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Synthesis and characterisation of Zn(II) and Cu(II) complexes with a Schiff base ligand

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Abstract: A new schiff base ligand, 1H-5-chloro-indole-2-one-3-(2-methyl-4-nitro)-anilinimine was synthesised by the condensation of 2-methyl-4-nitro aniline with 5-chloro isatin. Cu(II) and Zn(II)metal complexes were prepared by refluxing the above synthesised ligand with Chlorides, nitrates and acetates of the respective metals. The complexes were characterised by various physicochemical techniques such as elemental analysis, FT IR, ^{1}H NMR, UV visible, TGA, conductivity and magnetic susceptibility measurements. The ligand acts as bidentate in which the carbonyl oxygen and the azomethine nitrogen are involved in chelation. Square planar geometry for Cu(II) complexes and Tetrahedral geometry was proposed for Zn(II) complexes. The ligand and complexes were screened for their antimicrobial and antifungal activities.

Keywords: Chloro isatin, Schiff base, antibacterial, antifungal.

1. INTRODUCTION

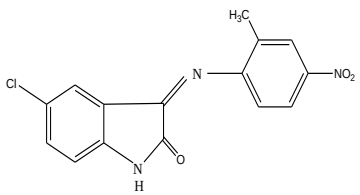
Schiff base ligands are important coordinating molecules and can exhibit variety in the structure of their metal complexes [1]. Schiff base metal complexes have been the subject of extensive research due to their novel properties and their industrial and biological importance. The chemistry of coordination compounds with Schiff base ligand can be formed by the condensation of primary amines with aldehydes and ketones. Isatin (1H-indol-2, 3-dione) is a synthetically versatile substrate, where it can be used for the synthesis of a large variety of heterocyclic compounds. Coordination compounds derived from isatin have been reported to have antibacterial, antifungal, analgesic and anti-inflammatory activity [2-4]. The present paper describes the synthesis and characterization of Zn(II) and Cu(II) complexes of the Schiff base derived from the condensation of 2- methyl -4- nitro aniline with 5-chloro isatin.

2.MATERIALS AND METHODS

All the chemicals used were of AR grade. Zinc (II)) and Copper (II) used as chloride, nitrate,and acetate salts were of Merck. The CHN analysis were performed using CHNS Analyzer: ELEMENTAR Vario EL III. The electronic spectra were recorded on UV-Visible spectrophotometer. The proton NMR data was recorded on Bruker Advance DPX spectrometer at 400 MHz. The thermogravimetric analysis was carried out using TGA-DTA Hitachi STA7000. KBr discs were used in IR spectrum analyses using a Perkin Elmer Spectrum RX-I FTIR spectrophotometer. The Melting points were determined in open capillaries and are uncorrected. Gouy balance is used to measure the magnetic susceptibility, and molar conductance measurements in DMSO were conducted using 10^{-3}M solutions of the complexes at room temperature.

2.1. Synthesis of Schiff Base ligand, 1H-5-chloro-indole-2-one-3-(2-methyl-4-nitro)-anilinimine

Schiff base ligand was prepared by refluxing the ethanolic solutions of 5 – Chloro isatin (0.01 mol, 1.82 g) in 50 ml and 2-methyl-4-nitro aniline (0.01 mol, 1.52 g) for about 1-2 hours. The reaction mixture was evaporated to a small volume and left to cool. The resulting Schiff base ligand precipitated on cooling and then was filtered off, washed with ethanol and recrystallized from ethanol [5].



Schiff base ligand (L)

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2.2. Synthesis of Zn(II) and Cu(II) complexes

The metal complexes of chlorides, nitrates and acetates of Zn(II) and Cu(II) were prepared by adding solution of respective metal salts (1 mol) in 50 ml of absolute ethanol to 50 ml of an ethanolic solution of the ligand (1 mol) and refluxed at 80-90°C for about 2 to 4 hours. The reaction mixture was then concentrated into a small volume by evaporation method. On cooling, the metal complexes crystallizes out. The crystallized metal complexes were filtered and washed thoroughly with ethanol and dried under vacuum over fused calcium chloride.

2.3. Biological activities

Antibacterial and antifungal activity of newly synthesized ligand and the complexes were investigated by the agar diffusion method. The samples were used for the determination of zone of inhibition or sensitivity, against bacteria E.Coli, Bacillus sp and Pseudomonas sp strain and against fungi Aspergillus terreus, Penicillium brocae and Trichoderma sp. The results were correlated with standard antibiotics drugs like gentamycin and fluconazole.

