



# Photoluminescence Properties of Sm(III) Complex with β-hydroxy ketone Ligand Amplified by usage of 5-Nitro-1, 10-phenanthroline

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#### ABSTRACT

Ternary Sm(III) ion complex, Sm(HDMPE)<sub>3</sub>.nphen is synthesized through usage of solution precipitation method which is further characterized by numerous techniques such as elemental analysis, <sup>1</sup>H-NMR, FT-IR and photoluminescence spectroscopy. We have also probed luminescence decay time as well as color coordinates of the evolved complex. When the excitation of the evolved complex is monitored at 373 nm in solid state at room temperature, it showcased pure sharp emission bands from f-f transitions of samarium ion,  ${}^{4}G_{5/2} \rightarrow {}^{6}H_{5/2}$ ,  ${}^{4}G_{5/2} \rightarrow {}^{6}H_{7/2}$  and  ${}^{4}G_{5/2} \rightarrow {}^{6}H_{9/2}$  at 564 nm, 600 nm and 646 nm respectively. The Sm(III) ion can be efficiently sensitized alone by 1-(2-hydroxy-4,6-dimethoxyphenyl)ethanone (HDMPE) but the introduction of adjuvant ligand 5-Nitro-1, 10-phenanthroline (nphen) have outcomed in amplified photoluminescence. The photoluminescence emission spectra showcased that ternary complex might be a promising candidate for usage in advanced displays and lighting systems.

Keywords: Samarium complex, characterization, amplified photoluminescence properties, NMR, IR.

#### **INTRODUCTION**

ecently, Ln<sup>3+</sup> ion complexes have garnered significant thoughtfulness of research fraternity. These complexes have exhibited extraordinary solicitations in numerous arenas such as biomedical sensors as well as bio-medical imaging. Here, when doped in polymers, these complexes can be used for sensing minute quantities of biomolecules which then can have various solicitations such as assessing physical state of any patient<sup>1, 2</sup>, for usage inorganic light emitting diodes<sup>3-8</sup> or for amplification of optical properties<sup>9, 10</sup>. These Ln<sup>3+</sup> ion complexes have showcased distinctive photoluminescent characteristics like high color purity, narrow emission bandwidths in the visible as well as nearinfrared regions of the spectra, large Stokes shifts and lifetimes up to several milliseconds<sup>11</sup>. Nevertheless, direct excitation of Ln<sup>3+</sup> ion is not efficacious as these ions exhibits feeble fluorescent properties primarily owing to its low quantum yield as well as small absorption crosssection<sup>12</sup>. Henceforth, any strong absorbing organic chromophore like  $\beta$ -diketone,  $\beta$ -hydroxy ketone or  $\beta$ aminoketone were employed as light gatherer for sensitizing Ln<sup>3+</sup> ion through antenna effect<sup>13-15</sup> which will ultimately enhance Ln<sup>3+</sup> ion emission. Finally, 1-(2hydroxy-4,6-dimethoxyphenyl) ethanone (HDMPE)is picked as prime ligand primarily owing to its dual role of bridging varied building blocks as well as chelating central metal ion which outcomes in amplified photoluminescence as well as quantum yield.

Adjuvant ligands like biquinoline or 1,10-phenanthroline or bathophenanthroline or bipyridine are recurrently picked up by most researchers for carrying out synthesis of ternary lanthanide metal complexes as these bidentate chelating ligands replaces water molecules from coordination sphere of central metal ions<sup>16-18</sup>, thereby satisfies coordination number of lanthanide ions which will ultimately lead to amplified luminescence properties. Consequently, for seeking newer photo luminescent material showing outstanding luminescence properties, 5-Nitro-1, 10-phenanthroline (nphen) is picked here as adjuvant ligand for synthesizing ternary Sm(III) ion complex "Sm (HDMPE)3.nphen" through usage of 1-(2hydroxy-4,6-dimethoxyphenyl) ethanone (HDMPE) as prime ligand. The complex formed is characterized by analysis and numerous elemental spectroscopic techniques. The photoluminescence emission spectrum of this complex shows the introduction of adjuvant ligand 5-Nitro-1, 10-phenanthroline (nphen) have outcomed in amplified photoluminescence. Hence this ternary complex might be a promising candidate for usage in advanced displays, organic light emitting devices and lighting systems.

