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SYNTHESIS, CHARACTERIZON AND ANTIBACTERIAL STUDIES OF SOME METAL COMPLEXES OF A SCHIFF BASE DERIVED FROM BENZALDEHYDEAND SULFONAMIDE

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Abstract: Neutral complexes of Cu(II),Ni(II), and Co(II) with a schiff base derived from benzaldehyde and sulfonamide have been synthesized and characterized the basis of on FTIR, UV-VIS, molar conductance and atomic absorption spectroscopic data. These metal complexes were also screened for their antibacterial activity against two bacterial species *E.coli and Salmonella typhae.* The metal complexes showed enhanced antibacterial activity as compared to uncomplexed ligand.

Key Words: Schiff base, metal ions, antibacterial activity.

Introduction

Schiff bases and their metal complexes have exhibited biological activity as antibiotics, antiviral and antitumor agents because of their specific structure [1-2]. Heteronuclear Schiff base complexes have found applications as magnetic materials, catalysts and in the biological engineering [3-7]. Sulpha drugs are a group of compounds used for eliminating a wide range of infections in human and other animal systems. Many chemotherapeutically important sulpha drugs like sulphadiazine, sulphathiazole, sulphamerazine etc. possess SO_2NH moiety which is an important toxophoric functional group.

Sulfonamides were the first drugs found to act selectively and could be used systematically as preventive and the therapeutic agents against various diseases .Sulfur ligands are widespread among co-ordination compounds and are important components of biological transition metal complexes which posses many applications such as diuretic, antiglaucoma or antiepileptic drugs among others [8-10]

First row transition metals such as Cu, Zn, Co etc. compounds have attracted much attention due to their biological importance [11]. It has been reported that the biological activity of sulfur containing ligand increases on complexation. Many aldehydes are known to be potential inhibitors for DNA synthesis; therefore, if a salicydehyde, benzaldehyde and furfural moiety is coupled with sulfonamide the resulting compounds sulfonamide-imine may show enhanced biological activity. These factors prompted us to carry out a study for synthesis of sulfonamide-imine (schiff base) and its complexes with Cu (II), Ni (II), and Co (II) metal ions.

Aim of this investigation is to synthesize the sulfonamide-imine derived from benzaldehyde and sulfonamide, study their complexation behavior and antibacterial activities in complexed and uncomplexed states against ***E.coli and Salmonella thphae.***

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EXPERIMENTAL WORK

Materials and Reagents

All chemicals used were of analytical grade (AR) and of higest purity available which included benzaldehyde (BDH), sulfonamide (Sigma), copper (II) chloride dihydrate, nickel (II) chloride hexahydrate, cobalt (II) acetate tetrahydrate (United laboratory chemical works) and Sulfuric acid (United laboratory chemical works). Organic solvents used included ethanol, methanol, Carbon tetrachloride, Chloroform, dimethylformamide (DMF) and dimethylsulphoxide (DMSO) (Merck).

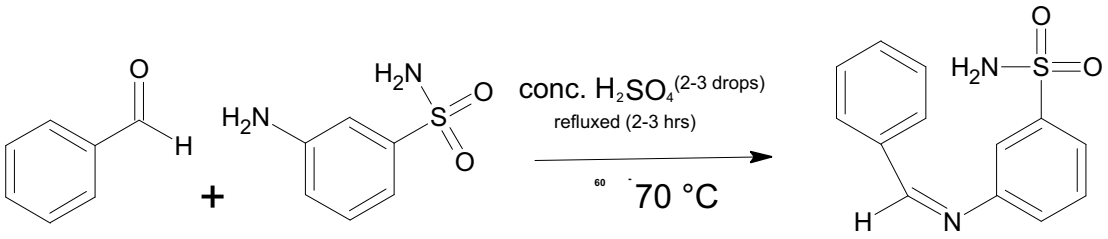
Instrumentation

The spectrophotometric measurements in solution were carried out using various instruments. Liquid state UV-Vis spectra of ligand and its metal complexes were recorded on a Hitachi U-2000 spectrophotometer using DMSO as a solvent in the range of 200-600 nm. The solid state FTIR spectra of the ligand and its metal complexes were recorded on Perkin-Elmer spectrum-RXI FTIR spectrophotometer using KBr pellets in the range of 4000-500 cm^{-1}. The amounts of metals present in the complexes were estimated by Varian AA-1275 spectrophotometer.

