Research Article

Photoluminescence, Antimicrobial and Antioxidant Properties of New Binary Samarium (III) complex with 1-(2-hydroxy-4,6-dimethoxyphenyl)ethanone

Poonam[1], Rajesh Kumar[2], S. P. Khatkar[1], V.B.Taxak[1*]
[1]Department of Chemistry, Maharshi Dayanand University, Rohtak, India.
[2]Department of Chemistry UIET, Maharshi Dayanand University, Rohtak, India.
***Corresponding author's E-mail:** v_taxak@yahoo.com

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ABSTRACT

A new binary complex of samarium (III) ion has been synthesized with organic ligand 1-(2-hydroxy-4,6-dimethoxyphenyl)ethanone (HDMPE). The attained results of Infrared spectroscopy, ^{1}H nuclear magnetic resonance spectra and elemental analysis confirmed the structures of the ligand and complex. The powder X-ray diffractometer (XRD) suggested the crystalline nature of the complex. At the same time, photoluminescence excitation, emission spectra and decay curve were used to characterize the luminescence properties of the complex. The PL spectra demonstrated that the complex could be excited effectively in the near Ultraviolet light region at 392 nm. From the photoluminescence spectra, emission transition at 564 nm ($^4G_{5/2} \rightarrow {}^6H_{5/2}$) is more prominent than the normal orange emission transition at 605 nm ($^4G_{5/2} \rightarrow {}^6H_{7/2}$). Furthermore, the synthesized ligand and complex have been tested for *in vitro* antimicrobial activity against gram-positive bacteria: *S. aureus*, *B. subtilis* and gram-negative bacterium: *Escherichia coli* and fungal strains: *C. albicans* and *A.niger* by tube dilution method are reported. The obtained results of the antimicrobial activity suggests that complex $Sm(HDMPE)_3.2H_2O$ is a potent antimicrobial agent. In addition the antioxidant activity tests *in vitro* by using DPPH method indicated that the complex has considerable antioxidant activity.

Keywords: Sm(III)complex, Photoluminescence, Antimicrobial, Antioxidant

INTRODUCTION

The lanthanide complexes are of great interest due to their striking optical properties such as sharp emission band for high color purity, large stokes shifts, long life time and high quantum yield.[1-6] Lanthanide ions are good triplet quenchers so, and they play a key role in the development of lighting devices. The unique luminescent properties of lanthanide complexes find enormous technological application particularly in electroluminescent materials in organic light emitting diodes, OLEDs[7-9], magnetic resonance imaging (MRI) contrast agents[10], X-ray fluorescence spectrometry[11], flow injection analysis[12], analytical chemistry and biomedical devices.

Usually, in the luminescent lanthanide complexes, the chelating organic ligand act as a photosensitizer, which efficiently absorb and transfers light to the central metal ion by an antenna effect.[13,14] The β-hydroxyketone ligand is an important chelating ligand which has a strong tendency to absorbtion within a large wavelength range for its ability to sensitize the luminescence of lanthanide ions.[15-18] Among the lanthanide ions, Sm(III) ion producing intense orange light which shows bright emission with identified emission bands by the intra 4f transition of Sm(III) ion such as $^4G_{5/2}$ to ^6H_j (j=5/2,7/2,9/2,11/2). The transition $^4G_{5/2} \rightarrow {}^6H_{5/2}$ (564 nm) and $^4G_{5/2} \rightarrow {}^6H_{7/2}$ (605 nm) are magnetic dipole transition, while the transition $^4G_{5/2} \rightarrow {}^6H_{9/2}$ (649 nm) is electric dipole transition, which from the practical point of view is most suitable source for lighting and display devices. The literature study suggests that many lanthanide complexes also exhibited interesting antimicrobial activity.[19] Hence it is significant to search new lanthanide complexes as potential antimicrobial.

Looking into it the present work has been undertaken to synthesis and investigate luminescent properties and antimicrobial activity of samarium complex with organic ligand 1-(2-hydroxy-4,6-dimethoxyphenyl) ethanone (HDMPE).

MATERIALS AND METHODS

Generals

The glass wares were washed with nitric acid, after that thoroughly washed with deionised water and dried in hot air oven. All the solvents used were of analytical grade. Samarium nitrate (99.9), benzene-1,3,5-triol and dimethyl sulphate were purchased from Sigma Aldrich, INDIA and used as received.

