



SYNTHESIS AND PHOTOLUMINESCENCE PROPERTY OF Eu^{3+} $(\text{TTA})_3$ PHEN AND ITS BLENDED THIN FILMS EMBEDDED IN PMMA FOR OPTOELECTRONIC PLLCD APPLICATION

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ABSTRACT:

Colour purity of lanthanide complexes is tough criteria for red emission display devices. Synthesis of Eu^{3+} $(\text{TTA})_3$ Phen (TTA : Thenoyl Trifluoro Aceton) and its blended thin films with polymethyl methacrylate (PMMA) for red emission is reported here. Stability of Eu^{3+} $(\text{TTA})_3$ Phen against Eu^{3+} $(\text{TTA})_3$ is discussed in this paper. Fourier transform infrared spectroscopy (FTIR) analysis and Photoluminescence property of Eu^{3+} $(\text{TTA})_3$ Phen and blended thin film of Eu^{3+} $(\text{TTA})_3$ phen introduced in polymethyl methacrylate (PMMA) is studied at room temperature and compared. At room temperature, composition of typical Eu^{3+} ion red emission obtained from synthesized composite Eu-complex. Photoluminescence emission line of pure Eu^{3+} $(\text{TTA})_3$ Phen and Eu^{3+} $(\text{TTA})_3$ Phen /PMMA obtained at 612 nm due to $^5\text{D}_0 \rightarrow ^7\text{F}_2$ electric -dipole transition. Absorption peak in near UV region is the interest of using this complex for PLLCD application.

Keywords: PMMA, Eu^{3+} $(\text{TTA})_3$ Phen, Blended thin film, PLLCD.

INTRODUCTION:

To achieve the optimum performance from the PLLCD device, specially engineered phosphors are required. A narrow band, near UV emitting source is required with a peak emission wavelength close but not extending into the visible region. Lanthanide complexes are extensively used as emitter in luminescent material for the wide range of application particularly in optoelectronics display devices. Optoelectronic organic electroluminescent (EL) devices becoming more and more popular among the researchers as their application area is wide in the field of light emitting displays. F-block lanthanides based phosphors are the interest of researchers as they are imparting a role of f-f transition. Eu^{3+} complex is found to be red emitting activator. The main source of red emission is f-f transition of Eu^{3+} ion. Intensified light collection and security of

ligands, further upgrade of emission of Eu ion are possible for such a metal complex by introduction of second ligand may be the solution which enhanced the absorption [1-6]. This concept improves mechanical and thermal stability [7]. Hui Zhang et al [8] proves that thermal stability of $\text{Eu}(\text{TTA})_3(\text{phen})$ when dispersed in polymethyl methacrylate (PMMA) was found to be better than original $\text{Eu}(\text{TTA})_3(\text{phen})$. This is due to the fact that there is a chemical bonding of $\text{Eu}(\text{TTA})_3(\text{phen})$ with oxygen atom of carbonyl group of polymethyl methacrylate. Here in this report we study blended thin film of Eu^{3+} $(\text{TTA})_3$ Phen introduced in (PMMA) as a second ligand.

MATERIALS AND METHODS

Experimental

A volatile Eu complex, $\text{Eu}(\text{TTA})_3(\text{phen})$ (TTA = thenoyltri fluoroacetone, phen = 1, 10 -

phenanthroline), was synthesized by solution techniques.

For the synthesis of $\text{Eu}(\text{TTA})_3(\text{phen})$. following starting chemicals were used.

- i) EuCl_3 purity 99.5 % ,M.W.= 351.92 ,Indian Rare Earth Limited, Rare Earth Division, Udyogamandal, Kerala, India.
- ii) Ethanol absolute ($\text{C}_2\text{H}_5\text{OH}$), GR, purity 99.98 % from Merck,. 1 litre = 0.79 Kg, M = 46.07 g/mol.
- iii) 2-Thenoyltrifluoroacetone (TTA) $\text{C}_8\text{H}_5\text{F}_3\text{O}_2\text{S}$, M.W. = 222.09 g/mol ,(AR) purity 99.5 %, Burgoyne urbidges &co. India.
- iv) 1, 10 phenonthroline (monohydrate), $\text{C}_{12}\text{H}_8\text{N}_2\text{H}_2\text{O}$ M.W. = 180.0784 – purity 99.5 %.,Burgoyne urbidges &co. India.
- v) KOH – purity 99.98 % purchase from Merck
- vi) HCl ,69% GR grade, Merck make

The stoichiometry of chemical compounds for the formation of $\text{Eu}(\text{TTA})_3(\text{Phen})$ as follows.

