

# Synthesis, Characterization And Biological Significance Of Novel Schiff-Base 3d Metal Complexes

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## Abstract

The chemistry of metal complexes with tailor made Schiff base ligands and their applications, have aroused considerable interest mainly because of preparative accessibility and structural variability. Complexes of Co(II), Ni(II) and Cu(II) with the Schiff base ligands derived from 5-bromo-2-hydroxybenzaldehyde with 2-amino-4-chlorophenol (BHAP) and 2-aminothiazole (BHAT) have been synthesized and characterized by various physicochemical methods viz. micro-analytical data, UV-VIS, FT-IR, ESR Spectroscopy molar conductance and magnetic susceptibility measurements. All the complexes are colored and stable towards air and moisture. The Schiff base and metal complexes show a good activity against the bacteria; *B. subtilis*, *E. coli* and *S. aureus* and fungi *A. niger*, *A. flavus* and *C. albicans*. The antimicrobial results also indicate that the metal complexes are better antimicrobial agents as compared to the Schiff bases.

**Keywords:** Synthesis, FAB-mass, IR, UV-Vis, ESR, Biological activities.

## INTRODUCTION

Transition Metal complexes of Schiff bases have played a central role in the development of coordination chemistry and have many applications in various fields.[ 1-8] Metal complexes of Schiff bases containing nitrogen and sulphur as donor ligand atoms are of interest in different field like industry, pharmacy and biology.[9-14] Preparation of the new ligands is perhaps the most important step in the development of metal complexes, which exhibit unique desired properties and novel reactivity. Such ligands and their metal complexes have a variety of applications in biology and industry due to their role in catalysis with inorganic & organic synthesis.[15-20] In present paper, the synthesis and characterization of the complexes of Co(II), Ni(II) and Cu(II) involving the Schiff base ligands derived from 5-bromo-2-hydroxybenzaldehyde with 2-amino-4-chlorophenol (BHAP) and 2-aminothiazole (BHAT) have been reported.

## Experimental

All the used chemicals and solvents were of A.R. grade. All the reagents used for the preparation of the Schiff bases were obtained from Sigma Aldrich. Electronic spectra in ethanol were recorded on Perkin Elmer Lambda-2B-spectrophotometer. Molar conductance measurements were conducted using  $10^{-3}$  M solutions of the complexes in ethanol on Elico-CM 82 Conductivity Bridge at room temperature. Magnetic susceptibility measurements were carried out on a Gouy balance at room temperature using  $\text{CuSO}_4$  as the calibrant. FT-IR spectra were recorded in KBr medium on a Perkin Elmer RX1 spectrophotometer in wave number region  $4000-400\text{ cm}^{-1}$ .

## Synthesis of Schiff Bases

BHAP and BHAT Schiff bases were synthesized by the condensation of equimolar ratio of 5-bromo-2-hydroxybenzaldehyde with 2-amino-4-chlorophenol (BHAP) and 2-aminothiazole (BHAT) dissolved in ethanol. The resulting reaction mixture was stirred well, refluxed for 5-6 h and then allowed to cool overnight. The coloured solid precipitate of Schiff base obtained was filtered, washed with cold ethanol and dried in air at room temperature and finally stored under reduced pressure in a  $\text{CaCl}_2$  desiccators. The purity of synthesized compounds was checked by TLC using silica gel G (Yield: BHAP = 83.40% and BHAT = 81.62%).

## Synthesis of Metal Complexes

The metal complexes were prepared by the mixing of (50 ml) ethanol solution of  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ / $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ / $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$  with the (50 ml) ethanol solution of Schiff bases (BHAP/BHAT) in 1:2 (metal:ligand) ratio. The resulting mixture was refluxed on water bath for 6-8 h. A coloured product appeared on standing and cooling the above solution. The precipitated complexes was filtered washed with ether and recrystallized with ethanol several times and dried under the reduced pressure over anhydrous  $\text{CaCl}_2$  in a desiccator. It was further dried in electric oven at  $60-70\text{ }^\circ\text{C}$ .

## RESULTS AND DISCUSSION

All the metal complexes are coloured, solid and stable towards air and moisture at room temperature. They decompose on heating at high temperature, more or less soluble in common organic solvents. Analytical data of the compounds, together with their physical properties are consistent with proposed molecular formula are given in Table 1. All the metal chelates have 1:1 or 1:2 (metal:ligand) stoichiometry. The observed molar conductance value of the complexes in methanol at room temperature is consistent with the non-electrolytic nature of the complexes.

