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### SYNTHESIS, AND SPECTROPIC CHARACTERIZATION OF SCHIFF BASE COMPLEXES OF CINNAMALDEHYDE HYDRAZONES

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#### ABSTRACT

The synthesis and structural features of some Bidentate Schiff base complexes are reported. The ligand was derived by the condensation of Cinnamaldehyde hydrazone with different benzaldehyde . The co-ordination of the metal salts based on this ligand with transition metal ions viz,  $Cu^{+2}$ ,  $Ni^{+2}$ ,  $Mn^{+2}$ ,  $Co^{+2}$  and  $Fe^{+2}$  were prepared and studied for their metal : ligand ratio (M/L), it has been found that ligand forms complexes in 1:2 ratio. The metal complexes were characterized by elemental analysis, molar conductance, magnetic momentum measurements, melting point , IR, spectral data and uv-visible spectral data . An octahedral geometry was proposed for all metal complexes. The conductivity data show that all these complexes are non electrolytes. The ligands and their metal complexes were screened for Gram positive bacteria *Bacillus Substlis* and Gram negative bacteria *E.Coli*. **Keywords**: Hydrazones, Cinnamaldehyde , Antibacterial activity, Antifungal activity, Transition metal complexes and IR studies .

#### INTRODUCTION

In recent years, the study of co-ordination Bidentate Schiff base complexes has been made much progress [1,2]. Coordination Schiff base mostly derived from bichelating ligands in which metal ions and chelating agents arrayed alternatively. Most of bichelating ligands are derived from well known chelating agents like salicylaldehyde, 8-hydroxy guinoline, hydroxyl acetophenone, salicylic acid etc [3-4]. The joining segment of these similar ligands are mainly -N=N-,-SO<sub>2</sub>,- CH<sub>2</sub>,-O- or -CH<sub>2</sub>-S-CH<sub>2</sub>- etc [3-5]. Structural studies have shown that Schiff base exhibit different types of bonding depending on the nature of the metal ion and the reaction condition [6,7,9]. The interest in such complexes continues increasing due to the possibility of their use as models to explain some intricate reaction in biological systems such as- antiviral, antimalarials, antiulcer, anticancer etc ,[8,10,]. Hydrazones have been demonstrated to possess, among other, antimicrobial, anticonvulsant, analgesic, antiinflammatory, antiplatelet antitubercular, and antitumoral activities. For example, Epilepsy is a common neurological disorder and a collective term given to a group of syndromes that involve spontaneous, intermittent, abnormal electrical activity in the brain. The pharmacotherapy of epilepsy has been achieved during the last decade . Furthermore, new antiepileptic drugs have been introduced into although for the last twenty years clinical practice, the maximal electroshock (MES) test and the subcutaneous (scPTZ) test are the most widely used animal models of epilepsy to pentylenetetrazole characterize the anticonvulsant activity. The biological results revealed that in general, the

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acetylhydrazones provided good protection against convulsions while the oxamoylhydrazones were significantly less active[11]. Fifteen new hydrazones of (2 – oxobenzoxazoline – 3-yl)acetohydrazide were synthesized and their antiepileptic activity was tested in scPTZ test. The 4 – fluoro derivative was found to be more active than the others [12]. 4 – Aminobutyric acid (GABA) is the principal inhibitory neurotransmitter in the mammalian brain . GABA hydrazones were designed and synthesized and evaluated for their anticonvulsant properties in different animal models of epilepsy such as MES , scPTZ , subcutaneous strychnine (scSTY) and intraperitonal picrotoxin (ipPIC) induced seizure tests . Some of the compounds were effective in these models . A new hydrazone ligand, HL, was prepared by the reaction of 7-chloro-4-hydrazinoquinoline with o-hydroxy benzaldehyde by Mustafa M El-Behry and his co-workers. The ligand behaves as monoprotic bidentate. The ligand reacted with Cu (II), Ni (II), Co (II), Fe (III), and UO<sub>2</sub> (II) ions to yield mononuclear complexes. The HL and metal complexes were tested against one stain gram positive bacteria (*Staphylococcus aureus*), gram negative bacteria (*Escherichia coli*), and fungi (*Candida albicans*). The tested compounds exhibited higher antibacterial activities [13].