1:10 phenanthroline Europium chloride 2-thenoyltrifluoroacetone

(Phen)	(EuCl_3)	(TTA)
1 Mol	1 Mole	3 Mol

The synthesis route for the formation of $\text{Eu}(\text{TTA})_3\text{phen}$ is shown in the following fig (1)

Stoichiometric molar calculation for syntheses of $\text{Eu}(\text{TTA})_3\text{Phen}$ is as follows

$$[\text{EuCl}_3] + 3* [\text{TTA}] + [\text{Phen}] \{ \text{M.W. of } \text{EuCl}_3 \} + \{ 3* \text{M.W. of TTA} \} + \{ \text{M.W. of Phen} \} \{ 258.32 \} + \{ 3*222.19 \} + 198.224 \quad 258.32 + 666.57 + 198.224$$

We had taken

- 1) TTA= 2 gram.
- 2) Phen= $(2*198.224) / 666.57 = 0.5947$ gram
- 3) $\text{EuCl}_3 = \{(2*258.32) / 666.57\} = 0.7750$ gram.
- 4) Potassium hydroxide of 1 N solution

1 N of KOH= 5.611 g. in 100 ml distilled water.

PROCEDURE

Synthesis of $\text{Eu}(\text{TTA})_3\text{Phen}$ can be performed in following steps

Step-1 :-

0.5947 gram of 1, 10 phenonthroline (Phen) and 2grams of 2-thenoyltrifluoroacetone (TTA) were dissolved in 20ml of ethanol.

Step -2:-

P_H value of dissolved solution was observed around 5-6. P_H value was adjusted to 7 by adding KOH solution drop by drop. (If P_H value become greater than 7, it means solution become more alkaline. In this case add HCL drop by drop and every drop check P_H)

Step-3 :-

0.7750 gram. of EuCl_3 was dissolved in a 10ml of distilled water. The P_H value of obtained solution was adjusted to 3.48 by pouring dil. HCl and KOH as stated in step 3. Crystal transparent solution was again observed

Step-4 :-

The solution of EuCl_3 was added into the solution of TTA and Phen drop by drop. The entire mixture was heated to 60°C with continuous stirring for 1hr. Pale –coloured precipitate was obtained.

Step-5 :-

The obtained precipitate was filtered out and washed with distilled water. The resulting precipitate was then dried for 2 hrs at 80°C in an oven. In this way $\text{Eu}(\text{TTA})_3\text{Phen}$ complex was synthesized.

RESULTS AND DISCUSSIONS

3.1 Fourier transform infrared spectroscopy (FTIR) Analysis

To confirm the molecular structure of synthesized $\text{Eu}(\text{TTA})_3\text{Phen}$ by knowing vibrational bonding, FTIR analysis is carried out. Fig (3) represents FTIR image of $\text{Eu}(\text{TTA})_3\text{Phen}$. Image indicate that stretching vibration of TTA , regarding C=O bond at 1624 cm^{-1} , C=C bond at 1535 cm^{-1} . It is observed that stretching vibration of TTA is found to be shifted to lower side from original wavenumber [9]. Peak at 493 cm^{-1} and 461 cm^{-1} resembles to stretching vibration of Eu-O bond. As a result coordination bonds are formed between Eu and TTA [10]. Secondly , stretching vibration peaks of phen regarding C-H bond obtained at 724 cm^{-1} and 843 cm^{-1} and C=N bond at 1601 cm^{-1}

1. These peaks are also shifted to lower side of wavenumber. This indicates the participation of coordinate reaction [11-13].

3.2 Morphology of $\text{Eu}(\text{TTA})_3 \text{Phen}$

SEM images of $\text{Eu}(\text{TTA})_3 \text{Phen}$ is shown in fig (4). Apparently this picture indicate rectangular shaped crystals and size is about approximately 10 μm . Diameter of crystal is not uniform. Large surface area leads to increase emission center of Eu^{3+} that can exposed to near uv excitation. Ultimately, more photoluminescence emission is expected.