**Table 1.** Analytical and physical data of the compounds

Compounds (Molecular Formula)	Colour/ (Mol. Wt)	Yield (%)	Elemental Analysis Found/(Calcd.)%				
			C	H	N	M	$\Lambda_M$
BHAP (C <sub>13</sub> H <sub>8</sub> BrClNO <sub>2</sub> )	Yellow (295.8)	83.40	47.5 (47.8)	2.5 (2.8)	4.1 (4.3)	-	-
[Co(C <sub>13</sub> H <sub>8</sub> BrClNO <sub>2</sub> ) (H <sub>2</sub> O) <sub>3</sub> ]	Purple (437.57)	72.60	35.2 (35.7)	2.7 (3.0)	3.1 (3.2)	13.0 (13.5)	18.9
[Ni(C <sub>13</sub> H <sub>8</sub> BrClNO <sub>2</sub> ) (H <sub>2</sub> O) <sub>3</sub> ]	Coffee brown (437.30)	73.00	35.3 (35.7)	2.5 (3.0)	3.2 (3.2)	13.1 (13.4)	14.5
[Cu(C <sub>13</sub> H <sub>8</sub> BrClNO <sub>2</sub> ) (H <sub>2</sub> O)]	Brownish yellow (406.12)	72.70	38.2 (38.5)	2.0 (2.2)	3.1 (3.5)	15.3 (15.7)	9.6
BHAT (C <sub>10</sub> H <sub>07</sub> BrN <sub>2</sub> OS)	Orange (283.16)	78.00	51.8 (51.9)	3.0 (3.1)	8.0 (8.1)	-	-
[Co(C <sub>10</sub> H <sub>07</sub> BrN <sub>2</sub> OS) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]	Brown (854.5)	69.90	45.6 (45.8)	2.9 (3.0)	7.2 (7.1)	7.0 (7.4)	11.2
[Ni(C <sub>10</sub> H <sub>07</sub> BrN <sub>2</sub> OS) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]	Yellowish Brown (804.03)	69.50	45.5 (45.8)	2.9 (3.0)	6.9 (7.1)	7.2 (7.4)	12.5
[Cu(C <sub>10</sub> H <sub>07</sub> BrN <sub>2</sub> OS) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]	Yellow Turmeric (746.80)	79.70	45.2 (45.4)	3.0 (3.1)	6.7 (7.1)	7.3 (8.1)	9.4

$$*\Lambda_M = (\text{ohm}^{-1} \text{cm}^2 \text{mol}^{-1})$$

### IR spectra:

The data of the IR spectra of Schiff base ligand and its metal complexes are listed in Table 2. The IR spectra of the complexes were compared with those of the free ligand in order to determine the involvement of coordination sites in chelation. Characteristic peaks in the spectra of the ligand and complexes were considered and compared.

The IR spectrum of synthesized (BHAP) Schiff base shows absorption band at 1623 cm<sup>-1</sup> due to (>C=N) azomethine group. It has shifted towards lower frequency (1599-1602 cm<sup>-1</sup>) in the complexes suggesting coordination through azomethine nitrogen. The ligand spectrum shows bands at 3361 and 1361 cm<sup>-1</sup> due to (the stretching and deformation vibration) phenolic-OH, which are absent in the spectra of complexes. An intense ligand band at about 1263 cm<sup>-1</sup> (phenolic C-O) shift to higher frequency (1277-1280 cm<sup>-1</sup>) in the complexes. This suggests deprotonation of the phenolic OH group after its chelation with metal ion. The appearance of broad band around 3331-3361 cm<sup>-1</sup> in the spectra of complexes suggests the presence of water molecules. A band of weak intensity observed at 806-820 cm<sup>-1</sup>  $\nu(\text{OH})_{\text{Rocking}}$ , suggests presence of coordinated water in the complexes. The IR spectra of the complexes show some new bands at 521-529 and 479-491 cm<sup>-1</sup> which are probably due to  $\nu(\text{M-O})$  and  $\nu(\text{M-N})$  modes, respectively.

The IR spectra of the BHAT Schiff base shows band at about 1620 cm<sup>-1</sup> due to  $\nu(\text{C=N})$  (azomethine) group. The complexes display lower side shift by about 15-20 cm<sup>-1</sup> suggesting coordination through azomethine nitrogen. The ligand spectrum shows bands at 3311 and 1358 cm<sup>-1</sup> due to the stretching and deformation of phenolic -OH group, respectively. These are absent in the spectra of complexes. A medium intensity ligand band at about 1254 cm<sup>-1</sup> (phenolic C-O) has shifted higher and appeared at 1277-1287 cm<sup>-1</sup> in the metal complexes. This indicates its involvement in chelation. The appearance of broad bands around 3361-3393 cm<sup>-1</sup> in the spectra of complexes may be due to water molecules. A band of medium intensity at 800-823 cm<sup>-1</sup> ( $\nu(\text{OH})_{\text{rocking}}$ ), suggest the presence of coordinated water in all complexes. The new bands of weak intensity at 536-541 and 481-491 cm<sup>-1</sup> in the metal complexes have tentatively been assigned to  $\nu(\text{M-O})$  and  $\nu(\text{M-N})$  modes respectively. The  $\nu(\text{C=N})$  at 1560 cm<sup>-1</sup> and  $\nu(\text{C-S-C})$  at 755 cm<sup>-1</sup> of the thiazole ring remain unchanged suggested that thiazole group does participate in coordination.

