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Synthesis, characterization and biological activities of Schiff base and it's transition metal complexes

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ABSTRACT

Biivalent transition metal Mn(II), CO(II), Ni(II) and Cu(II) complexes of 3,4-{[-benze 1-sulphacetamide] imino] methyl} furan complexes derived from heterocyclic furfural and aniline sulphacetamide, have been synthesized and characterized by elemental, magnetic moment and electrochemical such as electronic spectra, IR Spectra and ESR Spectral studies. The ligand and metal complexes were screened for biological activity against Gram (+) and Gram (-) bacteria by the agar well diffusion method.

Keywords: Schiff base, Transition Metal Complexes, Spectral Studies, Antibacterial Studies, hetrocycles.

INTRODUCTION

Compound containing hetrocyclic structures have high degree of binding affinity to biological system (Rajiv et al, 2011). The metal complexes of Schiff bases showed higher biological activity in composition to Schiff bases. A large number of Schiff base transition metal complexes have been synthesized, which showed antibacterial, antimalerial, antileukemia activity, DNA binding, DNA cleavage, anticancer and antioxidant activities (Ebrahimpour et al, 2015; Rosu et al, 2010; Tabassum et al, 2013; Wu et al, 2015). Schiff base containing hetrocycles possess hypnotic activity (Vachala et al, 2012). In past few decades, researchers have focused towards transition metal complexes of heterocyclic aromatic Schiff bases having, nitrogen, oxygen and sulphur donar atoms are due to their therepentic importance (Sridhar et al, 2001; Valverde et al, 2005). Its complexes have also been found to be active against HIV and tumor cells, as good anti-tubercular, anti-inflammatary, anticoagulant, anticonvulsant and as chemotherepentic agents in cancer and infectious disease research (Malathy et al, 2015; Osowole et al, 2012; 2010a; 2011b; 2012c). According to the search material, no work has been carried out on this ligand and its transition metal complexes. I herein report the synthesis, characterization and biological activity of new Schiff

base ligand 3,4{[-benzene 1-sulphacetamide] imino} methyl} furan and its bivalent transition metal Mn(II), CO(II), Ni(II) and Cu(II) complexes. In present study, metal co-ordination with ligand, takes place via imino nitrogen and the oxygen of sulphacetamde >C=O group. Ligand and all complexes are characterized by elemental, magnetic moment and spectral data studies.

METHODS AND MATERIALS:

All the chemicals used were of analytical and GR-grade and purchaged either from BDH, E-Merk, S.D.Fine's and Sisco chemical industries, Bombay. The solvents and liquid reagents were carefully purified by distillation, while solid reagents and metal salts were used as such. The complexes were recrystallized with the solvents, depending upon its solubilities, and its purity was checked by TLC.

Physical measurements:

Magnetic susceptibility measurements of the prepared complexes were carried out on EG & G model 155 VSM at room temperature. The infrared spectra of the Schiff base, 3,4- {[-benzens 1-sulphacetamide] imino] methyl} furan and its complexes were recorded on Perkin Elmer Spectrometer in the FT-IR region using KBr Pellets. The electronic spectra in solution EtoH/DMF were recorded on ELICO SL 171 Spectrophotometer at room temperature. Mass spectra of the prepared Schiff base ligand was taken on MASPEC System (MSW/9629) using 200^{o}C intel temperature. The ESR spectra of Cu(II) complex was recorded on a varian X-band spectrometer E_4. An elemental analysis of carbon, hydrogen and nitrogen was done on at RSIC, Chandigarh. Estimation of sulphur in ligand and complexes were determined by standard method (Weicher, 1965) and estimation of halogen was estimated by Volhard's method (Weicher, 1965a). Metal content of complexes were determined by standard methods (Flascuke, 1954; Kalthof et al, 1963; Tradwell, 1968; Vogel, 1961; Wathrich, 1965).

Synthesis of 3,4- {[-benzene 1-sulphacetamide] imino] methyl} furan (SB_1) $C_{13}H_{12}N_2O_4S$

An equimolar solution of the titeled Schiff base was prepared by refluxing equimolar solution of aniline-sulphacetamide (10mmol, 2.14g in 25ml alcohol) and 2-furfuraldehyde (10mmol, 0.96ml in 25ml alcohol) on water bath for 3-4 hour and then cooling, a yellow solution resulted, was stirred for 2-3 hour. The solvent was slowly evaporated. A solid colored powder is obtained and washed with ethanol and dried in vaccum. Light yellow crystals were obtained.

