



Synthesis, characterization and antimicrobial activity of a bivalent schiff base derived from salicylaldehyde and 4-methoxyaniline and its lanthanides (III) complexes

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ABSTRACT

The synthesis of new divalent Schiff base on the Lanthanide (III) complexes like La (III), Ce(III), Nd(III), Sm(III), Gd(III), Tb(III) and Dy(III) were derived by the condensation of salicylaldehyde with 4-methoxyaniline in alcoholic medium with forming 2-(E)-(4-methoxyphenylimino) methyl phenol (MPMP) The structures of the Schiff base ligands and Lanthanide (III) complexes have been proposed by the analytical and spectral data by FTIR, ¹HNMR, Molar conductance, XRD and Magnetic studies. The XRD studies indicate that orthorhombic crystal system for Ce(III), Nd(III), Tb(III), Dy(III) and Gd(III) complexes and other were tetrahedral crystal system for La(III) and Sm(III) complexes. The diameters inhibition zone were measured after 24 hrs of incubation [Gd(MPMP)₂(H₂O)₂NO₃] were found to be effective against Staphylococcus aureus in 500 ppm solution. The MPMP ligands metal complexes have been tested for their antibacterial activity against Staphylococcus aureus, Bacillus subtilis species and antifungal activity Aspergillus Niger, Fusarium Oxysporum species at the same concentration 250 ppm and 500 ppm.

Key words: MPMP ligands, antibacterial and antifungal activity, Magnetic properties.

INTRODUCTION

The wide variety of application of Schiff base derived from aromatic amine and aldehyde is a field of inorganic, analytical and organic chemistry [1-5]. The synthesis of bioactive transition metal complexes reported in previous report [6]. The oxygen, nitrogen of Schiff base metal complexes has played an important role in the development of coordination chemistry [7-10]. In the present work synthesis of biologically active lanthanide (III) complexes containing azomethine group (-CH=N-) containing carbon nitrogen double bonds known as Schiff base. They are usually prepared by condensing primary amines with active carbonyl compounds. The synthesis of asymmetrical bidentate Schiff base formed by the condensation of salicylaldehyde with 4-methoxyphenylamine (Scheme-I). The Schiff base MPMP ligand were allowed to react with La(III), Ce(III), Nd(III), Sm(III), Gd(III) Tb(III) and Dy(III) ions by using ethanol as a solvent in the molar ratio 1:2 (metal : ligand).

EXPERIMENTAL SECTION

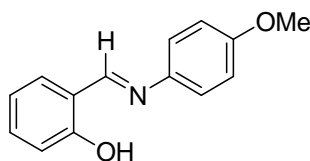
All chemicals and solvents of A.R. grade. Lanthanide (III) nitrates obtained from Rare Earth Ltd. (India) were used without further purification. Salicylaldehyde and 4-methoxyaniline was obtained from Alfa Acer Chemicals and solvents were obtained from Aldrich Chemical Company. ¹H-NMR spectra of ligand and metal complexes were

recorded on a spectra were taken in d_6 -DMSO and the chemical shifts were given in ppm with respect to tetramethylsilane (TMS) used as internal standard. The magnetic measurements were carried out at room temperature with a Gouys balance and Mercury tetrathiocyanato Cobalt (II) was used as a standard for calibration.

1.2 Synthesis of 2-(E)-(4-methoxyphenylimino) methyl phenol (MPMP) Schiff base ligand

The syntheses of Schiff base MPMP ligand were prepared by total amount of solution prepared by 50 ml ethanol contain 0.01mol (0.122g) of salicylaldehyde, and 0.01mol (0.123 g) of 4-methoxyaniline were stirred for 4 hrs at room temperature as show in (Scheme-I). Fine shining

Gray precipitate of the Schiff base ligand (2-(E)-(4-methoxyphenylimino) methyl phenol) formed was filtered off, washed with ethanol and stored in vacuum desiccators over anhydrous calcium chloride. The purity of MPMP ligand was checked by using thin layer chromatography (TLC) by using silica gel plates. The product was purified and recrystallized with hot ethanol. Yield obtained were 80%.