Optical properties of $\text{Eu}^{3+}(\text{TTA})_3 \text{Phen}$

In spite of efforts of several groups to apply europium complexes as red emitters in electroluminescent devices, only marginal achievement has been obtained [14]. Because of shielding effect of electrons in the outer shells, inner 4 f electronic transitions of the RE ions give rise to a narrow emission band [15]. The mean energies of Eu^{3+} emission along with the possible transition during Eu^{3+} emission is given in Fig(5) Most europium complexes used in electroluminescent devices absorb UV light and emit red light at 612 nm due to the unique mechanism of excitation explained in the following steps: -

- 1) Excitation of ligands under UV absorption to form excited singlet state S1 of the ligands.
- 2) Relaxation from singlet state S1 of the ligands to triplet state T1 of the ligands by intersystem crossing.
- 3) Energy is transformed from triplet state of ligands to the center RE (in this case Eu) by intramolecular energy transfer i.e. center RE ion is excited, which rises the 4f electron of the ion to excited energy level[16].
- 4) Finally, radiative electronic transitions back to the ground state or to other lower state give off photons.

From the above mechanism it is clear that RE ions primarily accept the excitation energy of triplet but not that of siglet state of the ligands. As a result 100% efficiency of energy transfer from ligands to

the center ion is only possible when the efficiency of the ligands internal conversion from singlet to triplet is unity.

$\text{Eu}(\text{TTA})_3$, (TTA= thenoyltrifluoroacetone) is efficient red emitter complex, but it is non-volatile and hence it is difficult to fabricate thin film by vacuum thermal deposition. Hence there is a need to attach another complex with $\text{Eu}(\text{TTA})_3$ using the concept of synergistic effect. Synergism effect is the introduction of a second ligand into metal complexes to neutrilize and saturates the co-ordination number of metal complex. Co-ordination number is the no. of attachment of ligands to center metal Re ions. The non- volatile nature of $\text{Eu}(\text{TTA})_3$ originates from its high polarity due to unsaturated co-ordination number polarity. The polarity is decreased when the second ligand “Phen” (Phen=1, 10 – phenanthroline) is introduced into $\text{Eu}(\text{TTA})_3$, so as to form $\text{Eu}(\text{TTA})_3\text{Phen}$.

1:10 phenanthroline and thenoyltrifluoroacetone are bidentate (Bidentate=point of attachment to Eu is 2). Eu^{3+} is associated with 3 molecules of TTA and 1 molecule of phen. Hence Eu^{3+} has got a eight coordinate (six with TTA and two with phen). Higher the co-ordination number, more chemically stable will be the complex. Hence $\text{Eu}(\text{TTA})_3\text{Phen}$ is more chemically stable than $\text{Eu}(\text{TTA})_3$

Fig 7(a) & (b) shows the PL excitation and emission spectra of $\text{Eu}(\text{TTA})_3\text{Phen}$ respectively. As shown in figure 5 (a) the excitation spectrum of the pure $\text{Eu}(\text{TTA})_3\text{phen}$ complex exhibits a broad excitation band (BEB) between 200 and 500 nm ($\lambda_{\text{max}} = 385 \text{ nm}$), which can be assigned to the $\pi\text{-}\pi^*$ electron transition of the ligands. A peak at 467 nm is observed due to the f-f absorption transition ($7\text{F}_0 \rightarrow 5\text{D}_2$) of Eu^{3+} ion. This transition is weaker than the absorption of the organic ligands and overlapped by BEB, which proves that luminescence sensitization via excitation of the ligands is much more efficient than the direct excitation of the Eu^{3+} ion absorption level. The maximum emission peak was observed at 612 nm

with smaller peak 581 nm, 592 nm and 652 nm when excitation wavelength is 385 nm. [17-19]. As no emission from the ligands is detected, the emission spectrum (excited at 385 nm) displays only the intra- 4f_6 , $^5D_0 \rightarrow ^7F_J$ ($J = 0, 1, 2, 3, \text{ or } 4$) transitions of Eu^{3+} ion; this energy transfer process seems to be very efficient for the $\text{Eu}(\text{TTA})_3\text{phen}$ complex. The observed number of Stark components for $^5D_0 \rightarrow ^7F_J$ ($J = 0, 1, \text{ and } 2$) transitions is 1, 3, and 5, respectively. The other transitions are negligible since their intensities are weak and their Stark components are not completely resolved. The stark splitting reveals an ordered “crystalline” rare-earth ion environment, rather than an amorphous one[20].

3.4 CIE Coordinate of $\text{Eu}^{3+}(\text{TTA})_3\text{Phen}$

CIE (International commission on illumination) is standard reference for measurement of colour space. 612nm red emission of $\text{Eu}(\text{TTA})_3\text{Phen}$ shows CIE colour two-dimensional coordinate ($x=0.64, y=0.33$) in the following fig. (6). CIE colour coordinate shows exact matching of colour bonding.

