



Synthesis, spectral characterization and some biological studies of transition metal complexes with Schiff base ligand containing N,O and S as donor atoms

Anil Kumar M R^{1*}, Shanmukhappa S¹, Rangaswamy B E² and Revanasiddappa M³

¹Department of Chemistry, Bapuji Institute of Engineering and Technology, Davangere – 577 004.

²Department of Biotechnology, Bapuji Institute of Engineering and Technology,

³Department of Chemistry, PESIT – Bangalore South Campus, Hosur Road, Bangalore 560 100.

*Corresponding author: anilshyagale@gmail.com

ABSTRACT: Transition metal complexes of Cu(II), Co(II), Ni(II), Zn(II), Cd(II) and Mn(II) have been synthesized with the Schiff base ligand 5-Sub-N-(2-mercaptophenyl)salicylideneimine. Elemental analysis of these complexes suggest that these metal ions forms complexes of type ML(H₂O)_{stoichiometry} for Cu(II), Co(II), Ni(II), Zn(II), Cd(II) and Mn(II). The ligand behaves as tridentate and forms coordinate bonds through O, S and N atoms. Magnetic susceptibility, IR, UV – Visible, Mass and ESR spectral studies suggest that Cu(II), Ni(II) complexes possess square planar geometry, whereas Co(II), Zn(II), Cd(II) and Mn(II) complexes possess tetrahedral geometry. The complexes were tested for their antimicrobial activity against the bacterial strains *Staphylococcus aureus* and *Bacillus subtilis*. The Schiff base metal complexes evaluated for their antifungal activity against the fungi *A. niger* and *C. oxysporum*. The DNA cleavage studies of Schiff base complexes were studied using Calf – Thymus DNA by agarose gel electrophoresis method.

Key words: Schiff base, metal complexes, antimicrobial activity, antifungal activity, DNA cleavage

1. INTRODUCTION:

In the Year 1864, azomethines were synthesized by Hugo Schiff which are generally called as Schiff bases since they can act as ligands¹. General syntheses of the Schiff bases involve the condensation reaction between carbonyl compounds (aldehydes or ketones) with aromatic primary amines by the elimination of water molecule. These Schiff bases possess the general formula R – CH = N – R₁, where R and R₁ are alkyl or aryl or heterocyclic group. The C=N group is called as imine group or azomethine². With several derivatives, Schiff bases form complexes with transition metals through N, O and S they can form many kinds of complexes with different metal ions³. Generally Schiff bases can act as chelating ligands since they contain groups such as –OH, –SH, –NO₂ etc when present closer to azomethine group. The preparation of Schiff bases was simple and this enables the preparation of number Schiff bases and their metal complexes⁴.

Many of the Schiff bases and the derivatives of Schiff bases possess various biological activities such as antimicrobial, antifungal, anticancer etc. Since the bacterium can easily achieve the resistance to many antibiotics through the morphological modifications the study of antibacterial activity is more predominantly done than the study of antifungal activity⁵. The presence of azomethine group and sulfonamide group in Schiff bases was mainly responsible for their antimicrobial activity and that activity can be varied by the change in the type of substituent attached to the aromatic ring. Most of the research works shows that redox potential of the Schiff bases is directly related to their dioxygen binding ability and nucleophilicity⁶.

2. EXPERIMENTAL:

2.1 - Solvents and Metal Salts: The solvents like ethanol, methanol, dimethyl sulphoxide and diethyl ether were used for the synthesis of Schiff bases and their complexes. Analytical grade metal salts like MnCl₂, CoCl₂, NiCl₂, CuCl₂, ZnCl₂ and CdCl₂ were used. Molar conductivities of prepared 10⁻³M solutions were carried out using EM 183 EC conductivity meter. Melting points for the compounds are obtained from Electric melting point apparatus. Elemental analysis data for the prepared complexes are obtained from microanalysis of Carbon, Oxygen, Hydrogen, Nitrogen and sulphur using Thermo Finnigan FLASH EA 1112 CHNS analyzer. The FT - IR spectra were recorded on a FTIR-ATR spectrometer with 4 cm⁻¹ resolution in the frequency range 400–4000 cm⁻¹. The proton magnetic resonance spectra of complexes were recorded on a Bruker Ac 300 F, 300 MHz in the range of 0 – 15 ppm down field of TMS. DMSO – d⁶ was used as solvent and tetramethyl silane (TMS) as the internal standard.