Scheme-I. Structure of Schiff base ligand 2-(E)-(4-methoxyphenylimino) methyl phenol (MPMP)

1.3 Synthesis of Lanthanide (III) complexes

Lanthanide (III) complexes were synthesized by taking the mixture of (0.01mol) ligands 2-(E)-(4-methoxyphenylimino) methyl phenol dissolve in 50ml ethanol solution simultaneously and other lanthanide (III) nitrate (0.01mol) 25 ml of ethanol was added under constant stirring for 3 hrs at room temperature. The precipitated complexes were filtered off, washed with ethanol, diethyl ether and dried under vacuum over anhydrous calcium chloride ($CaCl_2$). Decomposition points of lanthanide (III) complexes were above $250^\circ C$.

RESULTS AND DISCUSSION

2.1 1H -NMR spectra of ligands:

The 1H -NMR spectra of MPMP ligands in $CDCl_3$ at room temperature shows the signals at 10.85δ (s, 1H, phenolic OH), $7.5-9.3\delta$ (m, 7H, Ar-H), 7.4δ (s, 1H, N=C-H), 3.94δ (s, 3H, OCH_3).

2.2 Molar conductance:

Molar conductivity data for all the complexes in DMF solution at room temperature were in the range of $11-129\text{ cm}^2\text{ mol}^{-1}$ reported for 1:1 electrolytes [11]. Conductance measurements of 10^{-3} M solutions of the complexes in DMSO were carried out on a conductivity meter by using conductivity cell Type Cd-10, cell $K=1.0\pm 10\%$. The conductivity values were suggests in Table 2. All the lanthanides (III) complexes showed low conductivity i.e. non electrolytic behavior.

2.3 Magnetic Measurement:

Study of unpaired electron of lanthanide (III) complexes by using magnetic susceptibility measurements of the powdered complexes were carried out by employing Guoys balance method at $28^\circ C$ temperature. Mercury tetrathiocyanato Cobalt (II) was used as a standard for calibration. The effective magnetic moment μ_{eff} of the metal ions were calculated by using the following formula

$$\mu_{\text{eff}} = 2.83 (\chi_m T)^{1/2} \text{ BM}$$

χ_m = molar susceptibility, T = absolute temperature and BM = Bohrs Megatons The μ_{eff} of the metal ions were calculated in the present lanthanide (III) complexes were summarized in Table-2. The data shows that lanthanide (III) complexes were diamagnetic in nature, as expected from its closed shell electron configuration and absence of unpaired electrons [12]. All other triple positive charged lanthanide complexes were paramagnetic due to the presence of 4f-electrons, which are effectively shielded by $5s^2$ and $5p^6$ electrons [13].

Table- 1. Physical and analytical data of MPMP ligand Lanthanide (III) complexes

Ligand/ Complexes	M.wt.	M.P. Temp(°C)	Yield (%)	Elemental analysis found (calculated)				
				C	H	N	O	M(Ln)
MPMP	227.25	85	80	5.77 (5.71)	6.16 (6.11)	14.28 (14.8)	27.25 (27.53)	-
[La(MPMP) ₂ (H ₂ O) ₂ NO ₃]	691.46	≥250	54	48.64 (48.21)	4.37 (4.45)	6.08 (5.65)	20.82 (20.12)	20.09 (19.95)
[Ce(MPMP) ₂ (H ₂ O) ₂ NO ₃]	693.52	≥250	58	48.84 (47.97)	4.30 (4.42)	6.98 (5.92)	20.49 (20.35)	20.39 (20.04)
[Nd(MPMP) ₂ (H ₂ O) ₂ NO ₃]	696.79	≥250	56	48.26 (48.21)	4.34 (4.12)	6.03 (5.65)	20.67 (20.23)	20.70 (20.36)
[Sm(MPMP) ₂ (H ₂ O) ₂ NO ₃]	702.91	≥250	57	47.84 (47.51)	4.30 (4.11)	5.98 (5.39)	20.49 (20.25)	21.39 (21.23)
[Gd(MPMP) ₂ (H ₂ O) ₂ NO ₃]	709.82	250	60	47.38 (47.98)	4.26 (4.36)	5.92 (6.31)	20.29 (19.89)	22.15 (22.55)
[Tb(MPMP) ₂ (H ₂ O) ₂ NO ₃]	711.48	250	55	47.27 (47.12)	4.25 (4.26)	5.91 (5.89)	20.24 (20.56)	22.34 (22.68)
[Dy(MPMP) ₂ (H ₂ O) ₂ NO ₃]	715.05	250	57	47.03 (47.40)	4.23 (4.21)	5.88 (5.90)	20.14 (20.63)	22.73 (21.89)