3. RESULTS AND DISCUSSION

All the metal (II) complexes synthesised are non hygroscopic, highly stable at room temperature and can be stored for longer time. They are insoluble in water and in common organic solvents but are soluble in DMF and DMSO.The elemental analysis are found to be in agreement with the calculated values. The conductivity of 10^{-3} M solutions of all the complexes in DMSO are measured and is tabulated. The observed molar conductance for the complex 1a,1b and 2b are found to be in the range 31-34 Scm^2mol^{-1} and therefore is considered to be non electrolytes. On the other hand the complexes 1b, 1c, 2a and 2c possess the conductance values in the range 65-76 Scm^2mol^{-1} and are considered to be 1:1 electrolytes(Table 1)

Table-1 Physicochemical and analytical data of ligand and complexes

Ligand/ Complex	Yield %	Elemental Analysis (%)Found (Calculated)			Melting Point ^0C	Mol Cond $Scm^2\ mol^{-1}$
		Carbon	Hydrogen	Nitrogen		
$C_{15}H_{10}N_3O_3Cl$(L)	91%	57.54 (57.05)	3.28 (3.16)	13.75 (13.31)	136	---
$[Zn(L)Cl_2]$ (1a)	92.0	39.94 (39.83)	2.11 (2.21)	9.43 (9.29)	215	32
$[Zn(L)(NO_3)_2]$ (1b)	94.5	35.53 (35.65)	1.30 (1.18)	13.71 13.86)	210	31
[Zn(L)(OAC)]OAc (1c)	90.5	45.84 (45.70)	3.42 (3.20)	8.32 (8.41)	219	76
$[Cu(L)ClH_2O]Cl$ (2a)	88	39.05 (38.46)	2.78 (2.56)	9.14 (8.97)	220	65
$[Cu(L)(NO_3)_2]$ (2b)	89.5	35.11 (35.78)	1.74 (1.98)	13.72 (13.88)	210	34
[CU(L)OAc]OAc (2c)	88.0	46.09 (45.87)	3.57 (3.21)	8.56 (8.45)	203	69

3.1 IR Spectra

The IR spectra gives very interesting information about the bonding in complexes. The IR spectrum of the free ligand is characterized by the strong bands at 3349, 1734 and 1633cm^{-1} which are attributed to the stretching frequencies of ν NH (isatin), ν C=O and ν C=N(azomethine) groups respectively. The IR spectra of the complexes exhibited a lower shift of wave numbers for azomethine (C=N) group by about 18 cm^{-1} and also showed a lower shift of wave numbers by 20 cm^{-1} for (C=O) moiety of the isatin. This confirms the chelation of the ligand to the metal through the azomethine and carbonyl groups. The IR spectra of the complexes of Zn(II) and Cu(II) exhibit new bands at 534-542cm^{-1} for M-O , 326-329 cm^{-1} for M-Cl and 462-480 cm^{-1} for M-N stretching modes respectively. The complexes 1b and 2b shows bands at 1518, 1285, 1056 cm^{-1} and 1514, 1284, 1061cm^{-1} respectively corresponding to the monodentate nitrate group [6,7]. The spectrum of complexes 1c and 2c exhibited bands which could be assigned to the coordination mode of the acetate anion [8,9]. A correlation between the infrared antisymmetric and symmetric absorption frequencies of the acetate group and the type of acetate group has been developed. The carboxylate ion in solution is characterized by bands at 1578 and

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1411 $cm^{-1,}$ which are commonly assigned to the antisymmetric (ν_{as}) and the symmetric (ν_{sym}) stretching vibrations of the carboxylate group. The difference in these frequencies, $\Delta\nu = \nu_{as} - \nu_{sym}$, have been used to confirm the coordination modes of the acetate group. According to Deacon and Phillips [10] a difference larger than 200 cm^{-1} indicates a monodentate coordination, where as a difference smaller than 150 cm^{-1} indicates a bridging coordination mode. However, it is also accepted that a values of $\Delta\nu$ smaller than 200 cm^{-1} can indicate a bidentate coordination mode. In complexes 1c and 2c, the frequencies of the vibration ν_{as}(COO) appears in the range 1564–1584 cm^{-1}, while those characteristic of the ν_{sym} (COO), ranges from 1397 to 1415 cm^{-1}. For these complexes, $\Delta\nu$ is >150 cm^{-1}, which leads to the conclusion that the acetate group is bidentate in nature. The Selected IR frequencies of ligand and its metal complexes are represented in the Table 2.

Table 2.Selected IR frequencies, UV-Visible and Magnetic moment of the Ligand and its complexes.

