Research Article CODEN: IJPRNK ISSN: 2277-8713
M Dhanalakshmi, IJPRBS, 2013; Volume 2(6): 109-119 IJPRBS

INTERNATIONAL JOURNAL OF
PHARMACEUTICAL RESEARCH AND BIO-SCIENCE

SYNTHESIS,CHARACTERIZATION AND ANTIMICROBIAL ACTIVITY OF SOME MIXED LIGAND METAL COMPLEXES

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Accepted Date: 26/10/2013; Published Date: 27/12/2013

Abstract: Schiff base transition metal complexes plays an important role in medical field. So, we have prepared the mixed ligand transition metal complexes of the metal ions Fe(II), Cu(II), Ni(II), Co(II), and Zn(II) using the Schiff base derived from salicylaldehyde and p-methoxy aniline as one of the ligand and 1,10-phenanthroline as another ligand. The Schiff base was first prepared by condensing salicylaldehyde and p-methoxy aniline. The structure of the ligand was confirmed by IR, UV-Visible and H^1NMR spectra. The shift in the spectral bands of the ligands upon complexation was also confirmed by taking the spectra of complexes. Using this we have proposed the possible common structure for the complex. The anti microbial activity of mixed ligand Schiff base transition metal complexes against the bacterias Pseudomonas fluorescence, Klebsilla pneumonia, Bacillus, Salmonella and Escheriacoli and the fungi Aspergillusniger and Aspergillusflavus was accessed by finding out their zone of inhibition using disc diffusion method. We have observed antimicrobial activity for all complexes So, these transition metal complexes can be used in medicinal field.

Keywords: Schiff base, 1,10-phenanthroline

PAPER-QR CODE

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Access Online On:

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How to Cite This Article:

M Dhanalakshmi, IJPRBS, 2013; Volume 2(6): 109-119

INTRODUCTION

Antibiotics are one of our most important weapons in fighting bacterial infections and have greatly benefitted the health related quality of humanlife. The increasing incidence of infection caused by the rapid development of bacterial resistance to most of the known antibiotics is a serious health problem. As multidrug resistant bacterial starins proliferate, the necessity for effective therapy has stimulated research into the design and synthesis of novel antimicrobial molecules. Isolation, identification and application of organo-sulphur and nitrogen containing compounds resulted in their useful scientific, technical and industrial growth, which has led to the development of heterocyclic chemistry during the last two decades.Coordination compounds exhibit different characteristics properties which depends on the metal ion to which they are bound, the nature of the metal as well as the type of ligand etc. These metal complexes have found extensive application in various fields of human interest. Synthesis, characterization and antimicrobial activity of cobalt(II) and nickel(II) complexes of acetyl derivatives of urea and thiourea metal complexes are monodentate and bind to the central metal atom through the oxygen and sulphur donor atoms. Lliterature studies reveals that Schiff base complex have a large number of roles as drug,catalyst,etc.In the present work,complexes of Cu(II),Co(II),Ni(II),Zn(II) and Fe(II) with Schiff base derived from salicylaldehyde and p-methoxy aniline as one of the ligand and 1,10-phenanthroline as another ligand. have been synthesized, and characterized by Elemental analysis,UV,IR and H^1 NMR spectral analysis.The antimicrobial activity of prepared transition metal complexes were determined.

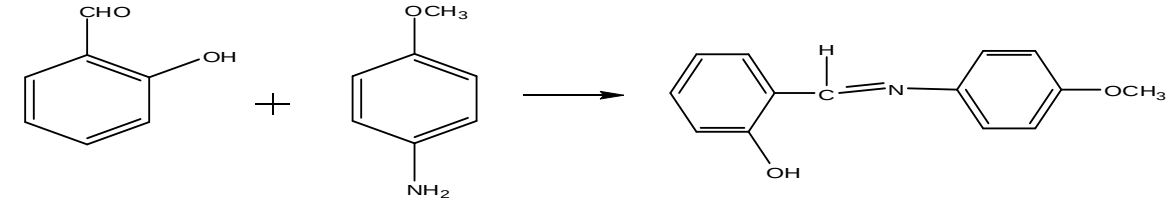
MATERIALS AND METHODS:

Metal salts like Copper sulphate, Cobalt chloride, Zinc chloride, Nickel sulphate and Ferrous sulphate were purchased fromE.mercklimited .Salicylaldehyde , p- methoxy aniline and 1,10-phenanthroline were purchased fromLobachemie limited. The antibacterial activity of synthesized Schiff base metal complexes was determined by **Disc Diffusion Method.**

Preparations of Schiff base ligand(L_1):

20ml of ethanol solutions of salicylaldehyde(0.0244g;0.01M) and the same volume of ethanolicsolution of p-methoxyaniline(0.0246g; 0.01M) were mixed. The mixture was reflected for 4-5hours. This solution was evaporated and dried in vaccum to remove the solvent. The product after filtration was washed several times with ethanol and recrystallized from hot ethanol and dried. The reaction can be represented as follows.

