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Research Article

SYNTHESIS AND CHARACTERIZATION OF SCHIFF BASE ANILINE WITH 5-BROMO -2-HYDROXYL BENZALDEHYDE AND THEIR METAL COMPLEXES

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ABSTRACT

A series of transition metal complexes of Cu (II), Co (II), Mn (II), Fe (II), Ni (II) and V (II) were prepared from bidentate schiff base. The Schiff base ligand synthesized from the condensation of 5-Bromo 2-Hydroxy Benzaldehyde (NEELIMA, D, KULKARNI; P, K, BHATTACHARYA. Can. J. Chem. 1987, 65, 348) and Aniline in an alcohol medium. These metal complexes were characterized on the basis of their analytical data like IR, NMR. The ligand and their metal complexes were screened antibacterial activity against various bacteria like Escherichia coli, B. subtilis. The result indicated that the complexes exhibited good antibacterial activities.

INTRODUCTION

Schiff bases derived from an amino compound and carbonyl compound that coordinate to metal ions via azomethine nitrogen atom (1). Azomethine (C=N) has been reported to possess remarkable antibacterial, biological activities (2-3) but copper complex with ligand have wide applications in food industry, dye industry, analytical chemistry, catalysis, fungicidal, agrochemical, anti-inflammable activity, antiradical activities and biological activities (4). The complexes of Schiff bases with metal ions are very important in the development of coordination chemistry field. These complexes are also known to have anticorrosion properties and biological activities such as antimicrobial (5-21). Importance of Schiff bases and their metal complexes are important as biochemical (22), electrochemical (23), biological antibacterial activities (24-28), redox catalysts (29, 30). The role of chlorophyll, hemoglobin, carbonic anhydrase, vitamin B_{12}, xanthine oxides and haemocyanin, illustrates the intimate linkage between inorganic chemistry and biology (31-37). The coordinate chemistry field, a lot of Schiff bases operates as ligands (38-40). Some of the Schiff bases complexes combinations with a metal ion are used as insecticides, fungicides, herbicides (41).

MATERIALS AND METHODS

Synthesis of Bromo 2-Hydroxy Benz Aldehyde

A solution of bromine 0.02 moles in glacial acetic acid is added slowly to a stirred solution of 0.02 mole Salicylaldehyde in glacial acetic acid. During the addition, the reaction mixture is kept in a cold-water bath. After the addition is over the reaction mixture will be yellow colored due to excess of bromine. (42) The reaction mixture is allowed to stand for 15-20 minutes at room temperature and poured into crushed ice. Water is added to remove colour. Separated product is filtered, washed with cold water and crystallized from diluted alcohol. yield is approximately 25 gm. and M.P. 106-107^{0}C. (Figure 1**)**

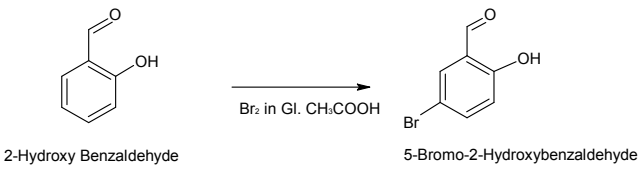

Figure 1 Synthesis of 5-Bromo 2-Hydroxybenzaldehyde

Synthesis of ligand

The ligand was prepared by a modification of the reported methods. The Schiff base ligand has been synthesized by

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refluxing a mixture of 0.01 mole of 5-Bromo 2-Hydroxy Benzaldehyde and 0.01 mole of Aniline in 50 ml super dry ethanol refluxed for about 3hr. Schiff base thus formed was cooled to room temperature and collected by filtration, followed by recrystallization in ethanol and dried.(Figure 2**)**

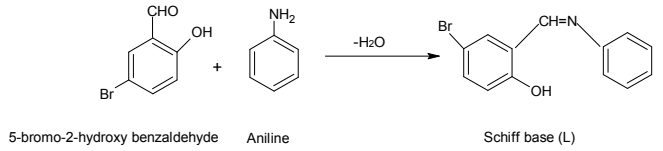

Figure 2 Synthesis of ligand

Synthesis of metal complexes

Cu, Ni, Mn, V, Fe, & Co was used as metals for the derivation of metal complexes. In the process, 0.02 mole Schiff base was dissolved in 25 ml ethanol. To this solution, 0.01 mole of the metal salt solution was added. This mixture was allowed to stir and refluxed for 3 hours. The product was cooled and washed with cool water. Obtained precipitates were then dried for further application. **(**Figure 3)