Conductometric measurements of metal complexes were taken from Cybar Scan 500 conductometer, using DMSO as solvent. Melting points were determined by digital Gallenkamp apparatus.

Synthesis of Ligand

0.1M solution of sulfonamide (25 ml) was added to 0.1 M solution of benzaldehyde (25 ml), then 2-3 drops of Conc. H_2SO_4 were added in reaction mixture and refluxed for 2 to 3 hours at 60 to 70˚C. The resulting solution was concentrated up to 10 ml on a water bath, and allowed to cool on ice bath. Pale yellow precipitate formed was filtered, washed with Carbon tetrachloride, recrystalized with methanol and dried in *vacuo* to furnish the required Schiff base.



benzaldehyde

(0.1M/25ml)

sulfonamide

(0.1M/25ml)

sulfonamide-imine

Synthesis of Metal Complexes

Metal complexes of schiff base were prepared by refluxing the 0.2 M (25 ml) of schiff base ligand with 0.1M (20 ml) of Ni, Co and Cu salts for 2 hours .The resulting solution was concentrated to 10ml on a water bath and allowed to cool on ice bath, colored precipitates formed were filtered, washed with Carbon tetrachloride, recrystalized from methanol and dried in vacuo.

Antibacterial Studies

Antibacterial activity of the complexes/ ligand was carried out at the Department of Microbiology, University of the Punjab, Lahore. Antibacterial activity agianst two bacterial species*, E.coli and Salmonella typhea* was determined using paper Disc diffusion method [12]

The nutrient agar medium (Peptone, Beefextract, NaCl and Agar-Agar) and

5mm diameter paper disc (whatman No.1) were used. Compounds were dissolved (30 μg in DMF (0.01ml).

Filter paper disc was soaked in the solution of ligand as well as complexes, dried and then placed in Petri plates previously seeded with the test organism. Plates were incubated for 24-30 hrs at 37˚C. Zone of inhibition (diameter in mm) was then measured around the disc.

Results and Discussion

Structure of the ligand was established with the help of FTIR and previous analytical data available in the literature [1-7, 11]. Metal complexes of this ligand were analyzed for their elemental composition. Color change of the complexes along with decomposition point >200˚**C** shows characteristic differences between schiff base and metal complexes. Upon heating the ligand melts at 194˚**C** without decomposing, whereas color change of the complexes is observed due to decomposition of the complexes above 200˚**C**. This shows that thermal energy accumulated in the complex not only changes its physical characteristics, i.e. melting, but also breaks chemical bond i-e decomposition. The same kind or phenomenon has been observed in starch and starch like biopolymers. Schiff bases and its complexes are soluble in ethanol, methanol, DMSO and DMF (**Table 1**).

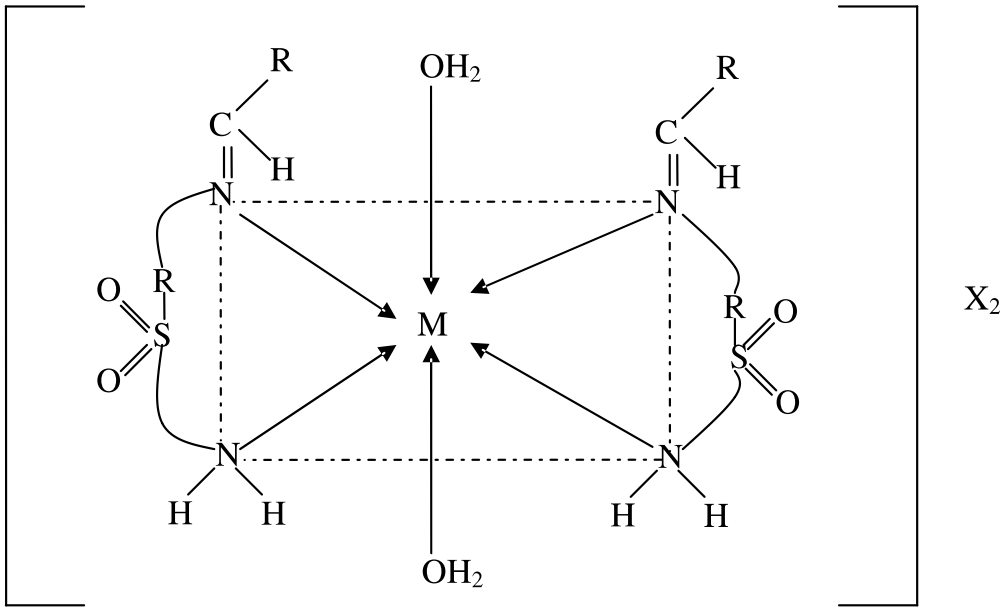
Conductometric measurement of metal complexes were recorded, chelates are found to have molar conductance values in the range of 63-343.98 (μs) indicating that they are electrolyte (**Table 1**).