UV – Visible spectra were recorded on UV spectrophotometer. The magnetic susceptibility was measured at room temperature using Gouy balance. The Electron spin resonance spectra of Copper (II) complex in poly crystalline state were recorded on Varian X – band ESR spectrometer diphenyl picryl hydrazine (DPPH) free radical of “g” marker (g = 2.0027) at room temperature. Mass spectra were recorded on a JEOL SX 102/DA 6000 Mass spectrophotometer by using argon/xenon (6 kV, 10 mA) as the FAB gas. The DNA cleavage activity of all the compounds was done by agarose gel electrophoresis method by using Calf-Thymus DNA. Antibacterial activity and the antifungal activities of the Schiff base and metal complexes were studied by agar diffusion method.

2.2 - Synthesis of Metal Complexes: The preparation of the metal complexes was done by mixing of about 50 mL of 2.5 mmol solution of MnCl₂.xH₂O, CoCl₂.6H₂O, NiCl₂.6H₂O, CuCl₂.6H₂O, ZnSO₄.7H₂O and CdCl₂.5/2H₂O in ethanol/methanol with about 50 mL of 2.5 mmol solution of Schiff base ligands in ethanol in the metal: ligand ratio of 1:1.



The mixture was subjected for refluxing on a water bath nearly about 4-5 hours. The coloured product produced when the reaction mixture was allowed to stand and then after cooling the above obtained solution. The precipitated complex compound in solid state was filtered and then subjected to recrystallization with ethanol and then dried in an electric oven⁷⁻⁸. Yield: 70-80%.

3. RESULTS AND DISCUSSION

3.1 – Elemental Analysis and Molar Conductivity:

The analytical data of complexes and their molar conductance values are given in table 1. All these complexes are analyzed. The stoichiometry of the ratio 1:2 of the type ML(H₂O)

Table-1: Elemental analysis, colour, melting point and conductance data of Ligand and metal complexes

Sl. No	Molecular formula	Elemental analysis, found(calculated)						Colour	M.P (°C)	Molar conductivity (S _{cm} ² mol ⁻¹)
		C	H	O	N	S	M			
1	C ₁₃ H ₁₁ NOS Ligand	68.10 (68.12)	4.70 (4.80)	6.88 (6.99)	6.08 (6.11)	13.88 (13.97)	----	Yellow	165	----
2	Co(C ₁₃ H ₁₁ NOS)(H ₂ O)	50.86 (50.99)	4.10 (4.24)	10.36 (10.45)	4.44 (4.58)	10.34 (10.45)	19.12 (19.26)	Brown	280	10.88
3	Ni(C ₁₃ H ₁₁ NOS)(H ₂ O)	50.88 (51.03)	4.16 (4.25)	10.34 (10.46)	4.48 (4.57)	10.32 (10.46)	19.08 (19.19)	Yellow	340	20.08
4	Cu(C ₁₃ H ₁₁ NOS)(H ₂ O)	50.06 (50.23)	4.06 (4.18)	10.24 (10.30)	4.42 (4.50)	10.20 (10.30)	20.28 (20.46)	Dark Brown	330	10.33
5	Zn(C ₁₃ H ₁₁ NOS)(H ₂ O)	49.76 (49.93)	4.10 (4.16)	10.16 (10.24)	4.34 (4.48)	10.14 (10.24)	20.62 (20.93)	Yellow	> 300	8.44
6	Cd(C ₁₃ H ₁₁ NOS)(H ₂ O)	43.18 (43.40)	3.04 (3.16)	8.74 (8.90)	3.68 (3.89)	10.72 (8.90)	31.12 (31.27)	Yellow	216	7.20
7	Mn(C ₁₃ H ₁₁ NOS)(H ₂ O)	51.54 (51.66)	4.16 (4.30)	10.44 (10.59)	4.42 (4.63)	10.50 (10.59)	18.06 (18.19)	Brown	> 300	8.98

3.2 – IR Spectral Data:

In the present work, in the IR spectra of the complexes, the characteristic stretching frequency of azomethine group that was to be observed around 1600 cm⁻¹ is shifted towards lower frequency side which confirms the coordination of the nitrogen of azomethine group to the metal⁹. The band that was observed in ligand around 1200 cm⁻¹ is due to the presence of hydroxyl group attached to aromatic ring, but in the spectra of all the complexes derived from this ligand the band due to the stretching frequency of C-O is shifted towards longer wavelength side or lower frequency side which confirms the coordination of the O atom with the metal ion by the deprotonation of phenolic group. All the metal complexes exhibit broad bands in the frequency range of 3400 – 3785 cm⁻¹ and weak bands around 850 and 700 cm⁻¹ shows the presence of water molecules coordinated to the metal¹⁰⁻¹². The presence of bands in our synthesized complexes lie in the region ≈ 500-600 cm⁻¹ and 450-550 cm⁻¹ are due to M-O and M-N vibrations respectively¹³. The presence of a weak band in the region 742 – 765 cm⁻¹ was observed corresponding to the νC-S in the ligands and in the complexes this band was shifted to lower wave number side and that supports the involvement of S in coordination with the metal ion¹⁴. In the present work we observed the bands in the IR spectra of the all complexes synthesized by the ligand around 700 cm⁻¹ and by this we can confirm the involvement of S in coordination with the metal ion. The IR spectral data of complexes is given in table 2.

Table-2: IR spectral data (cm⁻¹) of complexes

Sl. No.	Complexes	ν _{M-O}	ν _{M-N}	ν _{M-S}
1	Co(C ₁₃ H ₁₁ NOS)(H ₂ O)	590.1	517.8	727
2	Ni(C ₁₃ H ₁₁ NOS)(H ₂ O)	591.1	518	700
3	Cu(C ₁₃ H ₁₁ NOS)(H ₂ O)	601.7	520.7	702.0
4	Zn(C ₁₃ H ₁₁ NOS)(H ₂ O)	576.6	536.1	727
5	Cd(C ₁₃ H ₁₁ NOS)(H ₂ O)	629.9	515.9	735
6	Mn(C ₁₃ H ₁₁ NOS)(H ₂ O)	577.6	518.8	727

3.3 – Electronic Spectra:

The electronic spectra of Co (II) complex exhibit band at 25477 cm^{-1} and that can be assigned to LMCT transition¹⁵. Presence of band at 35050 cm^{-1} can be assigned to $\pi \rightarrow \pi^*$ transition¹⁶⁻¹⁷. Another very less intensity band observed at 19996 cm^{-1} can be assigned to ${}^4A_2(F) \rightarrow {}^4T_1(P)$ ¹⁸. Based on the spectral data and the magnetic moment value of 4.36 B.M the Co (II) complex is expected to have tetrahedral geometry.

Ni (II) complex exhibits mainly two bands at 29886 cm^{-1} and 23629 cm^{-1} which can be assigned for metal to ligand charge transfer band and due to ${}^1A_{1g} \rightarrow {}^1B_{1g}$ respectively¹⁹. Another band observed at 36258 cm^{-1} and can be assigned to $\pi \rightarrow \pi^*$ transition. Based on this data we propose presence of square planar geometry for Ni(II) complex²⁰.

Cu (II) complex exhibit a band at 29753 cm^{-1} can be attributed to metal to ligand charge transfer transition and the band observed at 38714 cm^{-1} is due to $\pi \rightarrow \pi^*$ transition²¹. Bands present at 40209 cm^{-1} and 46040 cm^{-1} can be assigned to the intraligand charge transfer transitions²². Absence of any band less than 10000 cm^{-1} excludes the possibility of tetrahedral geometry around for Cu (II) complex²³. Based on the spectral data and the magnetic moment value of 1.72 BM, the Cu (II) complex is expected to have square planar geometry.

In the electronic spectra of Mn (II) complex, a band is observed at 29403 cm^{-1} can be assigned to ${}^6A_1 \rightarrow {}^4E_1(F)$ transition and the band that appears at 26109 cm^{-1} can be assigned to d-d transition²⁴⁻²⁵. Based on the spectral data and the magnetic moment value of 5.62 BM, the Mn (II) complex is expected to have high spin tetrahedral geometry.

