

Research Article



Augmented Photoluminescence and Biological Properties of Sm(III) Complex with β -Hydroxyketone Ligand by Usage of 2,2'-bipyridine as Ancillary Ligand

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ABSTRACT

Newer ternary Sm(III) ion complex, Sm(HDMPE)₃.bipy is synthesized through usage of solution precipitation method which is further characterized by numerous techniques such as elemental analysis, ¹H-NMR, FT-IR and photoluminescence spectroscopy. Here, 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, ⁴G_{5/2}→⁶H_{5/2}, ⁴G_{5/2}→⁶H_{7/2} and ⁴G_{5/2}→⁶H_{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 ancillary ligand 2, 2'-bipyridine (bipy) have outcomed in augmented photoluminescence. The photoluminescence emission spectra showcased that ternary complex might be a promising candidate for usage in advanced displays and lighting systems. Furthermore, we have probed antimicrobial properties as well as antioxidant properties of the ligand and their Sm(III) ion complex.

Keywords: Sm(III) complex, augmented photoluminescence properties, antimicrobial activities, antioxidant activities.

INTRODUCTION

Today, Ln³⁺ ion in combination with organic ligands have generated complexes which have garnered significant thoughtfulness of research fraternity. These complexes have exhibited extraordinary solicitations in numerous are in as 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^{1,2}, for usage inorganic light emitting diodes³⁻⁸ or for amplification of optical properties^{9,10}. These Ln³⁺ ion complexes have showcased distinctive photoluminescent characteristics like high color purity, narrow emission bandwidths in the visible as well as near-infrared regions of the spectra, large Stokes shifts and lifetimes up to several milliseconds¹¹. Nevertheless, direct excitation of Ln³⁺ ion is not efficacious as these ions exhibits feeble fluorescent properties primarily owing to its low quantum yield as well as small absorption cross-section¹². Henceforth, any strong absorbing organic chromophore like β -diketone, β -hydroxyketone or β -aminoketone were employed as light gatherer for sensitizing Ln³⁺ ion through antenna effect¹³⁻¹⁵ which will ultimately enhance Ln³⁺ ion emission.

Finally, 1-(2-hydroxy-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 augmented photoluminescence as well as quantum yield.

Ancillary ligands like 1,10-phenanthroline or bathophenanthroline 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 ions¹⁶⁻¹⁸, thereby satisfies coordination number of lanthanide ions which will ultimately lead to augmented luminescence, antimicrobial properties as well as antioxidant properties. Consequently, for seeking newer photoluminescent materials having outstanding luminescence properties, 2, 2'-bipyridine (bipy) is picked here as ancillary ligand for synthesizing one newer ternary Sm(III) ion complex "Sm(HDMPE)₃.bipy" through usage of 1-(2-hydroxy-4,6-dimethoxyphenyl)ethanone (HDMPE) as prime ligand. The complex formed is characterized by numerous spectroscopic techniques which are further validated through elemental analysis. Also, here photoluminescence behaviour of the evolved complex is further probed along with antimicrobial properties as well as antioxidant properties of the ligand and their Sm(III) ion complex.

MATERIALS AND METHODS

2, 2'-bipyridine, Sm(NO₃)₃·6H₂O (99.9), benzene-1,3,5-triol, dimethyl sulphate, potassium carbonate, xylene orange and 1,1-Diphenyl-2-picrylhydrazyl radical (DPPH) were purchased from Sigma-Aldrich and used as received without additional purification. The microorganisms used in antimicrobial activities were purchased from Institute of Microbial Technology, Sector 39-A, Chandigarh, India. Nutrient broth medium, nutrient agar medium, subour and dextrose agar medium and subour and dextrose broth medium were purchased from Hi-Media Pvt Ltd. The synthesized ligand HDMPE was recrystallized 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 $^1\text{H-NMR}$ spectra were measured on Bruker Avance II 400 spectrometer using tetramethyl silane (TMS) as an internal reference (chemical shift in δ ppm).

Infrared spectra were recorded (Perkin Elmer spectrum 400) from 4000–400 cm^{-1} in KBr pellets.

The excitation and emission spectra was 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.