UV-Vis spectra of the schiff base and metal complexes showed that in case of schiff base there is a broad band at λmax 315nm (n-π*) and in case of metal complexes occur a blue shift of the n-π* 315 to 310, 304 and 311nm, which is due to the formation of bond between nitrogen atom of the ligand the metal ions (**Table 1**).

FTIR spectra of schiff base and its metal complexes are listed (**Table 1**). FTIR spectra of the complexes were compared with those of the free ligand to determine the co-ordination sites that may have been involved in the bonding. Upon comparison it was found that azomethine ν(C=N) stretching vibration which was found in the free ligand at 1621.2cm^{-1} band is shifted to lower wave number 1589-1594 cm^{-1} in the complexes indicating the participation of the azomethene nitrogen in coordination (M-N). In addition there are some bands in the region of 3600-3800 cm^{-1} in the FTIR spectra of all metal complexes, which indicates the presence of co-coordinated water. Two molecules of water are co-ordinated to the 5th and 6th co-ordination sites of the metal ion as the ligand is bidentate.

The atomic Absorption studies of the metal complexes were recorded using the Varian AA-1275 Spectrophotmeter, in order to determine M-L ratio. The calculated and observed values for different geometries of different metal complexes are given in (**Table 1)**. Geometries of all complexes are found to be octahedral, with configuration [M $(L)_2$. $(H_2O)_2$].Cl_2 where M= Cu (II), Ni (II) or Co (II) And L= Schiff-base ligand.

The title schiff base and its metal chelates were evaluated for their antibacterial activity aganist bacterial species *E.coli and Salmonella thpha.* The compounds were tested at a concentration of 30μg/0.01ml in DMF solution paper disc diffusion. The diameters of the susceptibility zones were measured (in mm) and the result are presented in (**Table 2)**. It was observed that the metal chelates are more antibacterial than



uncomplexed ligand. On the basis of the above observation, it is concluded that the process of chelation [13] dominantly affects the biological behavior of the compounds that are potent against some bacterial strains.

Table1: Physico-analytical data of ligand and its metal complexes

S.NO.	Color	solubility	Melting point (°C)	Conductance (µs)	U.V/Vis. λ (nm)	I.R. cm^{-1}	% metal	
							Found	Calculated
Ligand	Pale yellow	CH_3OH,C_2H_5OH DMSO,DMF	194	-	247, 315	1621.2 (C=N), 3291.3(N-H) 1376.1 Sym, 1154.8 Asy (SO_2)	-	-
$[Cu(L)_2(H_2O)_2]Cl_2$	Dark green	CH_3OH,C_2H_5OH DMSO,DMF	209	169.9	233.5,310, 477	1592(C=N), 3276(N-H) 3729.5, 3804(co-ord.H_2O) 1376.8 sym, 1154.7 As (SO_2)	10.6	9.2
$[Ni(L)_2(H_2O)_2]Cl_2$	Light green	CH_3OH,C_2H_5OH DMSO,DMF	220	343.98	246, 304	1589(C=N), 3293.7(N-H) 3839.7(co-ord.H_2O),1376.8 sym, 1153.9 Asy (SO_2)	8	8.5
$[Co(L)_2(H_2O)_2](CH_3COO)_2$	Pink	CH_3OH,C_2H_5OH DMSO,DMF	202	63	311,547	1593.2(C=N),3280.2(N-H) 3662, 3821.9 (co-ord.H_2O) 1376.3 sym,1151.3 Asy (SO_2)	9.3	8.6

***DMSO = Dimethyl sulphoxide, *DMF= Dimethyl formamide.**

Table 2: Antibacterial activity data of ligand and its metal complexes

Schiff base complex	Microbial activity	
	E.coli	*Salmonella typhae*
Ligand	+	++
$[Cu(L)_2(H_2O)_2]Cl_2$	+++	+++
$[Ni(L)_2(H_2O)_2]Cl_2$	++	++
$[Co(L)_2(H_2O)_2](CH_3COO)_2$	+	++

Inhibition Zone (mm) = + ≅ 14-16, ++ ≅ 16-18, +++ ≅18-20

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