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SYNTHESIS AND CHARACTERIZATION OF SCHIFF BASE DERIVED FROM VANILLIN WITH VARIOUS AMINE AND FORMATION OF Co(II), Cu(II) and Ni(II) METAL COMPLEXES WITH DERIVED SCHIFF BASE.

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ABTRACT

The Schiff base ligand synthesized from the Condensation of vanillin with primary amines. These Schiff base were used to prepare metal complexes of Co(II), Cu(II) & Ni(II). All synthesized compound where characterized by TLC, M.P., Solubility, Spectroscopic techniques show that Schiff base behave as a bidentet Ligand and metal complexes purposed to octahedral geometry where metal to ligand ratio is (1:2). The synthesized ligand and metal complexes are screened for antibacterial activity against E-coli, B-Subtilus, Pseudomonas and antifungal activity against Aspergillus niger.

KEYWORDS: Vanillin, Amine, Synthesis, Characterization, metal complex etc.

INTRODUCTION

The Schiff base and their metal complexes have special important in the field of co-ordination chemistry. Schiff base compound (-CH=N-) are usually formed by the condensation of primary amines with chelating ligands having, N,S and O as a donor atoms are attracting attention due to their versatile nature of metal binding mode. Transition metal have a strong tendency to form Co-ordination complex due to small size high charge densities and vacant (n-1) d orbital. The synthesis and application of Schiff base and their Coordination compounds have been highly considered in inorganic and bio inorganic field similar to some

of the biological system.

EXPERIMENTAL

All the chemicals and solvent used were A.R. grade and were used without further purification.

Preparation of 3methoxy 4{[(2nitropheny)imino]methyl}phenol (L1)

0.05 mole 2-nitroanilline in hot ethanol was added to an ethanolic solution of vanillin (0.05 mole). The resulting mixture was stirred under reflux for 2hours on a water bath product is obtained. It was allowed to cool and separated, filtered, washed with ethanol and dried in natural condition for 4-6 hours.

Preparation of 3methoxy 4{[(3nitropheny) imino] methyl}phenol (L2)

0.05 mole 3-nitroanilline in hot ethanol was added to an ethanolic solution of vanillin (0.05 mole). The resulting mixture was stirred under reflux for 2hours on a water bath product is obtained. It was allowed to cool and separated, filtered, washed with ethanol and dried in natural condition for 4-6 hours.

Preparation of 3methoxy 4{[4nitropheny)imino]methyl}phenol (L3)

0.05 mole 4-nitroanilline in hot ethanol was added to an ethanolic solution of vanillin (0.05 mole). The resulting mixture was stirred under reflux for 2hours on a water bath product is obtained. It was allowed to cool and separated, filtered, washed with ethanol and dried in natural condition for 4-6 hours.

SYNTHESIS OF METAL COMPLEXES

Hot solution of metal chloride (0.01mole) in an ethanol-water mixture (1:1) added to the hot solution of Schiff base (L2) (2mole) in same Solvent. The resulting mixture was stirred under heating for 1 hour till the product is obtained. Then product was cooled by filtration washed with (1:1) (ethanol : water) and then dried in natural condition for 4-6 hours.

Antimicrobial Activity

The in vitro antimicrobial properties of the Schiff base ligand and metal Complexes were assayed with the following bacteria, E-coli, B-subtilus, Pseudomonas and fungal species Aspergillus niger. Agar diffusion assay was carried out to evaluate the antimicrobial activity of some synthesized compound. The plates were incubated at 37^0C for 24 hours during which activity was pridenced by the presence of a zone of inhibtion surrounding the well and

antibacterial and antifungal activity was expressed as mean of diameter of inhibi- tion zones (mm) product by the synthesised compound when compared to controls.

RESULT AND DISCUSSION

The analytical data along with some physical properties of ligand L^1, L^2, L^3 and Schiff base (L2) on interaction with metal Co(II), Cu(II) & Ni(II) synthesized metal Complexes ligand L^1, L^2,L^3 having some physical properties and their analytical data is summarized in table No.1. All synthesized Complexes are air stable and high yield products.

Physical characteristics and analytical data for the Schiff base ligand and metal (II) complexes.