3.5 Development of blended film from $\text{Eu}(\text{TTA})_3\text{Phen}$ with PMMA

Blended thin film of $\text{Eu}(\text{TTA})_3\text{phen} : \text{PMMA}$ (polymethyl methacrylate) at concentration ratio **1:100** can be obtained in the following way:-

1 gram of PMMA was taken & dissolved it in 20 mL of chloroform in a stainless steel plate. To this paste like solution, the solution of **0.01gram** of $\text{Eu}(\text{TTA})_3\text{phen}$ in 10 mL chloroform was added. The entire mixture was stirred well and kept as it is for several hrs. for evaporation to dryness. Blended thin film of $\text{Eu}(\text{TTA})_3\text{phen} : \text{PMMA}$ at concentration ratio 1:100 was obtained. Blended thin film of $\text{Eu}(\text{TTA})_3\text{phen} : \text{PMMA}$ at concentration ratio 10:100, 0.1:100 were obtained similarly.

3.6 PL Tuning of $\text{Eu}(\text{TTA})_3\text{phen}$

In recent years, a substantial amount of work has been carried out in the area of polymer matrices doped by low molecular weight materials. We have investigated the photoluminescence characterization of $\text{Eu}(\text{TTA})_3\text{phen}$ embedded in polymethyl methacrylate (PMMA) at different

concentration. We have recorded PL characteristics of blended film of $\text{Eu}(\text{TTA})_3\text{phen} : \text{PMMA}$ at concentration ratio of 1:100, 0.1:100.

In Fig 10, no noticeable change is observed in emission spectra of 1% $\text{Eu}(\text{TTA})_3\text{Phen}$ in blended film with acrylic from original powder of $\text{Eu}(\text{TTA})_3\text{Phen}$. At an excitation wavelength =380nm, maximum emission peak in blended film for all concentration of $\text{Eu}(\text{TTA})_3\text{phen}$ is same and it is 612nm. This indicates that there is no emission from the ligands (TTA & Phen). Entire emission is observed from electronic transitions of Eu^{3+} ion. In Fig 9, We observed a blue shift in the excitation spectrum of $\text{Eu}(\text{TTA})_3\text{phen}$ blended films and it is significant between $\lambda = 320-390\text{nm}$. Emission intensity of blended film is less than the powder $\text{Eu}(\text{TTA})_3\text{Phen}$ but emission wavelength is same in both cases, because emission occurs due to $^5D_x \rightarrow ^7F_x$ transition of Eu^{3+} ions.

CONCLUSION

$\text{Eu}(\text{TTA})_3\text{Phen}$ absorbed near UV light of 385 nm and emit red light at 612 nm due to $^5D_0 \rightarrow ^7F_2$ electric -dipole transition. Hence, $\text{Eu}(\text{TTA})_3\text{Phen}$ is the best candidate for red emitting PLLCD phosphor. Concentration quenching effect is not observed due to homogeneous dispersion of Eu ion. Secondly, PMMA does not play any role either in emission or in energy transfer. But it increases the stability of the complex. When sample is excited at 385nm, the energy absorbed by TTA of $\text{Eu}(\text{TTA})_3\text{Phen}$ and transfer to Eu^{3+} . No energy is absorbed by PMMA.

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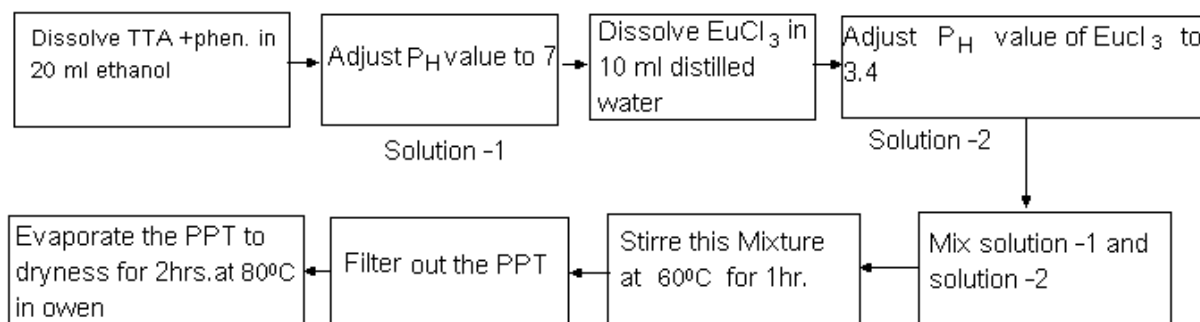