Ligand/Complex	ν_{O-H}	ν_{N-H}	$\nu_{C=O}$	$\nu_{C=N}$	ν_{NO3}/ ν_{SO4}/ ν_{OAC}	M-Cl / M-O	M-N	Free oAc	μ_{eff} BM	λmax cm^{-1}
$C_{15}H_{10}N_3O_3Cl$	---	3349	1734	1633	---	---		---	-----	25740 38837
$[Zn(L)Cl_2]$ (1a)	---	3336	1704	1620	---	329	480	---	DIA	23364 28011
$[Zn(L)(NO_3)_2]$ (1b)	---	3344	1712	1620	1518, 1285, 1056	541	464	---	DIA	23282 28650
[Zn(L)(OAC)]OAc (1c)	---	3349	1711	1623	1397, 1564	534	462	665,911, 1460,2921	DIA	23484 27868
$[Cu(L)ClH_2O]Cl$ (2a)	3445	3341	1704	1620	---	326	470	---	1.85	23361 16007
$[Cu(L)(NO_3)_2]$ (2b)	---	3236	1711	1620	1514,1284, 1061,	537	465	---	1.89	23255 15624
[CU(L)OAc]OAc (2c)	---	3231	1712	1618	1415, 1584	542	466	661,898, 1460,2923	1.95	24038 15560

UV-Visible and Magnetic susceptibility Measurements

The UV-Visible spectral data of ligand and its metal complexes have been tabulated in the Table 2. In the complexes 1a,1b and 1c, Zinc (II) ion has d^{10} configuration and hence the the absorption at 23364, 23282 and 23484cm^{-1} could be assigned to a charge transfer transitions. Hence by considering the spectra and the diamagnetic property of these complexes, a tetrahedral geometry can be assumed [5,11].

The electronic absorption spectra exhibits bands at 23361 cm^{-1}, 16007cm^{-1} (2a), 23255cm^{-1}15624cm^{1} (2b) and 24038cm^{-1} 15560cm^{-1} (2c) for the copper complexes which can be assigned to $^2B_{1g} \rightarrow {}^2A_{1g}$ and $^2B_1g \rightarrow {}^2E_1g$ transitions. These transitions, and also the measured value of the magnetic moment (1.85-1.95BM) depicts a square-planar structure for Cu(II) complexes.

3.2 ^{1}H NMR Spectra

The proton NMR spectrum of the ligand and the complexes were recorded in DMSO and the data are tabulated in Table 3. The proton NMR spectrum of the ligand revealed two singlets at 11.06 and 2.45ppm, which are attributed to protons of indole NH, and three hydrogens of methyl group of amine respectively, while the protons at 7.47-7.81 ppm range were for the aromatic ring. In the diamagnetic complexes 1a,1b and 1c the protons of indole (N-H) are attributed to singlet proton signals at 12.01, 11.43, and 11.22 ppm, respectively, while the signals at 2.58 ,2.55 and 2.60 ppm are attributed to the methyl group of amine[12]. The aromatic protons reverberated as a multiplet in the range of 7.51-7.84, 7.54-7.89 and 7.5-7.86 ppm respectively

Table 3. ^{1}H NMR spectral studies

Ligand/ Complexes	(N-H) Isatin	Methyl group	Aromatic Hydrocarbons
L-$C_{15}H_{10}N_3O_3Cl$	11.06	2.45	7.47-7.81
$[Zn(L)Cl_2]$ (1a)	12.01	2.58	7.51-7.84
$[Zn(L)](NO_3)_2]$ (1b)	11.43	2.55	7.54-7.89

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[Zn(L) OAc]OAc (1c)	11.22	2.60	7.5-7.86

3.3 Thermogravimetric studies

TGA studies of the complexes were carried out in nitrogen atmosphere at the rate of 10°C per minute up to 700°C and the data is presented in Table 4. A general pattern is observed In the thermal decomposition studies of the complexes . The thermal decomposition occurs in two stages for the complexs 1a and three stages for the complex 2a [5]. For 1a, in the first stage there is decomposition of two coordinated Cl atoms followed by the loss of ligand in the second stage leaving finally the metal oxide. In 2a, removal of uncoordinated Cl atom occurs in first stage, removal of coordinated Cl and H_2O occurs in second stage followed by ligand in the third stage. In the complexes of 1b and 2b, there is removal of two nitro groups in the first stage followed by the ligand in the second stage [7]. In the complexes 1c and 2c also there is loss of two acetates in the first stage followed by the the loss of ligand leaving behind finally the metal oxide.

Table 4. Thermoanalytical data of the metal complexes.