Therefore the synthesis and characterization of Hydrazones and their metal complexes are of tremendous importance. The literature survey reveals that the metal complexes of hydrazones demonstrated more potent bactericidal and fungicidal properties than their corresponding ligands. In this paper, we are reporting the synthesis, characterization and biological activities of Schiff base (derived from Cinnamaldehyde hydrazone with different benzaldehyde) against various microorganisms and their coordination behavior with bivalent metals like  $Cu^{+2}$ , Ni<sup>+2</sup>, Co<sup>+2</sup>, Mn<sup>+2</sup> and Fe<sup>+2</sup>.

#### MATERIALS AND METHOD

Cinnamaldehyde was obtained from Sigma – Aldrich Chemical Ltd , and used without further purification . The metal salts i.e.  $CuSO_{4.}5H_2O$ , NiSO\_{4.}6H\_2O, CoSO\_{4.}7H\_2O, MnSO\_{4.5H\_2O}, and FeSO\_4.6H\_2O and the benzaldehyde compounds were obtained from BDH Chemicals Ltd , England . The metal content of the complexes were estimated gravimetrically, copper metal was estimated by ammonium thiocynate gravimetrically. Nickel, cobalt and manganese were estimated by gravimetrically using dimethylglyoxime, ammonium thiocynate and iron with triethanolamine respectively [14]. Conductivity measurements were made on1x  $10^{-3}$  M solutions of the complexes in ethanol using Equiptronics model no Eq-660A conductivity meter, provides with a dip type cell having cell constant 1.0. Magnetic susceptibility of complexes was measured at room temperature on a faraday balance using Hg [Co (SCN) 4] as a calibrant. Electronic spectra were recorded using digital spectrophotometer in ethanol. The IR spectra of ligands and their complexes were recorded as KBr pellets in the region 4000-400cm-1 on FT IR spectrophotometer Shimadzu 8201. The Melting points were determined in an Electrothermal 9200.

#### **Preparation of the Ligands**

The ligand was synthesized in two steps. The first step is the synthesis of Cinnamaldehyde monohydrazone according to the reporting method [14], followed by the Cinnamaldehyde hydrazones (II) in the second step from monohydrazone.

#### Cinnamaldehyde – (2 – bromo, 3 – methoxy benzylidene) hydrazide

Cinnamaldehyde monohydrazone (2gm, 1m mol) (69% yield, mp 1200c, yellow crystal) was dissolved in absolute ethanol (10ml). To this solution 2 - bromo - 3- methoxy benzaldehyde (1.42ml) were added for preparing L1 [ Cinnamaldehyde - (2 - bromo, 3 - methoxy benzylidene) hydrazide]. The reaction mixture was refluxed for 4hrs. After cooling, the formed yellow precipitate was collected, filtered and finally washed with absolute ethanol (10ml) and purified by recrystalization from ethanol (80% yield mp 1400c).

#### Cinnamaldehyde – (2 – chloro, 3 – methoxy benzylidene) hydrazide

Cinnamaldehyde monohydrazone (2gm, 1m mol) (77% yield, mp 1200c, yellow crystal) was dissolved in absolute ethanol (10ml). To this solution 2 - chloro - 3- methoxy benzaldehyd (1.40ml) were added for preparing L1 [Cinnamaldehyde – (2 - chloro, 3 - methoxy benzylidene) hydrazide]. The reaction mixture was refluxed for 4hrs. After cooling, the formed yellow precipitate was collected, filtered and finally washed with absolute ethanol (10ml) and purified by recrystalization from ethanol (88% yield mp 1200c). (Scheme 1.)



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 $R_1 = L \ 1 = Br$ ,  $R_2 = L \ 2 = Cl$ Scheme 1. Schematic representation of Ligands formation of compounds

### **Preparation of metal complexes :**

# Complex : Cinnamaldehyde – (2 – bromo, 3 – methoxy benzylidene) – hydrazide copper (II) A

Copper(II) sulphate pentahydrate (1 mmol) was dissolved in absolute ethanol (10 ml). To this, (3mmol) Cinnamaldehyde – (2 - bromo, 3 - methoxy benzylidene) – hydrazide (L1) in ethanol (10 ml) was added. The mixture was stirred magnetically at room temperature. The precipitated complexes were filtered, washed with ethanol and dried. All other complexes were prepared in similar manner i.e. Complex 2, 3, 4, 5,