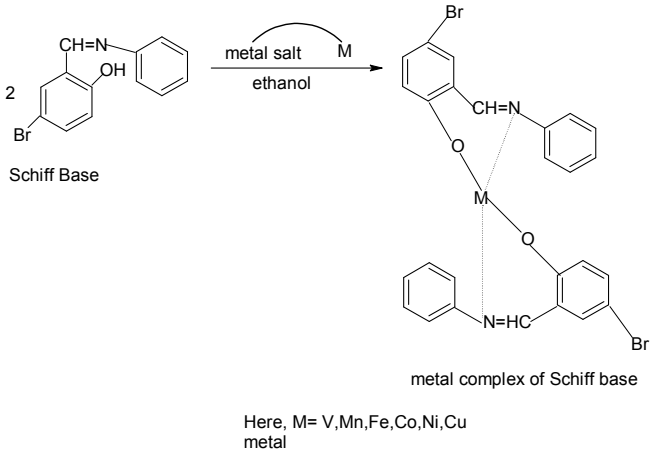

Figure 3 Synthesis of metal complexes

RESULTS AND DISCUSSION

Schiff bases of 5-Bromo 2-Hydroxy Benzaldehyde and Aniline and its complexes have a variety of applications including biological, clinical and analytical. The coordinating possibility of 5-Bromo 2-Hydroxy Benzaldehyde has been improved by condensing with a variety of carbonyl compounds. An attempt has been made to synthesize Schiff bases from 5-Bromo- 2-Hydroxy Benzaldehyde with Aniline. Physical characteristics, TLC data, IR data, and NMR data, of metal complexes are given in (Table 1 and 2).

Table 1 Physical data of Schiff base and their metal complexes

Compound	Molecular weight (g)	M.P.(°C)	Color	Yield % (w/w)	RF Value
Schiff base(L)	275.9 gm.	163	Yellow	98.08	0.57
L-Cu metal	612.52 gm.	253	Blue	81.02	0.65
L-Co metal	576.99 gm.	179	light green	78.33	0.73
L-Fe metal	604.82 gm.	201	Brown	92.56	0.54
L-V metal	599.92 gm.	186	pale yellow	30.54	0.69
L-Ni metal	607.67 gm.	212	Yellow	62.12	0.71
L-Mn metal	603.91 gm.	191	pale yellow	71.00	0.78

Table 2 FTIR Spectral Data

	IR FUNCTIONAL GROUP DATA				
Compound	ν (C=N)	ν (N-H)	ν (M-O)	ν (M-N)	ν(C-Br)
Schiff base(L)	1614	3438	-	-	1122
L-Cu metal	1610	3448	526	456	1168
L-Co metal	1616	3249	558	452	1116
L-Fe metal	1616	3437	543	459	1179
L-V metal	1614	3438	527	439	1170
L-Ni metal	1600	3439	557	457	1167
L-Mn metal	1616	3427	526	438	1169

The analytical data of complexes revel 2:1 molar ratio (ligand: metal) and corresponds well with the general formula ML2 (where M=Cu(II),Ni(II),Co(II),Mn(II), V(II) and Fe(II).(Table 1)

FTIR spectrum analysis

The IR spectra of the complexes are compared with that of the ligand to determine the changes that might have taken place during the complexation. The bands at 3438, 1587, 1614, 1181, and 1275 cm-1 assignable to ν OH (intermolecular hydrogen bonded), C=C(aromatic), ν C=N (azomethine), ν AR-Br and ν C-O (phenolic) stretching modes respectively(43-45).The absence of a weak broadband in the 3200-3400 cm-1 region, in the spectra of the metal complexes, suggests DE protonation of the intermolecular hydrogen bonded OH group on complexation and subsequent coordination of phenolic oxygen to the metal ion. This is further supported by a downward shift in υ C-O (phenolic) with respect to free ligand [46]. On complexion, the ν (C=N) (47) band is shifted to lower wave number with respect to free ligand, denoting that the nitrogen of azomethine group is coordinated to the metal ion. The ν C-N band is shifted to lower wave number with respect to free ligand, The IR spectra of metal chelates showed new bands in between the 500-800 and 400-500 cm-1 regions which can be assigned to ν M-O and M-N vibrations (48) respectively The IR spectra of Cu (II), Co (II) Mn (II) Fe (II) Ni (II) and V (II) show a strong band in the 3050-3600 cm-1 region, suggesting the presence of coordinated water in these metal complexes. (Table 2)

NMR spectrum analysis

^{1}H NMR spectrum

The ^{1}H NMR spectrum of the Schiff base ligand was recorded in Acetone-d6. In the ^{1}H NMR spectra of Schiff base ligand, a peak appeared at 5.35 ppm was assigned to the proton of the phenolic group and a singlet peak appeared at 8.87 ppm was assigned to protons of azomethine group. (Figure 4**)**

