



**SYNTHESIS, SPECTRAL CHARACTERIZATION OF BIOLOGICALLY ACTIVE NOVEL SCHIFF BASE COMPLEXES DERIVED FROM 2-SULPHANILAMIDOPYRIMIDINE**

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**ABSTRACT**

New Schiff base has been prepared from 2-sulphanilamidopyrimidine and 2-hydroxy-3-methoxy benzaldehyde in ethanolic media and then complexed with Mn(II), Co(II), Ni(II), Cu(II) and Zn(II) ions. The synthesized ligand and complexes were characterised by Elemental analysis, Molar conductance, Magnetic susceptibility, IR, UV,  $^1\text{H}$  &  $^{13}\text{C}$  NMR, EPR, and. The Spectral data confirms well coordination of Schiff-base ligand with the metal through imine nitrogen and oxygen atoms of Schiff base moiety. The Schiff base ligand and the complexes were screened for antimicrobial activity and fluorescent behaviour. From the analytical and spectral data, the stoichiometry has been found to be 1:2 for all the complexes. An octahedral structure has been proposed. All the new complexes were found to be active against bacteria and fungi.

**KEYWORDS:** Schiff base, 2-sulphanilamidopyrimidine, 2-hydroxy-3-methoxybenzaldehyde, Metal complexes, Biological activity, Fluorescent behaviour.



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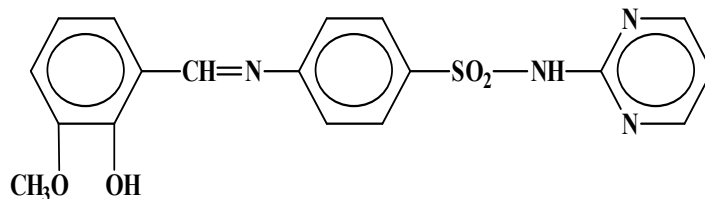
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## INTRODUCTION

**Sulpha** drugs are chemotherapeutic agents whose molecular structures contain a 4-aminobenzene sulfonamide moiety. The antimicrobial activity of these drugs is believed from the structural resemblance between sulfanilamide group and p-amino benzoic acid where the sulpha drug mimics this metabolite and block folic acid synthesis in bacteria, there by causing cell death. Many sulpha drugs like sulphadiazine, sulphamethoxazole, sulphamerazine possess  $\text{SO}_2\text{NH}$  moiety as an important toxophoric function<sup>1</sup>. It has been reported that the biologically active compounds show greater activity when administered as metal complexes than as free compound. The efficacy of the sulpha drugs can be enhanced upon co-ordination with a suitable metal ion<sup>2</sup>. Schiff base compound containing an imino group ( $-\text{RC}=\text{N}-$ ) are usually formed by the condensation of primary amine with an active carbonyl group. Schiff bases are regarded as privileged ligands. Schiff base and their metal complexes are very popular due to their diverse chelating ability. They play important role in both synthetic and structural research because of their preparative accessibility and structural diversity<sup>3</sup>. Metal complexes of Schiff bases are extensively studied due to synthetic flexibility, selectivity and sensitivity towards a variety of metal atoms. Schiff bases are used as pigments and dyes, catalysts, intermediates in

organic synthesis and as polymer stabilizers. A number of Schiff's base molecules show biological activities including antibacterial, antifungal, antidiabetic, antitumour, antiproliferative, anticancer, anti-corrosion and anti-inflammatory activities<sup>4-7</sup>. The topical application of metal complexes of sulphadiazine has recently revived the usefulness of these compounds in medicine<sup>8</sup>. Indeed metal sulphadiazine complexes are now widely used to prevent bacterial infection during burn treatments. The interest in metal based sulfonamides was stimulated by the successful introduction and preparation of Ag(I) and Zn(II) sulphadiazine complexes to prevent various bacterial infections<sup>9</sup>. Paralleling the same idea, this paper records the synthesis and characterization of Mn(II), Co(II), Ni(II), Cu(II) and Zn(II) complexes derived from 2-sulphanilamidopyrimidine and 2-hydroxy-3-methoxy benzaldehyde. The structures of the ligand and its metal complexes were characterized by Elemental analysis, Molar conductance, Magnetic susceptibility, IR, UV,  $^1\text{H}$  &  $^{13}\text{C}$  NMR and ESR. The biological activities are also studied against gram positive and gram negative bacteria and fungi organisms for Schiff base ligand and their complexes. The structure of Schiff base ligand confirmed in the present work is given in Figure 1