# Complex : Cinnamaldehyde – (2 – chloro, 3 – methoxy benzylidene) – hydrazide copper (II) B

Complex (B) was synthesized in a similar manner to that used for complex (A), reacting a mixture

of Cinnamaldehyde – (2 – chloro, 3 – methoxy benzylidene) – hydrazide (L2) (3 mmol) and copper(II) sulphate pentahydrate (1 mmol) in ethanol (10 ml). The analytical data of ligands and their metal complexes are given in table-I. (Scheme 2)

 $MSO_4.nH_2O + 2LH \rightarrow ML_2 + H_2SO_4 + nH_2O$ (n = 5 ,6 , or 7) Journal of Physical Sciences and Innovation



Scheme 2. Schematic representation of metal complex formation of compounds

Comp. No	Emperical formulae	M.P °c	Mag. Mom.	Yield %	Color	Mol. cond. Am	Elemental Analysis Calculated ( found )			Form. Wat	
						Cm <sup>2</sup> mol- 1ohm-1	%C	%N	%Н	M%	5
							59.09	8.11	4.92	-	
L1	$C_{17}H_{17}N_2OBr$	126	-	65	Yellow	11	(59.05)	(8.11)	(5.41)	-	345.24
L2	C <sub>17</sub> H <sub>17</sub> N <sub>2</sub> OCI	130	-	77	Dark yellow	12	67.83 (67.85)	9.31 (9.30)	5.65 (5.61)	-	300.77
					Dark brown		69.99	6.87	3.92	15.57	
L1 – Cu <sup>+2</sup>	C <sub>17</sub> H <sub>16</sub> N <sub>2</sub> OBrCu	165	1.83	68		23	(69.98)	(6.85)	(3.90)	(15.53)	407.77
_					Brown		50.03	6.95	3.97	14.64	
L1 – Ni <sup>+2</sup>	C <sub>17</sub> H <sub>16</sub> N <sub>2</sub> OBrNi	145	2.73	64	Black	22	(50.01)	(6.90)	(3.95)	(14.66)	402.94
							50.60	6.95	3.97	14.63	
L1 – Co <sup>+2</sup>	C <sub>17</sub> H <sub>16</sub> N <sub>2</sub> OBrCo	189	4.10	79	Black	26	(50.64)	(6.97)	(3.96)	(14.62)	403.16
L1 – Mn <sup>+2</sup>	C <sub>17</sub> H <sub>16</sub> N <sub>2</sub> OBrMn	188	5.75	79	Dark Black	35	51.11 (51.21)	7.01 (7.02)	4.01 (3.99)	13.78 (13.76)	399.17
L1 – Fe <sup>+2</sup>	C17H16N2OBrFe	166	5.21	88	Brownish	28	50.99 (50.98)	7.00 (7.03)	4.00 (4.01)	14.00 (13.97)	400.08
	-1, 10 2		-		Dark		56.15	7.71	4.40	17.48	
L2 – Cu <sup>+2</sup>	C <sub>17</sub> H <sub>16</sub> N <sub>2</sub> OClCu	143	1.82	80	Brown	21	(56.17)	(7.66)	(4.45)	(17.49)	363.30
L2 – Ni <sup>+2</sup>	C <sub>17</sub> H <sub>16</sub> N <sub>2</sub> OCINi	156	2.68	75	Brownish Black	26	56.91 (56.89)	7.81 (7.84)	4.46 (4.55)	16.46 (16.56)	358.47
							57.51	7.89	4.51	15.51	
L2 – Mn <sup>+2</sup>	C <sub>17</sub> H <sub>16</sub> N <sub>2</sub> OCIMn	187	5.77	64	Black	33	(57.50)	(7.84)	(4.50)	(15.49)	354.70
L2 – Co <sup>+2</sup>	C <sub>17</sub> H <sub>16</sub> N <sub>2</sub> OCICo	199	4.01	87	Black	24	56.87 (56.89)	7.81 (7.84)	4.46 (4.49)	16.45 (16.43)	358.69
L2 – Fe <sup>+2</sup>	C <sub>17</sub> H <sub>16</sub> N <sub>2</sub> OCIFe	143	5.23	62	Brownish	20	57.37 (57.39)	7.87 (7.88)	4.50 (4.53)	15.75 (15.71)	355.61

### Characterization of the complexes:

The given formulae (Table-1) are based on analytical data.

