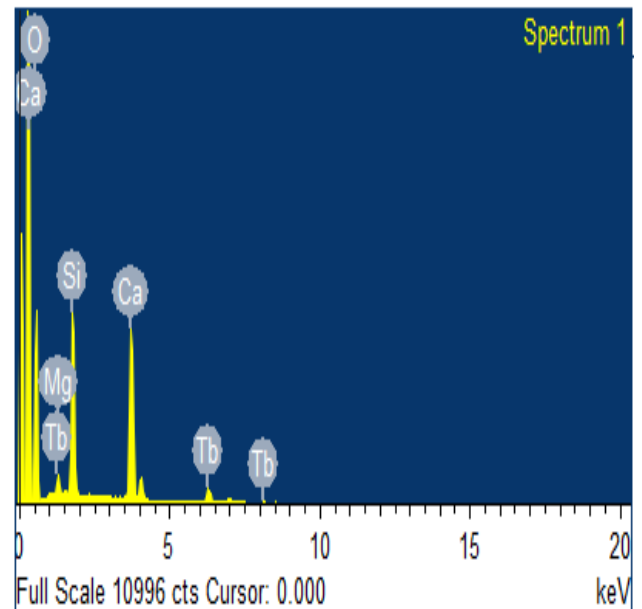Fig. 3(ii): EDX spectra of $CaSiO_3:Tb^{3+}$ phosphor.

Table 1. Chemical Composition of $CaSiO_3:Er^{3+}$ and $CaSiO_3:Tb^{3+}$ phosphor

S .no	$CaSiO_3:Er^{3+}$			$CaSiO_3:Tb^{3+}$		
	Element	Weight%	Atomic%	Element	Weight%	Atomic%
1	SiK	34.00	49.17	O K	59.02	78.48
2	Ca K	45.17	45.77	Mg K	0.83	0.73
3	Er L	20.83	5.06	Si K	12.96	9.82
4	Total	100.00	100.00	Ca K	18.46	9.80

3.4. Photoluminescence (PL)

Fig.4(a)shows the excitation spectra of CaSiO3:Er3+phosphor. The excitation spectra were observed within the range 420- 480nm. From fig 4(a) it is clear that $CaSiO_3:Er^{3+}$exhibit a broad band in the UV region centred at about 440 nm. The broad band between 420 and 450 nm, called charge transfer state (CTS) band it is due to the erbium oxygen interactions, which is caused by an electron transfer from an oxygen 2p orbital to an empty 4f shell of erbium and the strongest excitation peak is at about 440 nm. Fig. 4(b) shows the emission spectra of $CaSiO_3:Er^{3+}$phosphor in the range of 640 to 720nm, it is composed of broad band, corresponding to transitions from the excited states $^2H_{11/2}$ to the ground state. Such emissions in the luminescence spectra suggest the local environment of $Er^{3+}$is affected by the phase mixture, evidenced by the XRD patterns. The Er^{3+}ions in the $^4F_{7/2}$level decay non-radiatively to the $^4S_{3/2}$, $^2H_{11/2}$ level and benefit the transition to the $^4F_{9/2}$ level, rather than to the $^4S_{3/2}$ level, which results in the green emissions and red emissions. On the other hand, the variation in the luminescence efficiency might also be attributed to the divergence of the local environment surrounding Er^{3+}ions and surface defects in the particles of the host material. The various transition of Er^{3+}in the $CaSiO_3$system indicated in figure 4(c).
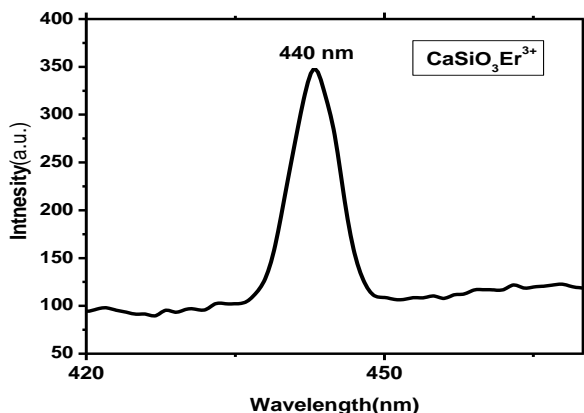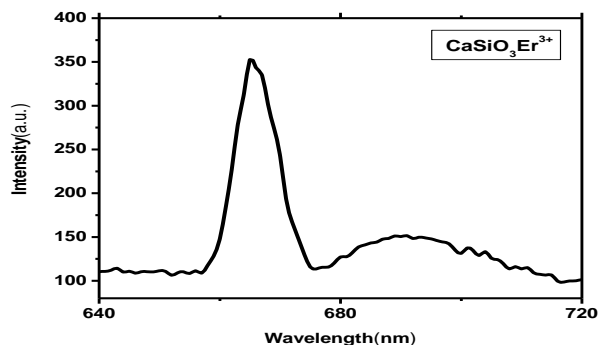