**Figure 1**  
**Structure of the ligand**

## EXPERIMENTAL

All chemicals used were of analytical reagent grade (AR) and of highest purity available. Solvents were purified and dried according to

the standard procedures. All metal (II) compounds were used as acetate salts. IR spectra of the complexes were recorded in KBr pellets with a Perkin Elmer RX1 FT-IR

Spectrophotometer in the 4000-400 $\text{cm}^{-1}$  range. The electronic spectra were recorded in DMF on a Perkin Elmer Lambda 35 spectrophotometer in the 190-1100 nm range. The  $^1\text{H}$  &  $^{13}\text{C}$  NMR spectra were recorded on a Bruker 400MHz FT-PMR spectrometer ( $\text{DMSO-d}_6$ ). Elemental analysis of the ligand and complexes were obtained using Elementar Vario EL CHN rapid analyser. The X-band EPR Spectrum were recorded on a Bruker ESP X-band EPR Spectrometer using powdered samples at a microwave frequency 9450MHz. Magnetic susceptibilities were measured on a Auto magnetic Susceptibility meter (MSB-Auto) at room temperature. Melting points were determined using melting point apparatus (Elico) and are uncorrected. Conductivity measurements for the complexes were carried out on Elico Conductivity Bridge and a dip conductivity cell using dimethyl formamide as solvent. Fluorescence spectra were detected using Perkin Elmer LS 45 Spectrofluorometer.

### Synthesis of Schiff base ligand

(L) The Schiff base was prepared by the condensation of equimolar amounts of 2-sulphanilamidopyrimidine and 2-hydroxy-3-methoxy benzaldehyde in minimum quantity of ethanol. The resulting mixture was then refluxed on a water bath for 5 hours. A pale orange coloured solid mass separated out on

cooling was filtered, washed and dried over anhydrous  $\text{CaCl}_2$  in a desiccator. The purity of the ligand was checked by melting point, TLC and spectral data. The ligand is insoluble in some common organic solvents viz. acetone, benzene and soluble in polar solvents viz. DMF, DMSO.

### Synthesis of metal complexes

Metal complexes were synthesized by mixing the hot solution of ligand (0.004 mole) in minimum quantity of dimethyl formamide and ethanolic solution of metal acetates (0.002 mole). The resulting mixture was then refluxed in a water bath for 6 hours. The complexes obtained in each case were cooled, filtered and washed with ethanol several times to remove any excess of the ligand. Finally complexes were washed with anhydrous diethylether and dried in a desiccator.

## RESULTS AND DISCUSSION

The Schiff base ligand is synthesized by using equimolar quantities of 2-sulphanilamidopyrimidine and 2-hydroxy-3-methoxy benzaldehyde and its complexed with metal acetates according to the following equation:



The metal complexes derived vary in their colour. All the complexes are stable, non-hygroscopic and coloured solids. The physical characteristics and micro analytical data of the ligand and metal complexes are given in Table 1 and Table 2