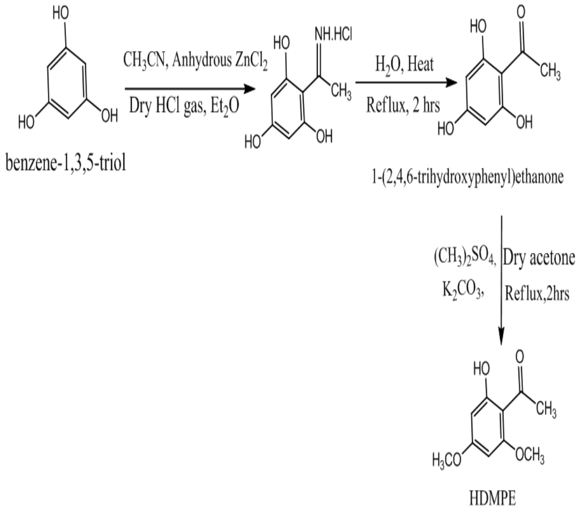
Analytical Methods

Carbon, Hydrogen and Nitrogen content of the complex were analysed by using thermo scientific flash 2000 elemental analyzer, the metal content of the complex was determined by complexometric titration with EDTA, using xylenol orange as an indicator. The IR spectra were recorded on a Perkin Elmer spectrum 400 FT-IR spectrometer with KBr pellet technique at room temperature in the region 4000-400 cm^{-1} at resolution 4 cm^{-1}. The ^{1}H-NMR spectra of the ligand and the complex were recorded on a Bruker avance II 400 MHz spectrometer operating at 400.13MHz using dimethylsulfoxide (DMSO) as solvent. Luminescent measurements were performed with a Xe flash lamp based Hitachi F-7000 fluorescence spectrophotometer with excitation and emission slit at about 2.5nm. The solid samples were placed in the integrating sphere and the Xe lamp was employed as the light source to pump the samples. Thin-layer chromaticity (TLC) was used for monitoring the progressive step of the reaction in the ligand by using silica gel plates and spot were visualized by exposure to iodine vapours.

General Procedure for the Synthesis

Synthesis of ligand

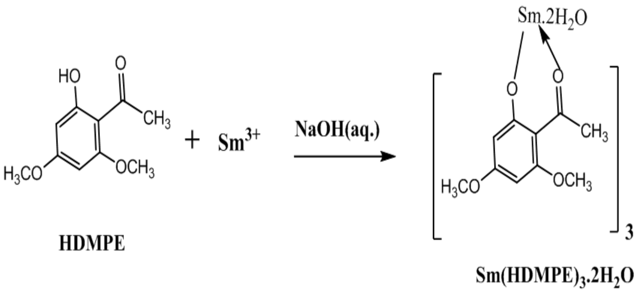
The global scheme (Scheme 1) demonstrates the synthetic route for the ligand 1-(2-hydroxy-4,6-dimethoxyphenyl)ethanone (HDMPE). The ligand was synthesised by following the conventional method as per literature.[20]



Scheme 1: The synthetic route of the ligand HDMPE.

Synthesis of complex Sm(HDMPE)$_3$.2H$_2$O

The ethanol solution of HDMPE (3mmol) with an aqueous solution of $Sm(NO_3)_3.6H_2O$ (1mmol) was constantly stirred on magnetic stirrer. The pH of mixture was adjusted to 6.5-7 with 0.05M NaOH solution. The white precipitates of the complex were formed. These precipitates were stirred for 3 hrs at 35^{o}C and then allowed to stand for 1 hr. The precipitates were filtered, washed with deionised water, ethanol, dried in air and then in vacuum desiccators to obtain complex. The complex was white solid with 94% yield.



Scheme 2: The synthetic route of $Sm(HDMPE)_3.2H_2O$

Antimicrobial Properties

HDMPE and their corresponding Sm(III) ion complex have been studied for their antibacterial activities against gram-positive bacteria: *B.subtilis, S.aureus* and gram-negative bacterium: *Escherichia coli* using ciprofloxacin as the standard drug for reference. The dilutions of synthesized complex as well as standard drug were prepared in double strength nutrient broth I.P. The standard and complex were dissolved in DMSO to give concentration of 100µg/mL. The samples were incubated at 37°C for 24 h.

