



Research Article

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Synthesis and microbial evolution of Co(II), Ni(II), Cu(II) and Zn(II) complexes of bidentate Schiff base derived from 2-amino-4-phenyl thiazole

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ABSTRACT

Schiff base derived from 2-hydroxy-5-chloroacetophenone and 2-amino-4-phenyl thiazole with Complexes of Co(II), Ni(II), Cu(II) and Zn(II) have been synthesized and characterized on the basis of elemental analysis, Infrared, ^1H NMR, molar conductance and magnetic susceptibilities analysis. The Schiff base acts as a monobasic bidentate ligand commonly coordinates through the oxygen atom of phenolic OH group and the nitrogen atom of azomethine group, which is confirmed by IR spectral data. All the complexes have been evaluated for their antimicrobial activity by agar cup-plate method against various organisms.

Keywords: Schiff base, Magnetic, Antimicrobial

INTRODUCTION

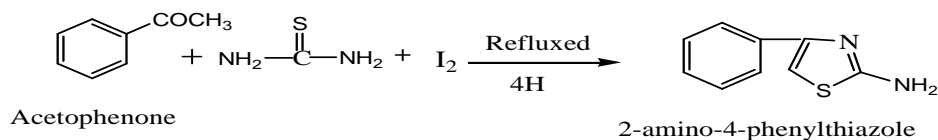
The development of coordination chemistry after the work of Werner was due to Linus Pauling who in 1931 extended the theory of valence bond. The theory was pushed into the background by the development of the ligand field theory which evolved out of the electrostatic crystal field theory. The study of a range of coordination compounds is linked to the coordination of metals with the Schiff base ligands. The hydrazone Schiff bases are widely used ligands to synthesize their metal complexes due to their facile synthesis, significant and good solubility in common organic solvents. Thus, they have played an important applicable role in research and development of coordination chemistry as they readily form stable metal complexes in different oxidation states[1]. Schiff bases or their metal complexes have many applications in different fields[2] Hydrazones, heteroaroyl hydrazones ligands and their metal complexes are biologically active. Heteroaroyl hydrazones forms stable metal complexes with transition metal ions and inner transition metal ions due to complexing ability of ligand through keto-enol tautomerism and availability of other donor sites in the ligand i.e. isonicotinoyl hydrazide is one of the drugs in chemotherapy of tuberculosis[3]. Thiazole Schiff base ligands and their metal complexes are biologically active[4]. Due to its biological potency, pharmacological properties and synthetic flexibility of Schiff base derived from isonicotinic acid hydrazide[5]. The aim of present investigation is to synthesize various transition metal complexes of Schiff base derived from 2-hydroxy-5-chloro acetophenone and 2-amino-4-phenyl thiazole

EXPERIMENTAL SECTION

All the chemicals were of A.R. grade and used as received. 2-hydroxy-5-chloro acetophenone (HCA) and 2-amino-4-phenylthiazole was prepared by known methods[6-9]. The solvents were purified by standard methods[10].

Synthesis of 2-amino-4-phenylthiazole; The synthesis of 2-amino-4-phenylthiazole prepared by known method[7-9]. The product was filtered and crystallized from 70% ethanol, after several minutes the golden coloured product of 2-amino-4-phenylthiazole was separated out.

Yield: 75%; m.p.: 148-150°C



Synthesis of 2-hydroxy-5-chloro acetophenone 2-imino-4-phenyl thiazole [HCAT]:

A solution of 2-hydroxy-5-chloro acetophenone (0.02M) in 25ml of ethanol was added to an ethanolic solution(25ml) of 2-amino-4-phenylthiazole(0.02M) and the reaction mixture was refluxed on a water bath for 4h. After cooling a pale yellow coloured crystalline solid was separated out. It was filtered and washed with ethanol, crystallized from DMF and dried under reduced pressure at ambient temperature. The purity of ligand was checked by elemental analysis and m.p. It was also characterized by IR and ^1H NMR spectral studies.