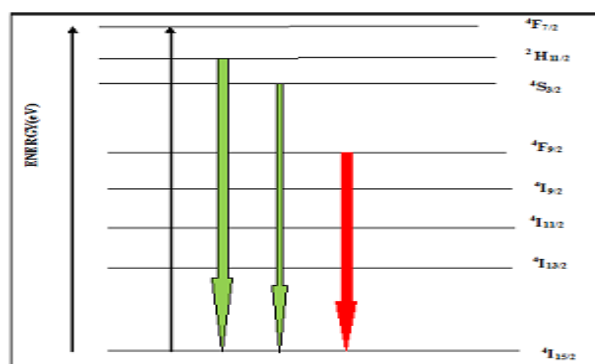Fig.4(b) Emission spectra of $CaSiO_3:Er^{3+}$ phosphor

Fig 4(c): Energy level of Er^{3+}in the $CaSiO_3$system indicating excitation and down-conversion

Fig. 5(a) shows excitation spectra and 5(b) shows the emission spectra of different mole % Tb^{3+} doped calcium silicate glasses excited to $^5D_3$ level (370 nm). The fluorescence spectra demonstrated the emission transitions arising from both $^5D_3$ and $^5D_4$ energy levels to ^7F_j ground state multiples. The emission peaks at 418 nm, 438 nm and 460 nm have been attributed to $^5D_3 \rightarrow {}^7F_{5,\,4,\,3}$ transitions and those located at 493 nm, 550 nm, 590 nm, 625 nm have been ascribed to $^5D_4 \rightarrow {}^7F_{6,\,5,\,4,3,\,2,\,1,\,0}$ transitions respectively. The intensity variation of blue ($^5D_3 \rightarrow {}^7F_4$ at 438 nm) and green ($^5D_4 \rightarrow {}^7F_5$ at 550 nm) emissions as a function of Tb^{3+} ions concentration has been graphically represented in Fig. 5(c).

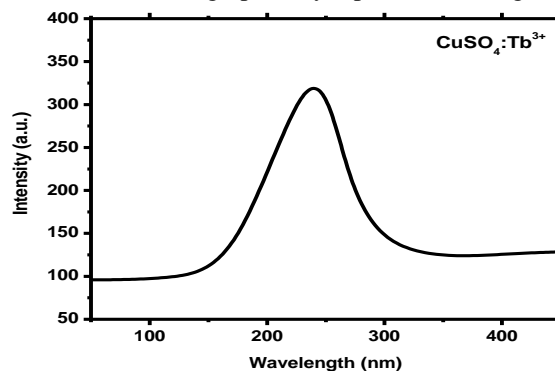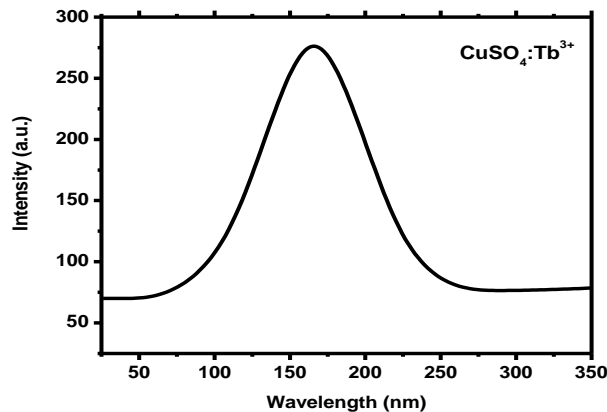Fig. 4(a): Excitation spectra of $CaSiO_3:Er^{3+}$ phosphor