The antifungal activities were carried out against *C. albicans* and *A.niger* by tube dilution method using fluconazole as the standard drug. Sabouraud dextrose broth I.P media were used in case of fungi. The incubation period for *A.niger* was 7 days at 25°C and in case of *C. albicans* 48h at 37°C. The results were recorded in terms of MIC (the lowest concentration of complex which inhibited the growth of microorganisms).

In vitro Antioxidant Activity

Free radical scavenging activity of synthesized complex against stable free radical 2, 2-diphenyl-2-picrylhydrazyl hydrate (DPPH), was determined spectrophotometrically. When DPPH reacts with antioxidant complex, which can donate hydrogen, it gets reduced. Following the reduction, its deep violet colour in methanol bleaches to yellow, shows a significant absorption decrease at 517 nm. Fifty millilitres of various concentrations (25, 50, 75, and 100) µg/ml of the complex dissolved in methanol was added to 5 ml of a 0.004% methanol solution of DPPH. After a 30 min 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 positive control. The DPPH scavenging activity was 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 and complex $Sm(HDMPE)_3.2H_2O$. The order of antioxidant activity of HDMPE and complex $Sm(HDMPE)_3.2H_2O$, according to their IC_{50} values is as follows: Standard Ascorbic acid> $Sm(HDMPE)_3.2H_2O$> HDMPE

Scavenging of DPPH free radical was calculated as:

DPPH scavenging activity (%) = [(Ac-At) / Ac] X 100

Where, Ac is the absorbance of the control reaction and At is the absorbance of the test sample.

RESULTS AND DISCUSSION

Solubility

The complex was white powder which was stable under atmospheric condition. The complex was completely soluble in DMSO, chloroform, dichloromethane and acetone where as sparingly soluble in methanol, ethanol and ethyl acetate but insoluble in benzene and hexane.

Elemental analysis, IR and ^{1}H-NMR Spectra

The elemental analytical data for ligand HDMPE ($C_{10}H_{12}O_4$) were found (calculated) % C, 60.19 (61.22); H, 6.20 (6.16); O, 32.78 (32.62). IR (KBr)cm^{-1}3448 (b),3090 (m),3002 (w),2945 (w),2850 (w),1640 (s),1456 (m),1367 (s),1324 (m),1271(s),1225 (s),1210 (s),1115 (m),1076 (m),1047 (w),895 (m), 840 (m),656 (m), 590 (s). ^{1}H-NMR (400MHz, DMSO): δ2.55 (s,3H,CH_3), 3.86 (s,6H,OCH_3), 6.08 (s,2H,Ar-H), 12.84 (s,1H,OH).

The data for $Sm(HDMPE)_3.2H_2O$ ($C_{30}H_{37}O_{14}Sm$)were found (calculated) % C, 47.06 (46.67); H, 4.69 (4.83); O, 28.86 (29.01); Sm, 19.38 (19.47). IR (KBr): cm^{-1} 3421 (b) 728 (m), 1596 (s), 1545 (s), 1385 (m), 1353 (s), 1271 (s), 1221 (s), 1156 (s), 1114 (s), 1079 (m), 837 (s), 596 (m), 469 (w), 439 (s), 424 (s). ^{1}H-NMR (400MHz, DMSO): 2.52-2.55 (bs,9H,-methyl), 3.32-3.35 (bs, 9H,-methoxy), 3.81-3.86 (bs,9H,-methoxy), 6.03-6.65 (bs,6H,Ar-H).

Abbreviations used to describe the IR spectra are: s = sharp, m = medium, w = weak and in the NMR spectra s = sharp and b = broad.

All chemical shifts are given in ppm with respect to tetramethylsilane (TMS).

The investigations from spectral data (IR and ^{1}H-NMR Spectra) and elemental analysis revealed that the Sm (III) ion are coordinated with the carbonyl and phenolic group of the ligand, as confirmed by the IR and ^{1}H-NMR Spectra.

The shift of characteristic stretching peak at 1596 cm^{-1} from 1640 cm^{-1} due to C=O group of the HDMPE in the complex, indicated that the C=O group of HDMPE participated in coordination with Sm^{3+}. Furthermore the broad peak at 3448 cm^{-1} of HDMPE due to O-H stretching vibration also changed to 3421 cm^{-1} in the complex, confirmed contribution of HDMPE in coordination of Sm^{3+} ion in complextation.