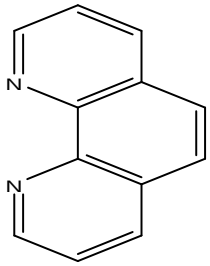
Preparations of Schiff base ligand(L_1)



Salicylaldehyde p-Methoxyaniline Schiff base Ligand (L_1)

Figure 1: The product Schiff base formed in this way was used as ligand 1 (L_1) for the preparation of transition metal complexes. 1,10-phenanthroline was used as ligand 2(L_2)

Structure of Schiff base ligand(L_2)



Schiff base Ligand (L_2)
[1,10-phenanthroline]

Figure 2: Structure of Schiff base ligend (L_2) from product of Ligend(L_1)

Preparation of Schiff base transition metal complexes

A Mixture of the solution of ligand (L_1) (0.01M ;0.0454 gram) in 20ml of ethanol, the same volume of aqueous solution of M(II) salt (M=Cu,Ni,Co,Zn,Fe 0.5M, Cu(II)-0.0498gram Ni(II)-0.0316gram,Zn(II)- 0.027gram, Fe(II)-0.0556gram and Co(II)-0.0474gram) and the same volume of aqueous solution of 1,10-phenanthroline (0.01M ;0.0396gram) were mixed. The mixture was stirred in a magnetic stirrer for 4-5hours.Diffferent colored precipitates were obtained in each case. The precipitates were filtered and washed several times with ethanol to remove excess metal ions. The precipitates were dried and stored in a dessicator over anhydrous $CaCl_2$ under vaccum.

COLOUR AND MELTING POINT:

The colour of the metal complexes was noted. The melting point of the ligand as well as the complexes were determined by placing the finely powdered sample in a glass capillary and heating by using **GallenKamp melting point apparatus**.

ELEMENTAL ANALYSIS(C, H, N analysis):

Elemental analysis of the Schiff base transition metal complexes were determined to get an idea about their composition. Elemental analysis was carried out by the use of **Elemental analyzer flash EA 1112.**

SPECTRAL STUDIES:

The transition metal complexes were characterized by following spectral studies.

INFRA RED SPECTRA:

FT-IR spectra of Schiff base and their metal complexes were obtained on a ***Shimadzu IR Affinity1 spectrophotometer.*** Generally a comparison of IR spectum of the ligand with that of its complex will be of much help to find out the atom through which the ligand is attached to the metal ion.

ELECTRONIC ABSORPTION SPECTRA:

The electronic absorption spectra of Schiff base and metal complexesin ethanol were recorded on the **UV 1601 Shimadzu spectrophotometer(Thermo vision pro software) .**

PROTON MAGNETIC RESONANCE SPECTRA:

The 300 MHz ^{1}H-NMR spectra of the Schiff base and Zinc complex were recorded on ***Bruker*(300MHz)** NMR instrument. The ^{1}H-NMR study is of help in the structural elucidation of the ligand as well as in locating precisely the donor site of the ligand. Usually ^{1}H-NMR spectral studies are carried out in this work to confirm the mode of coordination suggested from IR spectra.

ANTIBACTERIAL ACTIVITY OF SCHIFF BASE TRANSITION METAL COMPLEXES:

Antibacterial activity of the metal complexes were obtained by KIRBY-BAUER DISK-DIFFUSION METHOD.

RESULTS:

The colour, melting point and conductance of the Schiff base transition metal complexes are given in the **Table 1**

The percentage of C,H and N obtained from the elemental analyzer are compared with the percentages calculated using the assumed structure of 1:1:1(M:L_1:L_2) complexes. The proximity

of the values suggests that the ligand forms 1:1:1 complex with the metal ions. The results are tabulated in **Table 2**.

From the FT-IR spectral studies, observed that the schiff bases with metal ions The results of IR spectra of the metal complexes are collectively given in **Table 3**.

Comparison of the UV-Visible spectra of the ligand with the complexes reveal that, the band at 400nm in the ligand corresponding to the INCT bond, is shift to higher region of wave length in all the complexes. This confirms that the chromophoric group of the ligand is involved in bonding with the metal ions in all the complexes..The results of UV-Visible spectra of the metal complexes are collectively given in **Table 4**.