## MATERIALS AND METHODS

5-Nitro-1, 10-phenanthroline, Sm(NO<sub>3</sub>)<sub>3</sub>6.H<sub>2</sub>O (99.9), benzene-1.3-5-triol. dimethyl sulphate. potassium carbonate and sodium hydroxide were purchased from Sigma-Aldrich and used as received without additional purification. The synthesized ligand HDMPE was recrystalized three times with methanol before synthesis of complexes. The elemental analysis was performed using thermoscientific flash 2000 elemental analyzer. The percentage of Sm (III) was estimated by complexometric titration with EDTA. The <sup>1</sup>H-NMR spectra were measured Bruker Avance II 400 spectrometer using on tetramethylsilane (TMS) as an internal reference (chemical shift in  $\delta$  ppm). Infrared spectra were recorded (Perkin Elmer spectrum 400) from 4000–400 cm<sup>-1</sup> in KBr pellets. The excitation and emission spectra was



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measured using Hitachi F-7000 fluorescence spectrophotometer with excitation and emission slits of 2.5 nm and with PMT at 400 V at room temperature in solid state. The lifetime value of the complex was **Synthesis**  calculated by the software of the spectrophotometer (FL solution for F-7000) by monitoring  ${}^{4}G_{5/2} \rightarrow {}^{6}H_{7/2}$  emission line of Sm<sup>3+</sup> at room temperature in solid state.

#### Synthesis of ligand 1-(2-hydroxy-4,6-dimethoxyphenyl)ethan-1-one (HDMPE)

The ligand HDMPE was synthesized by adopting conventional method as per literature<sup>19</sup> and is given in Scheme 1 as follow:



#### Synthesis of complex Sm(HDMPE)<sub>3</sub>.nphen

Scheme 2 depicts the synthetic route of complex  $Sm(HDMPE)_3$ .nphen. The complex was synthesized by mixing ethanolic solution of 3 mmol HDMPE ligand, and 1 mmol nphen with ethanolic solution of 1 mmol  $Sm(NO_3)_3.6H_2O$ . Afterwards the pH of mixture was adjusted to 6.5 - 7, using NaOH (0.05 M) solution with constant stirring. This resulted into formation of white

precipitates. These precipitates were stirred for 3 h at about 40°C and then allowed to digest for 1 h. Finally, a suction filter was used to filter precipitates, washed with doubly distilled water and then with ethanol, dried in vacuum oven at 50°C. The obtained complex was white powder with 83 % yield. The powdered the complex was stored in sample tube in vacuum desiccator.

$$H_{3}CO \xrightarrow{HO \qquad OCH_{3}} + Sm^{3+}(aq) \xrightarrow{NaOH (aq)} nphen (alc)$$



Sm(HDMPE)<sub>3</sub>.nphen

Scheme 2. The synthetic route of Sm(HDMPE)<sub>3</sub>.nphen

## **RESULTS AND DISCUSSION**

## Solubility

The complex Sm(HDMPE)<sub>3</sub>.nphen was stable under atmospheric condition. The complex Sm (HDMPE)<sub>3</sub>.nphen was found to be soluble in dimethyl sulfoxide, dimethyl formamide, chloroform and acetone, sparingly soluble in methanol and ethanol but insoluble in benzene and hexane.

# Elemental analysis, <sup>1</sup>H-NMR and IR Spectra

The elemental analysis data for HDMPE ( $C_{10}H_{12}O_4$ ) were found (calculated) % C, 60.87 (61.22); H, 6.14 (6.16); O, 32.19 (32.61) IR (KBr) cm<sup>-1</sup> 3430 (b), 3099 (m), 3005 (w), 2943 (w), 2847 (w), 1640 (s), 1538 (s), 1457 (m), 1366 (s),

1324 (m), 1270(s), 1221 (s), 1207 (s), 1112 (m), 1079 (m), 1045 (w), 896 (m), 835 (m), 658 (m), 594 (s). <sup>1</sup>HNMR (400 MHz, DMSO): d 2.52 (s, 3H, CH3), 3.83 (s, 6H, OCH3), 6.02 (s, 2H, Ar-H), 13.84 (s, 1H, OH).

The elemental analysis data for Sm(HDMPE)<sub>3</sub>.nphen  $(C_{42}H_{40}O_{14}N_3Sm)$  was found (calculated) % C, 52.32 (52.48); H, 4.17 (4.19); N, 4.29 (4.37); O, 23.32 (23.38); Sm, 15.42 (15.64). IR(KBr):cm<sup>-1</sup>2934 (m), 2454 (m), 2326 (w), 1617 (m), 1587 (s), 1562 (s), 1486 (s), 1372 (s), 1325(m), 1236 (s), 1211 (m), 1142 (m), 1127 (s), 1056 (m), 907 (m), 874 (s), 841 (m), 823 (s), 787(m), 769 (s), 682 (s), 621 (m), 585 (m), 432 (m).<sup>1</sup>HNMR (400 MHz, DMSO): d 2.49 (bs, 9H, CH3), 3.47 (bs, 18H, OCH3), 6.22 (bs, 6H, Ar-



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H),7.66 (bd, H, nphen), 7.90 (bd, H, nphen), 8.36 (bd, H, nphen), 8.67 (bs, H, nphen), 8.90 (bd, H, nphen), 9.06 (bd, H, nphen), 9.12 (bd, H, nphen).