Fig (1) Flow chart of Eu(TTA)₃ phen synthesis

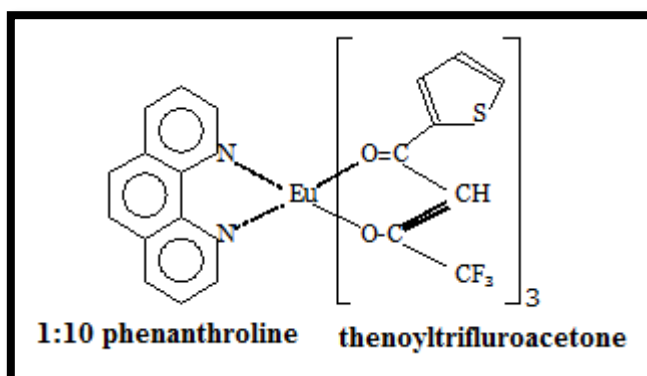


Figure (2): Chemical structure of Eu(TTA)₃(phen).

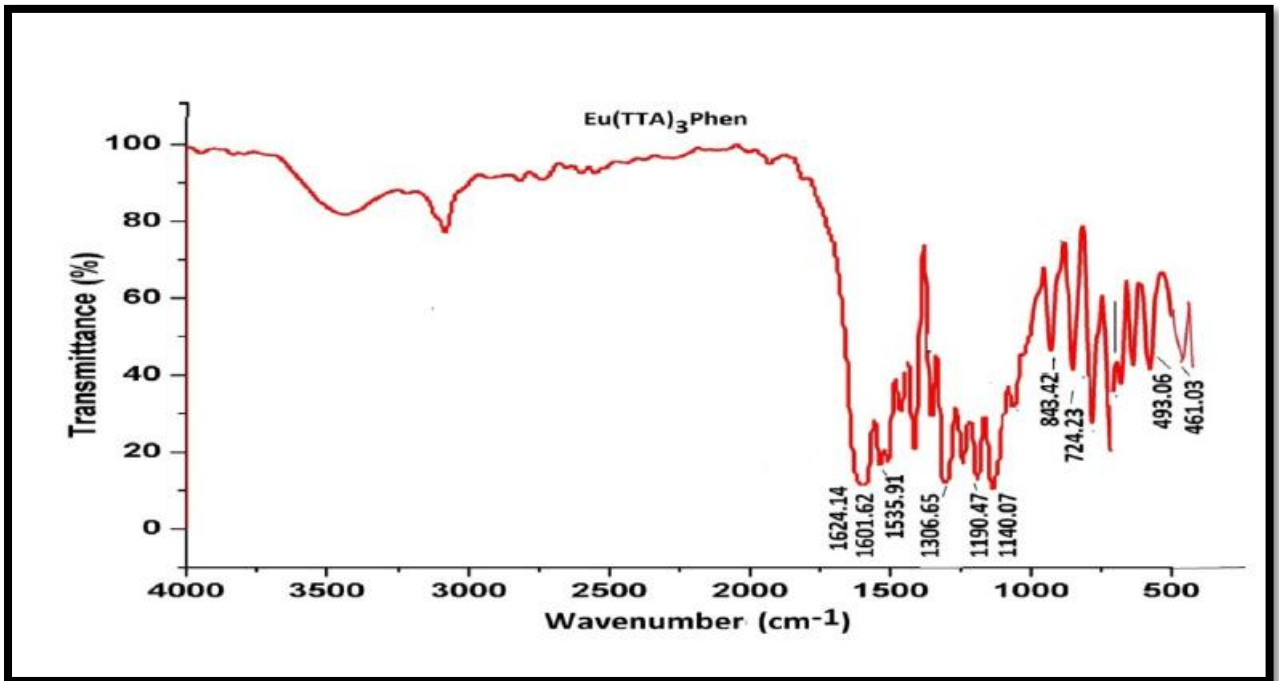


Fig (3) FTIR image of Eu(TTA)₃Phen

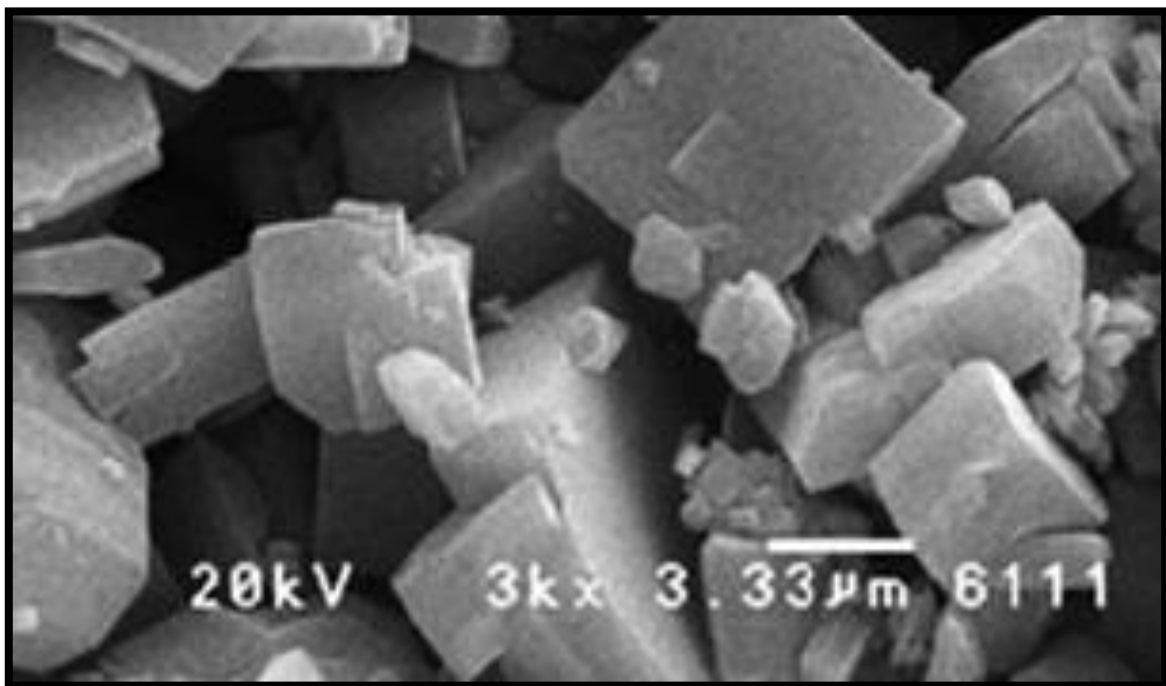


Fig (4) SEM image of Eu³⁺(TTA)₃Phen

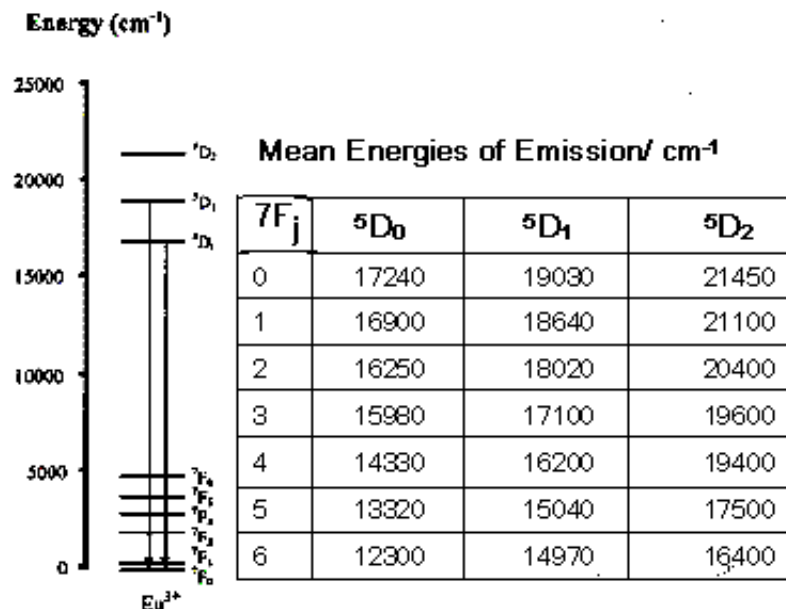


Fig (5) Possible transition during Eu³⁺ emission