Synthesis of metal complexes

The metal chloride (5mmol) in 25ml ethanol was added slowly to a solution of the Schiff base ligand (10mmol in 25ml ethanol). The resulting mixture was stirred for 30 minutes and then refluxes for 2-3 hour on water bath. The product was cooled and solvent was slowly evaporated, washed with ethanol, acetone and ether, dried in vacuum. The different colored crystals of different complexes with metal salts and ligand in 1:2 metal: ligand molar ratio have been isolated as $[Mn(SB_1)_2Cl_2]$, $[CO(SB_1)_2Cl_2]$, $[Ni(SB_1)_2Cl_2]$ and $[Cu(SB_1)_2Cl_2]$.

RESULTS AND DISCUSSION

The all prepared metal complexes were stable in dry air. They are soluble in DMF/DMSO but insoluble in most organic solvents. They were characterized with the help of physic-chemical methods such as magnetic susceptibility measurements, electronic spectra, IR spectra, ESR (Cu complex only) and elemental analysis. The analytical data and magnetic moment values are listed in table-1

Table-1 Analytical data and magnetic moment values of synthesized ligand and it's transition metal (II) complexes.

S.N.	Compound	Colour	Percentage of Elements (Found) Calculated							μ(B.M.)
			C%	H%	O%	N%	S%	Cl%	M%	
1	SB_1	Light Yellow	52.89 53.02	4.38 4.46	21.80 21.74	9.54 9.58	10.98 10.96	- -	- -	
2	$Mn(SB_1)_2Cl_2$	Yellowish Brown	43.58 43.64	3.85 3.92	18.02 17.94	7.96 7.92	8.98 8.96	9.92 9.88	7.82 7.78	5.74
3	$CO(SB_1)_2Cl_2$	Pink	43.54 43.50	3.90 3.86	17.78 17.82	7.80 7.81	8.90 8.94	9.84 9.86	8.36 8.26	4.5
4	$Ni(SB_1)_2Cl_2$	Greenish Brown	43.60 43.67	3.75 3.72	17.74 17.80	7.86 7.70	8.86 8.90	9.80 9.82	8.26 8.15	2.98
5	$Cu(SB_1)_2Cl_2$	Bluish Green	43.25 43.18	3.85 3.82	17.75 17.72	7.72 7.70	8.86 8.84	9.26 9.15	8.78 8.76	1.90

Table-2 Electron Spin Resonance Values of Cu (II) Complexes.

Parameter	G	g_{II}	$g_{\perp}$	g_{av}	G	A_{II}	$A_{\perp}$	Aav	λ/Δ_{II}	$\lambda/\Delta_{\perp}$	μ_{eff}
Complex $Cu(SB_1)_2Cl_2$	2.11	2.25	2.16	2.19	1.562	208	80	122.66	0.0309	0.0788	1.896

Magnetic measurements:

Complex of metal Mn(II) showed 5.74 BM. At room temperature, which is slightly lower than spin only value of 5.92 BM for high spin octahedral Mn(II) complex. CO(II) complex showed 4.5 BM, which is higher than the spin-only value of 3.89 BM, due to orbital participation. Therefore CO(II) complex is expected to have octahedral geometry, can be explained on the basis of octahedral symmetry involving a high degree of orbital contribution due to three-fold degeneracy of the ^4T_1g ground state. The magnetic moment values of Ni(II) complex is slightly higher than spin only moment of 2.83 BM. The magnetic moment values of Cu(II) complex is found in the range 1.90 BM at room temperature. According to found data, the Cu(II) complex can be assigned as distorted octahedral geometry (Figgish BN et al, 1960; Prabhu V et al, 1995) around Cu(II) ion as well as the presence of one unpaired electron on the metal ion. The geometry of all the complexes are further substantiated on the basis of spectral studies.