The results of H^1-NMR spectra of the metal complexes are collectively given in **Table 5.** Comparing the spectral data of metal complex and ligand,we can say that a shift the signal of azomethine proton is due to the bonding with the metal ion.

Comparsion of antibacterial activity of the different metal complexes against various bacteria can be obtained from the graph given in Fig 1-2.From this analysis, we find that some of the metal complexes are possessing antibacterial activity. The results of antibacterial activity and antifungal activity of the metal complexes are collectively given in **Table 6 and Table 7.**.

From the above spectral studies, we can propose the following structure for the transition metal complexes.

Table 1

Physical parameters of the complexes

S.No	Compound	Color	%yield	Meltingpoint(^oC)	Conductance (λm)$Ohm^{-1}cm^2mol^{-1}$
1	Ligand(L_1)	Silver	95	194	0.958
2	Ligand(L_2)	White	98	114-117	0.518
3	X_1	Green	92	82	0.798
4	X_2	Lightyellow	89	175	0.904
5	X_3	LightBrown	93	88	0.254
6	X_4	DarkYellow	92	190	2.38
7	X_5	Red	91	185	1.334

X_1 → Copper Complex

X_2 ⟶ Nickel Complex

X_3 ⟶ Cobalt Complex

X_4 ⟶ Zinc Complex

X_5 ⟶ Ferrous Complex

Table 2

Analytical data of C,H,N composition

S NO	COMPLEXES	ELEMENTS	C %		H %		N %	
1	X_1	Calculated found	83.14	83.16	11.54	11.57	0.96	0.98
2	X_2	Calculated found	81.88	81.96	10.92	10.98	0.88	0.92
3	X_3	Calculated found	83.65	83.72	11.12	11.18	0.92	0.96
4	X_4	Calculated found	84.44	84.56	11.02	11.14	0.78	0.82
5	X_5	Calculated found	82.18	82.34	11.72	11.76	0.94	0.98

Table 3

Analytical data for FT-IR spectra for Metal complexes

Compound	-C-O	The shift of absorptions – OHstretching vibrations	-C-H	The shift of absorptions- C=Nstretching vibrations	-C-C	-C-H	$-OCH_3$	M-N	M-O
Ligand L_1	1250	1290	3075	1635	1490	1025	2800	-	-
Ligand L_2	-	-	-	1675	-	-	-	-	-
X_1	1285	1250	3050	1610	1435	1025	2870	456	555
X_2	1250	1350	3010	1650	1440	1050	2850	480	550
X_3	1250	1285	3045	1610	1490	1030	2860	450	550
X_4	1200	1300	3050	1650	1440	1020	2850	470	550
X_5	1280	1230	3065	1630	1430	1110	2852	498	550

Table 4

UV-Visible Analytical data for the M(II) complexes

COMPOUND	SOLVENT	ABSORBTION (nm)	TRANSITIONS	STRUCTURE
Ligand L_1	ETHANOL	440	-	-
Ligand L_2	ETHANOL	272	-	-
X_1	ETHANOL	548	$^5T_{2g} \rightarrow {}^5E_g$	Square planar
X_2	ETHANOL	576	$^5T_{2g} \rightarrow {}^5E_g$	Square planar
X_3	ETHANOL	445	$^4A_{2g} \rightarrow {}^4T_{2g}$	Octahedral
X_4	ETHANOL	576	$^5T_{2g} \rightarrow {}^5E_g$	Square planar
X_5	ETHANOL	572	$^5T_{2g} \rightarrow {}^5E_g$	Square planar

Table 5

H^1NMR Spectrum of ligands and some mixed ligand M(II) Complexes

Complexes	Signal(ppm)	Multipicity observed signal	Inference
Ligand L_1	6.6-7.3	Multiplet	Aromatic proton
	3.7	Singlet	Methoxy proton
	13	Singlet	Phenolic proton
	8.6	Singlet	Azomethine proton
Ligand L_2	7.895	Multiplet	Aromatic proton
	9.1	Singlet	Azomethine proton
Fe(II)	6.943-7.407	Multiplet	Aromatic proton
	3.870	Singlet	Methoxy proton
	13.444	Singlet	Phenolic proton

	8.637	Singlet	Azomethine proton
Cu(II)	6.933-7.354	Multiplet	Aromatic proton
	3.830	Singlet	Methoxy proton
	13.414	Singlet	Phenolic proton
	8.599	Singlet	Azomethine proton

