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Synthesis, Spectral, Magnetic, Thermal and Antibacterial Studies of Cu(II) and Ni(II) Complexes of Schiff base Derived from 5-chloro-2-hydroxybenzophenone and p-Phenylenediamine

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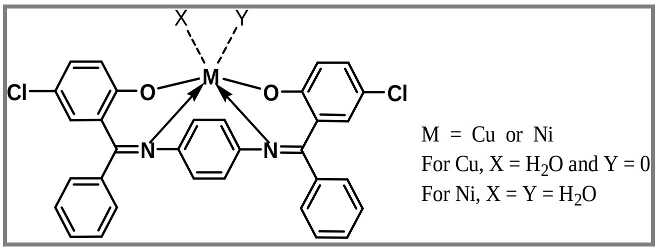
ABSTRACT

The complexes of Cu(II) and Ni(II) ions have been synthesized with Schiff base ligand derived by the condensation of 5-chloro-2-hydcoxybenzophonone and p-phenylenediamine in acidic medium. The synthesized compounds were investigated using different physicochemical techniques such as elemental analysis (C, H, N and Cl), FT-IR, ^{1}H NMR, diffuse reflectance spectroscopy, magnetic moment measurements and thermogravimetric analysis. The synthesized complexes have been assigned square planar geometries. The antibacterial activity of the Schiff base and its metal complexes have been also been tested, showing that the complexes are more active than the free Schiff base.

Graphical Abstract



M = Cu or Ni
For Cu, X = H_2O and Y = 0
For Ni, X = Y = H_2O

Suggested geometrical structure of Metal complex of CHBPPD

Keywords: Schiff base complex, Transition metal complexes, spectral and Thermal studies, antibacterial activities.

INTRODUCTION

Schiff bases are compounds containing imine or azomethine group (>C=N-) synthesized by the condensation of an amino compound with carbonyl compounds under specific conditions [1]. The Schiff bases are able to coordinate with many different metal ions and stabilize them in various oxidation states [2, 3]. Schiff bases and their metal complexes are very important class of compounds as they exhibit a broad range of biological activities such as anti-bacterial, antifungal, anti-tumour, anti-inflammatory, antiviral, antipyretic properties and are extensively used for industrial purposes [4-6]. Many Schiff base complexes exhibit excellent catalytic activities and have been widely used in catalytic reactions [7] and also used as models for biological systems [8]. In last few decades, considerable attention has been given to the chemistry of the metal complexes of Schiff bases containing nitrogen and oxygen donors because of their stability, biological activity and applications in many fields [9-13].

In the present work synthesis and characterization of Schiff base ligand derived by the condensation of 5-chloro-2-hydroxybenzophonone with p-phenylenediamine and their metal complexes with Cu(II) and Ni(II) ions has been reported. The characterization of Schiff base and its metal complexes carried out by different physicochemical and spectral techniques. The synthesized compounds were tested for antibacterial activities.

MATERIALS AND METHODS

All chemicals used in this work were ‘AR’ Grade. The solvents such as, DMF (dimethyl formamide), dimethylsulfoxide (DMSO), ethanol etc. were further purified by standard procedures, if required.

Preparation of Schiff base: The Schiff base ligand 2,2’-(1,4-phenylenebis(azan-1-yl-1-ylidene)) bis (phenylmethan-1-yl-1-ylidene) bis (4-chlorophenol) named as CHBPPD was synthesized by mixing an ethanolic solution of 5-chloro-2-hydroxybenzophonone to an ethanolic solution of p-phenylenediamine in 2:1 molar ratio. The reaction mixture was then refluxed for six hours on a water bath; the yellow precipitate formed was filtered, washed thoroughly with ethanol, recrystallized with petroleum ether and dried in desiccators over anhydrous calcium chloride. The yield was about 80% and the melting point of product was 290-295°C.