Fig. 5(a): Excitation spectra of $CaSiO_3:Tb^{3+}$ phosphor

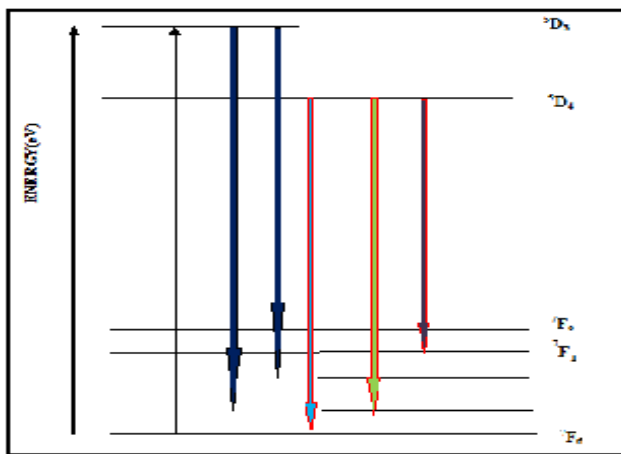Fig.5(b) Emission spectra of $CaSiO_3:Tb^{3+}$ phosphor

Fig 5(c): Energy level of Tb^{3+} in the $CaSiO_3$ system indicating excitation and down-conversion

3.5 Thermoluminescence (TL) studies

3.5.1 Er^{3+} concentration effect
TL is one of the most useful methods to study the trap centres and trap level in an insulator or semiconductor excited by radiation source. Fig.6(a) and Fig.6(b) shows the TL glow curve of UV- irradiated for different mole concentration(1,3,5 and 7mol%) of erbium and terbium with a constant heating rate of 3°C/sec. It is observed that the TL intensity increases with increasing concentration of Er^{3+} and attains a maximum value for 7mole % and it decreases with further increase in concentration of Er^{3+}. Similarly maximum intensity for Tb^{3+} the maximum intensity obtained at 7mole %. Due to increase in the activator concentration, the distance between the activators ions gets shorter. The interaction of the ions increases and the energy transfer takes place. On the other hand, a decrease in the activator concentration decreases the energy stored by the ions. Consequently, there is an optimum concentration of the activator.
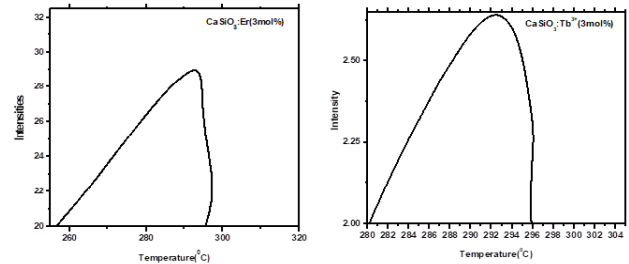

Fig. 6(a,b) : TL glow curve for different concentration at of Tb^{3+} and Er^{3+} in $CaSiO_3$

3.5.2 Calculation of kinetic parameters
TL is process to detect the recombination emission caused by de trapping of carriers thermally. The energy corresponding to the glow peak is equal to the trap depth. What we must point out is that traps and carriers (electrons and holes) may be produce by irradiation, but they are also able to be created during sample processing.

Chen's Peak shape method [14] was used for calculating kinetic parameters of phosphor .The shape of the TL glow curve is strongly influenced by the order of the kinetics. In half width method the temperature T_m, T_1 and T_2 are respectively, the peak temperature and temperature on lower(T_1) and upper(T_2) sides corresponding to half the peak intensity and dependent on the shape of the glow curve, are utilized to form equation to relate E to all or some of these temperatures. Using only the ascending part of a glow peak, one finds the value of E for the first order kinetics.

$$E = \frac{1.51(T_m T_1)}{(T_m - T_1)}$$

Whereas the descending part of a glow peak is used, the value of E is expressed as, $E = \frac{kT_m^2}{(T_2 - T_m)}$

The order of kinetics and the activation energy of glow curve was found using Chen's empirical formulae. Theoretically the form factor μ_g is found using formula

$$\mu_g = \frac{T_2 - T_m}{T_2 - T_1},$$

Where, T_m is the peak temperature at the maximum and T_1 and T_2 are respectively, the temperatures on either side of T_m, corresponding to half intensity.