Compound	Stage	Temp.°C	Species lost	Weight loss (%)Cal.(Obs.)	Residue
[Zn(L)Cl_2](1a)	1 2	110 320	Two Cl atoms Ligand	23.56 (23.55) 58.0(58.65)	ZnO
[Zn(L) (NO_3)$_2$](1b)	1	100-250 250-450	Two NO_3 groups $C_{15}H_{10}N_3O_2Cl$	24.56(24.05) 59.32(59.80)	ZnO
[Zn(L)(OAC)]OAc (1c)	1 2	100-220 220-450	Two OAc groups $C_{15}H_{10}N_3O_2Cl$	23.65 (23.15) 60.14 (60.86)	ZnO
[Cu(L)ClH_2O]Cl (2a)	1 2 3	100 210 320	Uncoordinate Cl Coordinated Cl and H_2O Ligand	7.5(7.79) 11.43(11.8) 63.9(64.2)	CuO
[Cu(L) (NO_3)$_2$] (2b)	1 2	100-250 250-450	Two NO_3 groups $C_{15}H_{10}N_3O_2Cl$	24.65 (23.35) 59.5 (60.08)	CuO
[CU(L)(OAc)]OAc (2c)	1 2	100-220 220-450	Two OAc groups $C_{15}H_{10}N_3O_2Cl$	23.74 (23.35) 60.26 (60.84)	CuO

3.4 Antibacterial and Antifungal activity

In vitro antibacterial and antifungal activities for the ligand and complexes were examined against bacteria Bacillus sp, Pseudomonas sp , .Coli and against the fungi Penicillium brocae, Aspergillus terreus and Trichoderma sp. Activitties were investigated by the agar diffusion method for the determination of zone of inhibition [13]. The antibacterial activities were performed on nutrient agar medium. Gentamycin and Fluconazole are used as the standard antibacterial and antifungal agents respectively. The nutrient agar were prepared, autoclaved at 121°C for 15 lbs, cooled and poured on sterilized petri plates and allowed for solidification. After solidification ,wells were made then the sample and standards are introduced in different plates. The plates were incubated at 37°C for 24 hours for bacteria and 26-28 °C for 48 to 72 hours for fungi. The incubated plates were observed for the zone of inhibition (in mm) and results are tabulated in Table 5 and 6.

Antibacterial screening for the newly synthesized metal(II) complexes showed better activity when compared to the free Schiff base ligand. The activity was enhanced with the complexation. Chelation theory [14] was used to explain the increase in activity for the complexes over the Schiff base ligand. The increase in the activity can be explained by the fact that ligands have azomethine (C=N) linkages. The positive charge of the metal ion is shared in part with the heteroatoms(O and N), and there may also be electron delocalization through out the chelating system[15]. As a result, metal chelates become more lipophilic and they are likely to pass through the lipid layer of bacterial membranes and block metal-binding sites in microorganism enzymes. Both antibacterial and antifungal activities were found to be enhanced in Cu(II) complexes when compared to Zn(II) complexes against selected microorganisms. With respect to the anion, the activity of the complexes follow the order $Cl^- > NO_3^- > CH_3COO^-$ [16]

Table 5. Antibacterial Activity

compound	Bacillus sp	E.Coli	Pseudomonas sp
L-$C_{15}H_{10}N_3O_3Cl$	1	---	---

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[Zn(L)Cl_2] (1a)	2	2	1
[Zn(L) (NO_3)$_2$] (1b)	1	2	2
[Zn(L)(OAC)]OAc (1c)	2	1	4
[Cu(L)ClH_2O]Cl (2a)	10	5	2
[Cu(L) (NO_3)$_2$] (2b)	8	3	4
[CU(L)OAc]OAc (2c)	3	2	4
Gentamycin	9	14	9

Table 6. Antifungal activity

compound	**Aspergillus terreus**	**Penicilliu m brocae**	**Trichoderma sp**
L-$C_{15}H_{10}N_3O_3Cl$	---	---	1
[Zn(L)Cl_2] (1a)	4	2	4
[Zn(L) (NO_3)$_2$] (1b)	2	1	2
[Zn(L)(OAC)]OAc (1c)	1	---	2
[Cu(L)ClH_2O]Cl (2a)	8	8	7
[Cu(L) (NO_3)$_2$] (2b)	3	3	5
[CU(L)OAc]OAc (2c)	---	3	---
Itraconazole	10	12	11

CONCLUSION

Newly synthesised Schiff base transition metal complexes of Zn(II) and Cu(II) are charecterised by various physicochemical methods and the ligand behaves as bidentate in nature with azomethine and carbonyl group as strong donors forming 1:1 complexes. Based on electronic study and magnetic moment measurements, tetrahedral geometry is proposed for Zn(II) complexes and square planar structure for Cu(II) complexes. The antibacterial and antifungal activities are enhanced in complexes compared to ligand. The activities are comparitively increased in Cu(II) complexes than in Zn(II) complexes.

Author's Note

Author's declares no conflict of interests**.**

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