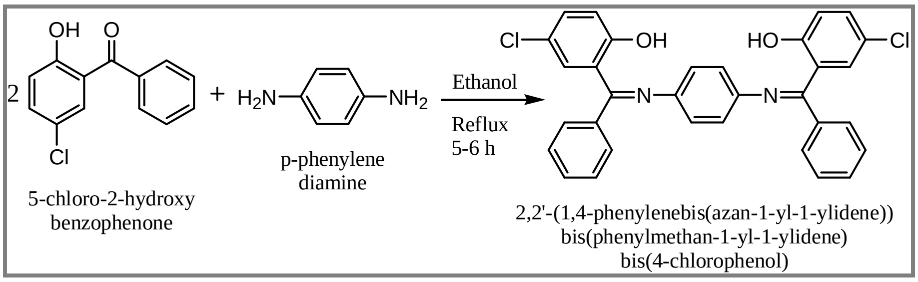


Figure 1. Preparation of Schiff base ligand CHBPPD.

Preparation of metal complexes: The metal complexes of Cu(II) and Ni(II) were prepared by mixing a solution of CHBPPD in DMF with an ethanolic solution of $M(CH_3COO)_2.nH_2O$ in 1:1 molar ratio. The reaction mixtures were refluxed for 5-6 h on a sand bath. The solid products obtained on cooling the reaction mixture were filtered and washed several times with DMF to remove unreacted ligand and metal acetates. Finally the complexes were dried in desiccators over anhydrous calcium chloride. The isolated complexes are coloured solids and quite stable in air. All complexes are insoluble in water and some common organic solvents, but considerably soluble in DMSO.

RESULTS AND DISCUSSION

The CHBPPD has been characterized by elemental analysis, ^{1}HNMR and FT-IR spectra while the metal complexes were studied by elemental analysis, FT-IR, magnetic studies, diffuse reflectance spectra and TGA. All the synthesized compounds were tested for antibacterial activities against two bacterial strains *E. coli* and *B. subtilis*.

Physicochemical analysis: The physicochemical analyses of the CHBPPD and its complexes are listed in table 1. The obtained elemental analyses data were in a good an agreement with the formation of complex of 1:1 [M:L] ratio.

Table 1. Physicochemical analysis CHBPPD and its complexes

Schiff Base/ Complex	Colour	M.P. (°C)	M.W.	Elemental Analysis % Found (Calculated)				
				M %	%C	%H	%N	%Cl
CHBPPD	Yellow	290-295	537.44	--	71.50 (71.51)	4.09 (4.13)	5.23 (5.21)	13.22 (13.19)
Cu-CHBPPD	Brown	- -	616.98	10.53 (10.30)	62.21 (62.29)	3.68 (3.59)	4.68 (4.54)	11.57 (11.49)
Ni-CHBPPD	Yellow	- -	630.14	9.37 (9.31)	60.85 (60.99)	3.78 (3.84)	4.51 (4.45)	11.32 (11.25)

^{1}H NMR spectra of CHBPPD (300 MHz, $CdCl_3$, δ in ppm): The ^{1}H NMR spectra of the ligand has been recorded at CDRI, Lucknow. The spectrum of the CHBPPD indicated that different non-equivalent proton resonates at different values of applied field [14-18]. The aromatic proton multiplet was observed between δ 7.69 – 7.71 ppm (10H) and the singlet at δ 6.65 ppm (4H). A broad singlet observed at δ 5.35 ppm (2H) is due to –OH groups. The other aromatic proton doublet observed at δ 6.96 (2H) and δ 7.35 (2H) while singlet at δ 7.74 (2H).

FT-IR spectra (KBr, cm^{-1}): The FT-IR spectrum of ligand CHBPPD compared with its metal complexes in order to identify the nature of coordinating group [19]. The spectra of ligand shows broad band at 3439 cm^{-1}, which may be assigned as ν(O-H) i.e. intramolecular hydrogen bonded O-H group. This band is absent in the spectra of complexes indicating coordination of deprotonated phenolic oxygen atom [20, 21]. A strong sharp band at 1626 cm^{-1} in the spectrum of ligand is assigned to ν(C=N) stretching. This band is shifted to lower frequencies in all complexes in the range of 1604-1612 cm^{-1} upon complexation, which indicates the coordination of azomethine nitrogen with the metal ion [22, 23]. The FT-IR data of compounds is depicted in table 2.