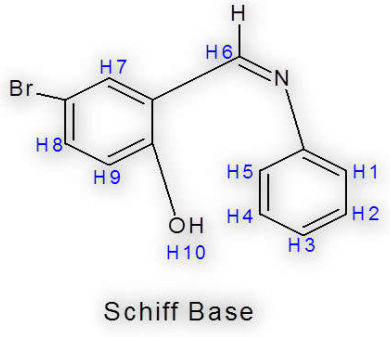

Figure 4. ^{1}H NMR spectrum

^{13}CNMR spectra

The ^{13}C NMR spectrum of the Schiff base ligand was recorded in Acetone-d6. In the ^{13}C-NMR spectra of the metal complexes, the signal appeared at 159.32 ppm (C7) was assigned to azomethine carbon atoms (C=N). A signal at 161.87 (C13) was assigned to phenolic carbon. Phenyl rings of ligand showed following signals 147.84(C1); 121.38(C2-C6), 119.01(C4), 129.45(C3-C5), 121.16(C8), 133.95(C9), 109.87(C10), 135.04(C11), 121.16(C12) ppm.. (Figure 5**)**

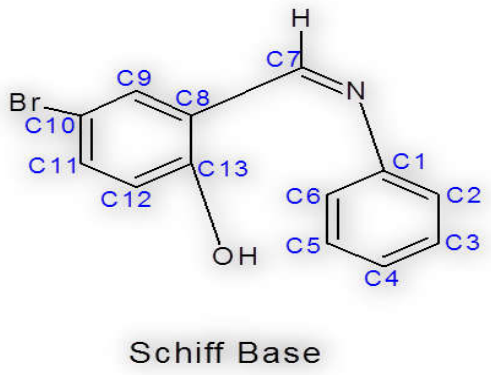

Figure 5 ^{13}CNMR spectra

Biological Applications

Antimicrobial Activity

The procedure used for screening is as reported. (49-51). The synthesized compounds dissolved in DMSO were examined using Well Diffusion method. In this method, all glass wares used were sterilized in a hot air oven. The gram-positive bacteria-B. megaterium and gram-negative-Escherichia coli were used. The viable bacterial cells were evenly swabbed onto the Nutrient agar plates, were soaked in the different test samples (concentrations 4, 15 & 30 mg/ml), drained and using sterilized forceps placed in the agar plates. The plates were then incubated for 48 hours at 37^{0}C. After the incubation period, the zones of inhibition were measured in mm. The ligands and the metal complexes show very good antimicrobial properties against Escherichia coli and B. megatherium. (Figure 6), (Table 3)

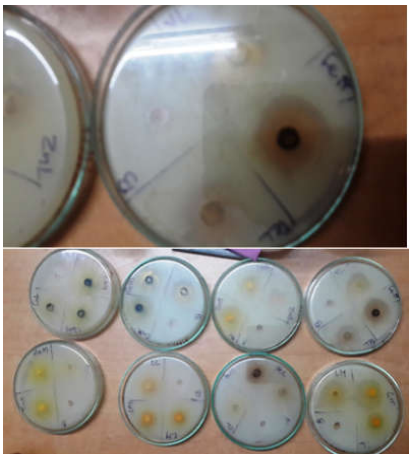

Figure 6 It was concluded on examination of zone that metal complexes of Schiff bases are more efficient antimicrobial agent than its native form. However, the antimicrobial potentialities of metal complex are highly dependent in the metal ion used for formation of group coordinated and metal complex

Table 3 Zone of inhibition

Compound	Concentration (mg/ml)	E. Coli (mm)	B. megaterim (mm)
Schiff base(L)	Low: 4	5.0	-
	Medium: 15	5.5	-
	High: 30	7.0	8.0
L-Cu metal	Low: 4	7.2	9.0
	Medium: 15	11.7	12
	High: 30	11.7	13.5
L-Co metal	Low: 4	11.8	9.5
	Medium: 15	12.4	12.0
	High: 30	14.3	14
L-Fe metal	Low: 4	12.5	11.0
	Medium: 15	13.5	12.5
	High: 30	16.0	15.0
L-V metal	Low: 4	12.0	9.5
	Medium: 15	14.0	11.0
	High: 30	18.0	14.0
L-Ni metal	Low: 4	5.0	8.0
	Medium: 15	8.88	9.5
	High: 30	11.0	12.0
L- Mn metal	Low: 4	10.0	-
	Medium: 15	12.0	8.0
	High: 30	16.0	12.0
Ampicillin	High: -	18	15

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