**TABLE 1**  
**Physical Characteristics of Schiff base ligand and their complexes**

S.No	Ligand/ Complexes	Colour	Molecular Formula	M.P °C	Yield %	$\mu_{\text{eff}}$ (BM)	CN
1	L	orange	$\text{C}_{18}\text{H}_{16}\text{N}_4\text{O}_4\text{S}$	203	75	-	-
2	$[\text{MnL}_{2-2\text{H}}(\text{H}_2\text{O})_2]$	Sandal	$\text{C}_{36}\text{H}_{34}\text{N}_8\text{O}_{10}\text{S}_2\text{Mn}$	235	65	5.6	6
3	$[\text{CoL}_{2-2\text{H}}(\text{H}_2\text{O})_2]$	Nut Brown	$\text{C}_{36}\text{H}_{34}\text{N}_8\text{O}_{10}\text{S}_2\text{Co}$	240	65	4.5	6
4	$[\text{NiL}_{2-2\text{H}}(\text{H}_2\text{O})_2]$	Brown yellow	$\text{C}_{36}\text{H}_{34}\text{N}_8\text{O}_{10}\text{S}_2\text{Ni}$	245	55	2.5	6
5	$[\text{CuL}_{2-2\text{H}}(\text{H}_2\text{O})_2]$	Green	$\text{C}_{36}\text{H}_{34}\text{N}_8\text{O}_{10}\text{S}_2\text{Cu}$	234	75	1.95	6
6	$[\text{ZnL}_{2-2\text{H}}(\text{H}_2\text{O})_2]$	yellow	$\text{C}_{36}\text{H}_{34}\text{N}_8\text{O}_{10}\text{S}_2\text{Zn}$	226	65	dia	6

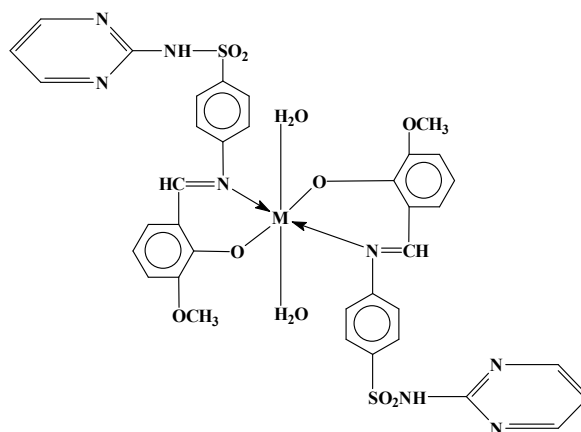
**TABLE 2**  
**Microanalytical data of Schiff base ligand and their complexes**

S.No	Ligand/ Complexes	Elemental Analysis (%) (calcd) found				%M (calcd) found	$\Lambda_m$ $\text{ohm}^{-1}\text{m}^2$ $\text{mol}^{-1}$
		C	H	N	S		
1	L	(56.25) 56.20	(4.17) 4.10	(14.58) 14.51	(8.33) 8.32	-	-
2	[MnL <sub>2-2H</sub> (H <sub>2</sub> O) <sub>2</sub> ]	(50.41) 50.41	(3.97) 3.96	(13.07) 13.0)	(7.47) 7.43	(6.42) 6.41	16.7
3	[CoL <sub>2-2H</sub> (H <sub>2</sub> O) <sub>2</sub> ]	(50.48) 50.45	(3.95) 3.95	(13.01) 12.99	(7.43) 7.43	(6.86) 6.84	3.44
4	[NiL <sub>2-2H</sub> (H <sub>2</sub> O) <sub>2</sub> ]	(50.19) 50.15	(3.95) 3.91	(13.01) 13.00	(7.44) 7.42	(6.82) 6.82	6.0
5	[CuL <sub>2-2H</sub> (H <sub>2</sub> O) <sub>2</sub> ]	(49.91) 40.91	(3.93) 3.91	(12.94) 12.93	(7.39) 7.39	(7.35) 7.34	7.86
6	[ZnL <sub>2-2H</sub> (H <sub>2</sub> O) <sub>2</sub> ]	(49.80) 49.79	(3.92) 3.91	(12.91) 12.90	(7.38) 7.38	(7.54) 7.54	2.9

### Molar Conductance and Magnetic Susceptibility Measurements

The observed molar conductances of all the complexes in  $10^{-3}$  DMF solution are found within the range of 2.9-16.7  $\text{ohm}^{-1}\text{cm}^2\text{mol}^{-1}$  showing their non-electrolytic nature. This in turn suggests that the water molecules are coordinated with the metal ions and present inside the coordination sphere in all the complexes. The magnetic data for Co<sup>II</sup> and Ni<sup>II</sup> complexes is consistent with octahedral

geometry around the metal ion for both complexes. The magnetic moment value of 1.95 BM for Cu (II) complex lies in the range expected for  $d^9$  system which contains one unpaired electron with octahedral geometry<sup>10</sup>. Zn (II) Complexes are found to be diamagnetic as expected. The observed magnetic moment value of 5.60BM for the Mn (II) complex suggests the octahedral geometry. The probable structure of complexes proposed in the present work is given in Figure 2.