Table 2. FT-IR spectra of CHBPPD and its complexes

Schiff Base/ Complex	IR Spectra (cm^{-1})						
	O–H	C=N	C–O	C–Cl	M–O	M–N	(H_2O)
CHBPPD	3439	1626	1222	700	--	--	--
Cu-CHBPPD	--	1604	1236	696	570	424	3357
Ni-CHBPPD	--	1612	1228	698	540	436	3385

The medium frequency band of phenolic ν(C-O) stretching of free ligand at 1222 cm^{-1} is shifted to higher frequency by 6-14 cm^{-1} in all the complexes further supporting the involvement of phenolic oxygen in coordination [24]. New bands appeared in the spectra of complexes at 540-570 cm^{-1} and 424-436 cm^{-1} have been tentatively assigned to υ(M-O) and υ(M-N) stretching frequency, respectively which confirmed the coordination of ligand through deprotonated phenolic oxygen and azomethine nitrogen atoms [25, 26]. The IR bands in the spectra of complexes at 3357 cm^{-1} and 3385 cm^{-1} are due to υ(H_2O) stretching suggesting hydrated complexes [27].

Magnetic moments and diffuse reflectance spectra: The magnetic moments and assignments of diffuse reflectance spectra of CHBPPD and its metal complexes are depicted in table 3. The electronic absorption spectra of the ligand show two bands at 282 and 395 nm which may be due to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions, respectively. The spectra of Cu(II) complexes display three bands each at 673, 580 and 370 nm which can be assigned to $^2B_{1g} \rightarrow {}^2A_{1g}$, $^2B_{1g} \rightarrow {}^2E_g$ and charge transfer transition, respectively suggesting square planar geometry for the complex [28]. The magnetic moment value 1.83 B.M. further supports the square planar geometry [29, 30].

The absorption spectrum of Ni(II) complexes show three bands at 758, 511 and 407 nm which are assigned to spin allowed $^1A_{1g}\rightarrow{}^1E_g$; $^1A_{1g}\rightarrow{}^1A_{2g}$ and $^1A_{1g}\rightarrow{}^1B_{1g}$ transitions, respectively indicating the square planar geometry [31]. The magnetic moment measurement shows diamagnetic nature of the complex which confirms the square planar environment around the Ni(II) ion [32].

Table 3. Magnetic moments and Assignments of diffuse reflectance spectra of CHBPPD and its complexes

Schiff Base/ Complex	μ_{eff} B.M.	Absorption band		Assignments
		(nm)	(cm^{-1})	
CHBPPD	--	395	25316	$n\rightarrow\pi^*$
		282	35460	$\pi\rightarrow\pi^*$
Cu-CHBPPD	1.83	673	14858	$^2B_{1g}\rightarrow{}^2A_{1g}$
		580	17241	$^2B_{1g}\rightarrow{}^2E_g$
		370	27027	C.T.
Ni-CHBPPD	diamagnetic	758	13192	$^1A_{1g}\rightarrow{}^1E_g$
		511	19569	$^1A_{1g}\rightarrow{}^1A_{2g}$
		407	24570	$^1A_{1g}\rightarrow{}^1B_{1g}$

Thermogravimetric analysis: TGA curves of Cu(II) and Ni(II) complexes show initial weight loss occurring within 100-120°C is interpreted as loss of lattice water molecules. The weight loss in Cu(II) complex corresponding to loss of one lattice water molecule while that of the Ni(II) complex corresponding to two lattice water molecules [33, 34].

Table 4. TGA data of BDBPC and Its Transition Metal Complexes

Compounds	Molecular Weigh	Degradation Temperature (°C)	% loss	Assignment
CHBPPD	537.44	>300	--	Parts of Ligand
[Cu(CHBPPD)].H_2O	616.98	105-115	2.92	1 H_2O
		>300	--	Parts of Ligand
[Ni(CHBPPD)].2H_2O	630.14	110-120	5.73	2 H_2O
		>300	--	Parts of Ligand

There is no further weight loss up to 300°C indicating absence of co-ordinated water molecules in complexes. The decomposition of complexes starts >300°C and undergo completion at about 600°C. Above this temperature was a horizontal curve and final product of decomposition at this temperature region corresponds to metal oxides [35]. The TGA data of CHBPPD and ITS metal complexes is given in table 4.

APPLICATION

Antibacterial activity: The ligand CHBPPD and its Cu(II) and Ni(II) complexes were tested against two species of bacteria *Escherichia coli* (Gram negative) and *Bacillus subtilis* (Gram Positive) to access their antimicrobial activity by Mueller-Hinton agar plates method [36]. The results are given in table 5 and table 6.