3.4 – ESR Spectra:

Powdered samples of Cu (II) complexes are used to record X-band ESR spectra of the complexes at room temperature. An ESR spectrum of complex shown is in figure 1. The value of $g_{\parallel} > g_{\perp} > g_e$ (2.0023) in the Cu(II) complex indicates the presence of an unpaired electron predominantly in the $d_{x^2-y^2}$ orbital. The main cause for the deviation of calculated g_{av} value of 2.175 (table-3) when compared to that of the free electrons (2.0023) can be due to the covalent character of metal – ligand bond. The g_{\parallel} values less than 2.3 indicates covalent environment in all the complexes. The high value of $g_{\parallel}/A_{\parallel}$ for our synthesized Cu(II) complex when compared to the $g_{\parallel}/A_{\parallel}$ value of octahedral Cu(II) complexes as reported indicates the presence of square planar environment in the Cu(II) complex²⁶⁻²⁷.

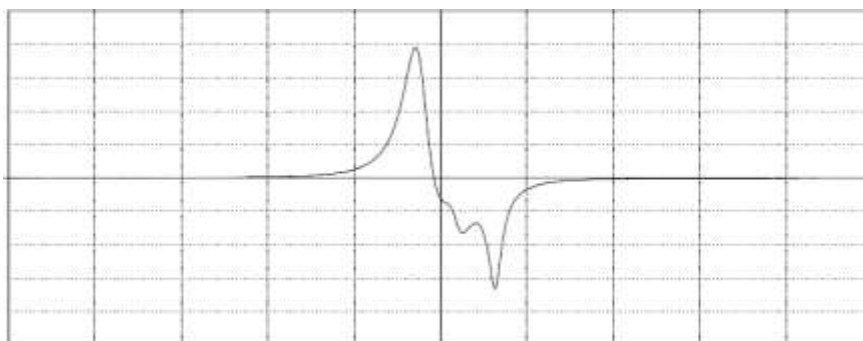


Figure 1: ESR Spectrum of Cu(II) complex

The electron spin resonance spectral data is given in table 3.

Table-3: Electron Spin Resonance Spectral Data

Sl.No	Complex	g_{\parallel}	g_{\perp}	g_{av}	A_{\parallel}	$g_{\parallel}/A_{\parallel}$
1	Cu(C ₁₃ H ₁₁ NOS)(H ₂ O)	2.28	2.07	2.175	126	0.0180

3.5 - Mass Spectral Data of Ni(II) Complex:

The mass spectra of the Ni(II) complex shows a molecular ion peak at m/z 292.1 which is due to the molecular weight of the complex (305) formed by the loss of water molecules which can happen due to fragmentation²⁸. The mass spectra (Fig: 2) of complex shows the presence of multiple peaks that represents the successive degradation of complex due to formation of various fragments. The fragments of species further undergo demetallisation to give a peak at m/z value of 172.1. The peaks of appreciable intensity observed at m/z values of 172.1 and 113.1 clearly indicate the fragmentation pattern²⁹. The fragmentation pattern of the Ni(II) complex can takes place in the possible ways as represented in fig. 3.