Compound	Molecular Formula	Colour	M.P.	Yield
L_1	$C_{14}H_{12}N_20_4$	Yellow	90^0C.	81.37%
L_2	$C_{14}H_{12}N_20_4$	Yellow	119^0C.	91.72%
L_3	$C_{14}H_{12}N_20_4$	Yellow	180^0C.	88.27%
Co-L_2	$Co(C_{14}H_{12}N_20_4)_2$	Brown	$>300^0$C.	80.32%
Cu-L_2	$Cu(C_{14}H_{12}N_20_4)_2$	Brown	$>300^0$C.	75.29%
Ni-L^2	$Ni(C_{14}H_{12}N_20_4)_2$	Brown	$>300^0$C.	70.04%

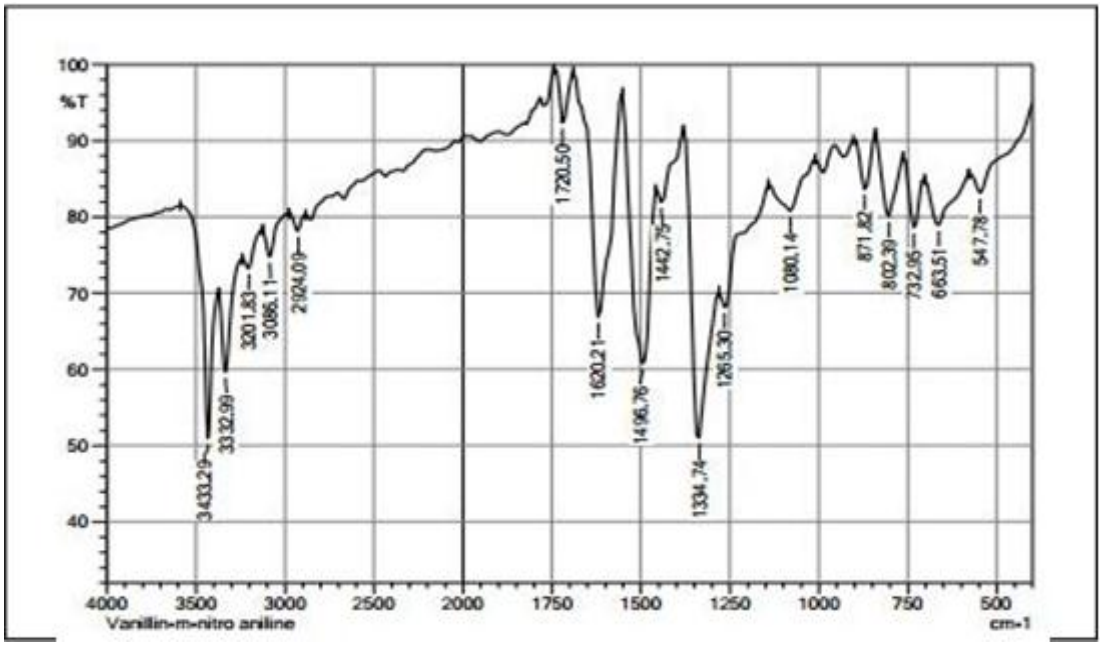
IR SPECTROSCOPY

The selected vibrational frequencies for the Schiff base ligand and its metal complexes are presented in Table No.2. The IR Spectra of the Complexes are compared with that of the ligand to determine the change that might have taken during the complexation.

Relevant infrared frequencies (cm-1) of the Schiff base ligands and their metal (II) complexes.

Compound	V (O H)	V (C=N)	V (-NO2)	V (-OCH3)	(Cu-N)
L1	3479.58 cm^{-1}	1589.34cm^{-1}	1427.32cm^{-1}	1157.29cm^{-1}	-
L2	3433.29 cm^{-1}	1620.21cm^{-1}	1427.32cm^{-1}	1265.30cm^{-1}	-
L3	3441.01 cm^{-1}	1666.50cm^{-1}	1427.32cm^{-1}	1296.16cm^{-1}	-
Cu-L2	3229.14 cm^{-1}	1647.21cm^{-1}	1427.32cm^{-1}	1207.44cm^{-1}	493.78cm^{-1}

The bands 3433.29 cm-1, 3086.11cm-1, 1620.21cm-1, 1496.76cm-1, 1265.30cm-1 assignable to V (OH) (Intra moleculor Hydrogen bonding), V(-C=H) (alkene), V(HC=N) (ozomethine), V(-NO_2) (nitrogroup) streching modes respectively of ligand (i.e. L_3).