**Table 2.** IR bands of Schiff base ligands and their complexes

Compound	$\nu(\text{C=N})$	$\nu(\text{O-H})$	$\nu(\text{C-O})$	$\nu(\text{C=N thiazole ring})$	$\nu(\text{C-S-C})$	$\nu(\text{H}_2\text{O})$	$\nu(\text{M-O})$	$\nu(\text{M-N})$
BHAP	1623	3376	1263	-	-	-	-	-
[Co(BHAP) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]	1600	-	1277	-	-	3340, 815	526	483
[Ni(BHAP) <sub>2</sub> ].2H <sub>2</sub> O	1599	-	1280	-	-	3361, 820	521	479
[Cu(BHAP) <sub>2</sub> ].2H <sub>2</sub> O	1602	-	1280	-	-	3331, 806	529	491
BHAT	1620	3311	1256	1560	755	-	-	-
[Co(BHAT) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]	1606	-	1277	1562	751	3373, 815	536	491
[Ni(BHAT) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]	1605	-	1287	1563	750	3361, 823	541	487
[Cu(BHAT) <sub>2</sub> ]	1598	-	1281	1559	748	3393, 800	539	481

### Electronic spectra and magnetic moments:

The nature of the ligand field around the metal ion has been deduced from the electronic spectra.

The electronic spectrum of the [Co(BHAP)(H<sub>2</sub>O)<sub>3</sub>] complex displayed two main bands at 15773 and 21121 cm<sup>-1</sup>. These have been assigned to <sup>4</sup>T<sub>1g</sub> (F)-<sup>4</sup>A<sub>2g</sub> (F) (ν<sub>2</sub>) and <sup>4</sup>T<sub>1g</sub> (F)-<sup>4</sup>T<sub>1g</sub> (P) (ν<sub>3</sub>) transitions. The ν<sub>1</sub> transition is expected to appear at larger λ (below 10000 cm<sup>-1</sup>) and hence it could not be observed. The magnetic moment value of complex is 5.05 B.M. The observed transitions are consistent with an octahedral geometry. The electronic spectrum of the [Ni(BHAP)(H<sub>2</sub>O)<sub>3</sub>] complex displayed three bands at 11283, 19961 and 23751 cm<sup>-1</sup>. These transitions have tentatively been assigned to <sup>3</sup>A<sub>2g</sub> (F)-<sup>3</sup>T<sub>2g</sub> (F) (ν<sub>1</sub>), <sup>3</sup>A<sub>2g</sub>-<sup>3</sup>T<sub>1g</sub> (F) (ν<sub>2</sub>) and <sup>3</sup>A<sub>2g</sub>-<sup>3</sup>A<sub>1g</sub> (P) (ν<sub>3</sub>), which are characteristic for hexa-coordinated Ni(II). Since the value of the magnetic moment is 3.08 B.M., hence a octahedral geometry for Ni(II) complex has been proposed. The electronic spectrum of the [Cu(BHAP)(H<sub>2</sub>O)] complex exhibited two bands at 14748 and 19533 cm<sup>-1</sup>, assignable to <sup>2</sup>B<sub>1g</sub>-<sup>2</sup>B<sub>2g</sub> and <sup>2</sup>B<sub>1g</sub>-<sup>2</sup>E<sub>g</sub> transitions. Since the value of magnetic moment is 1.94 B.M.; therefore, square planar geometry of complex has been suggested.

The electronic spectra of the [Co(BHAT)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>] complex displayed two main bands at 16841 and 20213 cm<sup>-1</sup>. These have been assigned to <sup>4</sup>T<sub>1g</sub> (F)-<sup>4</sup>A<sub>2g</sub> (F) (ν<sub>2</sub>) and <sup>4</sup>T<sub>1g</sub> (F)-<sup>4</sup>T<sub>1g</sub> (P) (ν<sub>3</sub>) transitions. The ν<sub>1</sub> transition is expected to appear at larger λ (below 10000 cm<sup>-1</sup>) and hence it could not be observed. The magnetic moment value of complex is 5.04 B.M. The observed transitions are consistent with an octahedral geometry. The electronic spectrum of the [Ni(BHAT)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>] complex displayed three bands at 11206, 19619 and 24527 cm<sup>-1</sup>. These transitions have tentatively been assigned to <sup>3</sup>A<sub>2g</sub> (F)-<sup>3</sup>T<sub>2g</sub> (F) (ν<sub>1</sub>), <sup>3</sup>A<sub>2g</sub>-<sup>3</sup>T<sub>1g</sub> (F) (ν<sub>2</sub>) and <sup>3</sup>A<sub>2g</sub>-<sup>3</sup>A<sub>1g</sub> (P) (ν<sub>3</sub>), which are characteristic for octahedral Ni(II)-complex. The value of the magnetic moment is 3.15 B.M., hence octahedral geometry for Ni(II) complex has been proposed. The electronic spectrum of the [Cu(BHAT)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>] complex a broad band appears at 13992 cm<sup>-1</sup> corresponding to transition <sup>2</sup>E<sub>g</sub>-<sup>2</sup>T<sub>2g</sub>. The value of magnetic moment is 1.90 B.M.; therefore, octahedral geometry has been suggested for this complex.