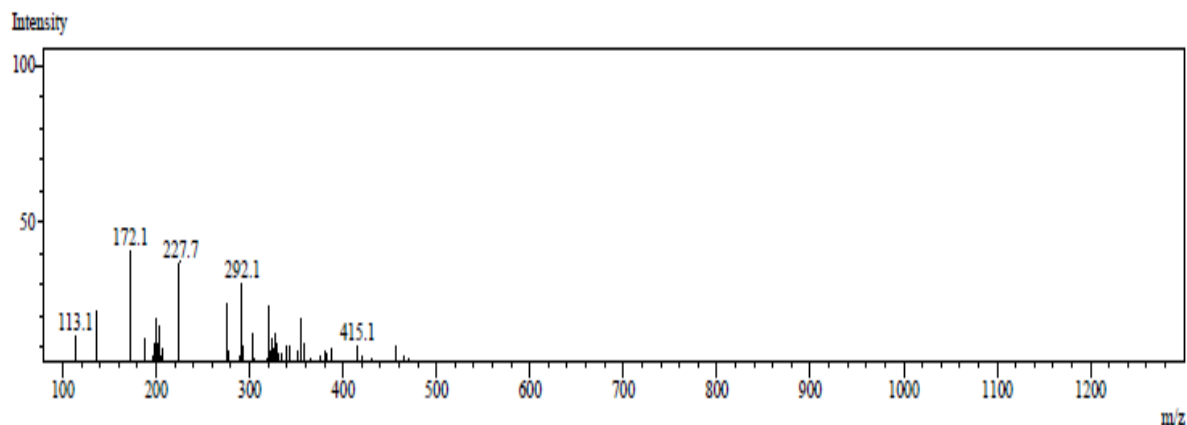


Fig. 2 – Mass Spectra of Ni(C₁₃H₁₁NOS)(H₂O) Complex

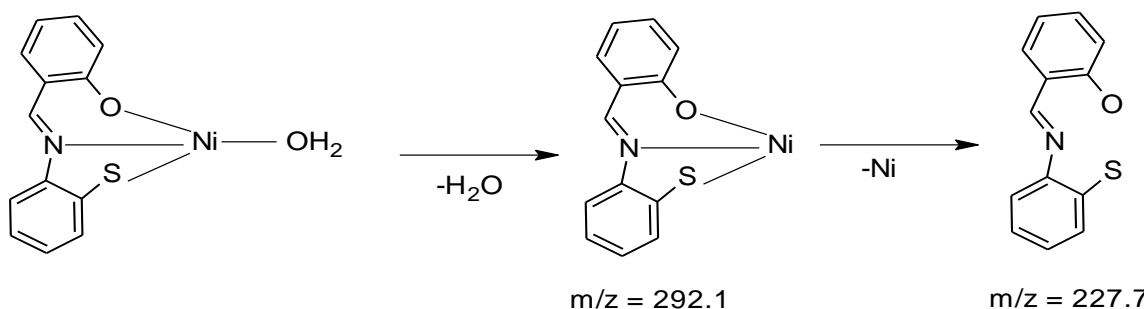


Fig. 3 – Fragmentation pattern of Ni(C₁₃H₁₁NOS)(H₂O) Complex

3.6 – Thermal Studies:

The thermal analysis technique is used to determine changes in weight with respect to changes in temperature. Differential thermal analysis (DTA) is a technique to determine changes of phases in materials with respect to temperature.

TG-DTA data of Co (C₁₃H₁₁NOS)(H₂O) complex:

The thermal study of Co (C₁₃H₁₁NOS)(H₂O) complex sample was conducted in the nitrogen atmosphere at a heating speed of 10°C/minute in the range 30-600 °C and the resulting thermogram is shown in Figure 4. The initial sample mass used for the study was 13 mg. The TG curve clearly shows that the Co (C₁₃H₁₁NOS)(H₂O) crystals started to undergo decomposition around 250 °C. The mass loss (0.9%) below the decomposition is due to the loss of coordinated water molecule. The considerable weight loss (98.52%) happens in the range 250 °C and 280 °C. Later, the resulting residue gives a weight loss for a small change from 280 °C to 500 °C. The DTA thermogram shows only one irreversible endothermic sharp peak at 278 °C corresponding to the melting point of Co (C₁₃H₁₁NOS)(H₂O) complex is clearly coincided with that of TGA curve. The narrowness of the endothermic peak represents the well crystalline nature³⁰⁻³¹. TG-DTA curve of Co (C₁₃H₁₁NOS)(H₂O) Complex is shown in figure 4.