2.4 Electronic spectra:

The UV–Visible spectrum of the solid ligand MPMP in Table-2 showed first bands at 215-279nm assigned to π - π^* transition within the aromatic ring (-C=C-) and second band at 346-355nm due to n - π^* transition within the azomethine (-C= N) group. The broad band is observed in the visible region in the spectra of the lanthanides (III) complexes of at 28.901 – 28.169 cm^{-1} and this band may be assigned to the strong ligand to metal charge transfer transition [14]. The absorption bands due to the f-f transitions of the lanthanide ions could not be identified in the visible region of all these complexes. This may be probably attributed to the fact that the f-f transitions are very weak, and the bands corresponding to these transitions are observed by the strong charge and transfer transition bands of the ligand [15].

Table-2. Magnetic moment and Molar conductance data of MPMP ligand Lanthanides (III) nitrate complexes at room temperature

Ligand / Metal Complexes	Magnetic moment (BM)	Molar conductance Mho($\text{cm}^{-2} \text{mol}^{-1}$)	λ_{max}	Band Assignment
	μ_{eff}	DMSO	nm	-
MPMP	-	-	279 346	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$
[La(MPMP) ₂ (H ₂ O) ₂ NO ₃]	Diamagnetic	44.01	230, 255 350	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$
[Ce(MPMP) ₂ (H ₂ O) ₂ NO ₃]	2.32	47.01	234, 275 352	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$
[Nd(MPMP) ₂ (H ₂ O) ₂ NO ₃]	3.83	54.01	264 351	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$
[Sm(MPMP) ₂ (H ₂ O) ₂ NO ₃]	1.45	47.00	215, 261 350	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$
[Gd(MPMP) ₂ (H ₂ O) ₂ NO ₃]	7.62	77.89	272, 292 351	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$
[Tb(MPMP) ₂ (H ₂ O) ₂ NO ₃]	9.56	40.01	275 345	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$
[Dy(MPMP) ₂ (H ₂ O) ₂ NO ₃]	10.35	66.20	215, 254 355	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$

2.5 FTIR Studies:

A comparative study of infrared spectra of free MPMP ligand and their respective lanthanide (III) complexes were recorded in the 4000-300 cm^{-1} range using KBr pallets. IR spectra of the Schiff base ligands were analyzed by comparison of the spectra of substituted benzene and the corresponding assignments were listed with comparing the IR spectra of previously reported data [16]. The partial IR data for ligand and their corresponding complexes are given in Table-3. These ligands behave as neutral bidentate and lanthanide (III) metals are coordinated through N and O of two azomethine groups and two aqua ion. The strong band observed at 1602 and 1636 cm^{-1} region in the free ligands have been assigned to ν (-C=N-) vibrations of azomethine groups. After complexation these bands were shifted to higher wave number by 14 cm^{-1} indicates a stronger double bonds character of the imines bonds and coordination other azomethine nitrogen atoms to the lanthanide (III) ion [17, 18]. The phenolic -OH indicates in the

presence of Ph-OH (ν_{OH}) group 1243 and 1255 cm^{-1} respectively present in metal (III) complexes. All the Schiff base ligands complexes show an additional peak in the region 2838-2970 cm^{-1} due to -C-H aromatic ring stretching. The lanthanide (III) metal nitrate complexes the occurrence of two additional strong absorption bands around at 1486 cm^{-1} , which were absent in the free ligand. The presence of non-ligand band in 804-884 cm^{-1} region, assignable to the rocking mode of water [19]. The IR spectra show a strong band in the 3593-3307 cm^{-1} region, suggesting that the presence of coordinated water in these lanthanide (III) complexes [20]. The FTIR spectra of metal complexes showed new bands in 550-574 cm^{-1} range assigned to (M-N) and 412-464 cm^{-1} range assigned to (M-O) mode [21].