Electronic spectral studies

The Mn(II) complexes have a half-filled d-shell due to d^5 configuration and are spherically symmetric. Being thus, it is unaltered by operations of the octahedral group and belongs to ^6A_1g. The bands, observed in this Mn(II) complex, are assigned to the transitions and energies in terms of Racah Parameters (Tomilso, 1969). The Mn(II) complex shows four bands 19840, 20600, 25100 and 26420 cm^{-1} due to the $^6A_1g \rightarrow {}^4A_1g$, $^6A_1g \rightarrow {}^4T_2g$, $^6A_1g \rightarrow {}^4Eg$ and $^6A_1g \rightarrow {}^4A_1g$ transitions, respectively. The transitions, energies and calculated values for various parameters B=803, C=2514, 10Dq=8833 and β%=7.0, β=0.93, F^2=1161, F^4=71.81, f=1103, λ=196, h=1.0 and π/B=2.17. Due to these values, the band fit into the Tanabe Sugano matrix. All the values of different ligand field parameters are in close agreement with those of known octahedral Mn(II) complex.

The electronic spectra of synthesized CO(II) complex showed three bands on 8050, 17200 and 19800 cm^{-1}, which may be assigned to the transitions $^4T_1g \rightarrow {}^4T_2g$ (F) ν_1, $^4T_1g \rightarrow {}^4A_2g$ (ν_2) and ^4T_1g (F)$\rightarrow {}^4T_1g$ bands closely resemble with a spectra of the other distorted octahedral CO(II) complexes (EI-Sonavati AZ, 1992). Using free ion value of B=971 CM^{-1}, The value of spectral parameters in CO (II) are as follows 10Dq=9150, B=857, β= B'/B= 0.88, β% = 12% and Π/B=1.22, C=1725, f=1015.65, h=0.500, F^2=1103.45, F^4=48.81 and π=21716.The value of ν_3/ν_1 is 2.45 and this value go down in the usual range (2.00-2.80) observed for the majority of octahedral CO(II) coordination compound (Patel P and Bhattacharya PK, 1993).The electronic spectra of synthesized Ni(II) complex have been analyzed by NSH Hamiltonian theory (Donini JC, 1977). To distinguish projected normalized parameters DQ, DS and DT were employed than the lower case Dq, Ds and Dt. The Ni (II) complex shows bands at 8200, 10460, 14100, 16670 and 25640 cm^{-1} due to the $^3B_1g \rightarrow {}^3B_2g$ (νB), $^3B_1g \rightarrow {}^3Eg$ (νE), $^3B_1g \rightarrow Eg$ (Sh), ^3A_2g (F)$\rightarrow {}^3T_1g$ (F) ν_2 (F)$\rightarrow {}^3T_2g$ (F)$\rightarrow {}^3T_2G$ (P) ν_3 transitions, respectively, suggesting, tetragonal structure of Ni (II) complex. The ratio, DT/DQ gives the amount of distortion, have been calculated and indicate that the present complex somewhere between square planar and octahedral structure. Since the limiting value of DT/DQ for a square planar complex is 0.4226 (Dhakarey R, 1980). The calculated electronic spectral parameters of Ni(II) complex summarized as Dq^{xy}=1045, Dq^z=590, Dt=256.28, Ds=480, -DS=3370, DQ=22546, -DT=3498, Dq^L=22541, DQ^z=30823, DQ_A=14270, DQ_E=26684, -DT/DQ=0.15, dσ=-1205, dπ=-70.05, Δ_1=155.86, Δ_2=9709.31, Δ_3=3217.95, Dq^E=1121.20 and Dt/Ds=0.54.

The Cu(II) complex possess two or more bands, which may be assigned to the transition $^2Eg \rightarrow {}^2T_2g$, a characteristic of the distorted octahedral Cu(II) complex (Lever ABP, 1968). The newly prepared Cu(II) complex with SB_1 ligand containing overlapping band. Each of the resolved spectra of Cu(II) complex show four bands at 11800, 13100, 15440 and 17300 cm^{-1}. Hence, the interpretation of the spectrum was carried out assuming that Cu(II) complex is tetragonaly distorted in D^{4th} symmetry. The band assignment was carried out considering the spin orbit interaction. Considering the electronic spectra of the synthesized one $Cu(SB_1)_2Cl_2$ complex exhibited band at 15440 and 17300 cm^{-1}, may be due to the $\Gamma=7^a$ (^2Eg) → $\Gamma 6^b$ (^2T_2g) and $\Gamma 7^a$ (^2Eg) → $\Gamma 7^c$ (^2T_2g) transitions. According to conventional theory, the spin orbit splitting of these bands should amount to