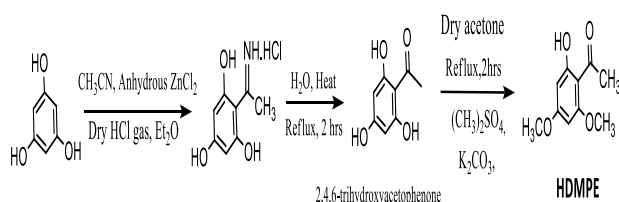
The lifetime value of the complex was calculated by the software of the spectrophotometer (FL solution for F-7000) by monitoring $^4\text{G}_{5/2} \rightarrow ^6\text{H}_{7/2}$ emission line of Sm^{3+} at room temperature in solid state.

Antimicrobial and Antioxidant activities were determined by tube dilution method and DPPH method respectively. All measurements were made at room temperature unless otherwise stated.

Synthesis

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

The ligand HDMPE was synthesized by adopting conventional method as per literature¹⁹ and is given in Scheme 1 as follow:



Scheme 1. The synthetic route of ligand HDMPE.

Synthesis of complex $\text{Sm}(\text{HDMPE})_3\text{.bipy}$

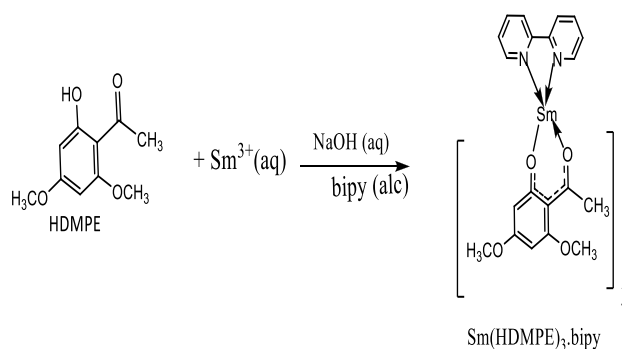
Scheme 2 depicts synthetic route of $\text{Sm}(\text{HDMPE})_3\text{.bipy}$. The complex was synthesized by mixing ethanolic solution of 3 mmol HDMPE ligand, and 1 mmol bipy with ethanolic solution of 1 mmol $\text{Sm}(\text{NO}_3)_3\text{.6H}_2\text{O}$.

Afterwards the pH of mixture was adjusted to 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 35°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 78 % yield. The powdered the complex was stored in sample tube in vacuum desiccator.



Scheme 2. The synthetic route and structure of $\text{Sm}(\text{HDMPE})_3\text{.bipy}$.

Biological Activity

Antimicrobial Activity

The assay was carried out on the synthesized ligand HDMPE and their corresponding Sm(III) ion complex ' $\text{Sm}(\text{HDMPE})_3\text{.bipy}$ ' using tube dilution method²⁰. The following bacteria were used for *in vitro* antibacterial activities, Gram-positive bacteria: *B. subtilis*, *S. aureus* and gram-negative bacterium: *Escherichia coli*. The following fungi were used for antifungal activity *C. albicans* and *A. niger*. The standard drugs ciprofloxacin and fluconazole²¹ have also tested for their antibacterial and antifungal activity at the same concentration under the same condition as that of the tested HDMPE and $\text{Sm}(\text{HDMPE})_3\text{.bipy}$. The dilutions of synthesized complex as well as standard drugs have been prepared in double strength nutrient broth I.P and sabouraud dextrose broth I.P media for bacteria and fungi respectively²². The standard, ligand and complex were dissolved in DMSO to give concentration of 100 $\mu\text{g}/\text{mL}$. The incubation period for HDMPE and $\text{Sm}(\text{HDMPE})_3\text{.bipy}$ were 24 h at 37 °C for bacteria, 48 h at 37°C for *C. albicans* and 7 days at 25 °C for *A. niger* respectively. The zone of inhibitions of the antimicrobial activity has been recorded in terms of minimum inhibitory concentration (MIC).

Antioxidant Activity

The antioxidant activities of the synthesized ligand HDMPE and complex $\text{Sm}(\text{HDMPE})_3\text{.bipy}$ were determined by using DPPH method²³. When DPPH reacts with antioxidant HDMPE and complex ' $\text{Sm}(\text{HDMPE})_3\text{.bipy}$ ', it shows a significant absorption decrease at 517 nm. Methanol was used as a solvent for preparation of various concentrations (25, 50, 75, and 100) $\mu\text{g}/\text{ml}$ of the materials. After a 30 minutes incubation period at room temperature, the absorbance was read against a blank at 517 nm. Tests were carried out in triplicate and ascorbic acid was used as a standard antioxidant. The DPPH scavenging activity is expressed as IC_{50} , whose concentration is sufficient to obtain 50% of maximum scavenging activity. Standard curve is plotted for different concentration of ascorbic acid, ligand and complex. Scavenging of DPPH free radical was calculated as:

$$\text{DPPH Scavenging Activity (\%)} = \left[\frac{(A_c - A_t)}{A_c} \right]$$

Where, A_c is the absorbance of the control reaction and A_t is the absorbance of the test sample.