**Figure 2**  
**Structure of the Complex**

**Infrared Spectra**

The infrared spectral data of the Schiff base and its metal complexes are recorded in Table 3. Schiff base showed a strong absorption band at  $1582\text{ cm}^{-1}$  characteristic of  $\nu(\text{C}=\text{N})$  whereas the broad band at  $3423\text{ cm}^{-1}$  characteristic of hydrogen bonded  $\nu(\text{O}-\text{H})$  stretching vibration<sup>11</sup>. The azomethine  $\nu(>\text{C}=\text{N})$  band at  $1582\text{ cm}^{-1}$  in Schiff base is shifted to higher frequency in Co(II), Mn(II), Ni(II), Cu(II), and Zn(II) by 15, 23, 12, 10 and  $12\text{ cm}^{-1}$  respectively which indicated the co-ordination of azomethine nitrogen on complexation<sup>12</sup>. The shifting of phenolic (OH) at  $3423\text{ cm}^{-1}$  in all the complexes suggests the coordination of phenolic oxygen after deprotonation<sup>13</sup>. The linkage with oxygen atom is further supported by the appearance of a band in the region around  $420\text{--}464\text{ cm}^{-1}$  which may be assigned to  $\nu(\text{M}-\text{O})$ <sup>14</sup>. A further

evidence of the coordination of the N atom of the Schiff base with the metal atom was shown by the appearance of a new weak frequency band at  $512\text{--}578\text{ cm}^{-1}$  assigned to the metal nitrogen  $\nu(\text{M}-\text{N})$ <sup>15</sup>. These new bands were observed only in the spectra of the metal complexes and not in Schiff base which confirmed the participation of the donor groups. The bands in the ligand due to  $\nu_{\text{as}}(\text{SO}_2)$  and  $\nu_{\text{s}}(\text{SO}_2)$  appear at  $1155\text{ cm}^{-1}$  and  $1332\text{ cm}^{-1}$  respectively. These bands almost remain unchanged in the complexes indicating that this  $-\text{SO}_2$  group is not participating in coordination. This is confirmed by the unchanged  $\nu(\text{S}-\text{N})$  and  $\nu(\text{C}-\text{S})$  modes appearing around  $945\text{ cm}^{-1}$  and  $843\text{ cm}^{-1}$  respectively. The ring nitrogen ( $=\text{N}-$ ) of the Schiff base does not take part in coordination, supported by unchanged band around  $1275\text{ cm}^{-1}$ .

**TABLE 3**  
**IR and Electronic spectral data**

Ligand/ Complexes	IR spectral data, $\text{cm}^{-1}$				Electronic spectral data, $\text{cm}^{-1}$
	$\nu(\text{O}-\text{H})$	$\nu(\text{C}=\text{N})$	$\nu(\text{M}-\text{O})$	$\nu(\text{M}-\text{N})$	
L	3423	1582	-	-	33333, 28601
[MnL <sub>2-2H</sub> (H <sub>2</sub> O) <sub>2</sub> ]	3446	1597	520	447	39205, 36639, 34230
[CoL <sub>2-2H</sub> (H <sub>2</sub> O) <sub>2</sub> ]	3440	1605	512	464	38165, 36777
[NiL <sub>2-2H</sub> (H <sub>2</sub> O) <sub>2</sub> ]	3450	1594	531	412	39064, 38329, 36846
[CuL <sub>2-2H</sub> (H <sub>2</sub> O) <sub>2</sub> ]	3446	1591	524	442	33976, 33065, 24915
[ZnL <sub>2-2H</sub> (H <sub>2</sub> O) <sub>2</sub> ]	3399	1594	531	431	38283, 36774