# Table 1. The physical and analytical data of the ligands and complexes

# **RESULTS AND DISCUSSION**

### Magnetic Measurements:

The magnetic moment values are useful in the evaluation of results provided by the other techniques of the structural investigation [14]. The magnetic moment data are presented in table- 1. The magnetic moment of the Cu(II) complex (1.82-1.83 B.M.) which is very close to the spin-only value (1.73 B.M.) expected for the one unpaired electron which offers the possibility of an octahedral geometry [15-16]. The magnetic moment value for Fe(II) complex is 5.21-5.23 B.M. at room temperature and this value is characteristic of high spin octahedral geometry for this complex [17]. The magnetic moment values for Co(II) complexes are 4.01-4.10 B.M. corresponding to three unpaired electrons which suggests an octahedral geometry [15-16]. The magnetic moment of the nickel complex at room temperature was observed 2.68-2.73 B.M . These values are in tune with a high spin configuration and show the presence of an octahedral environment around the Ni(II) ion in the complex [13,17]. The Mn(II) complex show magnetic moments is 5.75-5.77 B.M . at

room temperature corresponding to five unpaired electrons which suggest octahedral geometry [18].

## Table 2. IR and UV Spectral Bands of Ligands and Its Metal Complex

### I.R SPECTRA:

The infrared spectra of the complexes have been studied to characterize their structures. The IR spectra of the complexes register v(C-O) at about 1200 -1240cm<sup>-1</sup> [19-20]. The IR spectra of Schiff base shows a sharp band near 1610 cm<sup>-1</sup> which may be due to azomethine linkage and shows lowering in frequency in metal complex indicating the coordination of metal ion through azomethine linkage [21-23]. The sharp bend in the range 750-780 cm<sup>-1</sup> and 1190-1235 cm<sup>-1</sup> are due to aromatic v(C-H) and v(N-N) respectively. The frequencies in the range 1145-1165cm<sup>-1</sup> attributed to v(C-N) stretching [22]. Conclusive evidence of the bonding is also shown by the observation that new bonds in the spectra of the metal complexes appears at 440 - 512 cm<sup>-1</sup> these are assigned to v(M-N) stretching vibrations and are not observed in the spectra of the ligand [21-23]. The presence of sharp band corresponding to the remaining hydroxyl group at 3400 cm<sup>-1</sup> but it is obscured by the presence of water molecules bands. This was appeared for the most complexes and a very broad band at about 3100-3500 cm<sup>-1</sup> region, which was associated with coordinated or solvent water molecules [22].

S/n.	Compounds	IR spectral in KBr pellets, cm <sup>-1</sup>				U.V spectral nm		
		∨(C=N)	∨(N-N)	∨(M-N)	∨(C-O-C)	(-C=C-)	(-C=N)	
1.	L1	1613, 1603	1210	-	1200	250	300	
2.	L2	1606, 1604	1200	-	1207	245	310	
3.	L1 – Cu <sup>+2</sup>	1580, 1595	1199	440	1224	240	365	
4.	L1 – Ni <sup>+2</sup>	1570, 1567	1201	447	1220	250	350	
5.	L1 – Co <sup>+2</sup>	1572, 1568	1222	433	1224	240	358	
6.	L1 – Mn <sup>+2</sup>	1565, 1562	1245	462	1225	250	340	
7.	L1 – Fe <sup>+2</sup>	1596 1590	1190	512	1236	220	320	
8.	L2 – Cu <sup>+2</sup>	1594, 1588	1211	450	1214	245	372	
9.	L2 – Ni <sup>+2</sup>	1586, 1583	1232	461	1217	245	366	
10.	L2 – Mn <sup>+2</sup>	1571, 1567	1233	470	1229	240	344	
11.	L2 – Co <sup>+2</sup>	1575, 1566	1245	475	1225	245	350	
12.	L2 – Fe <sup>+2</sup>	1598 1593	1236	512	1242	210	360	