about 3/2λ in first order (Liehr, 1960). The energy separation between the ^2T_2g state bands (1858 cm^{-1}) is higher than that of the free ion value (1245 cm^{-1}). The large value of λ indicate the slight molecular distortion (Ortolano, 1964) in the complex formation, besides the spin orbit coupling. Two other band appeared at 11800 and 13100 cm^{-1} were correspond to $\Gamma 7^a$ (^2Eg) → $\Gamma 6^a$ (^2Eg) and $\Gamma 7^a$ (^2Eg) → $\Gamma 7^b$ (^2T_2g) transitions. Here, the root for d^9 complexes in first order becomes E [$\Gamma 7^b$ (^2T_2g)] = 4 Dq-2Ds+Dt, correspond to 10 Dq. From this equation λ can be easily calculated. The other parameters Dq, Ds and Dt were determined by using λ = -1240cm^{-1} and founded as 10Dq=13100, Ds=2197, Dt=602 and ν_4-ν_3=1860. All the above information indicated the distorted octahedral (Osowole et al, 2012a) geometry around Cu(II) metal ion.

Infrared Spectral studies:

The most important IR-spectra of the Schiff base and its metal(II) complexes were recorded in KBr and listed in table-4. The ligand exhibits intense frequency due to νC=N as sharp band at 1645 cm^{-1}, which consistent with the iminic absorption of free shiff bases (widad et al. 2014). In all complexes, these band shifted to upper wave numbers and was observed at 1649-1653 cm^{-1} indicating the involvement of azomethine nitrogen in the coordination with the metal ion (Kumar LS 2011). The band of νC=O in the lower region 1680-1709 cm^{-1} in the metal complexes with respect to Schiff base ligand at 1718 cm^{-1}, showing the confirmation of carbonyl oxygen involvement in coordination with the metal ion (Anacona et al, 2015; Cherchiaro et al, 2004; Murukan et al, 2007). It is also confirmed by the presence of a new band in the complexes at 537-547 cm^{-1} due to M-N and 434-446 cm^{-1} due to M-O respectively (Ferraro, 1971; Nakemato, 1970; Sonmez et al, 2006; Sonmez and Sekerci, 2003).

The medium bands at 1265 cm^{-1} assigned to νC-O-C group of furan ring. This band unaffected in the complex formation, which clearly shows the noninvolvement in chelation with all metal(II) ion. The ligand has strong band at 1334 cm^{-1} and at 1150 cm^{-1} due to -SO_2 group (Bellamy LJ, 1962). On complexation, these bands are unaffected with metal (II) ion, indicating the non-participation in coordination. Another lower frequency band at 290-330 cm^{-1} was also appearance in complexs due to M-Cl (Ferraro JR, 1971; Nakemato S, 1970).

The IR-spectra of bivalent metal Mn(II), CO(II), Ni(II) and Cu(II) complexes shows the bidentate nature of the ligand with oxygen of sulphacetamide >C=O oxygen and Schiff base linkage nitrogen acting as donar sites.

ESR Spectra of Cu (II) complex:

The ESR spectra of $Cu(SB_1)_2$ Cl_2 complex has been recorded at room temperature with two "g" values computed by Peisach and Blumberg's method (Vachala SD et al, 2012). The complex Cu (SB_1) Cl_2 showed g=2.11, g_{II}=2.25, g⊥=2.16, gav=2.19, G=1.562 respectively, which showed g_{II} > g_1 > ge for Cu(II) complex of sulphacetamide, which implies 3d unpaired electron of Cu(II) ion occupied the dx^2-y^2 orbital as the ground state. It would be characteristic of axial symmetry (Niswander RH et al, 1975) i.e., tetragonal distorted octahedral conformation (Bai LJ et al, 1982; Nohria L et al, 2001). The gs in a D^{4th} symmetry should be g_{II}=ge+8λ/Δ_{II}, and g_I=ge+2λ/Δ_I (ge=2.0023) listed in table-2, that for its complex λ/Δ_{II} < λ/Δt indicating the ^2Eg labile lies below the ^2B_2g level. It is seen from data that reduction in Δg_{II} values may be due to an increase in λ or a decrease in λ or a combination of both. An increase in Δg_{II}, Δ and or a decrease in λ will lead to decrease in Δg_{II}, Δg_I and Δav and increase in covalency of coordination bonding (Devies MB 1993; Prabhu V and Venkappaya D 1995) from metal to ligand and opposite it, shows increasing ionic character of the coordination bonding (Kivelson D and Neiman R 1961). The exchange coupling constt. (G < 4) indicated considerable exchange interaction between Cu(II) ions (Sonmez M and Hacryusufoglu 2006). Therefore, from the esr spectra of this complex, confirmed covalent character of the metal ligand band.