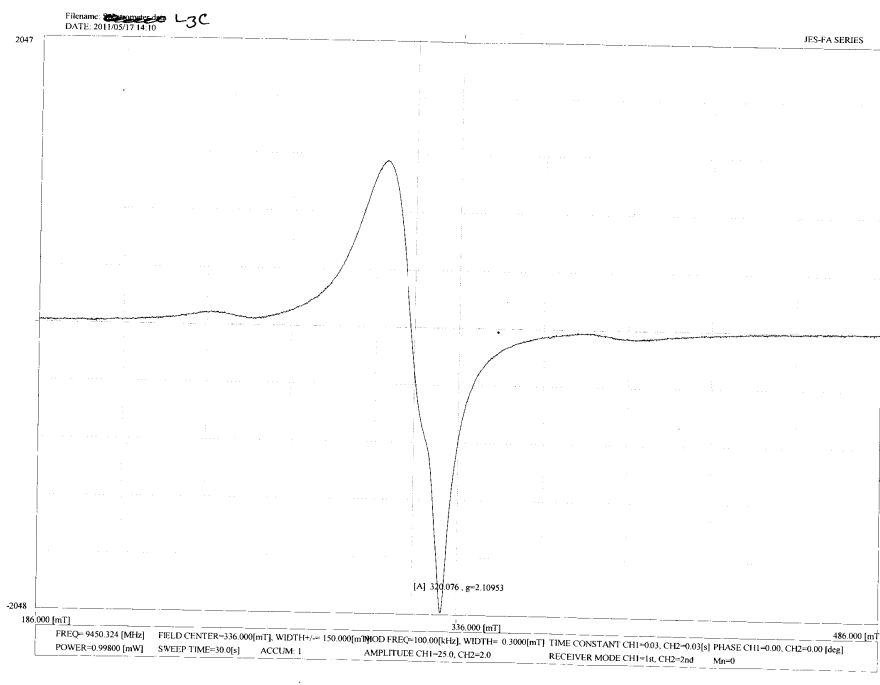
**Electronic Spectra:** Electronic spectrum of the ligand shows two high intensity bands at  $33,333\text{cm}^{-1}$  and  $28901\text{cm}^{-1}$  indicate  $n \rightarrow n^*$  and  $\pi \rightarrow \pi^*$  transitions respectively of the ligand moiety<sup>16</sup>. The electronic spectrum of the Mn(II) complex shows a band at  $39205\text{cm}^{-1}$  assignable to  $M \rightarrow L$  charge transfer spectra and the bands at  $36639$  and  $34230\text{cm}^{-1}$  are due to  ${}^6A_{1g} \rightarrow {}^4T_{2g}(D)$  and  ${}^6A_{1g} \rightarrow {}^4E_{g(D)}$ <sup>17</sup>. The electronic spectra of Co(II) complex displays bands at  $38165$  and  $36777\text{cm}^{-1}$ . The first band corresponds to intra ligand transition of the organic moiety<sup>18</sup> and the later corresponds to  ${}^4T_{1g}(F) \rightarrow {}^4T_{1g}(P)$  suggesting octahedral geometry of this complex. Ni(II) complex shows absorption bands at  $39064, 38329$  and  $36846\text{cm}^{-1}$ . The high intensity bands at  $39064$  and  $38329\text{cm}^{-1}$  is relatively attributed to  $L \rightarrow M$  charge transfer transitions whereas the band at  $36846\text{cm}^{-1}$  may be due to  ${}^3A_{2g} \rightarrow {}^3T_{2g}$ . The Cu(II) complex displays a band at  $33976, 33065$  and  $24915\text{cm}^{-1}$ . The first two bands are attributed to intra ligand transition and the third band at  $24915\text{cm}^{-1}$  corresponds to  ${}^2E_{g} \rightarrow {}^2T_{2g}$ <sup>19</sup>. Zn(II) complex displays high intensity bands at  $38283$  and  $36774\text{cm}^{-1}$ . This may be due to Ligand  $\rightarrow$  Metal charge transfer spectra<sup>20</sup>.