^{1}H-NMR spectra shows that a singlet of enolic OH proton at δ 13.84 ppm is exhibited in the spectra of ligand but not observed in the spectra of Sm (III) complex. Finally it can be concluded that the ligand HDMPE coordinated with Sm^{3+} ions through carbonyl group and the enolic OH group.

Antimicrobial activities

The synthesized ligand and its corresponding lanthanide complex were evaluated for their *in vitro* antibacterial activity against *B. subtilis, S. aureus, E. coli* and antifungal activity against C. albicans and A. niger by tube dilution method using ciprofloxacin (antibacterial) and fluconazole (antifungal) as reference standards and the results are tabulated in Table 1.

The results exposed that the ligand HDMPE was having insignificant antimicrobial activity against bacterial and fungal strains, where as $Sm(HDMPE)_3.2H_2O$ complex has shown to be effective bactericides and fungicides. The complex showed excellent antibacterial activity against all the gram-positive strains i.e. *S.aureus* and *B.subtilis* while in case of gram-negative strain i.e. *E.coli*, the complex was moderately active.

The antifungal activity of the synthesized $Sm(HDMPE)_3.2H_2O$ complex revealed excellent activity against *C.albicans* while the activity against *A.niger* was moderate due to the presence of m-directing electron withdrawing group, which enhanced the antimicrobial activity, also favoured by Sharma et al.[21].

Table 1: Minimum inhibitory concentration of HDMPE and its corresponding Sm(III) complex. The bold values indicate highest values of respective properties.

Complexes	Minimum Inhibitory Concentration (μM/mL)				
	MIC_{bs}	MIC_{sa}	MIC_{ec}	MIC_{ca}	MIC_{an}
HDMPE	31.8	31.8	31.8	31.8	63.77
$Sm(HDMPE)_3.2H_2O$	**8.09**	**8.09**	16.19	**8.09**	16.19
Standard	8.71[a]	8.71[a]	8.71[a]	10.09[b]	10.09[b]

[a]Ciprofloxacin [b]Fluconazole

Table 2: Percentage inhibition and IC_{50} value of DPPH radical scavenging activity of synthesized HDMPE and $Sm(HDMPE)_3.2H_2O$.

Compound	Conc.(μg/ml)				
	25	50	75	100	IC_{50}
HDMPE	23.12	43.02	60.08	80.83	60.42
$Sm(HDMPE)_3.2H_2O$	29.11	52.72	73.98	91.83	48.33
Std.	34.02	56.22	76.12	92.01	43.78

Antioxidant Activity

The capacity to transfer a single electron i.e. the antioxidant power of complex was determined by DPPH method. The IC_{50} was calculated for the synthesized complex from the graph plotted as percentage inhibition against concentration valve of the complex (shown in Table 2).

Tests were carried out in triplicate, and ascorbic acid was taken as standard compound. Standard curve is plotted for different concentration of standard ascorbic acid, ligand HDMPE and complex $Sm(HDMPE)_3.2H_2O$ shown in Fig. 1. The complex showed significant antioxidant activity while the ligand HDMPE shows poor antioxidant activity.

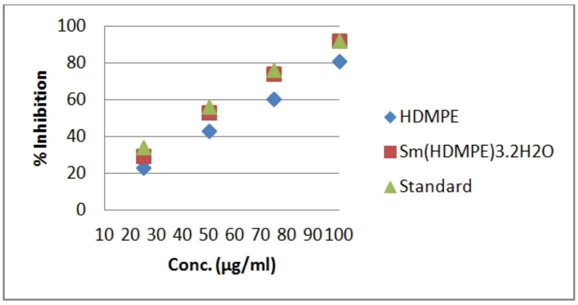


Figure 1: Percentage Inhibition value of HDMPE and $Sm(HDMPE)_3.2H_2O$ with respect to standard ascorbic acid.

XRD Measurement

Sm(III) complex having crystalline nature was confirmed by the powder X-ray diffraction pattern as depicted in Fig. 2. The XRD patterns clearly showed some characteristic crystal peaks at 2θ angle in the range 10-80° for lanthanide complex $Sm(HDMPE)3.2H_2O$, indicating the crystalline nature for complex**.** The eight identified peak appeared at 26.46°, 27.31°, 29.36°, 31.69°, 45.38°, 56.42°, 66.23°and 75.26°. By using the Scherrer's equation D = 0.941λ/β cos θ (where D is the average particle size, λ is X-ray wavelength, θ is diffraction angle and β is full width at half maxima (FWHM) of an observed peak, the particle size were calculated to be around 67.04, 86.02, 105.00, 96.23, 68.46, 94.55, 104.22, 68.25 nm respectively. The size of highest intense peak was 96.23nm at 31.69. The particle size and crystalline nature of the complex fulfil the condition for the fabrication of organic light emitting devices for luminescent materials.