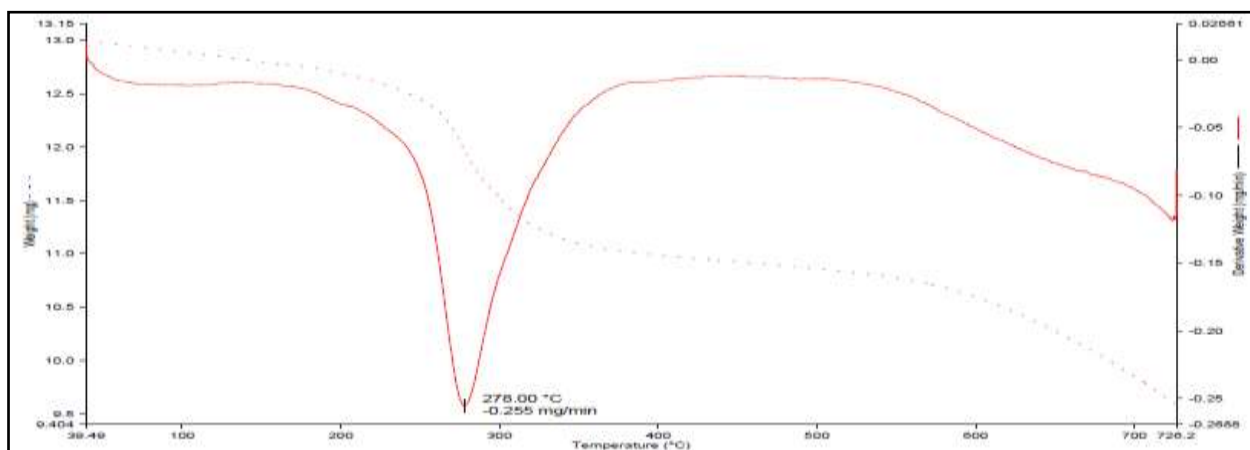


Fig – 4: TG-DTA curve of Co (C₁₃H₁₁NOS)(H₂O) Complex

3.7 – DNACleavage Studies:

The DNA cleavage activity of Co(II), Ni(II) and Cu(II), Zn(II), Cd(II) and Mn(II) complexes were studied by using agarose gel electrophoresis method with Calf-Thymus DNA. After the gel electrophoresis it was observed that the intensity of DNA is diminished which is due to cleavage of DNA by Schiff base metal complexes and this indicates the involvement of metal ions in the cleavage reactions. The difference observed in the bands of metal complexes obtained when compared to the control DNA was may due to molecular weight difference. This shows the control DNA cannot alone show any cleavage.

It is observed that the synthesized compounds can cleave DNA, thus we concluded that the many of our synthesized compounds inhibits the growth of the pathogenic organism due to cleavage of the genome³²⁻³⁴.

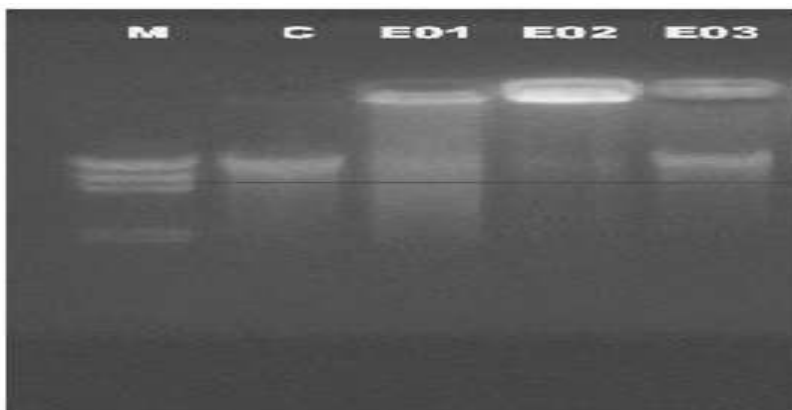


Figure 5: DNA cleavage analysis of metal complexes. [E01- Co(II), E-02- Ni(II), E-03- Cu(II)]

M- Standard DNA molecular weight marker C- Control DNA (untreated sample)

E01- Ni(C₁₃H₁₁NOS)H₂O

E02- Cu(C₁₃H₁₁NOS)H₂O

E-03- Co(C₁₃H₁₁NOS)H₂O

3.8 – Antibacterial activity:

The antibacterial activity of the Schiff base and their metal complexes against bacterial strains such as *Staphylococcus aureus* and *Bacillus subtilis* was investigated by agar diffusion method. The concentrations used for the two bacterial strains are 25, 50, 100, 200, 400 and 800 µg/ml. Standard antibiotic used was Gentamycin and DMSO is used as solvent. The antibacterial activities shown by ligand and the metal complexes against *Staphylococcus aureus* and *Bacillus subtilis* are shown in Table 4.