Table-3 Antibacterial study for ligand and their Metal Complexes

Sr. No.	Compounds	Diameter of growth of Inhibition Zone (mm)#			
		B.cereus	***S.aureus***	***E.coli***	***A.niger***
1	SB_1(L)	12	16	15	11
2	Mn $(SB_1)_2Cl_2$	16	18	20	14
3	CO $(SB_1)_2Cl_2$	20	17	17	15
4	Ni $(SB_1)_2Cl_2$	15	14	16	12
5	Cu $(SB_1)_2Cl_2$	18	17	19	16

Table-4 Characteristic Infrared Spectral Data of SB_1 ligand and it's Metal (II) Complexes

S.N.	Compound	νC=N	νC=O	νC-O-C	νM-N	νM-O	νSO_{2Sym}	νSO_{2Asym}
1	SB_1	1645s	1718s	1263s	-	-	1152m	1332m
2	$Mn(SB_1)_2Cl_2$	1653s	1709m	1262m	537s	434s	1153m	1330m
3	$CO(SB_1)_2Cl_2$	1659s	1705m	1263m	540m	438m	1150m	1329m
4	$Ni(SB_1)_2Cl_2$	1649s	1697m	1264w	543m	440m	1154w	1332s
5	$Cu(SB_1)_2Cl_2$	1652s	1680m	1262m	547s	446m	1153m	1330m

Biological Activity:

The microbial culture was procured from Microbial Type Culture Collection (MTCC). All synthesized compound was dissolved in DMSO. Biological activity of the synthesized Schiff base ligand and it's four metal complexes have been done by agar well diffusion method against 2 Gram (+) bacteria (*Bacillus cereus* and *S. aurens*) and one Gram (-) bacteria (*E.Coli*). All microbial culture was prepared by taking 10mg of compound in DMSO. An agar medium was taken into each petri dish with a same concentration of 20 ml. All synthesized compound was prepared with different concentration (100, 50, 40, 30, 20, 10, >μg mL^{-1}) in DMSO. These different concentrated compounds were swapped with 100 μL inocula of the test microorganisms tanking more than 15 minutes for adsorption. The petri disc incubated at 37°C for 48 hours. The microbial activities against synthesized complexes were writing down by the measurement of growth of Inhibition Zone with zone reader (Hi Antibiotic zone scale). DMSO was used as positive and negative control respectively. DMSO used as negative control for fungal strains. The procedure was performed triplicates for each microorganism (Aneja KR et al, 2011) and the inhibition growth was recorded. The minimum inhibitory concentration of each compounds was recorded as decreasing concentration range of 400 to 3.12 μg/Ml. The decreasing concentration of 100 μL volume was taken into wells in the agar petri dish, which have already seeded with same volume of standardized inoculums (10^6 cfu/mL), and recorded comparing with negative control and data have been in table-3.

CONCLUSION

The present study of Schiff base ligand (SB_1) -3,4{[-benze 1-sulphacetamide] imino] methyl} furan and it's metal Mn(II), CO(II), Ni(II) and Cu(II) Complexes have Octahedral geometry for Mn(II) and CO(II), distorted octahedral geometry for Ni(II) and Square planner geometry for Cu(II) complexes respectively. The synthesized ligand and all these metal(II) complexes are found to be active against Gram (+) bacteria (B. cereus and S. aureus), Gram (-) bacteria (*E.Coli*) and fungi (A. niger). Ligand (SB_1) has less reactive than it's all complexes with respect to Gram (+) *B.cereus* bacteria and fungal *A.niger*. But in complexes, Ni(II) complex is found to be less reactive against Gram (+), Gram (-) bacterial and fungal activity with respect to other metal(II) complexes.

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