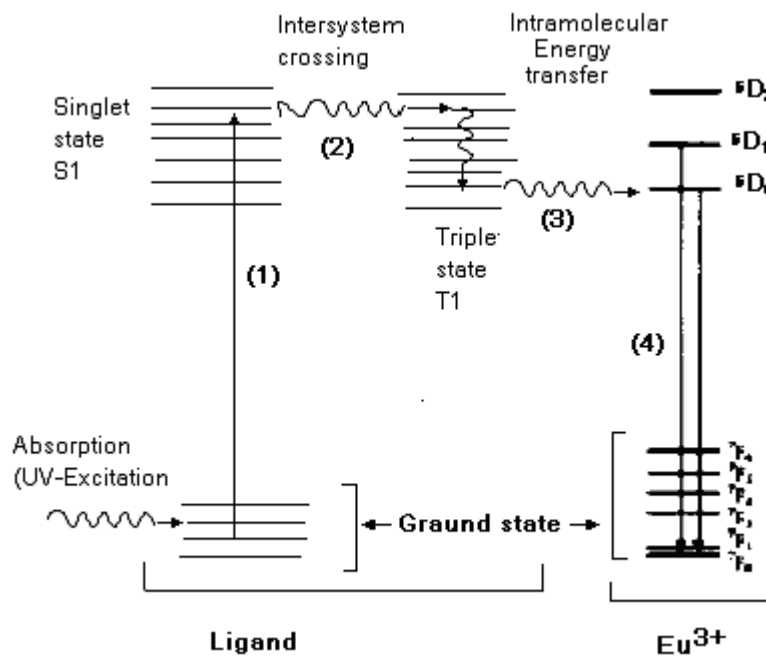


Fig (6) Excitation- Emission Mechanism of Eu³⁺ - complexes

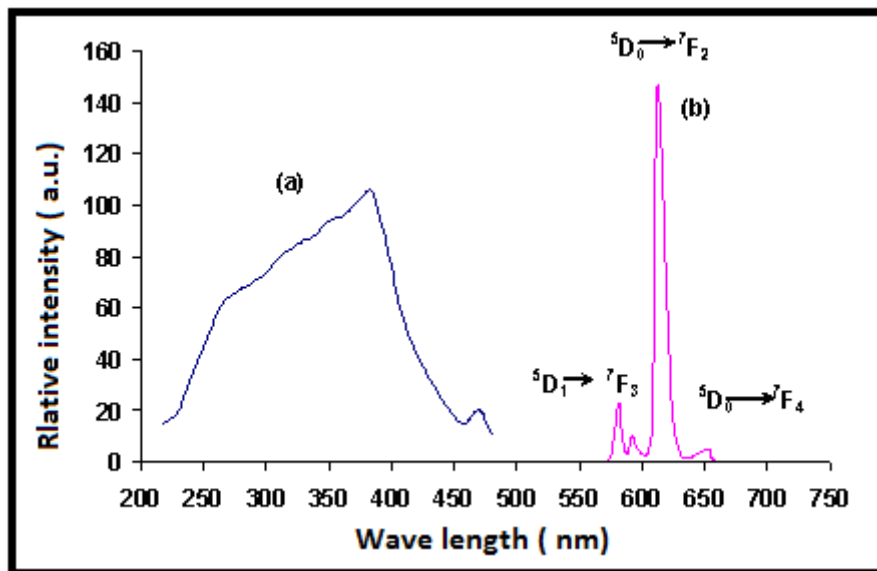


Fig 7 PL – spectra of $\text{Eu}(\text{TTA})_3\text{Phen}$ (a) Excitation spectra of $\text{Eu}(\text{TTA})_3\text{Phen}$ for emission wavelength =612 nm (b) Emission spectra of $\text{Eu}(\text{TTA})_3\text{Phen}$ showing maxima at 612nm with shoulder at 581 nm,592 nm and 652 nm for excitation wavelength= 385 nm

Table -1 Electronic transition of Eu^{3+} for obtained $\text{Eu}(\text{TTA})_3\text{phen}$ complex

Emission peak	Electronic transition
581 nm	$^5\text{D}_1 \rightarrow ^7\text{F}_3$
592nm	$^5\text{D}_0 \rightarrow ^7\text{F}_1$
612nm	$^5\text{D}_0 \rightarrow ^7\text{F}_2$
652nm	$^5\text{D}_1 \rightarrow ^7\text{F}_4$