Yield: 35%; m.p. 290°C

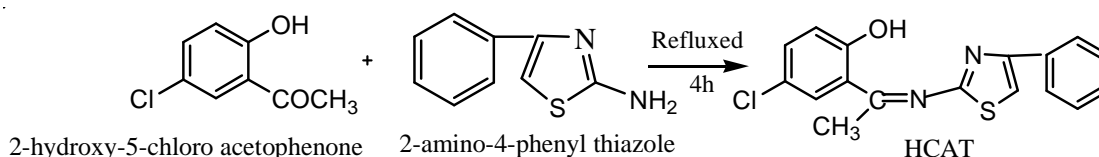


Table1. Analytical data of the Ligands

Sr. No.	Ligand	Molecular Formula	Formula Weight	Color and nature	Elemental Analysis				
					C% found (Cal.)	H% Found (Cal.)	N% Found (Cal.)	Cl% Found (Cal.)	S% Found (Cal.)
1.	HCAT	$\text{C}_{17}\text{H}_{12}\text{N}_2\text{OSCl}$	327.6	Yellow Crystalline	61.38 (62.27)	03.10 (03.66)	08.24 (08.54)	10.21 (10.83)	09.13 (09.79)

Preparation of complexes:

All the metal complexes were prepared in a similar way by following method. To a hot solution of ligand HCAT (0.02M) in 25ml of ethanol a suspension of respective metal salts [acetates of Co(II), Ni(II), Cu(II), and Zn(II)] was added drop wise with constant stirring. The reaction mixture was refluxed on a water bath for 4-6 h. The precipitated complexes were filtered, washed with ethanol followed by ether and dried over fused calcium chloride. Yield : 45-50%

The complexes are soluble in DMSO and DMF but insoluble in water and common organic solvents. The metal chloride content of complexes were analyzed by standard methods[11].

The ^1H NMR spectra of ligand was recorded and obtained from RSIC Chandigarh. IR spectra of the compounds were recorded on Perkin Elmer 842 spectrophotometer in the region $400\text{-}4000\text{cm}^{-1}$, Carbon, Hydrogen and Nitrogen analysis were carried out at RSIC, Punjab University, Chandigarh. The molar conductance of the complexes at 10^{-3} M dilution in DMF were determined using equiptronic digital conductivity meter EQ-660 with a cell constant 1.00 cm^{-1} at room temperature. The magnetic moment measurement were made on a Gouy balance at room temperature using $[\text{HgCo}(\text{SCN})_4]$ as the calibrant. The molecular weights of the complexes were determined by Rast method.

Table 2. Analytical data and molar conductance of the compounds

Compounds	Colour	Mol.wt.	Analysis % Found (calc.)						μ_{eff} B.M.	Λ_M ($\Omega^{-1} \text{cm}^2 \text{mol}^{-1}$)
			M	C	H	N	Cl	S		
[Co(L) ₂ (H ₂ O) ₂]H ₂ O	Brown	783.1	7.22 (7.52)	51.86 (52.10)	3.35 (3.57)	6.96 (7.15)	8.80 (9.06)	7.84 (8.20)	4.38	5.9
[Ni(L) ₂ (H ₂ O) ₂]H ₂ O	Green	782.9	7.20 (7.39)	51.78 (52.11)	3.38 (3.58)	6.86 (7.15)	8.90 (9.06)	7.79 (8.19)	3.1	8.4
[Cu(L) ₂ (H ₂ O) ₂]H ₂ O	Brown	787.7	7.20 (7.98)	51.60 (51.79)	3.15 (3.55)	6.79 (7.11)	8.82 (9.01)	7.95 (8.15)	1.90	7.2
[Zn(L) ₂ (H ₂ O) ₂]2H ₂ O	Reddish	807.6	7.80 (7.91)	50.11 (50.52)	3.48 (3.71)	6.12 (6.93)	8.30 (8.79)	7.54 (7.94)	Dia	8.4

RESULTS AND DISCUSSION

The Schiff base ligand HCAT and its complexes have been characterized on the basis of ¹H NMR, IR spectral data, elemental analysis, molar conductance, magnetic susceptibility measurements and thermogravimetric analysis data. All these values and analytical data is consistent with proposed molecular formula of ligand. All the compounds are coloured solid and stable in air. They are insoluble in water but soluble in coordinating solvents like DMF and DMSO. The molar conductance values in DMF(10⁻³M) solution at room temperature (Table 2) shows all the complexes are non electrolytes.