The above elemental analytical data indicate the stoichiometry of the ternary complex Sm(HDMPE)<sub>3</sub>.nphen to be 3:1:1 (HDMPE: Sm: nphen). The <sup>1</sup>H-NMR spectrum of the ligand HDMPE showed singlet at  $\delta$  13.84 due to phenolic proton which disappeared in the complex Sm(HDMPE)<sub>3</sub>.nphen was indicating that ligand is coordinated with Sm(III) ion through the oxygen atom of phenolic OH group of the ligand HDMPE. The FT-IR spectra of ligand HDMPE exhibits abroad absorption band at 3430 cm<sup>-1</sup> assigned to v(O-H) stretching vibration  $^{[20-22]}$ which disappeared in the IR spectra of complex the Sm(HDMPE)<sub>3</sub>.nphen. The ligand also displays the intense C=O stretching vibration band at 1640 cm<sup>-1</sup>, which was red shifted 23 cm<sup>-1</sup> in complex the Sm(HDMPE)<sub>3</sub>.nphen, indicating that phenolic and carbonyl group of HDMPE participated in coordination with Sm(III) ion<sup>23</sup>. The strong absorption band at 1587 cm<sup>-1</sup> in complex the Sm(HDMPE)<sub>3</sub>.nphen assigned to C=N stretching vibration, provided good evidence that the nitrogen atoms of nphen were coordinating with the Sm(III) ion<sup>18, 24</sup>. The strong absorption band at 1562 cm<sup>-1</sup> in complex the Sm(HDMPE)<sub>3</sub>.nphen assigned to N=O stretching vibration. The peak for Ph-O vibration of the ligand HDMPE present at 1270 cm<sup>-1</sup> showed a red shift of 34cm<sup>-1</sup> in the complex Sm(HDMPE)<sub>3</sub>.nphen, indicating that the phenolic group is involved in coordination with the Sm(III) ion. The appearance of absorption bands at 585cm<sup>-1</sup> and at 432 cm<sup>-1</sup> in the complex Sm(HDMPE)<sub>3</sub>.nphen was assigned to v(Sm-N) and v(Sm-O)<sup>18,25</sup> respectively, which affirms that the nitrogen atoms of the nphen and oxygen atoms of the ligand HDMPE participated in coordination with the Sm(III)ion. Finally, it can be concluded from the FT-IR and <sup>1</sup>H-NMR spectra of the ligand HDMPE and complex Sm (HDMPE)<sub>3</sub>.nphen, that the coordination of Sm(III) was through the oxygen atoms of phenolic and carbonyl group of ligand HDMPE and nitrogen atoms of the nphen.

## **Photoluminescent properties**

Figure 1 depicts the excitation profile of the Sm (III) complex Sm (HDMPE)<sub>3</sub>.nphen, recorded by monitoring  ${}^{4}G_{5/2} \rightarrow {}^{6}H_{7/2}$  transition, inset shows the excitation spectrum of ligand HDMPE. The excitation spectrum of ligand HDMPE shows a broad band extending from 260 to 435 nm centred at 350 nm assigned to  $\pi \rightarrow \pi^*$  transition. While the excitation spectra for the solid complex Sm(HDMPE)<sub>3</sub>.nphen, narrates the broad excitation band in the region 250 to 450 nm centered at 373 nm assigned to  $\pi \rightarrow \pi^*$  transition which shifted 23 nm as compared to free ligand indicated the larger conjugated system in the complex Sm(HDMPE)<sub>3</sub>.nphen. While excitation peaks centred at 401 nm appeared in the complex Sm(HDMPE)<sub>3</sub>.nphen assigned to the electronic transition  ${}^{6}H_{5/2} \rightarrow {}^{4}F_{7/2}$  of Sm(III). The excitation range from 345- 420 is fairly appropriate to meet the demands of UV LED. The most intense peak at 373 nm apparently show that the complex Sm(HDMPE)<sub>3</sub>.nphen effectively excited by near ultra violet light.



**Figure 1:** Photoluminescence excitation spectra of Sm(HDMPE)<sub>3</sub>.nphen, monitored at  $\lambda_{em}$  = 600 nm in solid state at room temperature and inset shows the excitation spectrum of ligand HDMPE.



Figure 2: Photoluminescence emission spectra of Sm(HDMPE)<sub>3</sub>.nphen in solid state at room temperature, monitored at  $\lambda_{ex}$  = 373 nm.