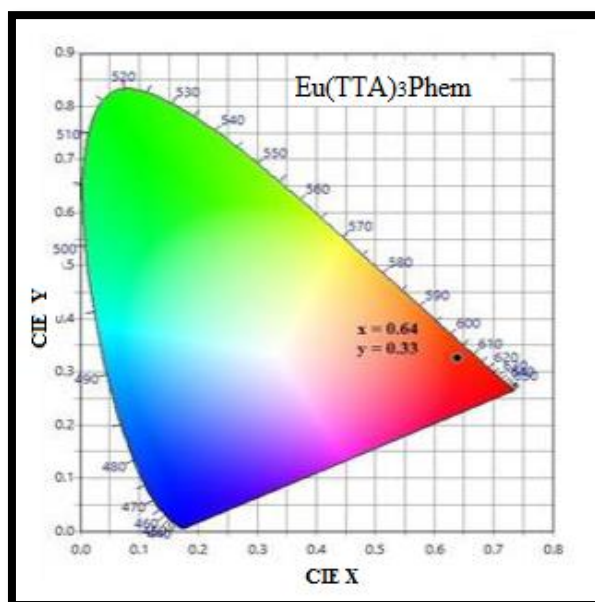


Fig (8) CIE Colour Coordinate of $\text{Eu}(\text{TTA})_3\text{Phen}$

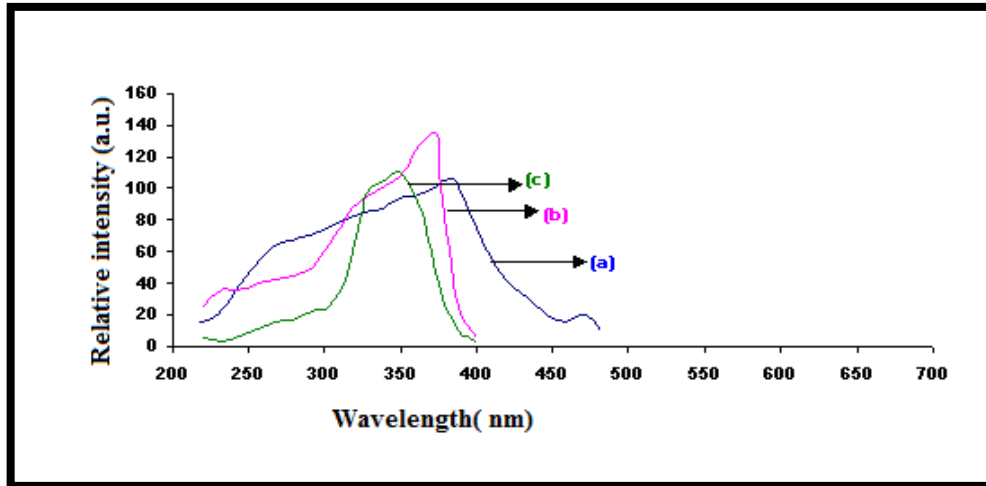


Fig 9 Excitation spectra for 612nm emission (a) Powder $\text{Eu}(\text{TTA})_3\text{Phen}$ showing max. peak at 385nm.(b) 1% $\text{Eu}(\text{TTA})_3\text{Phen}$ in blended film with acrylic showing max.peak at 374nm.(c) 0.1% $\text{Eu}(\text{TTA})_3\text{Phen}$ in blended film with acrylic showing max.peak at 347nm

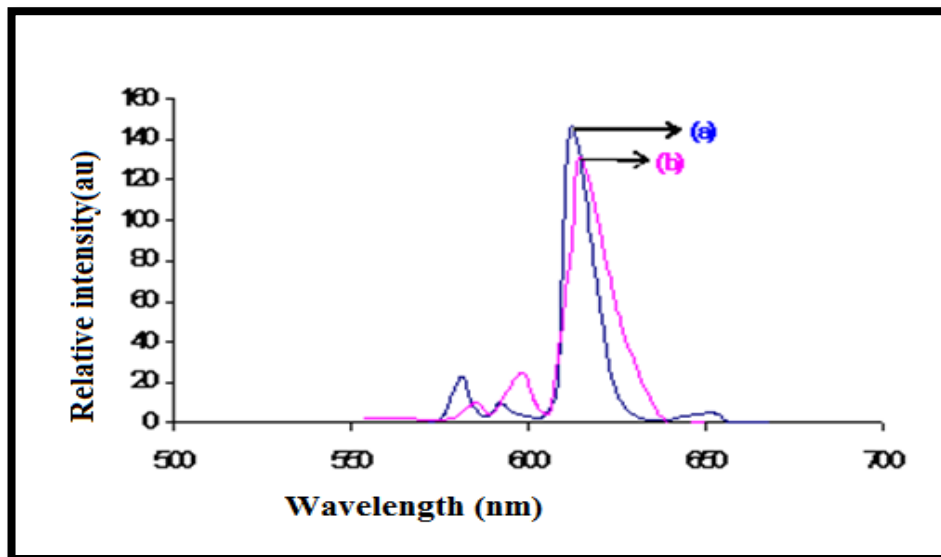


Fig 10 Emission spectra for 380nm excitation

(a) Powder $\text{Eu}(\text{TTA})_3\text{Phen}$ showing max.peak at 612nm.

(b) 1% $\text{Eu}(\text{TTA})_3\text{Phen}$ in blended film with acrylic showing max.peak at 612nm