Table -3. Major FTIR spectral data for the MPMP ligand and MPMP ligand Lanthanides (III) nitrate complexes (cm^{-1})

Ligand / Metal Complexes	H ₂ O	ν (C=N)	ν (Ar-O)	ν (M-O)	ν (M-N)	ν (C=C)	ν (C-O)	ν_1 NO ₃	ν_2 NO ₃
MPMP	-	1602	1244	-	-	1490	1150	-	-
[La(MPMP) ₂ (H ₂ O) ₂ NO ₃]	3426	1617	1243	435	557	1406	1149	1406	1243
[Ce(MPMP) ₂ (H ₂ O) ₂ NO ₃]	3307	1619	1248	423	554	1478	1146	1419	1298
[Nd(MPMP) ₂ (H ₂ O) ₂ NO ₃]	3596	1635	1254	453	572	1481	1174	1446	1285
[Sm(MPMP) ₂ (H ₂ O) ₂ NO ₃]	3593	1635	1254	464	573	1481	1147	1486	1286
[Gd(MPMP) ₂ (H ₂ O) ₂ NO ₃]	3593	1635	1254	412	550	1482	1147	1459	1288
[Tb(MPMP) ₂ (H ₂ O) ₂ NO ₃]	3593	1635	1254	436	574	1482	1174	1460	1289
[Dy(MPMP) ₂ (H ₂ O) ₂ NO ₃]	3594	1636	1255	438	574	1482	1175	1460	1291

2.6 X-ray diffraction study:

A good quality x-ray diffractogram of MPMP Ligands indicates semi crystalline as well as amorphous nature of lanthanide (III) complexes. All the reflection, has been indexed for h, k, l values using methods reported in the literature [22]. The comparative study on powder x-ray diffraction of lanthanide (III) complexes of Ce(III), La(III), Nd(III), Sm(III), Tb(III), Gd(III) and Dy(III) complexes were MPMP Ligand were scanned in the range of 5-65° and wavelength of 1.540598Å. The 2θ value each peak, relative intensity and diffractogram associated data depict the inter-planar spacing (d-values) and lattice constants a, b and c for each unit cell parameter showed in Table-4. In XRD pattern showed percentage of intensity vs, 2θ values in systematic arrangement of metal complexes. To calculate density values of the complexes were used specific gravity method [23]. The systematic arrangement of XRD studies on metal complexes are Lattice constant, Unit cell Volume, Crystal system, d-value, 2θ value [24].

The 'd' values of reflections were obtained using Bragg's equation.

$$n\lambda = 2d \sin \theta$$

To calculate the unit cell volume of Ce(III), Tb (III), Dy (III), Nd (III), and Gd (III), complexes for orthorhombic crystal system by the following equation was used.

$$V = abc$$

To calculate the unit cell volume of La (III) and Sm (III) complex for tetragonal crystal system by the following equation was used.

$$V = a^2c$$

2.7 Antibacterial Study:

The antibacterial activity of the newly prepared by MPMP ligands and some lanthanides (III) complexes were carried out successfully. Results of these studies are included in Table-5. The synthesized Schiff base ligand MPMP and their corresponding Ln (III) complexes were screened for antimicrobial activity against the gram positive (antibacterial activity) and gram negative (antifungal activity) pathogens namely *Bacillus Subtilis*, *Staphylococcus aureus*, *Aspergillus Nige*, *Fusarium Oxysporum* and their corresponding Ln (III) complexes by the reported method [25]. The stock solution (1 mg mL⁻¹) of the test chemical was prepared in DMF solution. The stock solution was further diluted with sterilised distilled water to different dilutions in 250, 500 ppm. The test chemicals of different dilutions were added to sterile blank antimicrobial susceptibility discs. The bacteria were sub- cultured in agar medium and the discs were kept onto the same. The Petri-dishes were incubated for 24 h at 28 °C. The Standard antibacterial drug (ciprofloxacin) was also screened under similar conditions for comparison. Activity was determined by measuring the zones of growth inhibition surrounding the discs. Growth inhibition was compared with the standard drug. From the results obtained, the antibacterial activity of Ln (III) Schiff base complexes is

found to be higher than that of a free Schiff base ligand, against the same microorganism under identical experimental conditions. Remarkable enhancement in activity was found for Gd (III) Schiff base complex against the species *Staphylococcus aureus* 500 ppm showed higher activity than the other Ln (III) complexes.