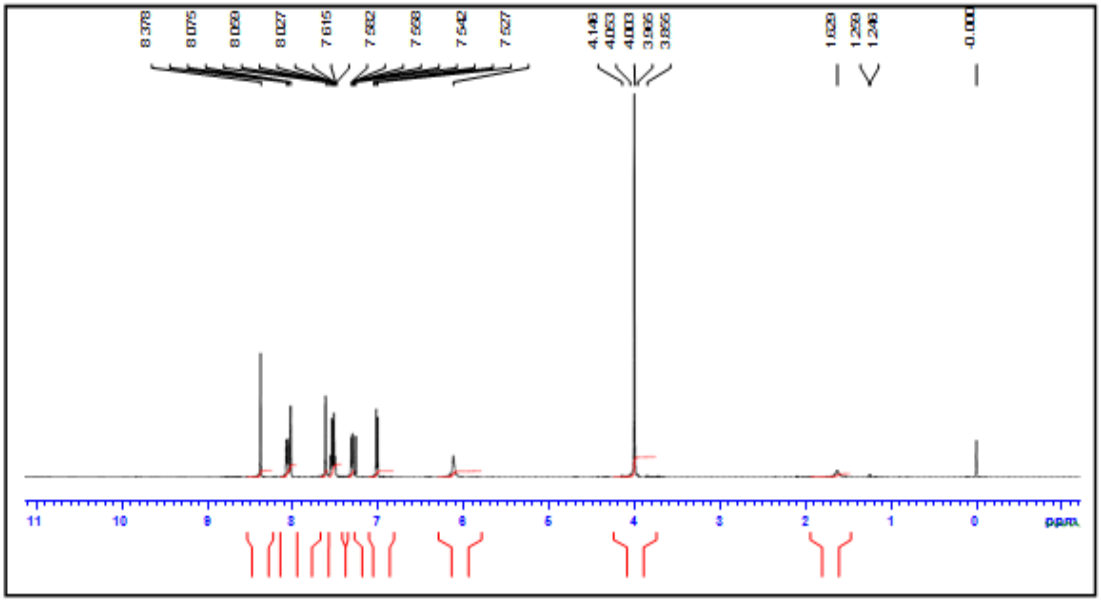
The IR Spectra of Cu-II metal complexes show a band 3329.14cm^{-1} region suggesting the presence of co-ordinating water in metal complex. The band 493.78 cm^{-1} give V of Cu-N streching mode. By comparising between IR frequencies of ligand and metal we observed that V-NO_2 increases while V- OCH_3 decreases.

1H-NMR

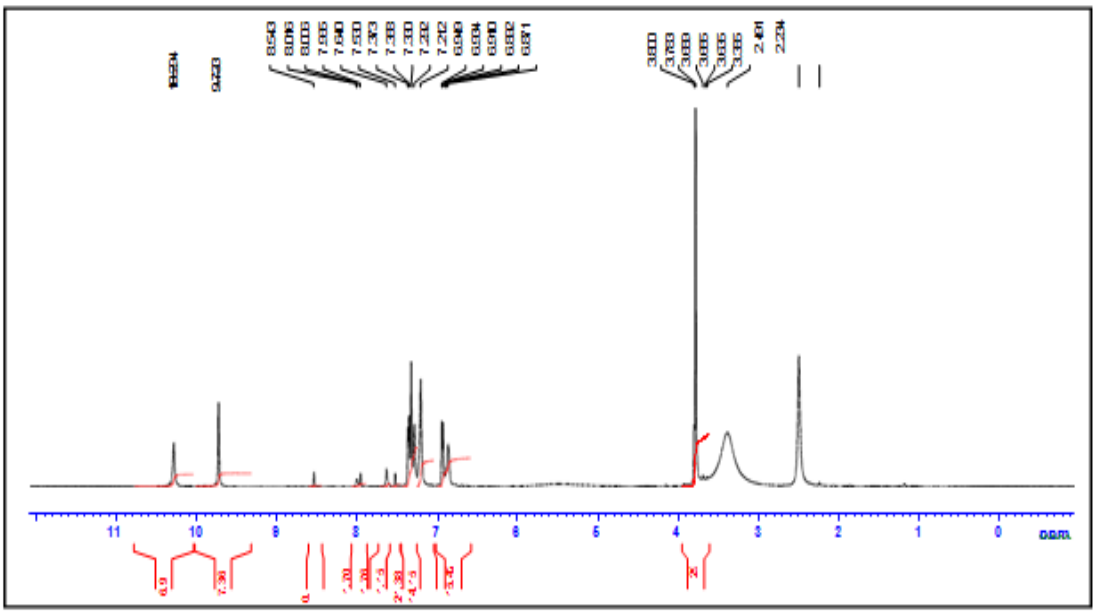
The 1H-NMR Spectra of ligand and its metal complexes are give some signals which are summerised in Table No. 3.