Table 2: The TL parameters of $CaSiO_3:Er^{3+}$ and $CaSiO_3:Tb^{3+}$ by Chen's empirical method

Sample $CaSiO_3:Er^{3+}$ and $CaSiO_3:Tb^{3+}$	Maximum peak temperature (T_m)		Order of kinetics(b_j)	Activation energy (E) eV							
				E_δ	E_δ	E_ω	E_ω	E_τ	E_τ	E_{avg}	E_{avg}
1 mol%	296	296	I	0.20	0.79	0.21	0.78	0.22	0.80	0.21	0.79
3 mol%	293	290	I	0.22	0.81	0.25	0.80	0.22	0.83	0.23	0.82
5 mol%	295	279	I	0.22	0.82	0.25	0.85	0.25	0.84	0.24	0.84
7 mol%	287	298	I	0.27	0.86	0.24	0.88	0.26	0.84	0.26	0.86
10 mol%	283	294	I	0.24	0.83	0.21	0.84	0.26	0.83	0.25	0.83

The trap depth or the thermal energy needed to free the trapped electrons can be calculated using the following equation ,

$$E_\alpha = c_\alpha (2 \frac{kT_m}{\alpha}) - b_\alpha (2kT_m) \ ,$$

$\alpha=\tau, \delta, \omega, \tau = T_m - T_1$ is the half width at the low temperature side of the peak $\delta = T_2 - T_m$ is the half width toward the fall-off side of the glow peak $\omega = T_2 - T_1$ is the total half-width $\mu = \delta/\omega$ is so called geometrical shape or symmetry factor

$c_\tau = 1.51 + 3.0 \ (\mu_g - 0.42)$,

$c_\delta = 0.976 + 7.3 \ (\mu_g - 0.42)$

$c_\omega = 2.52 + 10.2 \ (\mu_g - 0.42)$,

$b_\tau = 1.58 + 4.2 (\mu_g - 0.42)$,

$b_\delta = 0, \ b_\omega = 1$

Using the value of T_m, T_1 and T_2 from the experimentally obtained maximum TL glow curve the form factor of $CaSiO_3{:}Er^{3+}$ (7 mole %) is found 0.52, which shows first order kinetics in it. The kinetic parameters of $CaSiO_3{:}Er^{3+}$ by Chen's empirical method is shown in table 2.

IV. CONCLUSIONS

$CaSiO_3{:}Er^{3+}$ and $CaSiO_3{:}Tb^{3+}$ doped nano phosphor powders were prepared by the combustion reaction method at 700-800°C and several characterization techniques were studied for understanding their spectroscopic and luminescence properties. The XRD analysis of both phosphor revealed that the compounds are single and monoclinic phase. The EDX spectra confirm the present elements in $CaSiO_3{:}Er^{3+}$ and $CaSiO_3{:}Tb^{3+}$ phosphor. SEM images with different magnification shows that the surface morphology of the particles was not uniform and the aggregated tightly with each other. It can be observed that the prepared samples consists of particles with different size distribution .TL study shows that the optimum Er^{3+} concentration and Tb^{3+} the optimum concentration was found at 7mol% for each having values of activation energy 0.27eV and 0.86eV respectively. Analysis of the luminescence spectra for $CaSiO_3{:}Er^{3+}$ of the excited $^2H_{11/2}$, $^4S_{3/2}$ and $^4F_{9/2}$ levels for ground state $^4I_{15/2}$ revealed changes in the non-radiative relaxation and for and $CaSiO_3{:}Tb^{3+}$ the emission peaks at 418 nm, 438 nm and 460 nm have been attributed to $^5D_3 \rightarrow {}^7F_{5,\,4,\,3}$ transitions and those located at 493 nm, 550 nm, 590 nm, 625 nm have been ascribed to $^5D_4 \rightarrow {}^7F_{6,\,5,\,4,3,\,2,\,1,\,0}$ transitions respectively. The luminescence in systems with low Er^{3+} concentrations shows the dominance of a green band over a red band while the intensity variation of blue ($^5D_3 \rightarrow {}^7F_4$ at 438 nm) and green ($^5D_4 \rightarrow {}^7F_5$ at 550 nm) emissions as a function of mechanism are responsible for the luminescence quenching. Thus it is concluded that this phosphor exhibits efficient green emission and excellent colour stability, indicating that it has favourable properties for application as near ultraviolet LED conversion.

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