RESULTS AND DISCUSSION

Solubility

The complex $\text{Sm}(\text{HDMPE})_3\text{.bipy}$ was stable under atmospheric condition. The complex $\text{Sm}(\text{HDMPE})_3\text{.bipy}$ was found to be soluble in dimethylsulfoxide, dimethylformamide, chloroform and acetone, sparingly soluble in methanol and ethanol but insoluble in benzene and hexane.

Elemental Analysis, $^1\text{H-NMR}$ and IR Spectra

The elemental analysis data for $\text{Sm}(\text{HDMPE})_3\text{.bipy}$ ($\text{C}_{40}\text{H}_{41}\text{O}_{12}\text{N}_2\text{Sm}$) was found (calculated) % C, 53.76 (53.85); H, 4.72 (4.63); N, 3.07 (3.14); O, 21.59 (21.52); Sm, 16.81 (16.85). IR (KBr) cm^{-1} : 3006 (w), 2968 (w), 2939 (w), 2838 (w), 1612 (s), 1570 (s), 1538 (s), 1450 (s), 1418 (s), 1365 (s), 1325 (s), 1259 (s), 1220 (s), 1206 (s), 1158 (s), 1120 (s), 1076 (s), 1028 (s), 963 (s), 940 (m), 894 (m), 828 (m), 675 (m), 657 (m), 596 (s), 573 (m), 536 (m), 449 (w). $^1\text{H-NMR}$ (400MHz, DMSO): δ 2.70 (bs, 9H, CH_3), 3.81 (bs, 18H, OCH_3), 6.14 (bs, 6H, Ar-H), 7.29 (d, 2H, bipy), 7.47 (d, 2H, bipy), 7.88 (d, 2H, bipy), 8.41 (d, 2H, bipy).

The above elemental analytical data indicate the stoichiometry of the ternary complex $\text{Sm}(\text{HDMPE})_3\text{.bipy}$ to be 3:1:1 (HDMPE: Sm: bipy). The $^1\text{H-NMR}$ spectrum of the ligand HDMPE showed singlet at δ 13.84 due to phenolic proton which disappeared in the complex $\text{Sm}(\text{HDMPE})_3\text{.bipy}$ 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 broad absorption band at 3430 cm^{-1} assigned to $\nu(\text{O-H})$ stretching vibration²⁴⁻²⁶ which disappeared in the IR spectra of complex the $\text{Sm}(\text{HDMPE})_3\text{.bipy}$. The ligand also displays the intense C=O stretching vibration band at 1640 cm^{-1} , which was red shifted 28 cm^{-1} in complex the $\text{Sm}(\text{HDMPE})_3\text{.bipy}$, indicating that phenolic and carbonyl group of HDMPE participated in coordination with Sm(III) ion²⁷. The strong absorption band at 1570 cm^{-1} in complex the $\text{Sm}(\text{HDMPE})_3\text{.bipy}$ assigned to C=N stretching vibration, provided good evidence that the nitrogen atoms of bipy were coordinating with the Sm(III) ion^{18,28}. The peak for Ph-O vibration of the ligand HDMPE present at 1270 cm^{-1} showed a red shift of 11 cm^{-1} in the complex $\text{Sm}(\text{HDMPE})_3\text{.bipy}$, indicating that the phenolic group is involved in coordination with the Sm(III) ion. The appearance of absorption bands at 596 cm^{-1} and at 449 cm^{-1} in the complex $\text{Sm}(\text{HDMPE})_3\text{.bipy}$ was assigned to $\nu(\text{Sm-N})$ and $\nu(\text{Sm-O})$ ^{18,29} respectively, which affirms that the nitrogen atoms of the bipy 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 $^1\text{H-NMR}$ spectra of the ligand HDMPE and complex $\text{Sm}(\text{HDMPE})_3\text{.bipy}$, that the coordination of Sm(III) was through the oxygen atoms of phenolic and carbonyl group of ligand HDMPE and nitrogen atoms of the bipy.