Compound	H from azomethine group	H from aromatic group	H from Methoxy group	H from water molecule
L_2	8.378 ppm	7.6 - 7.0 ppm	3.855 ppm	-
Co-L_2	10.2 - 9.7 ppm	8 - 6.8 ppm	3.8 - 3.3 ppm	2.4 - 2.2 ppm
Cu-L_2	10.2 - 9.7 ppm	8 - 6.9 ppm	3.8 - 3.4 ppm	2.502 ppm
Ni-L_2	10.2 - 9.7 ppm	7.9 - 6.8 ppm	3.8 - 3.4 ppm	2.510 ppm

The 1H-NMR Spectra of ligand at room temperature shows the following signals 8.378 ppm (1H, hydrogen bonded to azomethine carbon) 7.61 - 7.0 ppm (H from aromatic carbon).



1H-NMR Spectra of Cu-metal complexes shows signals at 10.2ppm for (H from azomethine) group 8 -6.8 ppm for (H from aromatic region) 3.8 -3.4 ppm for (H from OCH3 group) and 2.4 - 2.5 ppm for (H from water molecule).



Antimicrobial activity

Antibacterial and antifungal activity of ligand and metal complexes were tested in vitro against bacterial and fungal by paper disc plate method. The compounds were tested at conc. 1% in ethanol. It is found that the inhibition by metal chelate is higher than that of ligand and Cu has more antimicrobial activity than Co & Ni metal complexes which are summarized in (table 4 & 5).

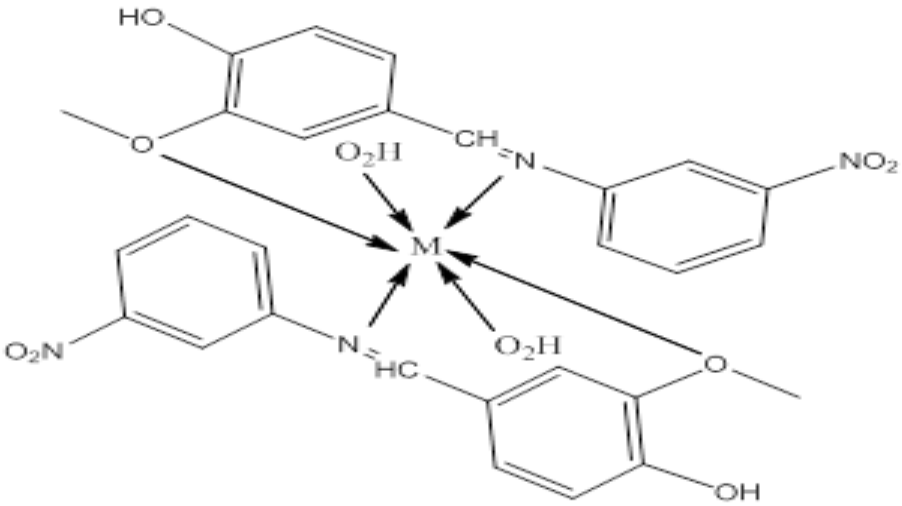
Antibacterial activity of ligand and their metal complexes.

Test Compound	Diameter in inhibition zone (mm)		
	E-coli	B-Subtlis	Pseudomonas
L_2	08 mm	03 mm	08 mm
Co-L_2	12 mm	08 mm	10 mm
Cu-L_2	17 mm	10 mm	15 mm
Ni-L_2	10 mm	05 mm	11 mm

Antifungal activity of ligand and their metal complexes.

Test Compound	Diameter in inhibition zone (mm)
	Aspergilus Niger
L2	10 mm
Co-L2	13 mm
Cu-L2	19 mm
Ni-L2	15 mm

STRUCTURE OF COMPLEX



GENERAL CONCLUSION

From above discussion we have propose octahedral geometry for CoII, CuII & NiII complexes on the basis of the physico-chemical and spectral data discussed above. One can assume that the ligand behave as bidentate ligand Co-ordinating Via -OCH3 group and imino nitrogen as illustrate in fig. 1. The complexes are biologically active and show enhanced antimicrobial activities compared to free ligand.

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