Electronic spectra: Electronic spectral data of the ligands and their transition metal complexes were recorded in ethanol solution .The absorption bands for the complexes will help to give an idea of their structure [24]. The electronic spectrum of the Fe(II) complex exhibit a band at 11,200 cm<sup>-1</sup>, assigned to the  $5T_2q \rightarrow 5Eq$  transition. The electronic spectra of Mn(II) complexes show the absorption bands in the range 16970–19540, 22280–24390 and 26109–27624 cm<sup>-1</sup>. These absorption bands may be assigned to the  $6A_1g \rightarrow 4A_1g$  (4G),  $6A_1g \rightarrow 4A_2g$  (4G), and  $6A_1g \rightarrow 4Eq$ ,  $4A_1g$  (4G) transitions, respectively. These bands suggest that the complexes possess an octahedral geometry [21]. Electronic spectrum of Co(II) complex exhibits absorption bands at 12,920, 16,260, and 23,450 cm<sup>-1</sup>, which may be assigned to  $4T_1(F) \rightarrow 4T_2(F)$ ,  $4T_1(F) \rightarrow 4A_2(F)$  and  $4T_1(F) \rightarrow 4T_1(P)$  transitions respectively which are in support of octahedral arrangement of binding centers around the metal ion [35]. The electronic spectra of the Ni(II) complex showed d-d bands in the region 10115 and 26280-26410 cm<sup>-1</sup>  $3A_2g(F) \rightarrow 3T_2g(F)$  and  $3A_2g(F) \rightarrow 3T_2g(P)$  respectively, consistent with their well-defined octahedral configuration [25,26,27]. Bands at the regions 675 nm (14815 cm<sup>-1</sup>) and 535 nm (18690 cm<sup>-1</sup>) for Cu(II) complex were assigned to the  $2B_1q \rightarrow 2B_2q$  and  $2B_2q \rightarrow 2Eq$  transitions respectively, found for octahedral Cu(II) complexes [22].

**Molar Conductivity:** Molar conductivity of ligand and metal complexes were recorded using 1x10 M solution of ethanol on Equiptronics Conductivity Meter EQ660A.The molar conductance measurements of the complex in ethanol is 9.1 W-1cm2mol<sup>-1</sup>, indicating their non-ionic nature. Table 1

**Antimicrobial activity:** The antimicrobial activity of the complexes against Gram positive bacteria *Bacillus Substlis* and Gram negative bacteria *E.Coli* have been done using the ["Agar diffusion method"]. The diameter of inhibition zone of the various compounds is recorded in table III. All the compounds have significant antibacterial activity at 1.0 x 104µgml-1 against bacteria. The compounds [M L1], [where M = Ni(II), Cu(II), Co(II), Fe(II), and Mn(II)] are more active as compared to the compounds [M L2], [where M = Ni(II), Cu(II), Cu(II), Cu(II), Co(II), Fe(II), and Mn(II)] against bacteria. All the compounds screened are more active against gram positive bacteria *Bacillus Substlis* than gram negative bacteria *E.Coli*.[23].

Table III . Antimicrobial activity

s/n	Compounds	Diameter of inhibition zone (mm) (% inhibition)				
		Bacillus Substlis	E.Coli			
1.	L1	11	12			
2.	L2	12	13			

3.	L1 – Cu <sup>+2</sup>	25	10
4.	L1 – Ni <sup>+2</sup>	23	16
5.	L1 – Co <sup>+2</sup>	25	20
6.	L1 – Mn <sup>+2</sup>	18	18
7.	L1 – Fe <sup>+2</sup>	22	18
8.	L2 – Cu <sup>+2</sup>	20	17
9.	L2 – Ni <sup>+2</sup>	21	21
10.	$L2 - Mn^{+2}$	27	12
11.	L2 – Co <sup>+2</sup>	28	11
12.	L2 – Fe <sup>+2</sup>	25	10

#### CONCLUSION

This study reports the successful synthesis of the title compounds in good yields and from the elemental analysis, molar conductivity, UV-Visible, magnetic, and IR spectral data it was possible to determine the type of coordination of the ligands in their metal complexes. In all the complexes, only one part of the ligand is coordinated to the metal ion resulting mononuclear complexes. The ligand coordinates through the nitrogen atoms (N-N) of the hydrazone moiety and oxygen atom from the methoxide fragment. The ligand acts as a monobasic, tridentate (FIGURE-1)

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