Photoluminescent Properties

Fig.1 depicts the excitation profile of the Sm(III) complex $\text{Sm}(\text{HDMPE})_3\text{.bipy}$, recorded by monitoring $^4\text{G}_{5/2} \rightarrow ^6\text{H}_{7/2}$ 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. While the excitation spectra for the solid complex $\text{Sm}(\text{HDMPE})_3\text{.bipy}$, narrates the broad excitation band corresponding to characteristic f-f transitions of Sm(III) in the region 250-450 nm. While two excitation peaks centred at 373 nm and 401 nm appeared in the complex $\text{Sm}(\text{HDMPE})_3\text{.bipy}$, which are assigned to the electronic transition $^6\text{H}_{5/2} \rightarrow ^4\text{D}_{1/2} + ^6\text{P}_{7/2}$ and $^6\text{H}_{5/2} \rightarrow ^4\text{F}_{7/2}$ respectively. The excitation range from 345-420 is fairly appropriate to meet the demands of UV LED. From the excitation spectra of complex $\text{Sm}(\text{HDMPE})_3\text{.bipy}$, it was found that the intensity of f-f transition at 373 nm was high as compared with the other transition and has been chosen for the measurement of emission spectra. The most intense peak at 373 nm apparently show that the complex $\text{Sm}(\text{HDMPE})_3\text{.bipy}$ effectively excited by near ultra violet light.

The emission profile of $\text{Sm}(\text{HDMPE})_3\text{.bipy}$ (Fig.2) shows three identified emission peaks corresponding to characteristic emission transition $^4\text{G}_{5/2} \rightarrow ^6\text{H}_{5/2}$ (magnetic dipole transition), $^4\text{G}_{5/2} \rightarrow ^6\text{H}_{7/2}$ (mixed magnetic-electric dipole transition) and $^4\text{G}_{5/2} \rightarrow ^6\text{H}_{9/2}$ (electric dipole transition) at 564 nm, 600 nm and 646 nm respectively³⁰⁻³² on monitoring the excitation at 373 nm in solid state at room temperature as shown in Table 1. Different from the ternary $\text{Sm}(\text{HDMPE})_3\text{.bipy}$ complex, the magnetic dipole transition $^4\text{G}_{5/2} \rightarrow ^6\text{H}_{5/2}$ is the strongest emission in case of binary complex^{27,33}. The magnetic-electric dipole transition $^4\text{G}_{5/2} \rightarrow ^6\text{H}_{7/2}$ was strongest in case of ternary complex $\text{Sm}(\text{HDMPE})_3\text{.bipy}$ while the electric dipole $^4\text{G}_{5/2} \rightarrow ^6\text{H}_{9/2}$ transition was subsidiary in most of Sm(III) complexes^{34,35}.

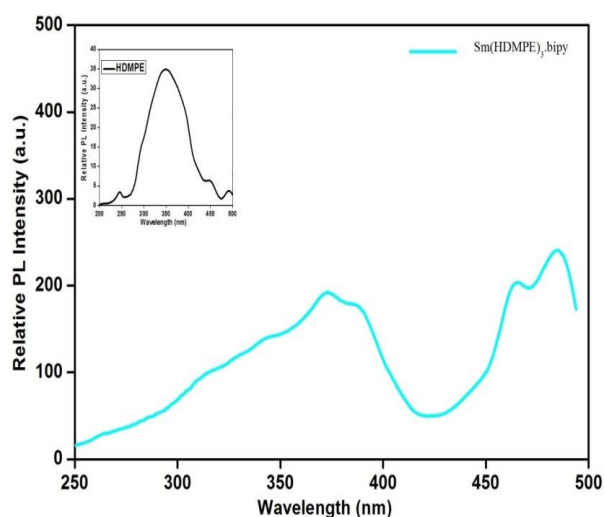


Figure 1: Photoluminescence excitation spectra of $\text{Sm}(\text{HDMPE})_3\text{.bipy}$, monitored at $\lambda_{\text{em}} = 600\text{ nm}$ in solid state at room temperature and inset shows the excitation spectrum of ligand HDMPE.

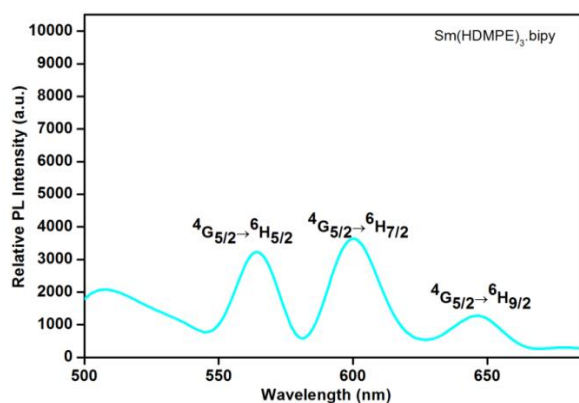


Figure 2: Photoluminescence Emission Spectra of Sm(HDMPE)₃.bipy in Solid State at Room Temperature, monitored at $\lambda_{ex} = 373$ nm.