Table-4. Lattice constant, Unit cell Volume, Crystal system, Inter-planar spacing of Ln(III) complexes

Unit cell of compounds	Lattice Constant			Unit cell Volume V (Å ³)	Edge length	Inter axial angle	Crystal system	2θ	d-value (Å)
	a (Å)	b (Å)	c (Å)						
La(III)	13.52	9.23	7.23	495.49	$a = b \neq c$	$\alpha = \beta = \gamma = 90$	tetragonal	33.81	2.64
Ce(III)	9.23	8.25	7.25	553.00	$a \neq b \neq c$	$\alpha = \beta = \gamma = 90$	orthorhombic	30.97	2.88
Nd(III)	10.25	9.25	8.24	782.29	$a \neq b \neq c$	$\alpha = \beta = \gamma = 90$	orthorhombic	58.62	1.57
Sm(III)	10.25	10.25	8.32	875.66	$a = b \neq c$	$\alpha = \beta = \gamma = 90$	tetragonal	64.92	1.43
Gd(III)	9.84	8.65	7.25	617.90	$a \neq b \neq c$	$\alpha = \beta = \gamma = 90$	orthorhombic	86.41	1.12
Tb(III)	9.32	8.42	7.86	617.91	$a \neq b \neq c$	$\alpha = \beta = \gamma = 90$	orthorhombic	70.18	1.33
Dy(III)	9.84	8.24	7.89	640.85	$a \neq b \neq c$	$\alpha = \beta = \gamma = 90$	orthorhombic	61.94	1.49

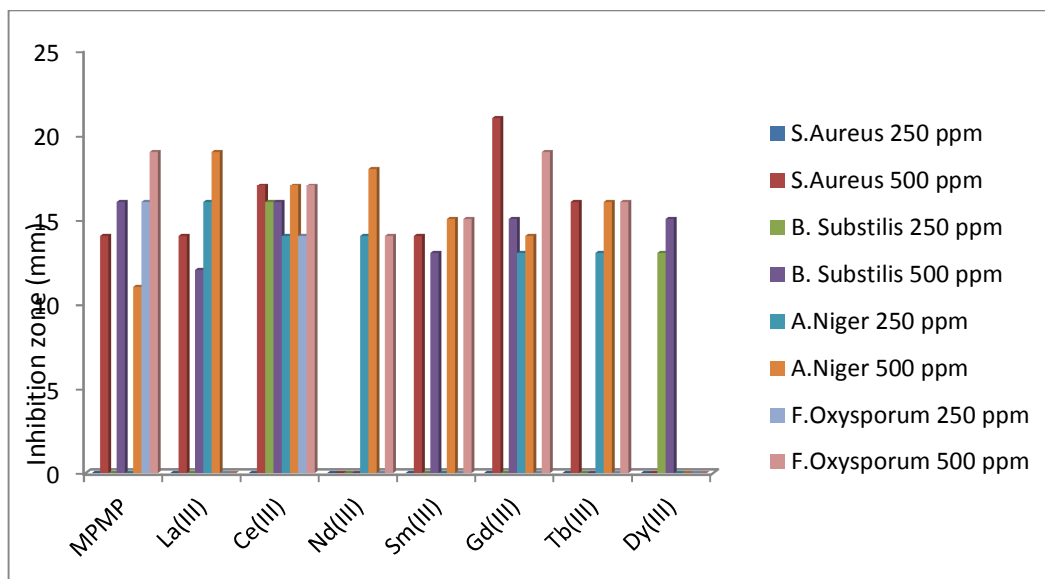
2.8 Antifungal Study:

The synthesized Schiff base ligand MPMP and their corresponding Ln (III) complexes were screened for in vitro antifungal activity according to the disc diffusion method. The assay was determined against the fungal species *Aspergillus Nige* and *Fusarium Oxysporum* by the reported method [26, 27]. The stock solution (1 mg mL⁻¹) of the test chemical was prepared in DMF solution. The stock solution was further diluted with sterilized distilled water to different dilutions in 250, 500 ppm. The test chemicals of different dilutions were added to sterile blank antimicrobial susceptibility discs. The fungi were sub cultured in potato dextrose agar (PDA) and the discs were kept onto the same. The Petri dishes were incubated for 24 h at 28°C. The standard antimicrobial drug (ciprofloxacin) was also screened under similar conditions for comparison. The results were recorded by measuring the zones of growth inhibition surrounding the discs. For the species *Fusarium Oxysporum* 500 ppm Gd (III) and *Aspergillus Niger* 500 ppm La (III) Schiff base complex showed higher activity than the other Ln (III) complexes.

Table 5. Antibacterial and Antifungal activity of MPMP ligands and MPMP ligand lanthanide (III) nitrate complexes [Diameter of inhibition Zone in (mm)]

Ligand / Metal Complexes	Antibacterial activity				Antifungal activity			
	<i>Staphylococcus aureus</i>		<i>Bacillus Subtilis</i>		<i>Aspergillus Niger</i>		<i>Fusarium Oxysporum</i>	
	250 ppm	500 ppm	250 ppm	500 ppm	250 ppm	500 ppm	250 ppm	500 ppm
MPMB	00	14	00	16	00	11	16	19
[La(MPMP) ₂ (H ₂ O) ₂ NO ₃]	00	14	00	12	16	19	00	00
[Ce(MPMP) ₂ (H ₂ O) ₂ NO ₃]	00	17	16	16	14	17	14	17
[Nd(MPMP) ₂ (H ₂ O) ₂ NO ₃]	00	00	00	00	14	18	00	14
[Sm(MPMP) ₂ (H ₂ O) ₂ NO ₃]	00	14	00	13	00	15	00	15
[Gd(MPMP) ₂ (H ₂ O) ₂ NO ₃]	00	21	00	15	13	14	00	19
[Tb(MPMP) ₂ (H ₂ O) ₂ NO ₃]	00	16	00	00	13	16	00	16
[Dy(MPMP) ₂ (H ₂ O) ₂ NO ₃]	00	00	13	15	00	00	00	00
ciprofloxacin	10	12	16	10	16	11	22	09

Graph-1. Comparative Antibacterial and Antifungal activity of MPMP ligands its metal complexes



CONCLUSION

To prepare new 2-(E)-(4-methoxyphenylimino) methyl phenol (MPMP) Schiff bases having N, O donor ligands using the derivatives of 4-Methoxyaniline with salicylaldehyde using rare earth metals like La(III), Ce(III), Nd(III), Sm(III), Gd(III), Dy(III) and Tb(III) complexes were distorted octahedral geometry. All the lanthanide (III) complexes showed low conductivity i.e. non electrolytic behavior. To study the applicability towards the antimicrobial activity of the rare earth metal Schiff base complexes derived from of 4-Methoxyaniline with salicylaldehyde. The complexes were biologically active like antibacterial *Staphylococcus aureus* in 500ppm are highly active of nutrient medium.

Acknowledgement

The authors are grateful to the Dr. Babasaheb Ambedkar Marathwada University, Aurangabad for the award of University Scholar Fellowship and Department of chemistry for providing the laboratory facilities.

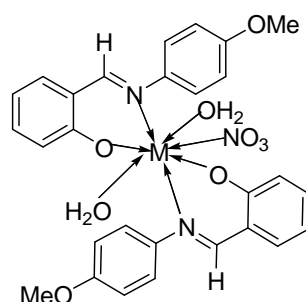


Figure-III. Proposed structure of MPMP ligand metal complexes

M= La (III), Ce(III), Nd(III), Sm(III), Gd(III), Tb(III) and Dy(III).

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