### **<sup>1</sup>H NMR and <sup>13</sup>C NMR Spectra**

The <sup>1</sup>H NMR Spectra of Schiff base and its complexes were recorded in DMSO (d<sub>6</sub>). The azomethine proton (-CH=N-) in Schiff base appeared at  $\delta = 8.5$  ppm has been shifted to downfield in metal complexes. This confirms the coordination by azomethine nitrogen<sup>21</sup>. The aromatic protons in Schiff base appeared in the range at  $\delta$  6.8 to 8.1 ppm and metal complexes in the range  $\delta$  6.5 to 8.5 ppm. The disappearance of phenolic -OH proton signal at  $\delta$  12.2ppm confirms the coordination by phenolic oxygen to metal ion<sup>22</sup>. The <sup>13</sup>C-nmr spectral data (imine at  $\delta$  160.0 ppm., aromatic C-OH at  $\delta$  119.1 ppm., CH pyrimidine at  $\delta$  157.9 ppm) for ligand and (imine at  $\delta$  158.04 ppm., aromatic carbon at  $\delta$  129.5., CH pyrimidine at  $\delta$  158.0 ppm) for complexes supports the proposed structure.

### **EPR Spectra**

The room temperature spectra of powdered samples were recorded at 9450MHz. Both parallel and perpendicular features of Cu are resolved in the spectra, which are characteristic of axial symmetry. The  $g_{\parallel}$  and  $g_{\perp}$  component for the complex were calculated as 2.22 and 2.04. The  $g$  value of Cu(II) complex is found to be 2.1095 confirms the presence of unpaired electron in the  $dx^2-y^2$  orbital of Cu(II). The  $g$  value are very close to those reported for a number of distorted Cu(II) complex. Moreover, the observed  $g$  value is less than 2.3, suggest the covalent nature of metal-ligand bonds in the complex.<sup>23</sup> The lines of this type usually observed are either due to the intermolecular spin exchange, which may broaden the lines or to the occupancy of the unpaired electron in the degenerate orbital. The nature and pattern of the EPR spectra (Figure 3) suggest an almost octahedral environment around the Cu(II)

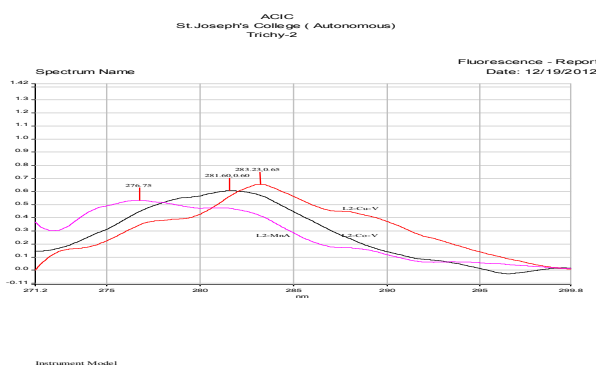


**Figure 3**  
**EPR spectra for Cu(II) Complex**

**Fluorescence Spectra**

The Photoluminescence properties of the azo Schiff base ligand and their complexes were studied at room temperature for  $10^{-4}$  M solution for all compounds in DMSO solution. Excitation and emission slit widths were set at 10nm with a scan speed of 500nm/min. The excitation spectra of the ligand show a maximum at 300nm and show an emission

peak at 338nm. Generally azo Schiff base systems exhibit fluorescence due to intraligand  $\pi-\pi^*$  transitions. Fluorescence spectra of the ligand solutions as  $10^{-4}$ M in DMSO obtained with the excitation at 300nm. The metal complex of Mn(II) exhibits the weakest quenching<sup>24</sup>. The strongest quenching has been obtained for the Cu(II) complex (Figure 4).



**Figure 4**  
**Fluorescence Spectra of Mn(II), Co(II) and Cu(II) Complexes**

These results show that the ligand has a potential for trace analysis of some transition metals with fluorimetric method. The fluorescence data of the complexes were summarized in Table 4.

**Table 4**  
**Fluorescence Characteristic of ligand and its complexes**

Complexes	Excitation Wavelength $\lambda_{\max}$ (nm)	Fluorescence $\lambda_{\max}$ (nm)	wavelength	Quantum Yield
L	300	338		0.88
[MnL <sub>2-2H</sub> (H <sub>2</sub> O) <sub>2</sub> ]	261	276		0.94
[CoL <sub>2-2H</sub> (H <sub>2</sub> O) <sub>2</sub> ]	270	281		0.96
[NiL <sub>2-2H</sub> (H <sub>2</sub> O) <sub>2</sub> ]	255	271		0.94
[CuL <sub>2-2H</sub> (H <sub>2</sub> O) <sub>2</sub> ]	270	283		0.95
[ZnL <sub>2-2H</sub> (H <sub>2</sub> O) <sub>2</sub> ]	260	274		0.94