The photoluminescence intensity of ternary complex was augmented as compared to the binary complex, because in the ternary complex OH groups was removed from the inner coordination sphere of Sm(III) ion. The replacement of water molecules by aromatic N-donors ancillary ligand 'bipy' results in augmented photoluminescence intensity and stability of complexes^{27,36}. Further, it was noticed that the ancillary ligands in the ternary complex augmented photoluminescence intensity greatly due to the coordination effect between the ligand and ancillary ligands which is beneficial for efficient intermolecular energy transfer from ligand to Sm³⁺ 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)₃.bipy possess low intensity ratio indicating that the Sm(III) ion occupies site symmetry with an inversion centre.

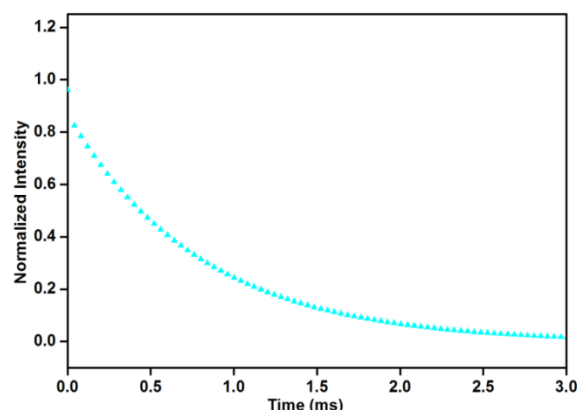


Figure 3: Luminescence Decay Curves for Sm(HDMPE)₃.bipy in Solid State at Room Temperature, Monitored at $\lambda_{ex} = 373$ nm and $\lambda_{em} = 600$ nm.

Fig.3 shows the photoluminescence decay curves for complex Sm(HDMPE)₃.bipy 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 $I = I_0 \exp(-t/\tau)$, where τ is the radiative decay time, I and I_0 are the luminescence intensities at time t and 0, respectively.

The life time value calculated for complex was found to be 0.796ms as presented in Table 1.

Table 1: Photoluminescence Data of Sm (HDMPE)₃.bipy in Solid State.

Complex	λ_{ex} (nm)	λ_{em} (nm)	Assignment	τ (ms)	x and y coordinates
Sm(HDMPE) ₃ .bipy	373	564	$^4G_{5/2} \rightarrow ^6H_{5/2}$	0.796	0.5251, 0.4725
		600	$^4G_{5/2} \rightarrow ^6H_{7/2}$		
		646	$^4G_{5/2} \rightarrow ^6H_{9/2}$		

Table 2: Minimum Inhibitory Concentration of HDMPE and Sm(HDMPE)₃.bipy

Compound	Minimum Inhibitory Concentration (μ M/mL)				
	<i>B. subtilis</i>	<i>S. aureus</i>	<i>E. coli</i>	<i>C. albicans</i>	<i>A. niger</i>
HDMPE	31.8	31.8	31.8	31.8	63.7
Sm(HDMPE) ₃ .bipy	14.0	7.00	14.0	7.00	14.0
Std.	8.71 ^a	8.71 ^a	8.71 ^a	10.09 ^b	10.09 ^b

^aCiprofloxacin^b Fluconazole

Table 3: Percentage inhibition and IC₅₀ values of DPPH radical scavenging activity of synthesized HDMPE and Sm(HDMPE)₃.bipy.

Compound	Concentration (μ g/mL)				
	25	50	75	100	IC ₅₀
HDMPE	23.12	43.02	60.08	80.83	60.42
Sm(HDMPE) ₃ .bipy	21.56	42.43	59.78	81.23	60.91
Ascorbic acid	34.02	56.22	76.12	92.01	43.78

With the help of Commission Internationale 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 0.5251, 0.4725 which lie in deep orange spectral region as shown in Fig.4, suggesting promising application of this complex in advanced display and lighting systems.