The ¹H NMR spectra of ligand HCAT shows signals at δ 11.26, (1H, s phenolic OH), 7.41, 7.40, 7.39 and 7.38 (4H, m, phenyl) δ 6.51, 6.50, and 6.58(3H, s Phenyl), 6.62 (1H s thiophene), and 2.16(3H, s, methyl)[12-15].

IR spectra of ligand and metal complexes shows $\nu(\text{C}=\text{N})$ peaks at 1618cm⁻¹ and absence of C=O peak at around 1700–1730 cm⁻¹ indicates the Schiff base formation[16-19].

Table 3. IR spectra of ligand and metal complexes

Compound	$\nu(\text{O-H})$ hydrogen bonded	$\nu(\text{C}=\text{N})$ Imine	$\nu(\text{C-O})$ phenolic	$\nu(\text{C-S})$	$\nu(\text{M-O})$	$\nu(\text{M-N})$
HCAT	3109	1618	1514	1122	-	-
[Co(L) ₂ (H ₂ O) ₂]H ₂ O	-	1606	1504	1098	470	430
[Ni(L) ₂ (H ₂ O) ₂]H ₂ O	-	1580	1465	1090	468	422
[Cu(L) ₂ (H ₂ O) ₂]H ₂ O	-	1602	1504	1110	509	410
[Zn(L) ₂ (H ₂ O) ₂]2H ₂ O	-	1503	1448	1106	470	425

Antimicrobial activity:

Antimicrobial inhibition Screening assay depends upon a comparison of the inhibition of growth of microorganism by measuring the concentration of the sample to be examined with the known concentration of standard antibiotic. For the antimicrobial analysis the agar cup-plate diffusion method has been employed[20-22]. In this study the ligand and their metal complexes were tested for their effect on certain human pathogenic bacteria such as Gram-positive. The inhibition effect of the ligand and its metal complexes on the growth of various bacterial species *B.subtilis*, *P.vulgaris*, *S.aureus*, *E.coli*, *P.fluorescen*, *A.aerogenes*, *B.megatherium*. are summarized in Table 4.

Table 4. Antimicrobial activity[23-30]

Ligand and its complexes	<i>B. subtilis</i> (mm)	<i>P. vulgaris</i> (mm)	<i>S. aureus</i> (mm)	<i>E. coli</i> (mm)	<i>P. fluorescen</i> (mm)	<i>A. aerogenes</i> (mm)	<i>B. megatherium</i> (mm)
HCAT	R	R	S ₁₆	R	S ₁₀	R	R
Co- HCAT	S ₈	R	S ₁₁	S ₈	S ₈	S ₉	R
Ni- HCAT	R	S ₉	S ₁₂	R	S ₇	R	S ₉
Cu- HCAT	S ₈	S ₁₂	S ₁₄	S ₇	R	R	S ₇
Zn- HCAT	R	S ₈	S ₁₀	R	R	S ₈	S ₇

CONCLUSION

A Synthesized ligand and complexes using thiazole has been characterized by spectral and analytical data. The results revealed that the ligands and their complexes show considerable antimicrobial activity. However, the zone of

inhibition of ligand varies with organisms as well as metal ions. Thus, it can be concluded that most of our ligands and their complexes possess antimicrobial activities. The structural changes have marked effect on the sensitivity and sensitivity varies with organisms.