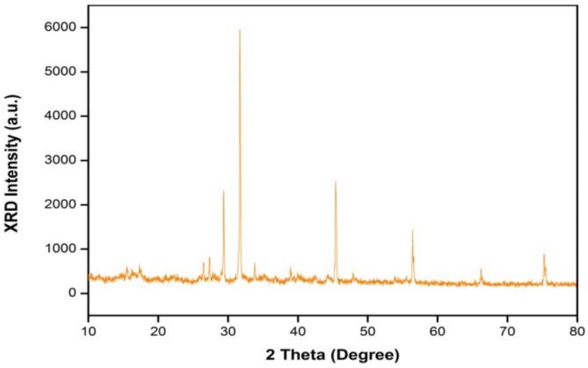


Figure 2: XRD profile of $Sm(HDMPE)3.2H_2O$

Photoluminescence Properties

Fig.3. depicts excitation spectra for HDMPE (350 nm) and the complex $Sm(HDMPE)3.2H_2O$ (392 nm) in solid state at room temperature, monitored at 488 nm and 605 nm emission intensity respectively. The excitation spectra of the HDMPE, displays a broad excitation band with strongest absorption at 350 nm, while the complex consist of a broad band in range 200-430 nm centered at 392 nm accompanied with two intense peaks in the longer wavelength at 470 nm and 488 nm. These may be attributed to electronic transition $^6H_{5/2}\rightarrow{}^4F_{7/2}$, $^6H_{5/2}\rightarrow{}^4F_{9/2}$ and $^6H_{5/2}\rightarrow{}^4I_{11/2}$ respectively. The excitation spectra clearly shows that peak maxima was red shifted by 42 nm after complexation due to chelation between the Sm(III) ion and ligand. Most of the lanthanide ions exhibit sharp excitation band in optical materials but sometimes stark splitting may cause broadening of these observed bands[22]. The excitation range from 345-420 nm (near-UV) is fairly appropriate to meet the demands of UV LED[23]. For the measurement of emission spectra of Sm(III) ions we select only one high up excitation band at 392 nm because on higher wavelength no significant results was obtained.

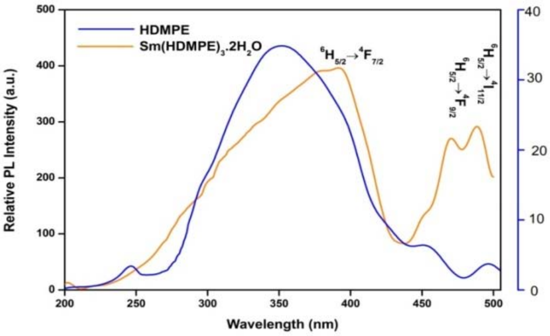


Figure 3: Photoluminescence excitation spectra of ligand HDMPE (350 nm) and complex $Sm(HDMPE)_3.2H_2O$ (392 nm) at room temperature in solid state.

The emission spectra of solid $Sm(HDMPE)_3.2H_2O$ complex consists of four transition peaks at 564 nm, 605 nm, 649 nm and 711 nm assigned to $^4G_{5/2}\rightarrow{}^6H_{5/2}$**,** $^4G_{5/2}\rightarrow{}^6H_{7/2}$, $^4G_{5/2}\rightarrow{}^6H_{9/2}$ and $^4G_{5/2}\rightarrow{}^6H_{11/2}$ respectively as shown in Fig. 4.

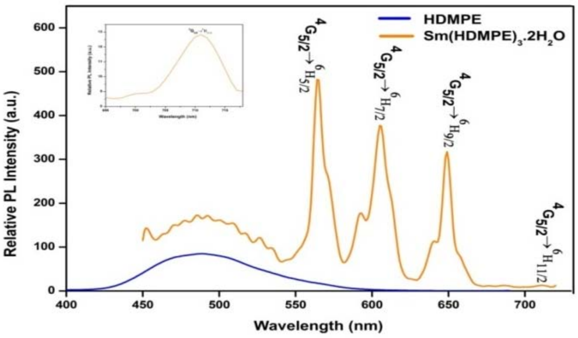


Figure 4: Photoluminescence emission spectra of HDMPE and complex $Sm(HDMPE)_3.2H_2O$.