Table 5. Antimicrobial results for *E. Coli*

S. No.	Compound	Zone of Inhibition (mm)			Average (mm)
		1	2	3	
1	CHBPPD	12	15	18	15.0
2	[Cu(CHBPPD)].H_2O	26	25	25	25.3
3	[Ni(CHBPPD)].2H_2O	19	20	20	19.7

Table 6. Antimicrobial results for *B. Subtilis*

S.No.	Compound	Zone of Inhibition (mm)			Average (mm)
		1	2	3	
1	CHBPPD	07	09	15	10.3
2	[Cu(CHBPPD)].H_2O	26	25	26	25.7
3	[Ni(CHBPPD)].$2H_2O$	21	22	21	21.3

It is observed that the Cu(II) and Ni(II) complex showed higher activity against *E. coli* bacteria, while the ligand showed lower activity. In case of *B. subtilis* strain also the Cu(II) and Ni(II) complex showed higher activity (Figure 2). The antimicrobial data reveal that the Cu(II) and Ni(II) complexes are more bioactive than the free ligand [37, 38].

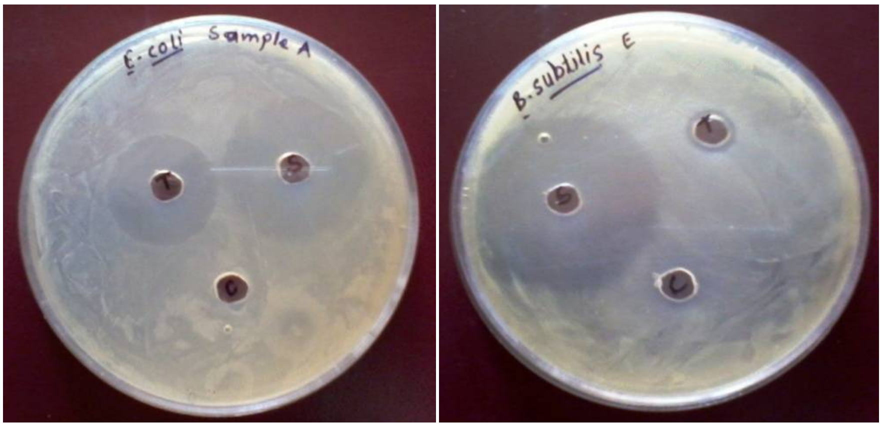
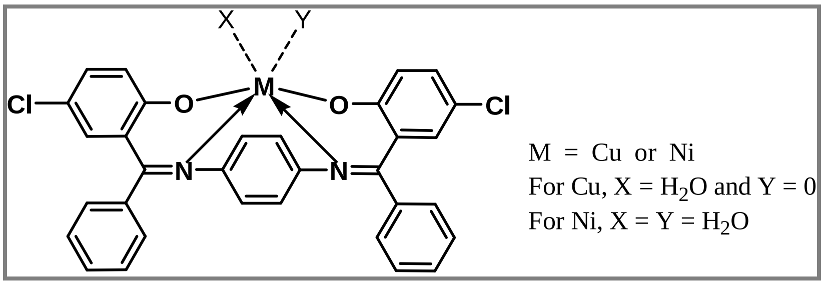


Figure 2. Photographs of Antibacterial Activity against *E. coli* and *B. Subtilis.*

CONCLUSION

The Schiff base ligand CHBPPD and its two transition metal complexes have been synthesized and characterized by analytical, spectral, thermal and magnetic measurements. The results of these investigations suggested that CHBPPD is a tetradentate ligand coordinating through azomethine nitrogen and phenolic oxygen atoms. The metal complexes with Cu(II) and Ni(II) ions are forming in 1:1 (M:L) ratio. On the basis of elemental, spectral, thermal and magnetic moment studies, four-coordinated square planar geometry has been assigned for Cu(II) and Ni(II) complexes. Biological activity of the Schiff base ligand and its metal complexes were studied and results shows that the activity of the metal complexes is higher than the free Schiff base ligand. Antimicrobial study showed there is enhancement of activity against given strains of bacteria after coordination. The suggested geometrical structure of complexes is shown below:



M = Cu or Ni
For Cu, X = H_2O and Y = 0
For Ni, X = Y = H_2O

Figure 3. Suggested geometrical structure of Metal complex of CHBPPD

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