The emission profile of Sm(HDMPE)<sub>3</sub>.nphen (Figure 2) shows three identified emission peaks corresponding to characteristic emission transition  ${}^{4}G_{5/2} \rightarrow {}^{6}H_{5/2}$  (magnetic dipole transition),  ${}^{4}G_{5/2} \rightarrow {}^{6}H_{7/2}$  (mixed magnetic-electric dipole transition) and  ${}^{4}G_{5/2} \rightarrow {}^{6}H_{9/2}$  (electric dipole transition) at 564 nm, 600 nm and 646 nm respectively<sup>26-28</sup> on monitoring the excitation at 373 nm in solid state at room temperature as shown in Table 1.

Different from the ternary Sm(HDMPE)<sub>3</sub>.nphen complex, the magnetic dipole transition  ${}^{4}G_{5/2} \rightarrow {}^{6}H_{5/2}$  is the strongest emission in case of binary complex<sup>22, 29</sup>. The magnetic-electric dipole transition  ${}^{4}G_{5/2} \rightarrow {}^{6}H_{7/2}$  was strongest in case of ternary complex Sm(HDMPE)<sub>3</sub>.nphen while the electric dipole  ${}^{4}G_{5/2} \rightarrow {}^{6}H_{9/2}$  transition was subsidiary in most of Sm(III) complexes<sup>30, 31</sup>.

The photoluminescence intensity of ternary complex was amplified as compared to the binary complex, because in the ternary complex OH groups was removed from the inner coordination sphere of Sm(III) ion.



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Complex	λ <sub>ex</sub> (nm)	λ <sub>em</sub> (nm)	Assignment	τ (ms)	x and y coordinates
Sm(HDMPE) <sub>3</sub> .nphen	373	564 600 646	${}^{4}G_{5/2} \rightarrow {}^{6}H_{5/2}$ ${}^{4}G_{5/2} \rightarrow {}^{6}H_{7/2}$ ${}^{4}G_{5/2} \rightarrow {}^{6}H_{9/2}$	0.795	0.5023, 0.4773

Table 1: Photoluminescence data of Sm(HDMPE)<sub>3</sub>.nphen in solid state.

The replacement of water molecules by aromatic Ndonors adjuvant ligand 'nphen' results in amplified photoluminescence intensity and stability of complexes <sup>[23, 32]</sup>. Further, it was noticed that the adjuvant ligands in the ternary complex amplified photoluminescence intensity greatly due to the coordination effect between the ligand and adjuvant ligands which is beneficial for efficient intermolecular energy transfer from ligand to  $Sm^{3+}$  ion. In the lanthanide complexes the intensity ratio of the electric dipole transition to the magnetic dipole transition act spectroscopic probe to determine the local environment around luminescent center. The Sm(HDMPE)<sub>3</sub>.nphen possess low intensity ratio indicating that the Sm(III) ion occupies site symmetry with an inversion centre.

Figure 3 shows the photoluminescence decay curves for complex Sm(HDMPE)<sub>3</sub>.nphen at  $\lambda_{em}$ = 600 nm and  $\lambda_{ex}$ = 373 nm in solid state at room temperature. The decay curve of this luminescent ternary complex obey single exponential curve, which can be represented by the equation *I*=*I*<sub>0</sub> *exp* (-t/ $\tau$ ), where  $\tau$  is the radiative decay time, I and I<sub>0</sub> are the luminescence intensities at time t and 0, respectively. The life time value calculated for complex was found to be 0.795 ms as presented in Table 1.



Figure 3: Luminescence decay curves for Sm(HDMPE)<sub>3</sub>.nphen in solid state at room temperature, monitored at  $\lambda_{ex}$  = 373 nm and  $\lambda_{em}$  = 600 nm.

With the help of Commission International De Eclairage (CIE) chromaticity coordinate diagram, the emission color of the luminescent complex have been analyzed and tabulated in Table 1. The CIE color coordinates (x, y) of the complex are located at 0.5023, 0.4773 which lie in deep orange spectral region as shown in Figure 4, suggesting promising application of this complex in advanced display and lighting systems.



Figure 4: CIE coordinate diagram of Sm(HDMPE)<sub>3</sub>.nphen.

## CONCLUSION

In this work, one newer ternary Sm(III) complex, Sm (HDMPE)<sub>3</sub>.nphen have been synthesized which is further characterized through various techniques like elemental FT-IR, <sup>1</sup>H-NMR and photoluminescence analvsis. spectroscopy. Variation in FT-IR and NMR spectra of free ligand (HDMPE) and evolved complex have indicated that oxygen atoms of both phenolic as well as carbonyl group of prime ligand and nitrogen atoms of adjuvant ligand (nphen) were effectively coordinated to Sm(III)ion. Furthermore, the preferred coordination environment is well-matched for sensitizing luminescence of Sm(III) ion with prime ligand HDMPE and adjuvant ligand, leads to highly efficient and saturated orange photoluminescence. In conclusion, this evolved complex Sm(HDMPE)<sub>3</sub>.nphen has show cased excellent luminescent properties, consequently can have potential solicitations for usage as light emitting materials in lighting systems.

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