REFERENCES

- [1] KS.Prasad; L.ShivaKumar; S.Chandan; B.Jayalakshmi and HD.Revanasiddappaa, *Spectrochim. Acta, Part A*, **2011**,81, 276.
- [2] M.Ramesh; KB.Chandrashekar and RK.Hussain, *Indian J. Chem.*, **2000**,39A,1337.
- [3] RP.Sharma; AK.Kothari and NK.Sharma, *Indian J. Derm. Vener. Lepr.*, **1995**.61,26.
- [4] S.Kumar, D.Dhar and P.Saxena, *J. Sci.and Indu.Research*, **2009**, 68, 181.
- [5] Hawlader MBH and Begum MS, *Indian J. Chem.*, **2004**, 43A, 2352.
- [6] A.Aswar, P.Bahad, A.Pardhi and N.Bhave, *J. Poym. Mater.* **1988**, 5, 232.
- [7] S.Pattan, M.Ali, J.Pattan, S.Purohit, V.Reddy and B.Nataraj, *Indian J. Chem.*, **2006**,45B, 1929.
- [8] D.Khrustalev, A.Suleimenova .and S.Fazylov, *Russian J. App. chem.*, **2008**, 81(5), 900.
- [9] H.Maradiya, and V.Patel, *J. Fibers and poly.*, **2002**, 3(1), 43.
- [10] B.Furniss, A.Hannaford, P.Smith & A.Tatchell, Vogel's practical organic chemistry 5thEd. (Logman Scientific Technical, John Wiley and Sons), **1989**.
- [11] AI.Vogel, "A Text book of quantitative inorganic chemistry" 3thEd., (ELBS,London,**1961**).
- [12] S.Sadigova; A.Magerramov and M.Allakhverdiev, *Russian J. Org. chem., chem.*, **2008**, 81(5), 900.
- [13] E.Campbell and S.Nguyen, *J. Tetrahedron*, **2001**, 42, 1221.
- [14] P.Pietikainen and A.Haikarainen *J. Mole. Catalysis.*, **2002**, 180, 59.
- [15] M.Kidwai, P.Poddar and k.Singhal, *Indian J. Chem.*, **2009**, 48B, 59.
- [16] S.Sonwane, S.Srivastava and S.Srivastava, *Indian J. Chem.*, **2008**, 47B, 633.
- [17] K.Patel and A.Mehata, *E. J. Chem.*, **2006**, 3(13), 267.
- [18] R.Maurya, D.Antony, S.Gopinathan, V.Puranic, S.Tavale and C.Gopinathan, *Bull. Chem. Soc. Jpn.* **1995**, 68, 2847.
- [19] D.Boghaei and S.Mohebi *J. Tetrahedron*, **2002**, 58, 5357.
- [20] AK.Kaura and M.Kaura, *Int. J. Chem. and Pharm. Sci.*, **2012**, 3(4), 24.
- [21] B.Sutariya; S.Mohan; S.Sambasiva and S.Rao, *Indian j. Chem.*, **2007**, 46B, 884.
- [22] P.Venkatesh, *Asian J. Pharm. Hea. Sci.*, **2011**, 1(1), 8.
- [23] CI Raj; M Christudhas; GA Raj, *J. Chem. Pharm. Res.*, **2011**, 3(6), 127.
- [24] SD Dhumwad; KB. Gudasiand; TR Gaudar, *Indian J. Chem.*, **1994**, 33A, 320.
- [25] UI Singh; RK Singh; WR Devi; CH Singh, *J. Chem. Pharm. Res.*, **2012**, 4(2), 1130.
- [26] S Prakash; VP Vaidya; KM Mahadevan; MK Shivananda1; PA Suchetan; B Nirmala; M Sunitha, *J. Chem.Pharm. Res.*, **2012**, 4(2), 1179.
- [27] IO Adeoye; OO Adelowo; OO Onawumi, *J. Chem. Pharm. Res.*, **2012**, 4(1), 1.
- [28] AK Mapari; KV Mangaonkar, *Int. J. ChemTech Res.*, **2011**, 3(1), 477.
- [29] M. Rajan; V. Kishor Kumar; P. Satheesh Kumar; K. Reddy Swathi, and S. Haritha, *J. Chem. Pharm. Res.*, **2012**, 4(6), 2860
- [30] P. Patel; D. Gor and PS. Patel, *J. Chem. Pharm. Res.*, **2012**, 4(6):2906.