Table 6

Antibacterial Activity for different metal complexes

Bacteria or Micro Organisms	Zone of inhibition(mm)				
	X_1	X_2	X_3	X_4	X_5
Klebsiella pneumonia	20	8	12	19	14
Pseudomonas fluorescence	-	-	-	-	11
Bacillus	15	-	6	6	4
Salmonella	14	3	5	4	-
Escherichia coli	10	-	6	2	3

Graph 1

Comparison of Antibacterial Activity of Schiff base metal complexes

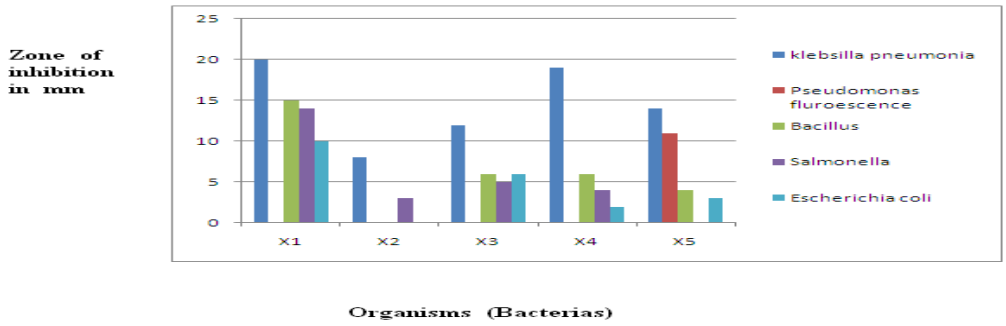


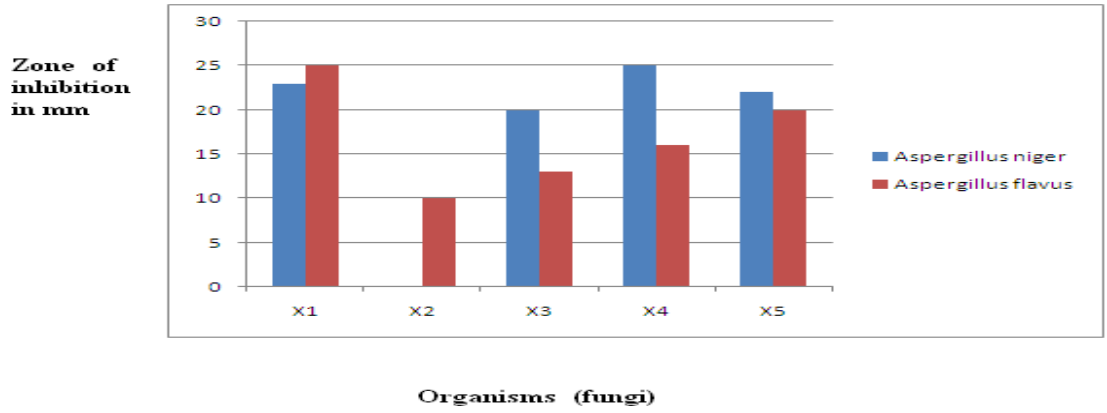
Table 7

Antifungal activity for different metal complexes

M (II) complexes	Aspergillusniger	Aspergillusflavus
X_1	23	25
X_2	-	10
X_3	20	13
X_4	25	16
X_5	22	20

Graph 2

Comparison of Antifungal activity of Schiff base metal complexes



DISCUSSION AND CONCLUSION

Schiff base transition metal complexes plays an important role in medical field. So, we have prepared the mixed ligand transition metal complexes of the metal ions Fe(II), Cu(II), Hg(II), Co(II), and Zn(II) using zone of inhibition using disc diffusion method. We have observed antimicrobial activity for all the Schiff base derived from salicylaldehyde and p-methoxy aniline as one of the ligand and 1,10-phenanthroline as another ligand. The Schiff base was first prepared by condensing salicylaldehyde and p-methoxy aniline. The structure of the ligand was confirmed by IR, UV-Visible and H^1NMR spectra. The shift in the spectral bands of the ligands upon complexation was also confirmed by taking the spectra of complexes. Using this we have proposed the possible common structure for the complex. he anti microbial activity of mixed ligand Schiff base transition metal complexes against the bacterias Pseudomonas fluorescence, Klebsilla pneumonia, Bacillus, Salmonella and Escheriacoli and the fungi Aspergillusniger and Asrpergillusflavus was accessed by finding out the zone of inhibition by disc diffusion method.Antibacterial activity was observed in many complexes. So, these transition metal complexes can be used in medicinal field.

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