The transitions $^4G_{5/2} \rightarrow {}^6H_{5/2}$ (564 nm) and $^4G_{5/2} \rightarrow {}^6H_{7/2}$(605 nm) were dominant due to magnetic dipole transition out of which $^4G_{5/2} \rightarrow {}^6H_{5/2}$ is more intense, while the electric-dipole $^4G_{5/2} \rightarrow {}^6H_{9/2}$ transition at 649 nm is subsidiary in case $Sm(HDMPE)_3.2H_2O$ complex. The magnetic dipole transitions obey the selection rule of J=0 and ± 1. In this complex, Sm^{3+} ion occupies a symmetry site with an inversion center. The luminescent decay dynamic behaviour of complex was affected by a number of factors like numbers of luminescent centres, energy transfer, defects and impurities present in the complex.

Fig.5. displayed the photoluminescence decay profiles for complex $Sm(HDMPE)_3.2H_2O$ monitored at λ_{em} =605 nm and λ_{ex} = 392 nm. The binary complex of Sm^{3+} ion can be fitted well by a single exponential function, which can be represented by the equation

$$I(t)=I_0 \exp(-\frac{t}{\tau})$$

Where, τ is the life time of the emission centre, I_0 is the initial emission intensities at t=0. The life time valve calculated for complex was found to be 0.64ms. The decay curve confirmed that the Sm^{3+} ion was being occupied by one symmetry site in the excited state, in this complex.

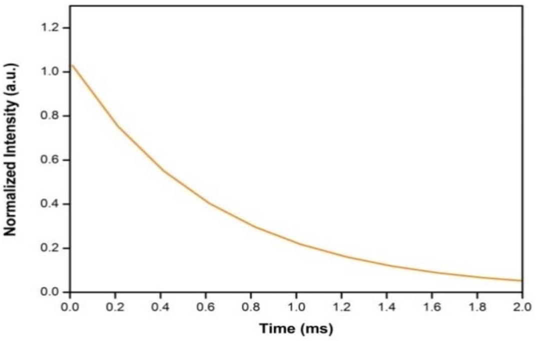


Figure 5: Luminescence Decay Curve of $Sm(HDMPE)_3.2H_2O$ of the $^4G_{5/2}$ emitting level monitoring at $^4G_{5/2} \rightarrow {}^6H_{7/2}$ transition at room temperature in solid state.

With the help of *Commission Internationale de Eclairage* (CIE) chromaticity coordinate diagram, the emission color of the luminescent complex has been analyzed. The CIE color coordinates (*x*, *y*) of the complex is located at 0.558, 0.440 in deep orange spectral region as shown in Figure 6, suggesting promising application of this complex in advanced display and lighting systems.

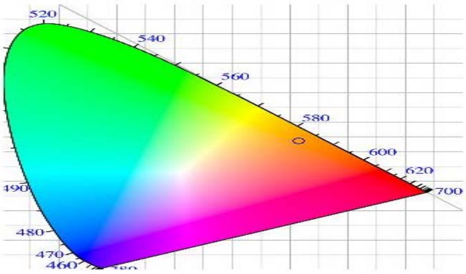


Figure 6: CIE diagram of $Sm(HDMPE)_3.2H_2O$

CONCLUSION

The prominent conformity was found with the proposed structure of complex $Sm(HDMPE)_3.2H_2O$. The complex has displayed a deep orange emission under UV source. From the results of photoluminescent properties, we suggest that it can be used as orange luminescent optical material.

The synthesized complex exhibited excellent *in vitro* antimicrobial and antioxidant profile. The high potencies against gram-positive bacteria and fungi were found hence, might be used as good antimicrobial agent.

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Corresponding Author’s Biography : Dr V.B. Taxak

Dr. V.B. Taxak is currently a Professor in Department of Chemistry, Maharshi Dayanand University, Rohtak, India. She postgraduated in Inorganic chemistry and received her doctorate degree from the same University. She has published more than 50 research papers. She has travelled to various places like South Korea, Las Vegas, Louisville, San Fransisco, Singapore for attending International Conferences and research activities. Her current research covers a wide range of areas including analytical chemistry, electrochemistry, material chemistry, nanomaterials and organic metal complexes.