### ESR spectra:

The ESR spectra of Cu(II) provide information about the extent of the delocalization of unpaired electron. The X-band ESR spectra of Cu(II) complexes were recorded in the solid state at room temperature and their g<sub>||</sub>, g<sub>⊥</sub>, Δg, g<sub>av</sub> and G have been calculated. The values of ESR parameters g<sub>||</sub>, g<sub>⊥</sub>, g<sub>av</sub>, Δg and G for Cu(II) complex of BABT are 2.205, 2.1351, 2.1351, 0.0699 and 1.5264 respectively.

The g<sub>||</sub>>2.3 is characteristic of an ionic environment and g<sub>||</sub><2.3 indicates a covalent environment in metal ligand bonding. The g<sub>||</sub> values for the complexes are less than 2.3 suggesting, the environment is covalent. According to Hathaway, if the value G is greater than four (G>4.0), the exchange interaction is negligible; whereas when the value of G is less than four (G<4) a considerable exchange coupling is present in solid complex. The G values for the Cu(II) complexes are less than four indicate, considerable exchange interaction in the complexes<sup>14,15</sup>.

### Pharmacology

Series of newly synthesized compounds were highly active against selected microorganisms. The minimal inhibition values were determined using the filter paper disc diffusion method and the concentrations have been used in ppm. All the final synthesized compounds have been screened in vitro for their antibacterial activity against *B. subtilis*, *E. coli* and *S. aureus* and antifungal activity against *A. niger*, *A. flavus* and *C. albicans*. Standards for antibacterial and antifungal activity Streptomycin and Griseofulvin were used respectively. Standards also screened under the similar conditions for comparison.

### Antibacterial activity

The above synthesized compounds were screened against some selected bacteria and examined for the inhibition of growth of the organism. The concentrations of the compounds were given in ppm. The diameter of the inhibition zones in (mm) are given in Table 3.

### Antifungal activity

The above synthesized compounds were screened against selected fungi and determined their minimal inhibition zones in (mm) were presented in Table 3 and concentrations of the compounds were given in ppm.

**Table 3.** Invitro antibacterial activity of compounds and their inhibition zone (%).

Compound	Antibacterial activity						Antifungal activity					
	<i>B. subtilis</i>		<i>E. coli</i>		<i>S. aureus</i>		<i>A. niger</i>		<i>A. flavus</i>		<i>C. albicans</i>	
	50 ppm	100 ppm	50 ppm	100 ppm	50 ppm	100 Ppm	50 ppm	100 Ppm	50 ppm	100 ppm	50 ppm	100 ppm
BHAP	11	16	15	20	13	18	11	19	16	20	13	20
Co(II)	22	31	21	31	21	30	20	31	23	32	19	31
Ni(II)	20	35	20	33	22	32	19	31	17	31	21	32
Cu(II)	21	30	21	28	20	31	20	32	22	32	19	32
BHAT	13	20	13	18	18	25	16	22	13	22	11	21
Co(II)	22	34	18	24	22	33	17	30	21	33	18	31
Ni(II)	24	32	21	32	16	32	22	32	20	30	16	30
Cu(II)	24	34	22	34	21	35	20	33	18	34	21	33
Streptomycin	27	36	25	33	27	36	-	-	-	-	-	-
Griseofulvin	-	-	-	-	-	-	22	31	21	34	25	37

## CONCLUSION

In the present research studies, our efforts are synthesized of some newly compounds. These synthesized compounds characterized by various physicochemical and spectral analyses. The IR data of both the Schiff base and its metal complexes show that the BHAP Schiff base is coordinated to the metal ion in tridentate manner with ONO donor sites while the BHAT Schiff base is coordinated to the metal ion in bidentate manner with ON donor sites of azomethine nitrogen and phenolic oxygen. FAB-mass data shows degradation pattern of the complexes. The antimicrobial data show that the metal complexes to be more biological active compared to those parent Schiff base ligand against all pathogenic species. The compounds also inhibit the growth of fungi and bacteria to a greater extent as the concentration is increased.

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