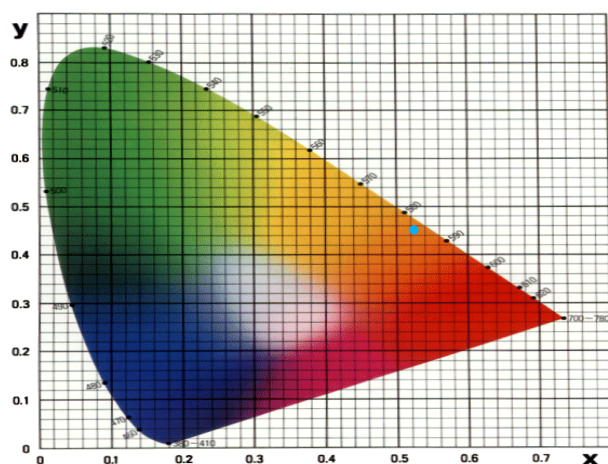


Figure 4: CIE Coordinate Diagram of $\text{Sm}(\text{HDMPE})_3\text{.bipy}$.

Antimicrobial Activity

The synthesized ligand HDMPE and $\text{Sm}(\text{HDMPE})_3\text{.bipy}$ were evaluated for their *in vitro* antimicrobial activity as tabulated in Table 2. The antimicrobial activity has been investigated by taking ciprofloxacin and fluconazole²¹ as standard drugs for antibacterial and antifungal activity respectively. The results revealed that the ligand HDMPE was having insignificant antimicrobial activity against bacterial and fungal strains, while $\text{Sm}(\text{HDMPE})_3\text{.bipy}$ showed moderate to good activity compared to the standard antibiotics and showed excellent activity against *S.aurius*.

Moreover, it was interesting to note that $\text{Sm}(\text{HDMPE})_3\text{.bipy}$ proved to be better than the standard ciprofloxacin. Further it was noticed that complex was excellently active in case of *C.albicans*, while moderately active in case of *A.niger*. The increase in antimicrobial activity of the complex may be due to the presence of Sm (III) ion coordinated with the donor atom of the ligand which leads to the π - electron delocalization over the chelate rings³⁷.

Antioxidant Activities

In DPPH free radical scavenging activity, antioxidant reacting with stable free radical 2,2-diphenyl-2-picrylhydrazyl (DPPH) produced colorless 2,2-diphenyl-2-picrylhydrazyl. The absorbance decreased, after receiving hydrogen radical.

The DPPH scavenging activity is expressed as IC_{50} . The IC_{50} value of ligand and $\text{Sm}(\text{HDMPE})_3\text{.bipy}$ were calculated from the graph plotted as inhibition percentage against concentration of HDMPE and $\text{Sm}(\text{HDMPE})_3\text{.bipy}$ as shown

in Table 3 and Fig.5. The results show that ligand HDMPE and complex showed poor activities compared to standard ascorbic acid ($\text{IC}_{50}=43.78\mu\text{g/ml}$).

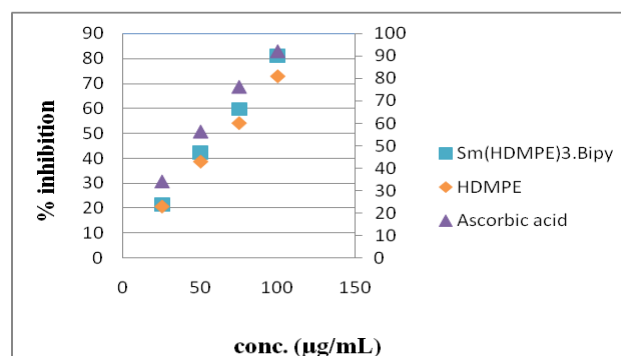


Figure 5: Percentage Inhibition of HDMPE and $\text{Sm}(\text{HDMPE})_3\text{.bipy}$ with respect to Standard Ascorbic Acid.

CONCLUSION

In this work, one newer ternary Sm(III) complex, $\text{Sm}(\text{HDMPE})_3\text{.bipy}$ have been synthesized which is further characterized through various techniques like elemental analysis, FT-IR, ¹H-NMR and photoluminescence 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 ancillary ligand (bipy) 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 ancillary ligand, leads to highly efficient and saturated orange photoluminescence. In conclusion, this evolved complex $\text{Sm}(\text{HDMPE})_3\text{.bipy}$ has showcased excellent luminescent properties, consequently can have potential solicitations for usage as light emitting materials in lighting systems. This evolved complex has showcased excellent *in vitro* antimicrobial against *S.aureus* and *C.albicans* but poor antioxidant profile as compared to standard ascorbic acid ($\text{IC}_{50}=43.78\mu\text{g/ml}$).

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