Table 4: Antimicrobial activity of compounds

Sl.No.	Compound	Inhibition zone diameter in mm											
		<i>Staphylococcus aureus</i>						<i>Bacillus subtilis</i>					
		25 µg	50 µg	100 µg	200 µg	400 µg	800 µg	25 µg	50 µg	100 µg	200 µg	400 µg	800 µg
1	C ₁₃ H ₁₁ NOS Ligand	0	0	0	2	8	12	0	0	0	4	8	9
2	Co(C ₁₃ H ₁₁ NOS)H ₂ O	0	1	2	4	8	12	0	1	3	5	9	15
3	Ni(C ₁₃ H ₁₁ NOS)H ₂ O	0	0	1	5	9	14	0	1	2	4	8	14
4	Cu(C ₁₃ H ₁₁ NOS)H ₂ O	0	0	1	4	10	15	0	1	3	5	9	15
5	Zn(C ₁₃ H ₁₁ NOS)H ₂ O	0	0	0	1	4	8	0	0	1	2	5	7
6	Cd(C ₁₃ H ₁₁ NOS)H ₂ O	0	0	0	1	3	7	0	0	2	3	5	7
7	Mn(C ₁₃ H ₁₁ NOS)H ₂ O	0	0	0	2	3	6	0	0	1	2	4	6
8	Gentamycin	13	18	21	25	27	34	8	10	15	19	22	25



It has been concluded that the antibacterial activity that was possessed by Schiff base metal complexes is more when compared to the antibacterial activity of the free ligand against the bacterial strains such as *Staphylococcus aureus* and *Bacillus subtilis*. And the same antibacterial activity that was possessed by the ligand and the metal complexes was found to be very much less than the activity that was shown by the standard antibiotic gentamycin³⁵⁻³⁶.

3.9 - Antifungal Activity:

The antifungal activity of synthesized complexes was studied by agar diffusion method against the fungi *Aspergillus Niger* and *Cladosporium Oxysporum*. Standard antibiotic used is Flucanazole and the solvent used is DMSO. The antifungal activity of the Schiff base ligand and its metal complexes against the fungi *Aspergillus Niger* and *Cladosporium Oxysporum* was shown in table 5.

Sl.No.	Compound	Inhibition zone diameter in mm									
		<i>Aspergillus Niger</i>					<i>Cladosporium Oxysporum</i>				
		0.12 5 mg	0.25 mg	0.5 mg	1.0 mg	2 mg	0.12 5 mg	0.25 mg	0.5 mg	1.0 mg	2 mg
1	C ₁₃ H ₁₁ NOS	0	0	1	2	4	0	1	2	4	5
2	Co(C ₁₃ H ₁₁ NOS)(H ₂ O)	0	1	2	4	8	0	2	3	4	9
3	Ni(C ₁₃ H ₁₁ NOS)(H ₂ O)	0	1	3	4	7	0	1	2	4	8
4	Cu(C ₁₃ H ₁₁ NOS)(H ₂ O)	0	0	2	5	7	0	1	3	5	7
5	Zn(C ₁₃ H ₁₁ NOS)(H ₂ O)	0	1	3	5	6	0	2	3	6	6
6	Cd(C ₁₃ H ₁₁ NOS)(H ₂ O)	0	0	2	3	4	0	1	2	4	6
7	Mn(C ₁₃ H ₁₁ NOS)(H ₂ O)	0	1	2	3	5	0	2	3	5	6
8	Flucanazole	0	3	5	7	12	0	4	5	6	10

Table 5: Antifungal activity of compounds

All the metal complexes were found to exhibit better antifungal activity when compared to antifungal activity of the free Ligand against the fungi *Aspergillus Niger* and *Cladosporium Oxysporum*. And the antifungal activity shown by ligand and its metal complexes was slightly less when compared to activity of shown by standard antibiotic Flucanazole³⁷⁻³⁸.

4. CONCLUSION

In the above mentioned work we have synthesized new metal complexes of Schiff base. The synthesized metal complexes were characterized by various methods. On the basis of the spectral data and other facts we propose the square planar geometry for Cu(II) and Ni(II) complexes and tetrahedral geometry for Co(II), Mn(II), Zn(II) and Cd(II) complexes. The electrical conductivity data of the synthesized complexes shows that these complexes behave as insulators at room temperature.

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AUTHOR DETAILS:



ANIL KUMAR M R
RESEARCH SCHOLAR
B.I.E.T, DAVANGERE
KARNATAKA – 577004



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