### Antimicrobial activity

Antibacterial and antifungal activity of Schiff base ligand and its manganese cobalt, nickel, copper, and zinc complexes have been tested by disc diffusion technique<sup>25,26</sup>. The various gram positive and gram negative bacterial organisms such as Gram negative bacteria *pseudomonas aeruginosa*, *E.coli* Gram positive bacteria *staphylococcus aureus*, *Klebsiella aerogenes* and fungi *aspergillus niger* and *Mucor* were used to find out the antimicrobial activity. (Table 5). Filter paper discs of diameter 6mm were used and the

diameters of zones of inhibition formed around each disc after incubating for a period of 72 hours at 25-30° C were recorded. Results were compared with standard drug Ciprofloxacin for bacteria and Nystatin for fungi at the same concentration. All the new complexes showed a remarkable biological activity against bacteria and fungus<sup>27</sup> (Figure 6). From the results it is clear that the metal complexes are found to have more antimicrobial activity than the parent ligand.

**TABLE 5**  
**Antimicrobial Activity of Schiff base ligand and complexes**

Antimicrobial activity of the ligand and complexes	Staphylococcus aureus	Klebsiella aerogenes	E.coli	Pseudomonas aeruginosa	Mucor	Aspergillus niger
Ligand (L)	++	+++	+++	++	++	++
[MnL <sub>2-2H</sub> (H <sub>2</sub> O) <sub>2</sub> ]	+++	+++	+++	+++	+++	+++
[CoL <sub>2-2H</sub> (H <sub>2</sub> O) <sub>2</sub> ]	+++	+++	+++	++	+++	+++
[NiL <sub>2-2H</sub> (H <sub>2</sub> O) <sub>2</sub> ]	+++	+++	+++	++	+++	+++
[CuL <sub>2-2H</sub> (H <sub>2</sub> O) <sub>2</sub> ]	+++	+++	+++	++	++	+++
[ZnL <sub>2-2H</sub> (H <sub>2</sub> O) <sub>2</sub> ]	+++	+++	+++	+++	++	+++

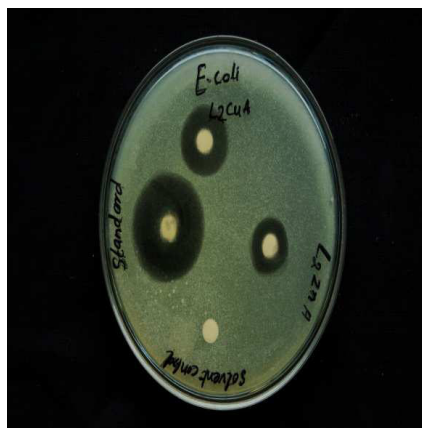
Standard= ciprofloxacin 5 g/ disc for bacteria ; Nystatin= 100 units/disc for fungi.

Highly active = +++ (inhibition zone > 15mm) ; Moderately active = ++ (inhibition zone > 10mm) ;

slightly active = + (inhibition zone > 5mm); Inactive = -- (inhibition zone < 5mm)



Figure 6



a) Activity of Cu(II) and Zn(II) complexes against gram positive bacteria E.coli



b) Activity of ligand and Mn(II), Co(II), Ni(II) complexes against fungus Mucor



c) Activity of ligand and Mn(II), Co(II), Ni(II) complexes against fungus Pseudomonas aeruginosa

## CONCLUSION

The coordination ability of the newly synthesized azo Schiff base has been proved in complexation reaction with Mn(II), Co(II), Ni(II), Cu(II) and Zn(II) ions. IR, UV,  $^1\text{H}$  NMR and magnetic measurements of the ligand and its complexes confirm the suggested coordination of the ligand through azomethine linkage. Based on these facts, an octahedral structure has been proposed for all complexes. The process of chelation dominantly affects the biological activity of the complexes that are potent against pathogens. In general, all the synthesized complexes can serve as potential

photoactive materials, as indicated from their characteristic fluorescence properties.

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